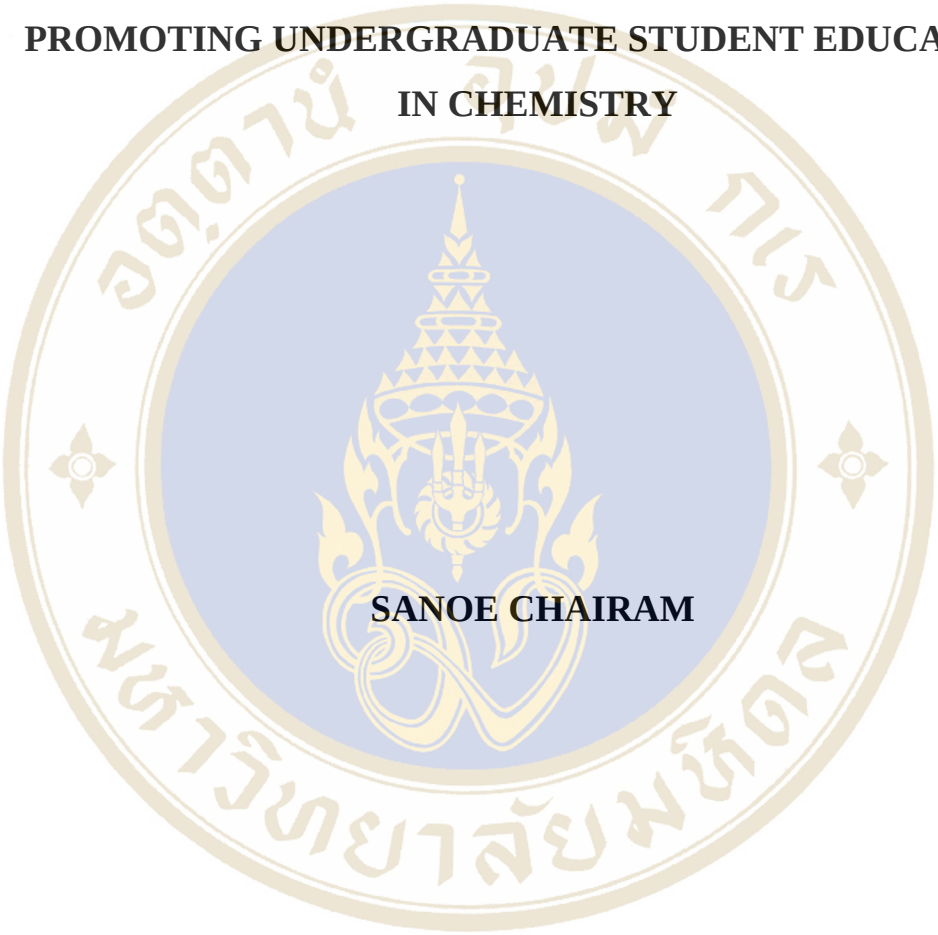
HANDS-ON FABRICATION OF MAGNETITE NANOPARTICLES ON MUNG BEAN VERMICELLI COATED WITH SILVER NANOPARTICLES BY GREEN METHOD FOR PROMOTING UNDERGRADUATE STUDENT EDUCATION IN CHEMISTRY



SANOE CHAIRAM

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR
THE DEGREE OF DOCTOR OF PHILOSOPHY
(SCIENCE AND TECHNOLOGY EDUCATION)
FACULTY OF GRADUATE STUDIES
MAHIDOL UNIVERSITY
2008

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Entitled

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Sanoe Chairam
.....
Mr. Sanoe Chairam
Candidate

.....
Asst. Prof. Ekasith Somsook
Ph.D. (Chemistry)
Major-Advisor

.....
Assoc. Prof. Bhinyo Panijpan
Ph.D. (Biophysics)
Co-Advisor

Darapond T.
.....
Lect. Darapond Triampo
Ph.D. (Materials Science and Engineering)
Co-Advisor

.....
Assoc. Prof. Richard K. Coll
Ph.D. (Chemistry), Ph. D. (Science Education)
Co-Advisor

B. Mahaisavariya
.....
Prof. Banchong Mahaisavariya
M.D.
Dean
Faculty of Graduate Studies

Pintip Ruenwongsa
.....
Assoc. Prof. Pintip Ruenwongsa
Ph.D. (Biochemistry)
Chair
Doctor of Philosophy Programme in Science and Technology Education
Faculty of Science

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was submitted to the Faculty of Graduate Studies, Mahidol University
for the Degree of Doctor of Philosophy (Science and Technology Education)
on
May 26, 2008

Sanoe Chairam
……
Mr. Sanoe Chairam
Candidate

……
Assoc. Prof. Bhinyo Panijpan, Ph.D.
Member

Pintip Ruenwongsa
……
Assoc. Prof. Pintip Ruenwongsa, Ph.D.
Chair

Darapond T.
……
Lect. Darapond Triampo, Ph.D.
Member

Ekasith Somsook
……
Asst. Prof. Ekasith Somsook, Ph.D.
Member

Pisarn Soydhurum
……..
Lect. Pisarn Soydhurum, Ph.D.
Member

……..
Assoc. Prof. Bhinyo Panijpan, Ph.D.
Director
Institute for Innovation and Development
of Learning Process
Mahidol University

B. Mahaisavariya
……
Prof. Banchong Mahaisavariya
M.D.
Dean
Faculty of Graduate Studies,
Mahidol University

……
Prof. Skorn Mongkolsuk, Ph.D.
Dean
Faculty of Science
Mahidol University

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ACKNOWLEDGEMENTS

I would like to express my gratitude to thank my major advisor, Asst. Prof. Dr. Ekasith Somsook, for continuously providing valuable advice and important guidance and challenging ideas in my Ph.D. research. I truly appreciate and thank him for his encouragements during the writing of this thesis. I also would like to thank my co-advisors, Assoc. Prof. Dr. Bhinyo Panijpan and Dr. Darapond Triampo, for their helpful comments, advice and their valuable time spent as member of the thesis examination committee. My sincere appreciation is also extended to Dr. Pisarn Soydhurum who was the external examiner of the thesis defence, and provided suggestions for improvement.

I wish to specially thank my co-advisor, Assoc. Prof. Richard K. Coll, for his support, advice, guidance and discussions about research methods in science education over a year of conducting the research at the Center for Science & Technology Education Research (CSTER), the University of Waikato, New Zealand. I also would like to thank CSTER director, Dr. Bronwen Cowie who provided an opportunity to do research in science education at New Zealand. I would like to thank all the staff and my great friends at CSTER when I was there.

I would like to especially thank all the staffs, at the Institute for Innovation and Development of Learning Process and the Department of Chemistry at the Faculty of Science, Mahidol University, who taught me throughout my Ph.D. degree. I would like to thank Asst. Prof. Dr. Juthamas Jitcharoen, the Department of Chemistry, the Faculty of Science, Ubon Rajatanee University, for encouragement during I have studied in Bangkok. Also, I would like to thank the Ubon Rajatanee University for research grant and financial support. Thanks also go to all first-year undergraduate science students who participated in this research.

Finally, my heartfelt thanks go to my family for their love, belief, support, care, and encouragements until I graduated.

Sanoe Chairam

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HANDS-ON FABRICATION OF MAGNETITE NANOPARTICLES ON MUNG BEAN VERMICELLI COATED WITH SILVER NANOPARTICLES BY GREEN METHOD FOR PROMOTING UNDERGRADUATE STUDENT EDUCATION IN CHEMISTRY

SANOE CHAIRAM 4637459 SCED/D

Ph.D. (SCIENCE AND TECHNOLOGY EDUCATION)

THESIS ADVISORS: EKASITH SOMSOOK, Ph.D. (CHEMISTRY), BHINYO PANIJPAN, Ph.D. (BIOPHYSICS), DARAPOND TRIAMPO, Ph.D.(MATERIALS SCIENCE AND ENGINEERING), RICHARD K. COLL, Ph.D. (CHEMISTRY), Ph.D. (SCIENCE EDUCATION)

ABSTRACT

Three novel chemical experiments have been developed consisting of two experiments in nanotechnology and one experiment for enhancing student education in chemical kinetics, namely: (i) magnetic iron oxide nanoclusters, (ii) silver and gold nanoparticles, and (iii) chemical kinetics of egg shells.

(i) A novel method for fabricating magnetic iron oxide nanoparticles was achieved by using a transparent vermicelli template consisting of mung bean starch as a new stabilizing material. The morphology of the as-prepared magnetic iron oxide that was deposited on the surface of vermicelli was observed as nanoclusters. The magnetization of the magnetic iron oxide nanoparticles at room temperature was decreased after carbonization at a temperature of 200°C. Furthermore, the thermal decomposition of iron oxide nanoparticles stabilized by starch vermicelli template yielded magnetic iron oxide/carbon nanocomposites with soft magnetic behavior, which is useful for biomedical applications.

(ii) Size- and shape-controlled syntheses of silver and gold nanoparticles were developed using a starch vermicelli template acting as nanoreactors for the growth of nanoparticles. Pale red colloidal solutions were observed for gold, while purple and yellow colloidal solutions were observed for silver. The carbonization at different temperatures was carried out to investigate nanoparticles embedded in vermicelli. The yellow color of silver was originated from silver nanoparticles while the purple color was originated from a mixture of silver and silver chloride nanoparticles. This approach offers a great potential for the large-scale synthesis of silver and gold nanoparticles for chemical and biological applications.

(iii) Chemical kinetics is an extremely important concept for introductory chemistry courses. The instruction in chemical kinetics is often teacher-centered at all levels. This research seeks to shift students from passive learning to more active, student-centered learning. The research involved first-year science students at Mahidol University, Thailand. Drawing on inquiry-based learning, the students were asked to design an experiment investigating the reaction of acids and bases. The research findings suggested that most students were able to explain changes in the rate of chemical reactions, and also develop a good conceptual understanding of chemical kinetics.

KEY WORDS: NANOSTRUCTURES / MAGNETIC IRON OXIDE/ GOLD AND SILVER NANOPARTICLES/ STARCH VERMICELLI/ EXPERIMENTAL DESIGN/ CHEMICAL KINETICS

188 pp.

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การสร้างอนุภาคที่มีสมบัติแม่เหล็กระดับนาโนบนวุ้นเส้นถั่วเขียวซึ่งเคลือบด้วยอนุภาคเงิน เพื่อใช้กระตุ้นการเรียนการสอนนักศึกษาในสาขาวิชาเคมี (HANDS-ON FABRICATION OF MAGNETITE NANOPARTICLES ON MUNG BEAN VERMICELLI COATED WITH SILVER NANOPARTICLES BY GREEN METHOD FOR PROMOTING UNDERGRADUATE STUDENT EDUCATION IN CHEMISTRY)

เสนอ ชัยรัมย์ 4637459 SCED/D

ปร.ด. (วิทยาศาสตร์และเทคโนโลยีศึกษา)

คณะกรรมการควบคุมวิทยานิพนธ์: เอกสิทธิ์ สมสุข, Ph. D. (Chemistry), ภิญโญ พานิชพันธ์, Ph.D. (Biophysics), ดาราภรณ์ เตรียมโพธิ์, Ph.D. (Materials Science and Engineering), Richard K. Coll, Ph. D. (Chemistry), Ph. D. (Science education)

บทคัดย่อ

การทดลองทางเคมี 3 ชนิด ที่ถูกพัฒนาขึ้นนี้ ประกอบด้วย 2 การทดลองในงานวิจัยนาโนเทคโนโลยี และ 1 การทดลองเพื่อใช้ส่งเสริมการเรียนการสอนของนักศึกษาในสาขาเคมี ได้แก่ (i) อนุภาคเหล็กออกไซด์ระดับนาโนที่มีสมบัติแม่เหล็ก, (ii) อนุภาคเงินและทองระดับนาโน และ (iii) จลนศาสตร์เคมีของเปลือกไข่

(i) วิธีการใหม่เพื่อสร้างอนุภาคเหล็กออกไซด์ระดับนาโนที่มีสมบัติแม่เหล็ก คือ การใช้วุ้นเส้นแป้งถั่วเขียวเป็นตัวช่วยเหลือตัวใหม่ รูปร่างของอนุภาคเหล็กออกไซด์ที่ถูกสังเคราะห์บนผิวของวุ้นเส้นมีลักษณะเป็นอนุภาคอยู่รวมเป็นกลุ่มระดับนาโน ความเป็นแม่เหล็กของอนุภาคเหล็กออกไซด์ที่อุณหภูมิห้องมีค่าลดลงหลังจากทำการเผาที่อุณหภูมิ 200 องศาเซลเซียส และได้คอมโพสิตระดับนาโนของอนุภาคเหล็กออกไซด์ที่มีสมบัติแม่เหล็ก/คาร์บอน ที่มีสมบัติทางแม่เหล็กที่อ่อน ซึ่งสามารถนำไปประยุกต์ใช้ทางการแพทย์ได้

(ii) การสังเคราะห์ที่ควบคุมขนาดและรูปร่างของอนุภาคเงินและทองในระดับนาโนถูกพัฒนาโดยใช้โครงสร้างของวุ้นเส้นเป็นตัวกำหนดการเติบโตของอนุภาค สารละลายคอลลอยด์สีแดงอ่อนพบเฉพาะสำหรับทองเท่านั้น ในขณะที่สีม่วงและเหลืองพบสำหรับเงิน การเผาที่อุณหภูมิต่างๆ เพื่อศึกษาอนุภาคระดับนาโนที่อยู่ในวุ้นเส้น พบว่า สีเหลืองเกิดจากอนุภาคระดับนาโนของเงิน ในขณะที่สีม่วงเกิดจากการผสมกันระหว่างอนุภาคระดับนาโนของเงินและเงินคลอไรด์ การสังเคราะห์อนุภาคระดับนาโนของเงินและทองในปริมาณมากนี้สามารถนำไปประยุกต์ใช้ในทางเคมีและชีววิทยา

(iii) จลนศาสตร์เคมีเป็นหัวข้อหนึ่งที่มีความสำคัญสำหรับเคมีพื้นฐาน การสอนจลนศาสตร์เคมีส่วนใหญ่จะเป็นแบบครูเป็นศูนย์กลาง งานวิจัยส่วนต้องการให้เป็นแบบผู้เรียนเป็นศูนย์กลางมากขึ้น โดยเกี่ยวข้องกับนักศึกษาคณะวิทยาศาสตร์ ชั้นปีที่ 1 บนพื้นฐานของการเรียนรู้แบบสืบเสาะ นักศึกษาจะถูกถามให้ออกแบบทำการทดลองเพื่อศึกษาปฏิกิริยาของกรดและเบส พบว่า นักศึกษาส่วนใหญ่สามารถอธิบายผลของการเปลี่ยนแปลงปัจจัยที่มีผลต่ออัตราการเกิดปฏิกิริยาเคมี และมีความรู้ความเข้าใจจลนศาสตร์เคมีดีขึ้น

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LIST OF ABBREVIATIONS

nm	Nanometer, 10^{-9} m
χ_M	Magnetic susceptibility
C_M	Curie constant
T_C	Curie temperature
μ	Magnetic moment
H	Magnetic field, Oe
μ_Z	Magnetic moment projection on the z-axis
μ_R	Remanent magnetic moment
τ	Relaxation time
V	Particle volume
K_{eff}	Anisotropy constant
T_B	Blocking temperature
K	Kelvin
°C	Degree Celsius
mol	Mole
J	Joule
a	Unit cell edge length
AuNPs	Gold nanoparticles
AgNPs	Silver nanoparticles
μm	Micrometer, 10^{-6} m
UV-Vis	UV-Visible
RT	Room temperature
Fe_3O_4	Magnetite
MRI	Magnetic resonance imaging
DI	De-ionized water

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LIST OF ABBREVIATIONS (CONTS.)

TGA	Thermal gravimetric analysis
DSC	Differential scanning calorimetric analysis
SEM	Scanning electron microscope
FE-SEM	Field emission scanning electron microscope
TEM	Transmission electron microscope
XRD	X-ray powder diffraction
VSM	Vibrating sample magnetometer
HR-TEM	High-resolution transmission electron microscopy
mL	Milliliter, 10^{-3} L
μm	Microliter, 10^{-6} L
θ	Angle
min	Minute
mg	Milligram
Fig	Figure
d	Crystal size
B_θ	Width of the peak at half height
λ	Wavelength
θ_B	Bragg angle
M	Magnetization, emu g^{-1}
ca	About
fcc	Face-centered cubic
JCPDS	Joint committee on powder diffraction standards
no	Number
h	Hour
s	Second

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LIST OF ABBREVIATIONS (CONTS.)

NRC	National Research Council
NSES	National Science Education Standards
MOE	Ministry of Education
CUAS	Central University Admission System
BEC	Basic Education Commission
ONPEC	Office of the National Primary Education Commission
MUA	Ministry of University Affairs
IPST	Institute for the Promotion of Teaching Science and Technology
POE	Prediction-observation-explanation
SD	Strongly disagree
D	Disagree
N	Neutral
A	Agree
SA	Strongly agree
TAs	Teaching assistants
r	Rate of reaction
t	Time
V	Volume
V_f	Final volume
$\bar{x}$	Mean
Std. Dev.	Standard deviation

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THE RELEVANCE OF THE RESEARCH WORK TO THAILAND

This research work consists of the preparation of nanomaterials by using mung bean vermicelli as a template, and an experimental design for promoting students in chemistry education. For the science part, as well known, Thailand is a world's top producer of starch with an annual yield of several million tons for global production over several past years. Starches produced in Thailand are usually exported to other countries in worldwide in form of a natural raw material, such as cassava and rice with a low price per kilogram. The use of starch production in other applications is still challenged for many researchers. Therefore, it would be good if we are able to develop starch production in the line of nanotechnology interest, and its value can be increased. For the science education part, the teaching of practical work or practical laboratory is a significant part for an effective pedagogy in science curriculum in worldwide including Thailand. Like physics and biology, chemistry is one of the most important science subjects to be concerned with the properties and reactions of substances. By doing the designed experiments in chemistry practical class, Thai students would be able to see what, how and why chemical reactions change.

The reasons are relevant to Thailand as follows.

(i) Knowledge of the chemistry of starch and methods of analysis will increase an understanding of the relationship between starch and its applications.

(ii) The use of starch production may help to create a novel material for increasing the diversity of the nanoparticle preparation in scientific laboratories.

(iii) Students are provided to investigate the scientific problems in a similar way to the real scientists.

(iv) Thai science teachers may wish to consider the teaching of science subjects based on inquiry-learning with simple reactions and materials related to everyday process. This may be used instead of more expensive chemicals in classrooms.

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Starch vermicelli template for synthesis of magnetic iron oxide nanoclusters

Sanoe Chairam[a], Ekasith Somsook[b,*]

[a]Institute for Innovation and Development of Learning Process, Mahidol University, Rama VI Road, Rachathewi, Bangkok 10400, Thailand
[b]Faculty of Science, Department of Chemistry, Mahidol University, Rama VI Road Rachathewi, Bangkok 10400, Thailand

Received 5 December 2007; received in revised form 26 January 2008

Abstract

A novel method for fabricating magnetic iron oxide nanoparticles was achieved by using transparent vermicelli template as a new stabilizing material. The morphology of the as-prepared magnetic iron oxide deposited on the surface of vermicelli was observed as nanoclusters. The magnetization of the magnetic iron oxide nanoparticles at room temperature was decreased after carbonization at 200 °C. Therefore the thermal decomposition of iron oxide nanoparticles stabilized by starch vermicelli template yielded iron oxide/carbon nanocomposites with the soft magnetic behavior which are useful for biomedical applications.
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Keywords: Magnetic iron oxide nanoparticle; Nanocluster; Starch vermicelli template

1. Introduction

Iron oxides with 16 phases are known and widespread in nature in the forms of oxides, hydroxides or oxide–hydroxides [1]. As known, nanometer-scale magnetic particles have been frequently synthesized for the nanomagnetism study due to a variety of potential applications in magnetism, catalysis, chemistry and biology [2–4].

The formation of magnetic nanoparticles has attracted considerable interest. To utilize magnetic iron oxide nanoparticles for highly sensitive magnetic nanodevices, several methods for synthesizing magnetic iron oxide nanoparticles have been developed. Among these, the solution-phase synthesis by co-precipitating of Fe^{2+} and Fe^{3+} ions in a basic media has been shown as the most effective method to prepare highly uniform magnetite (Fe_3O_4) nanoparticles in size and shape [5,6]. An alternative method is the thermal decomposition, which has been shown for the synthesis of high crystalline iron oxide nanoparticles. Many stabilizing agents with a long-chain structure [7,8] have been introduced to stabilize magnetite (Fe_3O_4) nanoparticles with the desired size. However, these compounds are highly reactive and potential environmental risks during the synthetic processes. To save the environment, biomaterials have been used as a stabilizing agent in the experimental procedures. Recently, Giri and coworkers reported the preparation of uniform sized magnetite (Fe_3O_4) nanoparticles stabilized by using phospholipids [6]. The development of new biomaterials is still a considerable challenge for *in vivo* applications. As known starch is one of the most widely used carbohydrate polymers, our interest here is the utilization of mung bean starch vermicelli as template may bring advantageous possibilities to stabilize magnetic iron oxide nanoparticles because vermicelli surface of starch which is full of polyhydroxylated functional groups [9], may assist the precipitation of the iron oxide nanoparticles on the surface of the vermicelli template.

In order to achieve the preparation of the soft magnetic materials for applications, it is required to reduce the strength of the applied magnetic field to be as small as possible. Herein, we have proposed a novel experimental procedure to prepare soft magnetite (Fe_3O_4) stabilized by mung bean starch vermicelli template after carbonization at a low temperature (200 °C). The phase transition

*Corresponding author. Tel.: +662 201 5123; fax: +662 354 7151.
E-mail address: scess@mahidol.ac.th (E. Somsook).

0304-8853/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jmmm.2008.02.168

Please cite this article as: S. Chairam, E. Somsook, J. Magn. Magn. Mater. (2008), doi:10.1016/j.jmmm.2008.02.168

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temperature of the sample is performed by using thermal gravimetric analysis (TGA) and differential scanning calorimetric (DSC) measurement. The morphology of the sample was investigated by field emission scanning electron microscope (FESEM) and transmission electron microscope (TEM), respectively. To investigate the crystalline phase, the as-prepared sample was characterized by using X-ray powder diffraction (XRD). Finally, the magnetic behavior was examined by vibrating sample magnetometer (VSM).

2. Materials and experimental procedures

2.1. Materials

The starting chemicals used in this experiment were commercially analytical graded reagents. Ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, $\geqslant 99.0\%$) was purchased from Fluka (Switzerland) and used without further purification. Hydrochloric acid (HCl, 37%) and ammonium hydroxide (NH_4OH, 25%) were obtained from Labscan Asia (Thailand). Transparent mung bean starch vermicelli (100%, Kaset brand) as a stabilizing material was purchased from a local supermarket in Thailand. All glassware in this experiment were cleaned with 10% nitric (HNO_3) solution and repeatedly washed with de-ionized water obtained from Nanopure® Analytical Deionization Water with an electronic resistance $\geqslant 18.2\,M\Omega\,cm$, and dried before use.

2.2. Preparation of magnetite (Fe_3O_4) nanoparticles stabilized by vermicelli template

The method described here for synthesizing magnetite (Fe_3O_4) nanoparticles was based on the literature [1]. The controlled size of particles was prepared with the addition of mung bean vermicelli template as a stabilizing agent according to the following procedure. Typically, required amount of 1.0 g transparent mung bean vermicelli was placed into a 100 mL round-bottom flask containing 50 mL of a 0.1 M $FeCl_2$ solution. The mixture solution was stirred vigorously under the presence of air and also heated at temperature 85–90 °C by placing the thermometer for temperature control in an oil bath. To form magnetite (Fe_3O_4) nanoparticles, the pH of the mixture solution was adjusted to 12 by a diluted ammonium hydroxide solution (NH_4OH). The color of the colloidal solution was changed immediately to black. The reaction system was maintained under magnetic stirring for 30 min before cooling down to room temperature. The magnetic behavior of the precipitate was observed and removed from the solution by applying a magnetic field from the bottom of the reaction flask, and the black precipitate powder settled down. After discarding the supernatant solution, the mung bean vermicelli coated with iron oxide nanoparticles was washed several times with a diluted hydrochloric solution and de-ionized water, respectively, in order to remove impurity ions. After washing, the solution was tested until the pH was neutral (usually pH~7). To avoid the phase transformation of the iron oxide [10], the sample is dried by incubating in an oven at 40 °C for 48 h.

2.3. Carbonization

A crucible alumina cup containing the as-prepared sample was placed in the central region of a furnace, Model 59256-P-Com Lindberg. The dry sample was carbonized at low heating temperature of 200 °C at a constant heating of 5 °C min^{-1}. The system was maintained at this temperature for 12 h, and subsequently cooled down to room temperature at the rate of 5 °C min^{-1}. The black precipitate powder was collected and dispersed in a solution by using an ultrasonic bath (ca. 5 min).

2.4. General characterization

The thermal properties of the dry sample were measured by using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) with the TA instrument-2960 SDT simultaneous analyzer. The sample was placed into an alumina crucible for TGA/DSC and heated from 30 to 700 °C with increasing temperature rate of 10 °C min^{-1} under a flowing argon gas at 20 $cm^3\,min^{-1}$. Scanning electron microscopy (SEM) was carried out by employing a JEOL JSM 6340F field emission scanning electron microscope (FESEM) by accelerating at a voltage of 5.0 kV. The sample was mounted on a Cu grid for SEM measurement and coated with gold to minimize charging. Transmission electron microscopy (TEM) was performed by using a Philip Tecnia G2 sphera transmission electron microscope with an acceleration voltage of 80.0 kV. The sample preparations for the TEM measurement were prepared by dispersing a drop of the sample solution on Formvar-coated Cu grids. The crystalline phase of samples was characterized by using XRD. A D8 Advance Bruker Analytic X-ray diffractometer was operated, the CuKα radiation at the wavelength of 0.15650 nm using 40 mA and 40 kV. The diffraction pattern over the range of 30–70° 2θ was recorded with the scan speed of 2 min/step and the scan rate of 0.015 2θ/s. Meanwhile, the coercivities (H_c), the remanent magnetizations (M_r) and the saturation magnetizations (M_s) of the as-prepared iron oxide nanoparticles were performed by using the vibrating sample magnetometer (VSM) and superconducting quantum interference device (SQUID) measurement (Lakeshore, Model 4500). The magnetic parameters (H_c, M_r and M_s) of each sample were determined from the hysteresis loops produced by the VSM. The saturation magnetization was reached at an applied field of 10 kOe. The magnetization data as a function of the applied field were plotted.

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3. Results and discussion

3.1. Thermal analysis

The thermal properties of the iron oxide samples were examined by TGA and DSC (see Fig. 1). The major transition due to the weight loss occurs over a narrow temperature range from 282.4 to 315.8 °C. The weight loss of the sample was 67.50% from the initial amount. Meanwhile, the DSC scan shows two endothermic peaks around 58.9 and 283.3 °C, respectively. The first peak is due to the release of the absorbed water of dehydration and the second peak is obtained from a phase transition of the iron oxide change. The main endothermic effect results from the combustion process of organic compounds in the as-prepared iron oxide consuming energy at 47.8 J/g and this may involve the oxidation, in the presence of oxygen, at high temperature in the TGA experiment. Magnetite (Fe_3O_4) can be easily oxidized to maghemite (γ-Fe_2O_3), which can be further transformed into hematite (α-Fe_2O_3) at high temperature [1], which is reported by Mikhaylova [11] and Wang [12]. No weight change was observed at temperature above 400 °C.

3.2. Electron microscopy (EM) analysis

The surface morphology of the as-prepared iron oxide crystals stabilized by mung bean starch vermicelli template was examined by the FESEM measurement. The FESEM micrograph shows a large quantity of formation of nanoscale iron oxide particles of spherical nanoclusters in shape (see Fig. 2). It was clear that spherical nanoclusters with a diameter of about 200–300 nm were formed by the assembly of many small iron oxide nanoparticles. The transmission electron microscopy (TEM) was further used to examine the morphology (see Fig. 3). After using an ultrasonic treatment to disperse the as-prepared samples, the TEM micrographs exhibit a region of iron oxide nanoclusters, which was found to be similar to the FESEM

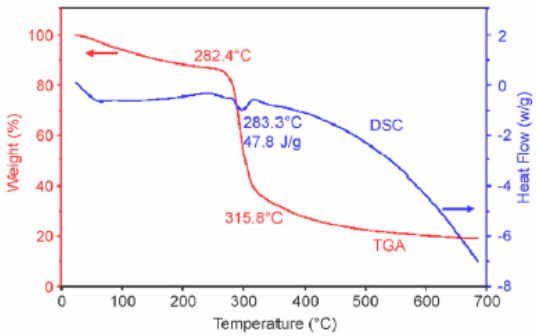

Fig. 1. Thermal analysis by using thermal gravimetric-differential scanning calorimetric (TGA-DSC) analysis of mung bean vermicelli deposited with the as-prepared magnetite (Fe_3O_4) nanoparticles.

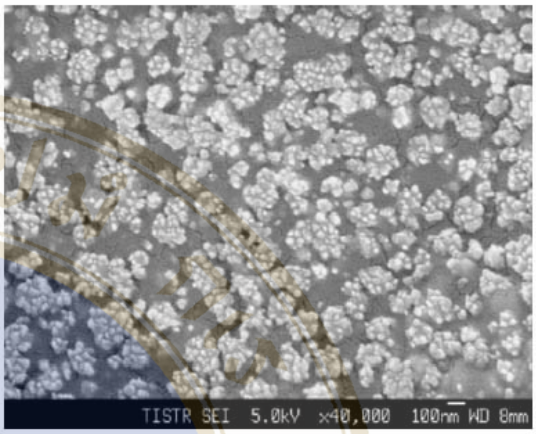

Fig. 2. The representative FESEM image of the as-synthesized iron oxide nanoparticles stabilized by mung bean vermicelli template.

results. Interestingly, it can be seen that wire-like nanoclusters with several hundred nanometers in distance were organized.

3.3. Powder X-ray diffraction (XRD) analysis

The characterization of the crystalline nature of iron oxide was carried out by using XRD analysis, which is essential for identification of the product [1,10]. The crystal structure and phase composition of the as-prepared samples was achieved by investigating the XRD patterns before and after carbonization (see Fig. 4). Before the carbonization process, the position and relative intensity of diffraction peaks from the XRD patterns of the as-prepared samples closely match with that of Fe_3O_4 powder diffraction data [1]. All peaks can be indexed closely to the cubic Fe_3O_4 nanoparticles, which are consistent with the data in the standard card (JCPDS file no. 19-629). The crystallite size of the as-prepared magnetic iron oxide nanoparticles can be calculated from the width of the representative peaks of the XRD patterns by using the Scherer's equation [13]

$$d = \frac{0.9\lambda}{B_\theta \cos \theta_B},$$

where B_θ is the width of the peak at half height and λ is the wavelength of the X-rays. θ_B is the Bragg angle. From the XRD results, the crystallite size of Fe_3O_4 nanoparticles is 14.8 nm after the carbonization. It is similar to Sun [8] who reported that high temperature reaction of the iron oxide could make smaller Fe_3O_4 nanoparticles from the larger ones.

3.4. Vibrating sample magnetometry (VSM) analysis

The saturation magnetizations were derived from the VSM measurement at room temperature by cycling the

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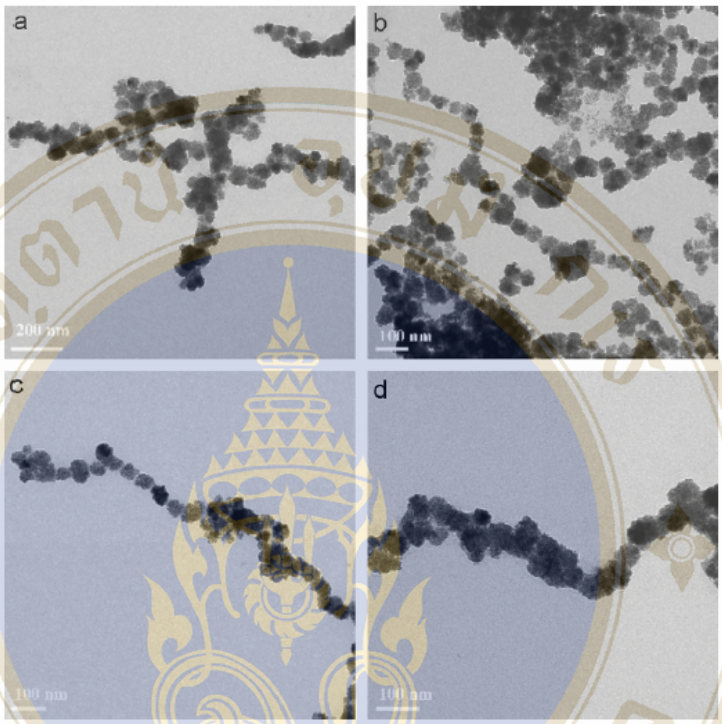

Fig. 3. Representative TEM images showing a region of a self-assembled chain of the as-prepared Fe_3O_4 nanoparticles. The bars of (a), (b), (c) and (d) correspond to 200, 100, 100 and 100 nm, respectively.

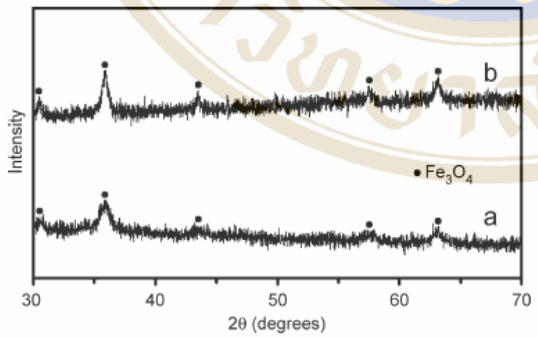

Fig. 4. XRD patterns of as-prepared iron oxide nanoparticles (a) before and (b) after carbonization. The XRD peaks for Fe_3O_4 were drawn from the JCPDS file 19-629.

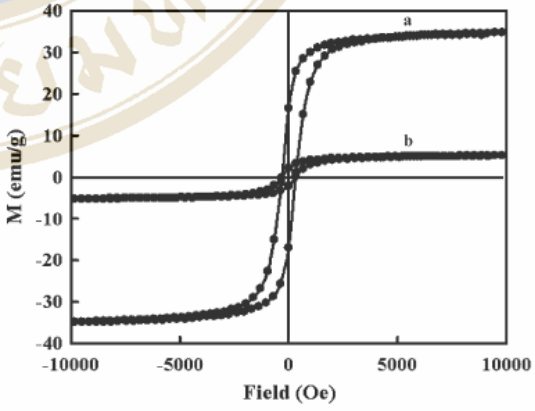

Fig. 5. Hysteresis loops at room temperature for the as-prepared iron oxide nanoparticles (a) before and (b) after carbonization.

magnetic field between −10 and 10 kOe (see Fig. 5). The saturation magnetization of the as-prepared iron oxide derived from pH 12 before carbonization was 34.8 emu/g, which is quite similar to the result reported by Wang [12]. In addition, the saturation magnetization of the final powder after carbonization was dramatically decreased to 5.1 emu/g. The saturation magnetization decreases when the crystallite size of nanoparticles is reduced as illustrated in the XRD analysis. In this case, the heating process may result to the major cracking of iron oxide nanoclusters to

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iron oxide nanoparticles [14,15]. The magnetic iron oxide nanoparticles stabilized by the organic structure of starch vermicelli template was carbonized in air into black carbon, which could form the magnetic iron oxide/carbon nanocomposites. This can be also inferred that the final product after thermal decomposition was magnetically softer. The results obtained here were found to be similar to Xiong and coworkers who recently reported the magnetic properties of iron oxide/carbon nanocomposites through the formation of the polymer with magnetic iron oxide nanoparticles by heating at different temperatures to carbonize the carbon matrix [16]. They suggested that the carbonization of magnetic iron oxide nanoparticles stabilized by carbon matrix to form iron oxide/carbon nanocomposites could result in the reduction of the magnetic properties of iron oxide nanoparticles. Therefore, this is commonly employed for the reduction of magnetic properties of magnetic iron oxide nanoparticles to obtain various magnetic nanoparticles/carbon composites by the carbonization process or pyrolysis method. In addition, the coercivity of the sample, after the carbonization remains unchanged at 298 Oe. The research findings here indicate that the carbonization can be attributed to make nanoclusters to nanoparticles. This may be used as component vehicles for magnetic field-directed drug delivery of biomagnetic applications as the carbonization process can reduce the strength of the magnetic field required to obtain the magnetic behavior.

4. Conclusion

In summary, we have demonstrated a novel method for synthesizing magnetic iron oxide nanoparticles by using transparent starch vermicelli template as a new stabilizing agent. The magnetite nanoparticles can be synthesized by using the precipitation of magnetic iron oxide nanoparticles by a wet chemical method. The morphology of the as-prepared iron oxide deposited on the surface of the vermicelli was the spherical nanoclusters in the form with a diameter of about 200–300 nm. The magnetic behavior of the as-prepared products is useful for a variety of biomedical applications.

Acknowledgments

This research was supported by the Center for Innovation in Chemistry: Postgraduate Education and Research Program in Chemistry (PERCH-CIC), the Thailand Research Fund (TRF), the Thailand Toray Science Foundation (TTSF) and Faculty of Science, Mahidol University. We thank Thailand Institute of Scientific and Technological Research (TISTR) for the FESEM measurement and the Center of Nanoimaging (CNI) at the Faculty of Science, Mahidol University for the TEM measurement.

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CHAPTER I
INTRODUCTION

This chapter is an introduction to the thesis which comprises (i) the world of nanotechnology involving nanostructures and the fabrication of nanomaterials, and (ii) the experimental design for enhancing students' learning in science education. The first part of the introduction in this research study begins with the world of nanotechnology through nanochemistry followed by the nanoparticles of iron oxide, gold and silver, respectively, and the significance of starch as a green material. Furthermore, the second part introduces the experimental design of chemistry laboratories to develop students' conceptual understanding of the nanotechnology experiment and chemical kinetics in the context of an inquiry-based learning. There are the background and justification for the practical class followed by the significance and context of the study in chemistry education.

1.1 The world of nanotechnology

In Greek, *nano* means dwarf, however in science *nano* means 1 billionth or $1x10^{-9}$. So if you are talking about 1 billionth of a meter, you are talking a nanometer. A nanosecond, one billionth of a second, is incredibly fast. A beam of light travels about one foot in a nanosecond.

After considering the current interest in nanotechnology, it deals with small structures or small-size materials, and seems appropriate to place the nanoworld in the context of the basic sciences. The prefix "nano" means one billionth. Therefore, one nanometer abbreviated 1 nm is one billionths of a meter, or 10^{-9} m. To get a sense of nanoscale, a human hair is about 50,000 nm across. The smallest seeable things with the human eyes are around 10,000 nm across. Therefore, in ten hydrogen atoms which make up to 1 nm, it is very small indeed in which the human eyes cannot observe. In

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general, nanoscale science here refers to a typically ranging dimension roughly between 1 and 100 nm [1].

Table 1.1 shows the prefixes used to describe metric system units. Nanoscale particles are smaller than can be seen. Scientists discovered that at the nanoscale, size matters. Nanoparticles exist at the size of single atoms, which are about 0.1 nm wide. So, 1 nm is almost equal to 10 hydrogen atoms, stretched end to end. (Remember that hydrogen is the smallest atom.)

Table 1.1 Most scientific data is reported in terms of metric measurements

Metric Prefix	**Multiples of Ten**	**Number**	**Name**
exa	10^{18}	1,000,000,000,000,000,000	quintillion
peta	10^{15}	1,000,000,000,000,000	quadrillion
tera	10^{12}	1,000,000,000,000	trillion
giga	10^{9}	1,000,000,000	billion
mega	10^{6}	1,000,000	million
kilo	10^{3}	1000	thousand
	10^{0}	1	
milli	10^{-3}	1/1000	thousandth
micro	10^{-6}	1/1,000,000	millionth
nano	10^{-9}	1/1,000,000,000	billionth
pico	10^{-12}	1/1,000,000,000,000	trillionth
femto	10^{-15}	1/1,000,000,000,000,000	quadrillionth
atto	10^{-18}	1/1,000,000,000,000,000,000	quintillionth

Because of the properties of nanomaterials, size matters can be uniquely different from the properties of materials in bigger bulk forms. There are two reasons for the differences in a material's nanoscale behavior. First, nanoparticles have a much great surface are per unit volume that is, a huge of metal, for example, has

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much more surface area when broken down into tiny particles than it does when whole. Since chemistry of solids occurs at these surfaces, more surfaces mean increased reactivity. Second, the smaller the particles get, the greater the changes in the particles' magnetic, optical, and electrical properties [2].

In contemporary, the particles of nanometer sizes began to attract scientists in different fields of science in the last 15-20 years. Materials in the micrometer scale mostly exhibit physical properties in the same as that of the bulk form. However, materials in the nanometer scale may exhibit these properties in distinctively different from that of the bulk materials. It is important to understand that the nanomaterials are not just small, but it is a special kind of small things. For example, a cube of gold that is 3 feet of each side. The cube of gold is now sliced to produce 8 little cubes, each 18 inches (approximately 50 mm) in a side. The properties of each of the 8 smaller cubes are exactly the same as the properties of the big one. Each will still be yellow, shiny and heavy. When cutting gold into the nanoscale size, everything will change including physical and chemical properties, such as color and melting point. The step cutting required to get the gold cube down to the nanoscale is called nanofabrication, or nanoscale manufacturing [3].

The important aspects are synthesis, physical properties, and chemical properties, but the most important of these at time is synthesis or as-called fabrication. The nanoparticles of interest are almost always prepared in the laboratory [4]. In the nanotechnology research, there are two methods to fabricate the nano-sized materials. Starting with a bulk form, it is cut down to the nanoscale. This kind is called *top-down nanofabrication* because we start from a large structure to make it smaller. In contrast, starting with individual atoms and building up to a nanostructure is called *bottom-up nanofabrication*. As mentioned above, the tiny gold nanostructures are called *quantum dots* or *nanodots* because they are dot-shape and have diameters at the nanoscale. This effect coupled with structural changes with size variation can lead to the chemical and physical properties, depending on size. Indeed, it has now been demonstrated that a host of properties depend on the size of such nanoscale particles, including magnetic properties, optical properties, melting points, specific heats, and

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surface reactivity. Furthermore, when such ultrafine particles are consolidated into macroscale solids, these bulk materials sometimes exhibit new properties [5].

It is accepted that nowadays a hung new field of science has been born. Nanotechnology research is now the study of the fundamental understanding and the technological advances resulting from new physical, chemical and biological properties of molecules and structures in that scale as described. Especially, the nanochemistry plays an important role in the field of nanoscience.

1.2 Approach to nanochemistry

In fact, nanoscience comprises closely interrelated concepts of chemistry, physics, and biology, which are aimed at the development of new fundamental knowledge. In the past 10-15 years, the progress in nanoscience has been largely associated with the elaboration of new methods for synthesizing, studying and modifying nanoparticles, and nanostructures. The extensive and fundamental development of these problems has been often determined by nanochemistry [6]. In turn, nanochemistry has two aspects. One is associated with gaining insight into peculiarities of chemical properties and the reactivity of particles comprising a small number of atoms, which lays new foundation of this science. Another aspect consists of applications of nanochemistry the synthesis, modification, and stabilization of individual nanoparticles and also for their direct self-assembling to give more complex nanostructures [7]. Moreover, the possibility of changing the properties of the synthesized structures by regulating the sizes and shapes of original nanoparticles deserves attention. Many aspects of synthesis, physiochemical properties, and self-assemble have been reviewed.

In nanochemistry which is in a stage of the rapid development, many questions associated with definitions and terms still arise [2]. The exact difference between such terms “cluster”, “nanoparticle”, and “quantum dot” has not yet been formulated in literature. The terms of “cluster” is largely used for particles that include small number of atoms, while the term of “nanoparticle” is applied for larger

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atomic aggregates, usually when describing the properties of metals and carbon. As a rule, the term “quantum dot” concerns semiconductor particles and the properties of which depend on quantum limitations on charge carriers and excitations [6]. In this research, no special significance will be attached to definitions, and the terms of “cluster” and “nanoparticle” will be considered as interchangeable.

Nanoparticles and metal clusters represent an important state of condensed matter. Such systems display many peculiarities, and physical and chemical properties that are never observed earlier. Nanoparticles may be considered as intermediate formations, which are limited by individual atoms on the one hand, the solid phase on the other. Thus, nanoparticles can be defined as entities measuring from 1 to 10 nm and built of atoms of one or several elements [4]. Presumably, they represent closely packed particles of random shapes with a sort of structural organization. One of directions of nanoscience deals with various properties of individual nanoparticles. Another direction is devoted to study the arrangement of atom within a structure formed by nanoparticles. Moreover, the stability individual part of this nanostructure can be determined by variations by kinetic and thermodynamic factors. Thus, the nanosystem are characterized the presence of various fluctuations [3].

Here again, one is up against a large number of different terms, such as nanostructures [5] or nanocomposites [8], which designate formulation built of individual, separate nanoparticles. Fore example, nanostructures can be defined as an aggregate of nanoparticles of definite sizes, which is characterized by the presence of functional bonds. In the reactions with other chemical substances, such limited-volume systems can be considered as nanoreactors. Moreover, nanocomposites [8] represent systems where nanoparticles are packed together to form a macroscopic sample, in which interactions between particles become strong, masking the properties of individual particles. Fore every type of interaction, it is important to know the properties of a sample change with its size, its shape, and its organization.

Nowadays, the attention of many scientists is focused on the development of new methods for the synthesis and stabilization of metal oxides or metal

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nanoparticles. The preparation of nanomaterials is a great challenge in the fields of synthetic chemistry and materials science because of the unusual properties that differentiate them from the bulk. The science of metal nanoparticles is well established and represents a vast body of knowledge of both fundamental and applied aspects of the subject [5, 9]. In this research, we consider the synthesis of metal oxides especially magnetic iron oxide, gold nanoparticles, and silver nanoparticles, respectively. The wide application of these nanoparticles may stem from their simplicity and availability. Furthermore, metallic nanoparticles are most suitable as nanoscale supports for binding biological materials to develop next-generation superstructures. It is noted that the ability to handle gold and silver nanoparticles like stable chemical compounds may greatly facilitates the study of their fascinating size-dependent properties to develop new applications. The introduction to each topic can be briefly discussed the following details.

1.3 Iron oxides

Metal oxides [10] belong to a class of widely used applications, such as catalysts. They can exhibit acidic and basic properties, which make them appropriate systems to be used as supports for highly dispersed metal catalysts or precursor of a metal sulfide, or chloride. Simple metal oxides range from essentially ionic compounds with electropositive elements to covalent compounds with nonmetals. However, taking into account the large variety of metal oxides, the principle objective of this research is to examine only the magnetic iron oxide that is attractive from magnetic properties. The interaction of the transition metal oxide with surface of the appropriate carrier or stabilization may develop well dispersed structure of this oxide. The crystal structure, composition, acid-base character are major ingredients of the chemistry investigated in this work. A new approach to the preparation of magnetic iron oxide is also included.

It is well known that metal oxides constitute a class of inorganic materials [11, 12] that have a wide range of properties and are therefore used in many technological applications, such as magnetism, dielectrics, superconductivity, which

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are remarkably sensitive to their chemical compositions, crystal structures, and also the applied external fields. As with most materials, many of the properties of metal oxides depend strongly on the preparation procedure employed in their synthesis. During the life of these materials, the properties of surface and interface usually play an important role in the overall properties and behavior; this holds both preparation and use. This is why chemical and structural features of surface and interface must be analyzed carefully in order to maintain a permanent control over reactivity. However, the properties of many metal oxides [10] including iron oxides [13] are still poorly known in comparison with metals. Therefore, there is currently great interest in exploring iron oxides in this huge field of nanoscience.

Iron oxides [13] have served human for centuries. Since the red and yellow ochres were first used to help produce prehistoric painting in caves, the role of iron oxides has expended enormously. Their applications as pigment and ability to catalyze various chemical reactions, their role as the precursors of iron, and steel and their activity as absorbants in the ecosphere are just a few examples of the contribution of these compounds to the well-being of human.

Iron oxides are common compounds which are wide dispersed in nature and readily synthesized in the laboratory. They are present almost all of the different compartments of the global systems, such as atmosphere, pedosphere, biosphere, and hydrosphere and take part in the manifold interrelationships between these compartments (see Fig. 1.1).

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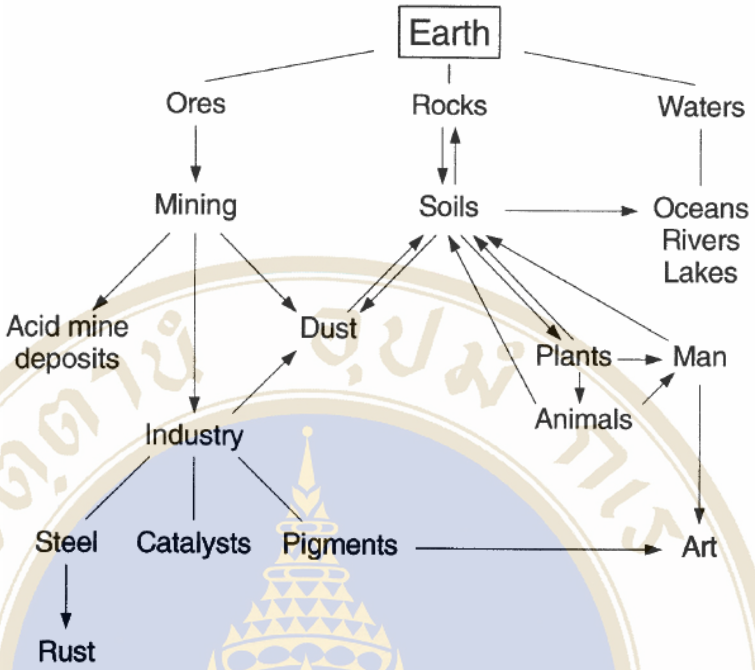

Figure 1.1 Iron oxides in the global system (adapted from the reference [13])

Initially, the formation of Fe^{3+} oxides involves aerobic weathering of magmatic rock (mainly on the earth's surface) in both terrestrial and marine environment. Such processes may involve mechanical transport by wind/water erosion from pedosphere into atmosphere or hydrosphere, or reductive dissolution followed by the migration of Fe^{2+} and oxidative reprecipitation in a new compartment. Man participates in these processes not only as a living organism, but also as a consumer of iron metal and iron oxides for various industrial purposes [14].

From the literature [13], there are 16 iron oxides (see Tab. 1.2). These compounds are oxides, hydroxides or oxide-hydroxides. The iron oxides are composed of Fe together with O and/or OH. In most compounds, iron is in the trivalent state, while three compounds - FeO, $Fe(OH)_2$, and Fe_3O_4 contain Fe^{2+}.

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Table 1.2 The iron oxides (adapted from the reference [13])

Oxide-hydroxides or hydroxides	**Oxides**
Goethite α-FeOOH	Hematite α-Fe_2O_3
Lepidocrocite γ-FeOOH	Magnetite Fe_3O_4 ($Fe^{2+}Fe^{3+}O_4$)
Akaganeite β-FeOOH	Maghemite γ-Fe_2O_3
Schwertmannite $Fe_{16}O_{16}(OH)_y(SO_4)_z \bullet nH_2O$	β-Fe_2O_3
δ-FeOOH	ε-Fe_2O_3
Feroxyhyte δ′-FeOOH	Wustite FeO
High pressure FeOOH	
Ferihydrite $Fe_5HO_8 \bullet 4H_2O$	
Bernalite $Fe(OH)_3$	
$Fe(OH)_2$	
Green rusts $Fe^{3+}{}_xFe^{2+}{}_y(OH)_{3x+2y-z}(A^-)_z$;	
$A^- = Cl^-, 1/2SO_4^{2-}$	

1.3.1 Types of magnetism of iron oxides

Diamagnetism substances involve a slight repulsion by a magnetic field. The magnetic susceptibility of a diamagnetic substance is small (10^{-6}), negative and independent of temperature. Iron oxides display additional types of magnetism.

Paramagnetic substances are attracted towards a magnetic field. Such substances possess unpaired electrons which are randomly oriented on different atoms. Each atom, ion or molecule of a paramagnetic substance can be visualized as a small magnet with its own, inherent magnetic moment. The application of a magnetic field causes (partial) alignment of these magnets parallel to the field. The magnetic susceptibility is positive and small (0 to 0.01). It varies with temperature and its behavior is described by the Curie-Weiss law,

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$$\chi_M = \frac{C_M}{T - T_C}$$

C_M and T_C are the Curie constant and the Curie temperature, respectively and T the temperature. The temperature dependence of χ_M is the result of two opposing tendencies; as the temperature rises, the increased alignment of the magnetic moment in the substance is opposed by the stronger thermal vibrations, hence, χ_M decreases.

Ferro- and *ferrimagnetic* substances are strongly attracted by a magnetic field. They contain unpaired electrons whose moments are, as a result of interactions between neighboring spins, at least partly aligned even in the absence of a magnetic field. The spin coupling energy is positive.

In a ferromagnetic substance, the alignment of the electron spins is parallel (see Fig. 1.2a). Such substances have an overall net magnetic moment, a large magnetic permeability and a large, positive susceptibility ($0.01\text{-}10^6$). In antiferromagnetic substance, the electron spins are of equal magnetic moment and are aligned in an antiparallel manner (see Fig. 1.2b). Such substances have zero overall magnetic moment, a positive permeability and a small positive susceptibility (0-0.1).

Ferrimagnetic substances consist, like antiferromagnetic materials of at least two interpenetrating sublactices and the alignment of spins is again antiparallel. In a ferrimagnetic substance, the different spins have however unequal moments, so that a ferrimagnetic material has a net magnetic moment (see Fig. 1.2c).

Ferromagnetic, antiferromagnetic and ferrimagnetic substances have a domain structures: only the particles in the range from 50 to 500 nm in size consist of a single domain. The spins within a domain are either parallel or antiparallel, but the different domains have different spin orientations. To eliminate the domains in a ferro- or antiferromagnetic substance, a sufficiently high magnetic field must be applied; as the applied magnetic field is increased, the spins in the domains become

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increasing aligned. At a high enough magnetic field, the saturation magnetization is reached, i.e. the spins of all the domains are parallel.

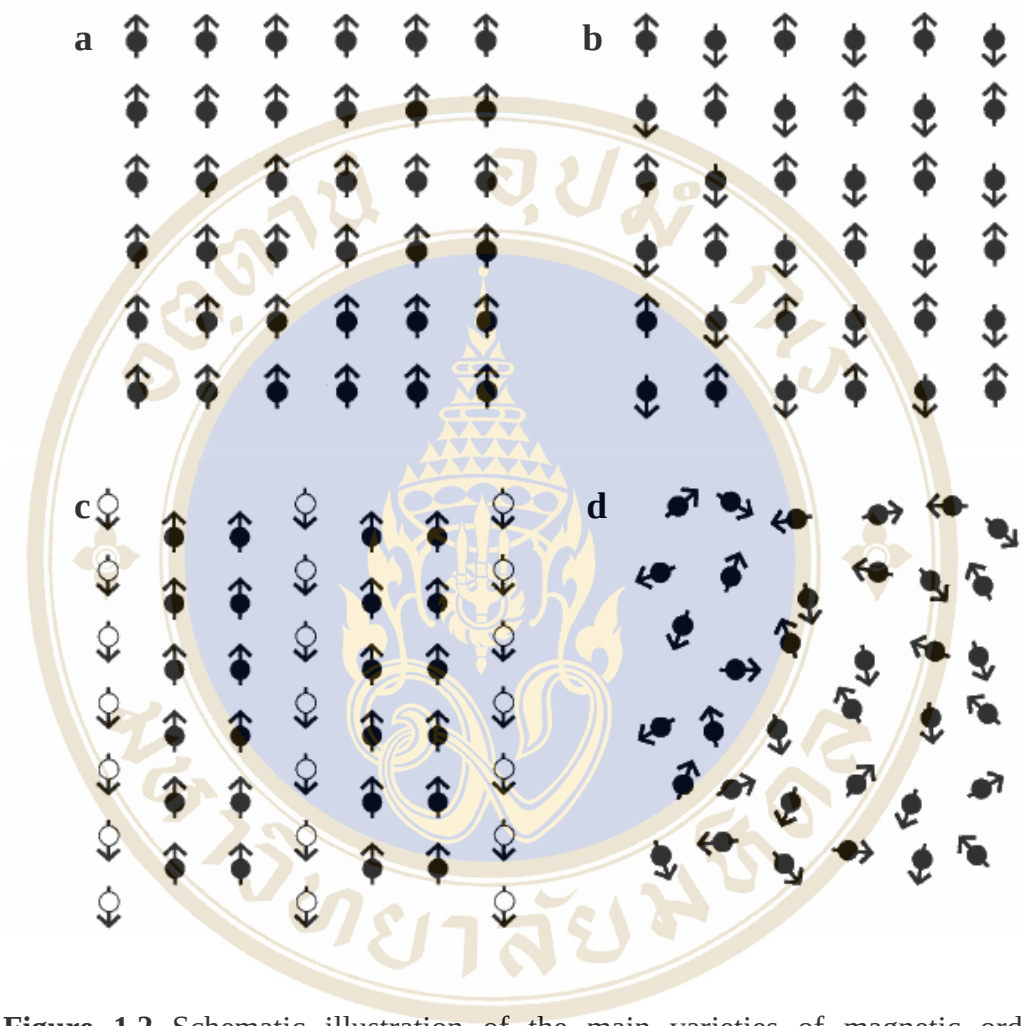

Figure 1.2 Schematic illustration of the main varieties of magnetic order: (a) ferromagnetism, (b) antiferromagnetism, (c) ferrimagnetism and (d) speromagnetism, where ○ and ● represent Fe atom and → is spin orientation (adapted from the reference [13])

A plot of magnetization against an applied magnetic field displays the hysteresis loop, the two branches of which correspond to the magnetization and demagnetization processes. The magnitude of the reverse field required to demagnetize a ferro- or ferrimagnetic substance is termed the *coercivity* [15]. Magnets with low coercivity are termed “soft”. “Hard” magnets have both a high coercivity

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and a high remanent magnetization; hence they are not easily demagnetized. Materials used in magnetic recording have coercivity ranging from 5.02 to 18.65 A m^{-1}.

If we apply an external field to a demagnetized (i.e., without a net magnetic moment) ferromagnetic sample, then a macroscopic magnetic moment $\mu = \int M dV$ will appear in this specimen. If we increase the field magnitude, then the magnitude of this moment also increases until at some certain field value H_{sat}, the so called the saturated state where all microscopic moments of a sample are aligned in the field direction will be achieved. The increase of the field intensity beyond this value will clearly not result in any further moment growth. The process described is termed the ‘‘magnetization process’’ which corresponds to the dependence of the total magnetic moment projection μ_Z on the field direction (which we will take as a positive direction of the z-axis) (see Fig. 1.3 below).

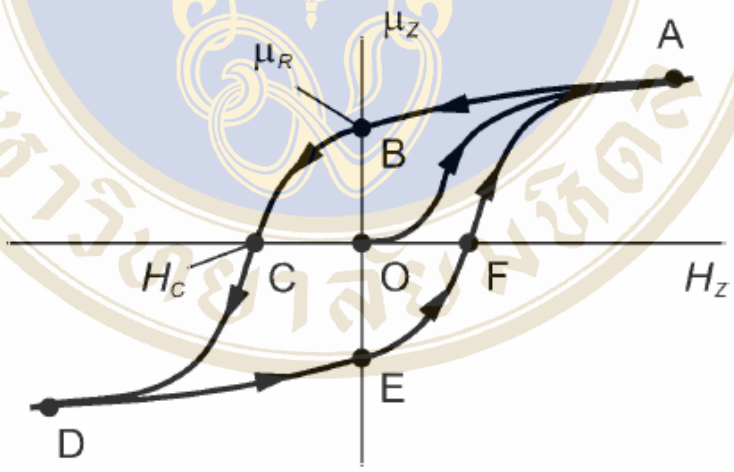

Figure 1.3 Typical hyteresis loop for magnetization; Initial curve O → A and hysteresis loop. A → B → C → D → E → F → A for ferromagnetic materials (adapted from the reference [15])

One of the most striking phenomena in ferromagnetism is the existence of the magnetization hysteresis or the irreversibility of the magnetization

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processes [15]. This means that if we decrease the field magnitude starting from the saturated sample state A, then the field dependence of the sample magnetic moment (shown by the curve AB) does not coincide with the corresponding dependence for the increasing field (curve OA). When we decrease the field to zero, there is still some substantial net magnetic moment left – this is called the remanent magnetic moment (μ_R). If we then reverse the field direction and again increase its magnitude, a certain (often quite large) nonzero value of this field Hc (called the coercive force) is needed to bring the net sample moment to zero (point C). Further increase of the field amplitude in the negative direction also leads to sample saturation in this direction (state D). Finally, by decreasing the field magnitude up to zero and then increasing it again in the positive direction, the magnetization of our sample will follow the curve D $\rightarrow$ E $\rightarrow$ F $\rightarrow$ A. So, during a complete field cycle $H_{sat} \rightarrow -H_{sat} \rightarrow H_{sat}$ the sample magnetic moment changes along the curve A $\rightarrow$ B $\rightarrow$ C $\rightarrow$ D $\rightarrow$ E $\rightarrow$ F $\rightarrow$ A, which is called the hysteresis loop.

Superparamagnetism arises as a result of magnetic anisotropy, i.e. the existence of preferred crystallographic directions along which the electron spins are most readily aligned and the substance most easily magnetized. The preferred direction for easy magnetization is along some crystallographic axis or set of axes, e.g. for magnetite, along the [111] directions. If sufficient energy is supplied, magnetism can be reversed along these axes. The time required for spin reversal, the relaxation time, τ depends on the height of the energy barrier between the forward and reverse spin states and the temperature, according to

$$\tau \propto \exp\left(\frac{K_{eff}.V}{kT}\right)$$

The height of the energy barrier between the forward and reverse states is the product of the particle volume, V, and the anisotropy constant K_{eff} (which is, to some extent, a function of particle size). Superparamagnetic relaxation occurs when thermal energy of the particles exceeds the activation energy barrier between

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the spin states and so allows rapid, spontaneous fluctuations between these states. The effect of these spin reversals is that the observed magnetic field is reduced or even absent.

Because the appearance of the superparamagnetic effect depends on the particle size on the anisotropy constant, it is often displayed at room temperature by iron oxides <10 nm in size, for example, soil iron oxides. Supeparamagnetic relaxation may be counteracted by lowering the temperature and thereby increasing τ. Supeparamagnetic particles will usually be ordered below a blocking temperature, T_B, which is:

$$T_B = \frac{K_{eff}.V}{25k}$$

Speromagnetism is a property of some amorphous or poorly ordered materials containing metal-O-metal bonds which, if the metal carries a magnetic moment, can support super-exchange relaxations (see Fig. 1.2d).

1.3.2 Magnetite (Fe_3O_4)

In this work, our interest of the iron oxide compounds is magnetite (Fe_3O_4), which can be described briefly below.

Magnetite (Fe_3O_4) is a black, ferrimagnetic mineral containing both Fe^{2+} and Fe^{3+} [10, 13]. The saturation magnetization at 300 K is in the range of 92-100 A m^2 Kg^{-1}. The structure is that of an inverse spinel. Magnetite is one of an important iron ore. It can be formed in various organisms in which it serves as an orientation aid. Other names of magnetite include black iron oxide, magnetic iron ore, $Fe^{3+,2+}$ oxide, loadstone (when natural polarity is present). Magnetite is attracted by a magnet but ordinarily, it cannot, itself, attract iron particles. It reflects the diversity of disciplines for which this mineral is of interest. The selected properties of magnetite are summarized in the Tab. 1.3 as shown below.

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Table 1.3 Melting point, boiling point, heat of fusion, decomposition, and vaporization of magnetite (adapted from the reference [13])

Melting point (°C)	Boiling point (°C)	Heat of fusion (kJ mol^{-1})	Heat of decomposition (kJ mol^{-1})	Heat of vaporization (kJ mol^{-1})
1583-1597	2623	138.16	605.0	298 at 2623 °C

Almost, all iron oxides, hydroxide, oxide-hydroxides are crystalline. The degree of the structural order and the crystal size are however variable, and depend on the conditions under which the crystals are formed. Especially, all iron oxides display a range of crystallinities except for ferrihydrite and Schwertmannite which are poorly crystalline. The main details of the structure of magnetite were established in 1915; this was one of the first mineral structures to which X-ray diffraction was applied. Magnetite has a face-centred cubic (fcc) unit cell based on 32 O^{2-} ions which are regularly cubic close pack along the [111] crystal direction [13]. The unit cell edge length is a = 0.839 nm. There are eight formula units per unit cell. Magnetite differs from other iron oxides in that it contains both divalent and trivalent iron. Its formula is written as $Y[XY]O_4$ where X = Fe^{2+}, and Y = Fe^{3+}. Eighth tetrahedral sites (T sites) are distributed between Fe^{2+} and Fe^{3+}, i.e., the trivalent ion occupies both tetrahedral and octahedral sites.

1.4 Gold nanoparticles

Colloidal gold solutions have a long history and were probably first described in the literature by Florentine glass maker and alchemist Antonio Neri in 1612. The first sound scientific studies of such materials were published much later by Michael Faraday in 1857. He developed new physical as well as wet-chemical preparative methods for colloidal gold, more importantly, was the first recognize that the gold colloidal suspension is present in metallic state. This conclusion was drawn from the observation that red or purple colored thin films of the dry material on a quartz surface could be compress mechanically so that they would exhibit the well-

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known optical properties of continuous thin films of metallic gold (thinly beaten gold leaf), which in transmittance appear green. Since no chemical reaction was involved in this process, Faraday concluded that the gold must always have present in the metallic state. With this discovery, he became the founder of the science of metal colloids, a field that continued to develop steadily, but at time of revolutionary changes in the physical sciences, appeared to be of but moderate interest [9].

Gold nanoparticles (AuNPs) [11, 16] are one of the most attractive nanoparticles in nanoparticle-based research due to their possess of the good properties, such as (i) identical size of all particles, (ii) identical shapes and morphology, (iii) identical chemical composition and crystal structure, and (iv) individually dispersed or monodispersed and no agglomeration. The gold nanoparticles are one of the most stable metal nanoparticles, and they present fascinating aspects such as their assembly of multiple types involving materials science, the behavior of individual particles, size-related electronic, magnetic and optical properties (quantum size effect), and their potential applications to catalysis and biology. Their promises are in the field of nanoscience as well as the bottom-up approach of nanofabrication, and they will be key materials and building block in the 21st century [17].

1.5 Silver nanoparticles

A large number of methods have been developed for the synthesis of metal nanoparticles since the discovery by Faraday on metal particles. Among them, silver nanoparticles (AgNPs) [11] are one of the most attractive noble metals in nanoparticle-based research due to their potential properties, such as catalysis. The synthesis of nanosized silver particles has attracted considerable interest in various fields of chemistry their unique optical, electrical, magnetic, and catalytic properties differing significantly from macroscopic metal phases and their potential applications in many areas including nanotechnology [6]. Especially, preparative methods have been widely applied to synthesize silver nanoparticles. For example, silver nanoparticles are prepared by the chemical reduction of a homogeneous solution

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containing silver salt and a large amount of stabilizers. The silver atoms and clusters are occurred from the formation of ultrafine particles in a solution. As known, silver has been known to exhibit strong toxicity to a wide range of microorganisms and has been used extensively in many antibacterial applications [16]. Therefore, the development of silver nanoparticles could be expected to open new avenues to fight and prevent disease.

1.6 Green chemistry

Frequently, many chemical reactions involve the use of reagents such as protecting groups and so-called catalysts that do not end up in the useful product. Organic solvents, often thought to be essential but sometimes not actually required at all, fall into this category. Some of these materials end up as waste and some are recovered, but in all cases valuable resources and energy are consumed that do not form part of the required product. These methods discussed spent some toxic chemicals which may cause problems to environment in synthetic nanoparticle processes. Therefore, the choice of the reducing agent has been a great concern in a green synthesis [18]. By using the concepts of green chemistry to integrate the chemistry and plant design, significant material savings can be made. When designing a process, there is currently growing interest in alternative sources of tools and materials that can target the specific bonds, and improve selectivity.

Chemistry has played, and continues to play, a fundamental role in almost every aspect of modern society, and, as the enormous populations in such China, India and the emerging nations demand western levels of healthcare, food, shelter, transport and consumer goods, so the demands on the chemicals industries will grow. The range of chemical products in today's society is enormous and these products make an invaluable contribution to the quality of our lives. Major surveys of public opinion throughout Europe in 2000 revealed that in no country were the majority of people favorably disposed towards the chemical industry [19]. This is the most critical for chemistry. We need to maintain a high level of interest and enthusiasm for chemistry at both secondary and tertiary education levels so that we can maintain the

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supply of a large number of highly intelligent, motivated and qualified young people for our industries, universities, schools and other walks of life.

Sustainable development is now accepted by governments, industry and the public as a necessary goal for achieving the desired combination of environmental, economic and societal objectives. The challenge for chemists and others is to develop new products, processes and services that achieve all the benefits of sustainable development. This requires a new approach whereby the materials and energy input to a process are minimized and thus utilized at maximum efficiency. The dispersion of harmful chemicals in the environment must be minimized or, preferably, completely eliminated [18]. We must maximize the use of renewable resources and extend the durability and recyclability of products, and all of this must be achieved in a way that provides economic benefit to the producer (to make the greener product and process economically attractive).

It has come to be recognized in past years that the science of chemistry is central to addressing the problems facing the environment. Therefore, there has an increased emphasis on the green chemistry in an area of education. In the late 1980s and early 1990s, there was a considerable amount of commitment to bring the concepts of waste reduction or elimination into the design of chemical processes and chemical productions [20]. Therefore, green and sustainable chemistry concerns the development of processes that result in more efficient chemical reactions that generate little waste and fewer environmental emissions than traditional chemical reactions do. The importance of green chemistry has been enhanced into the teaching of chemistry laboratory as a part of the current and future curriculum for young students. Therefore, the innovative approach to the experimental design of both clean chemical reactions and processes has been much considerable from colleagues applying twelve green chemistry principles [18] to a scientific laboratory as discussed following.

1. *Prevention*: It is better to prevent waste than to treat or clean up waste after it has been created.

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2. *Atom Economy*: Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product.

3. *Less Hazardous Chemical Syntheses*: Wherever practicable, synthetic methods should be designed to use and generate substances that possess little or no toxicity to human health and the environment.

4. *Designing Safer Chemicals*: Chemical products should be designed to affect their desired function while minimizing their toxicity.

5. *Safer Solvents and Auxiliaries*: The use of auxiliary substances (solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.

6. *Design for Energy Efficiency*: Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.

7. *Use of Renewable Feedstocks*: A raw material or feedstock should be renewable rather than depleting whenever technically and economically practicable.

8. *Reduce Derivatives*: Unnecessary derivatization (use of blocking groups, protection/deprotection, and temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.

9. *Catalysis*: Catalytic reagents (as selective as possible) are superior to stoichiometric reagents.

10. *Design for Degradation*: Chemical products should be designed so that at the end of their function they break down into innocuous degradation products and do not persist in the environment.

11. *Real-Time Analysis for Pollution Prevention*: Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.

12. *Inherently Safer Chemistry for Accident Prevention*: Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.

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1.7 Carbohydrates

There have been concerned using water-soluble materials for the synthesis of nanoparticles due to hydrophobicity of the capping agents. Among natural polymers, carbohydrates as the most common groups of natural products are construction materials of plants, such as cellulose, and they are also the source of energy and storage material in form of starch. Carbohydrates [21] are called *saccharides* (a term originally common for sugars), because the first-isolated compounds were sweet. As of 1999, the total annual biomass production is estimated to be 180 billion tons [22], which is divided into 75% carbohydrates, 20% lignin, and 5% other substances such as oils and fats, proteins, terpenoids, alkaloids, and nucleic acids (see Fig. 1.4). Of this biomass produced, 6 billion tons (3.5%) is presently used for human needs. The breakup of this amount is 3.7 billion tons for human food use; 2 billion tons of wood for energy use, paper, and construction needs; and 300 million tons to meet the human needs for nonfood raw materials such as clothing, detergents, and chemicals. The remainder of biomass produced is used in the natural ecosystems, namely as food for the wild animals, and the rest is lost as a result of the natural mineralization processes. The renewable raw materials mentioned here are almost obtained from agriculture and forestry.

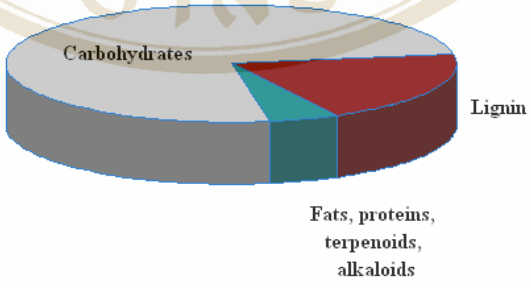

Figure 1.4 Renewable biomass 180 billion tons/year (adapted from reference [22])

The bulk of the annually renewable carbohydrate biomass is polysaccharides, yet their nonfood utilization is confined to the textile, paper, and coating industries, either as such or in the form of simple esters and ethers. Thus, carbohydrates are the major biofeedstocks from which to develop the industrially and economically viable

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organic chemicals that are to replace those derived from petrochemical sources. Organic commodity chemicals, however, are usually of low molecular weight, so they are more expediently obtained from low-molecular-weight carbohydrates than from polysaccharides [23]. Moreover, they are inexpensive, ton-scale accessible, and provide a resulting chemistry that is better worked out and more variable than that of their polymers. Also, carbohydrates are one of the most versatile materials available and it therefore is not surprising that carbohydrate-related technologies play a central role for the development of new products of food. Several of carbohydrates not only are utilized in food as components of ingredients or as additives, but also widely used to many technological applications [21], such as textiles, paper, pharmaceuticals, stabilizing agents, and biodegradable packing materials, due to their low cost, ton-scale availability and production from renewable resources.

The agricultural section of Thailand has significantly contributed the economic growth and the development of country in several past decades. It has not been recorded when mung bean (*Vigna radiata*) as one type of starch was first grown in Thailand, although it is believed that Thai people has grown and consumed mung bean for a long time [24]. The development of Thai mung bean production has been currently increasing in the terms of both volume and value of exports. Mung bean starch processing is in food manufacturing industries engaged in the production of vermicelli or transparent noodle, and the production varies with quality or grade of final products of vermicelli. Fore instance, the highest grade product is 100 % mung bean starch, whereas, the low grade is about 50 to 80 % mung bean starch mixed with other starch, such as potato and tapioca.

1.8 Synthesis and stabilization of nanoparticles

At present, there are many methods for synthesizing particles in various sizes. In principle, all methods for synthesizing nanoparticles can be however divided into two large groups [6]. The first group combines methods that allow preparation and studies of nanoparticles but do not help much in the development of new materials. They include condensation at superlow temperatures, certain versions of

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chemical, photochemical and radiation reduction, and laser-induced evaporation. On the other hand, the second group includes methods that allow preparation of nanomaterials and nanocomposites, based on nanoparticles. These are different versions of mechanochemical dispersion, condensation from the gas phase, plasmochemical synthesis, and certain other methods. Here, we will discuss them, for example, based on studies published, such as chemical reduction, reactions in micelles, emulsions, and dendrimers, and photochemical and radiation-chemical reduction, respectively.

1.8.1 Chemical reduction

Nowadays, the attention of many scientists is focused on the development of new methods for synthesizing and stabilizing of nanoparticles, including metals. Moreover, special attention is paid to monodispersed particles. Chemical reduction is used most extensively in the liquid phase including aqueous and nonaqueous media. Metals compounds are represented by their salts, while many compounds can serve as reducers, such as aluminohydrides, borohydrides, hypophosphites, formaldehyde, and salts of oxalic, citric and tartaric acids. The wide application of this method stems from its simplicity and availability [7].

As an example, Gold nanoparticles (AuNPs) (13 nm diameter) are prepared by the citrate reduction of $HAuCl_4$ gold precursor salt [16]. Briefly, all glassware used in the synthesis of AuNPs should be cleaned with aqua regia (3 parts HCl, 1 part HNO_3), rinsed with 18.2 mΩ nano-pore water, and then oven-dried prior to use. An aqueous solution of $HAuCl_4$ (1 mM, 500 mL) is brought to reflux while stirring, and then a solution of trisodium citrate (38.8 mM, 50 mL) is rapidly added such that the solution immediately changes color from pale yellow to deep red. After waiting 15 minutes, the mixture is allowed to cool to room temperature and subsequently filtered through a 0.45 μm filter. The colloidal gold is characterized using UV-Vis spectroscopy where the characteristic surface plasmon resonance band of monodisperse particle is located at 518-520 nm. Particles with more uniform size distributions have narrow absorption bands. Note that aggregated 13 nm colloid gold,

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as discussed above, displays a flattened and red-shifted absorption peak at approximately 570 nm.

Such organic compounds are preferred for the preparation of nanoparticles. They can perform stabilizing functions. Such solvents or surfactants sometimes play a key role in the synthesis of nanoparticles. They are bound to surface of growing nanocrystals via polar groups, form complexes with species in solutions, and control their chemical reactivity and diffusion to the surface of a growing particle. However, all mentioned processes depend on the temperature, surface energy of a nanocrystal, concentration of free particles in solution and their sizes, and the surface to volume ration of a particle. Stabilization processes accompany the preparation of different nanoparticles [6]. To prevent aggregation, active particle should not be allowed to contact one another. This is achieved by either presence of like charges or by steric repulsion of hydrophophic chains of the stabilizer.

1.8.2 Reactions in micelles, emulsions, and dendrimers

Synthesis of metal nanoparticles and their compounds involves using micelles, emulsions, and dendrimers as a sort of nanoreactors that allow synthesizing particles of definite sizes [3]. Fore example, nanoparticles of crystalline bismuth measuring less than 10 nm were obtained by reduction of aqueous solutions of bismuth salts. This process took place inside inverse micelles based on sodium diisooctylsulfosuccinate. Bismuth particles precipitated upon mixing and aging of the resulting mixture for several hours at room temperature (RT). The liquid phase was separated by centrifugation and dry deposit was redispersed in toluene or *n*-hexane.

At present, macromolecules can serve as matrices in synthesizing nanoparticles. In such method, the stabilizer molecules interact with surface of metal particle thus affecting their growth [3]. Fore example, reduction of copper (II) in the presence of poly-*N*-vinylpyrolidone produces particles with diameters of 7 ± 1.5 nm at a temperature of 11 °C. In recent years, much attention was drawn to monodispersed colloidal particles of polymeric materials due to their potential

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applications in biosensors, nanophotonics, porous membranes, and seed particles for core-shell and hollow structures. Fore example, polystyrene-based colloidal particles arrayed in porous structures were synthesized. Currently, for stabilization of nanoparticles, dendrimers based on polyamidoamines and their different modifications are used. A dendrimer represents a highly branched molecule, which includes a central nucleus, intermediate repetitive units, and terminal function groups. Dendirmers present a new type of macromolecules which combine the high molecular mass and low viscosity of their solutions, a three-dimensional molecular shape, and the presence of a special structure [6].

1.8.3 Photochemical and radiation-chemical reduction

Synthesis of metal nanoparticles influencing the chemical system by high energies is associated with the generation of highly active strong reducers like electrons, radicals, and excited species. Photochemical (photolysis) and radiation-chemical (radiolysis) reductions differ in energy [6]. Photosynthesis is characterized by energies below ~60 eV, whereas radiolysis uses energies 103–104 eV. Photochemical and radiation-chemical reduction methods have advantages over the chemical reduction method. Owing of the absence of impurities formed when chemical reducers are used, these methods produce nanoparticles of high purity. Moreover, Photochemical and radiation-chemical reduction make it possible to produce nanoparticles under solid-state conditions and at low temperature. Photochemical reduction in the solution is employed most frequently for synthesizing particles of noble metals. Such particles were obtained from solutions of corresponding salts in water, alcohols, and organic solvents.

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1.9 Teaching and learning in science laboratory

1.9.1 Teaching and learning in practical classes

A key purpose of science education is for students to come to understand science and how science works, by being engaged in doing science themselves. If they actually do some science, students may come to understand science, and to feel more confident with the science they are studying. In order to accomplish this, they should have 'personal experiences' with science concepts and gain scientific skills in hypothesizing, planning, designing, carrying out, and interpreting their own experiments in the laboratory [25].

Traditionally, most laboratory work at the tertiary level follows a 'cookbook' style, in which students are presented with aims, hypotheses, and detailed steps for carrying out the experiment. Observations and measurements are recorded, and questions are presented that help lead students to the required conclusion. Students may, or may not, learn something about the way scientists do things in such circumstances. However, science education research suggests that students can learn much more than practical science skills in the laboratory [26, 27]. According to the literature, students also should come to understand something of the nature of science.

The term *laboratory work* as used in the United States literature corresponds to the term *practical work* as used for the United Kingdom. The history of practical or laboratory work began in the nineteenth century. Practical science now has established a large and influential place in the teaching and learning of science in many countries. Practical work is an important part of the science curriculum in the United States and in Europe, particularly the United Kingdom, at both the secondary and tertiary level [28]. Practical laboratory activities in science are more common in England than in any other country, perhaps, because there are so many laboratories in England schools [26, 29]. Practical work when first introduced was seen as a significant and new conception with an important role in school science, and is now one of the key instruction processes at the secondary school and higher levels.

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The history of practical work began to be constructed at a number of institutions, King's College and University College London in 1845. Science education and the practical teaching of science within universities were argued for at the mid-nineteenth century. From the late 1950s, schools in the United Kingdom moved away from standard cookbook styles to practical work [28]. It is not difficult to criticize the standard practical experiments designed to lead students to the right way through prescribed worksheets. Such experiments do not lead to a good understanding of theory and do not allow students freedom to carry out science themselves. In the 1960s, the teaching in laboratory classes was much criticized and finally practical work began to be taught to students using scientific problems [29]. This signaled the beginning of practical work in school science.

A number of activities come under the umbrella term 'practical work' in science education. Woolnough [29] refers practical work as typical laboratory work where students encounter ideas and principles at first hand, that is, 'hands-on' science (p. 3.). Similarly, Millar, Marechal and Tiberghien [30] defined practical work as the teaching and learning activities in science which involve students at some point in handling or observing real objects or materials that they are studying (p. 36). Hegarty-Hazel [26] defined that student laboratory work is a form of practical work taking place in a purposely assigned environment where student engage in planned learning experiences, interact with materials to observe and understand the phenomena (p. 4). However, based on these sorts of definitions, the typical school laboratory is not a laboratory at all, because according to Osborn [31], students may attend practical classes, but instead of actually being engaged in hands-on work, they may instead watch teacher demonstrations (p. 40). In spite of some reservations, laboratory work is accepted by most authors as a key part of science education, and the science laboratory is thought to take a central role in the teaching and learning of science. This can be by way of encouragement of student interest, engaging them with experiences of concepts and developing their practical science abilities [32]. Practical work can be used to engage students in investigations, discoveries and problem-based solving [33], and the laboratory has been used for a variety of cognitive, practical and affective goals [28].

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1.9.2 Background and justification for the study

Developing a deep and long-term understanding of science concepts in students is an important aim of science teaching [34]. Most chemistry practical classes in science education, including those at Mahidol University - the context for this study, follow a 'cookbook style'. In laboratory instruction manuals, this cookbook approach is one of using the laboratory simply, with students following a detailed, pre-set laboratory procedure when conducting experiments. In rethinking laboratory activities, teachers often modify activities to take into account student prior knowledge and experiences, and seek to encourage student understanding [35]. If we are to shift students from cookbook approach towards inquiry-based teaching, teachers need to give students some instructions on how to conduct the experiments in their practical classes, but be much less directive [36]. Furthermore, students should not perceive that there is single way of approach their practical work, and a single, final, correct answer is to be attained. Inquiry-based practical classes are reported to improve the student mastery of science content, content retention, enhanced critical-thinking skills and laboratory skills, and to improve student attitudes towards chemistry when compared with traditional teaching methods. Thus, the science education suggests that teachers can enhance student learning and improve student attitude toward science when using inquiry-based teaching in practical classes [37]. This study thus investigates the use of inquiry-based learning for the teaching of chemical kinetics for first-year undergraduate science students in the chemistry laboratory at Mahidol University in Thailand**.**

1.9.3 Assumption and definition of terms

There are a number of assumptions that underpin this study of inquiry-based chemistry practical classes:

1. That students may develop sound science-inquiry skills if subjected to appealing learning experiences in their chemistry practical classes;

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2. That an inquiry-based approach to chemistry practical classes may enhance conceptual understanding for chemical kinetics;

3. The engagement of students in appropriate social interactions, student-student and student-teacher may serve to enhance student conceptual understanding for chemical kinetics; and,

4. The appealing classroom learning environment created by a teacher may be associated with improved student enjoyment and student learning outcomes in chemistry practical classes.

Likewise, a number of terms are used extensively throughout this thesis, and are defined as detailed below.

Scientific Inquiry: the diverse ways in which scientists study the natural world, and propose explanations based on the evidence derived from their work [38].

Conceptual Understanding: the ability of a learner to relate newly acquired concepts to prior existing knowledge in a substantive, non-arbitrary way [39].

Scientific Thinking: the distinctive thinking that involves both science content, and science process [40].

Perception: the process by which humans receive or extract certain information about their environment [41].

Group Work: the use of groups in education, and concerned with the practical aspects of starting, running, and finishing a social work group [42].

Practical Ability: designing and executing investigation and observation, recording data, and analyzing and interpreting results [32].

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1.9.4 Significance of the study

Practical classes are seen by many authors to serve a crucial role in the teaching and learning of science education at all levels including the tertiary level [26, 43]. As noted above, much of the work in practical classes at Mahidol University follows a familiar cookbook style. Students are expected by following the steps for carrying out the experiment, to gain understanding of what it is being done both in terms of content knowledge, and practical skills. Following on from this, it is considered that if students come to understand only chemistry content; they may not gain an understanding of scientific practice and scientific thinking. The significance from this research study seeks to show how the use of a scientific inquiry-based approach to chemistry practical classes enhances, or otherwise, student conceptual understanding of chemical kinetics for first year undergraduate science students. The study also explores the use of group work within this context. The findings from this thus will thus be of interest to tertiary chemistry teachers interested in the teaching of kinetics in practical; classes in Thailand and similar education contexts.

1.9.5 Context of the study

Thailand was known as Siam. Siam which has never been colonized was one of the few developing countries in the Southeast Asia. The structure of Thai education system was further refined in 1939, when five levels of education were created: pre-primary, primary, secondary, pre-university, and higher education. Based on the 1978/1981 curricula, an emphasis is placed on curricular programs that facilitate further advancement in scientist and technological fields and encourages various socioeconomic changes with country's transition from an agricultural to an agricultural-industrialized nation-state [44].

Education has always been regarded as an important means for the national development in Thailand, and has been controlled by the government through a number of planning instruments. The Ministry of Education (MOE) is responsible for the provision of the basic education nationwide [45], and the importance of

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science and technology is widely accepted. Thai is Thailand's official and national language. An emphasis is placed on curricular programs which facilitate further advancement in scientific and technological fields and encourage various socioeconomic changes with the country's transition from an agricultural to an agricultural-industrialized nation-state. Based on the 1990 revised National Curriculum, the basic Thai education system in Thailand is now four levels 'six-three-three-four': Students typically spend six years of primary, three years of lower secondary, three years of upper secondary education, and four years for higher education and students usually complete primary and secondary education by eighteen years of age [46]. Basic education places an emphasis on the learning process and the integration of knowledge and skills about social studies, science and technology, religion, culture, art, mathematics, and languages. Basic education institutions under the control of the Basic Education Commission (BEC) prescribe curricula relating to the needs of the community and the wider society. Compulsory education in Thailand is for nine years requiring children aged seven to enroll in basic education institutions until aged sixteen years with the exception of those who have already complete three-year lower secondary education.

One reality of schooling in Thailand is that the administrative system continues to be centralized in important respects: curriculum, budget, and personnel policies are decided in Bangkok. Similarly, the Office of the National Primary Education Commission (ONPEC) [46] maintains control over provincial, district, cluster, and school-level programs, through its authority to issue and enforce regulation. In Thailand, the primary education system has changed over recent decades. The basic research and implementation in developing education systems focused on a subset of policies to improve the quality of the teaching-learning and the management processes of schools in classroom [47]. The national curriculum, which is regarded as the educational standard, states that in any level of education, teaching-learning activities must emphasize 'learning to think, to do and to solve problems'. Teaching-learning activities are expected to address nine process skills:

1. Identifying the problem and the reason for it

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2. Thinking critically and creatively
3. Manipulating several alternatives
4. Evaluating and choosing among the alternatives
5. Defining the process of performance
6. Performing with cheerfulness
7. Evaluating the process of performance
8. Improving for better practice, and
9. Appraising with pride [48].

These process skills are emphasized as the features of the learning context along with some other processes: namely, science process skills; thinking processes; problem-solving processes; and, some processes in the affective and psychomotor domains [48]. In order to ensure the standard of education and to maintain national unity and identity, all schools in both the government and private sectors, are required to implement the national curriculum, and to follow guidelines in teaching and learning activities, learning materials, and the measurement of learning outcomes.

Higher education in Thailand is provided in universities, technical institutes, vocational college, or specialized-training institutions (i.e. nursing college, cadet schools and Buddhist universities). It is divided into two levels: non-degree level and degree level. Students seeking higher education may continue their education to either non-degree level or degree level. The non-degree level is mainly offered by colleges and institutes, public and private vocational colleges, as well as colleges of physical education, dramatic arts and fine arts. The major of courses offered are associated with vocational and teacher education and require two years of study [49].

A Bachelor degree normally requires four years of college-level course work. Undergraduate students leading to a Bachelor's degree are generally divided into two phase: a set of general course requirements in a broad range of subject areas and a concentrated or major program of study in one or more subject

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areas. Applicants to the Bachelor degree program in public universities are normally required to face an entrance examination and an interview. The Ministry of Education [50] plans to introduce a new system in 2006 called Central University Admission System (CUAS) to replace the existing entrance examination.

Another attempt in education reform emphasizes Thailand's need to adapt to challenges of surrounding globalization and internationalization because of the international education boom in Thailand [44]. Mahidol University originated from the first institution of higher learning in Thailand and, back in the late 1980s, opened an English-language International College. In 1889 the Medical School at Siriraj Hospital upgraded its teaching and service standards. The Rockefeller Foundation has also provided grants to the university for over a decade providing the Medical School at Siriraj Hospital with international recognition.

The Medical School at Siriraj Hospital has been combined into the University of Medical Science. In 1969, the University of Medical Science was given the new name 'Mahidol University'. Since then, Mahidol University has expanded into a comprehensive university and strived for academic excellence in a number of other fields, including the Faculty of Science. Nowadays, Mahidol University is one of the most prestigious universities in Thailand, recognized for its outstanding achievements in teaching, research and professional services. The faculty of science is responsible for teaching science to all first year undergraduate students of the university [51].

In the Faculty of Science [52] at Mahidol University, the Department of Chemistry offers a four-year program leading to the Bachelor of Science degree in Chemistry. The lecture courses are fully supported by laboratory classes. In addition to understanding the level of the science laboratories, the teacher reveals a number of suggestions in enhancing teaching and learning in the science laboratory experiences. By giving them the opportunity to experience practical work, students are actively engaged. By observing the students as they work, the teacher is able to gain important information about the student progress in skills, work habits,

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and attitudes. Most chemistry practical classes at Mahidol University are traditional in teaching approach, and as noted earlier science teaching is teacher-dominated and practical classes follow a cookbook style. The traditional views of teaching are that departmental staffs are to provide students information using a chalk-board, and to place strong emphasis on theoretical chemistry. Of particular relevance to this study, topics such as rates of reaction and kinetics are considered to be very important.

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CHAPTER II
OBJECTIVES

In scientific part, starch is one of the most widely used carbohydrate polymers. our interest here is that the utilization of mung bean starch vermicelli as template may bring advantageous possibilities to stabilize magnetic iron oxide, gold and silver nanoparticles because vermicelli surface of starch which is full of a plenty of polyhydroxylated functional groups may assist the precipitation of nanoparticles. In science education part, in past years, a teacher-centered approach played an important role in the educational system in Thailand. Science teachers in Thai education often concentrated on teaching theories rather than developing practical skills. Similarly most chemistry practical classes at Thai universities are traditional in teaching approach, meaning they are teacher-dominated and that practical classes follow a cookbook style. In an effort to change from a teacher-centered approach to learning, there is currently discussion in Thailand as how to change the teaching and learning approaches and strategies in order to facilitate the acquisition of the new types of knowledge.

2.1 Magnetic iron oxide nanostructures

Aim: The general purpose of this work is to prepare the soft magnetic iron oxide nanostructures for biological applications.

The objectives of this work are specified as:

- To prepare new inorganic/organic hybrid materials by fabrication of magnetic iron oxide nanoparticles stabilized by transparent mung bean vermicelli;

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- To carbonize the as-prepared magnetic iron oxide nanostructures stabilized by starch vermicelli into magnetic iron oxide/carbon nanocomposites at low temperature;

- To characterize the as-prepared sample before and after carbonization.

Scope: Nanometer-scale magnetic particles have been frequently synthesized for the nanomagnetism study due to a variety of potential applications in magnetism, catalysis, chemistry and biology. In order to achieve the preparation of the soft magnetic materials for applications, it is required to reduce the strength of the applied magnetic field to be as small as possible.

2.2 Gold and silver nanoparticles

2.2.1 Gold nanoparticles

Aim: The general purpose is that the helical structure of amylose or amylopectin containing glucose units could be utilized as nanoreactors for the fabrication of gold nanoparticles for potential applications**.**

The objectives of this work are specified as:

- To prepare new inorganic/organic hybrid materials by fabrication of gold nanoparticles stabilized by transparent mung bean vermicelli;

- To prepare size- and shape-controlled gold nanoparticles using transparent mung bean starch vermicelli;

- To characterize the as-prepared gold nanoparticles.

Scope: The starch transformation provides diverse structures and properties for numerous applications. In this study, mung bean starch vermicelli is of our interest due to higher percentage of amylose and its transparent structure so that the formation of color particles on the vermicelli can be readily observed. The scope of this work is

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to demonstrate a new approach for the preparation of uniform gold nanoparticles, using fine structure of transparent mung bean vermicelli as a template.

2.2.2 Silver nanoparticles

Aim: The general purpose is quite similar to that for gold nanoparticle synthesis. It would be interesting if amylose or amylopectin containing glucose units could be utilized as a template for the fabrication of silver nanoparticles for potential applications.

The objectives of this work are specified as:

- To prepare new inorganic/organic hybrid materials by fabrication of silver nanoparticles stabilized by transparent mung bean vermicelli;
- To prepare size- and shape-controlled silver nanoparticles and two colors of silver nanoparticles using transparent mung bean starch vermicelli;
- To characterize the as-prepared silver nanoparticles.

Scope: Among the noble metals, silver nanoparticles have been intensively investigated due to the exhibition of novel properties and technological application as advanced materials. The scope of this study is to demonstrate a new approach for the preparation of silver nanoparticles and dendritic silver nanoparticles, using fine structure of transparent mung bean vermicelli as a template.

2.3 Enhancing Thai student learning of chemical kinetics

Research questions:

This study sought to address the investigation of student learning of chemical kinetics by a means an inquiry-based chemistry laboratory based on the particular educational context, in this case Mahidol University in Thailand.

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The research questions for this inquiry are:

- Can an inquiry-based chemistry practical class enhance Thai students' learning of chemical kinetics?
- If so, how does an inquiry-based approach to chemistry practical classes enhance Thai students' conceptual understanding for chemical kinetics?
- Do Thai students enjoy an inquiry-based practical class more than their normal practical classes?

Research objectives:

The general purpose of the practical class reported in this work is to enhance the student understanding of kinetics by means of inquiry-based practical classes.

The research objectives are specified as:

- To investigate the use of an inquiry-based approach in practical chemistry classes for enhancing student conceptual understanding of chemical kinetics for first-year undergraduate science students;
- To evaluate the use of an inquiry-based approach in practical chemistry classes for enhancing student understanding of scientific thinking for first-year undergraduate science students;
- To evaluate student perceptions of the value of an inquiry-based approach in practical chemistry classes for first-year undergraduate science students.

Scope:

This study conceptualizes the scientific inquiry as the diverse ways in which students study scientific concepts, in this case about of the rate of a reaction and chemical kinetics, and purpose explanations based on the evidence obtained from their work in the laboratory. The participants in the group work are all first year undergraduate science students (at the age of 18-19 years) from the Faculty of

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Science, Mahidol University in Thailand. These students enrolled in a general chemistry laboratory course in the first semester. The course comprises 12 practical classes in which the chemical kinetics of acid-base reactions is one session. In general, the laboratory classes consist of practical experiments that complement the lecture material and the lectures are delivered before the related experiment. The practical classes overall try to teach basic practical chemistry techniques, including the use of pipette and burette, preparation of solutions, and redox chemistry. Data collection involved the use of quantitative instruments such as questionnaires. Qualitative data consisted of comprehensive student documentation, student discussion documented by writing, and the use of semi-structured interviews.

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CHAPTER III
LITERATURE REVIEW

This chapter presents the literature review for the thesis. It begins with the preparation of magnetic iron oxide nanoparticles, and then follows with gold and silver nanoparticles. The chapter ends with importance of the practical classes in science education, including the teaching and learning in practical classes and ways of developing the teaching and learning of chemical kinetics in practical classes.

3.1 Magnetic iron oxide nanoparticles

Iron oxides are commonly compounds, which are widespread in nature. Nowadays, there are sixteen iron oxides and these compounds are either in the form of oxides, hydroxides, or oxide-hydroxides, collected referred to the literature [13]. The preparation of iron oxide nanoparticles and their composite nanomaterials has attracted both fundamental and practical interest. As known, nanometer-scale magnetic particles have been frequently synthesized for studied of nanomagnetism because of a great potential for a variety of scientific and technological disciplines, such as magnetic, catalytic, chemical and biological applications [53-55]. Here, there are studies reported on the preparation of magnetic iron oxide nanoparticles.

In the past decade, several techniques for the formation of monodisperse magnetic iron oxide nanoparticles using different methods have attracted considerable interest. To use magnetic iron oxide nanoparticles for future highly sensitive magnetic nanodevices, several methods for synthesizing of magnetic iron oxide nanoparticles have been developed. Among these, the solution-phase synthesis by co-precipitating of Fe^{2+} and Fe^{3+} ions by a basic media has been generally used to be the most effective to prepare the highly uniform magnetite (Fe_3O_4) nanoparticles in size and shape [5, 6]. For example, Soo and coworkers [56] reported the synthesis of magnetite

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nanoparticles in aqueous solutions without any surfactant by co-precipitating of Fe^{2+} and Fe^{3+} ions in the presence of NaOH. Recently, an alternative method is that magnetite particles can be prepared by alkalinizing stoichiometric mixture of Fe^{2+} and Fe^{3+} with a diluted ammonium solution by partial oxidation of aqueous Fe^{2+} ions [57]. However this precipitation method is suitable for synthesizing of magnetic iron oxide nanomaterials, it is still difficult to control desired sizes and morphologies due to their aggregation.

In contrast, biological magnetite synthesis can be occurred in water at room temperature and close to neutral pH. Many laboratory studies on magnetite growth have focused exclusively on the use of magnetotactic bacteria and iron reducing bacteria, such as *Geobacter metallireducens* (a distant cousin of magnetotactic bacteria). In all studies to date, the disadvantage is that biosynthesis of magnetite was found to be extremely slow (often requiring 1 week) under strictly anaerobic conditions. Bharde and coworkers [58] discovered that the bacterium *Actinobacter* spp. is capable of magnetite synthesis by reaction with suitable aqueous iron precursors under fully aerobic conditions. The magnetite nanoparticles form extracellularly and show excellent magnetic properties. Magnetite is formed within 24 h by reaction of *Actinobacter* spp. with an aqueous potassium ferricyanide/ferrocyanide mixture under fully aerobic conditions, thus considerably simplifying the synthetic procedure. Faivre and coworkers [59] recently reported a cell-suspension assay for the growth-independent study of magnetite formation under highly controlled conditions. Magnetite formation could be induced in iron-starved nonmagnetic wild-type (WT) cells by the addition of either ferric citrate or ferrous ascorbate (see Fig. 3.1), indicating that the bacteria are capable of intracellular reduction or oxidation of extracellular iron to form the mixed-valence Fe_3O_4 crystals. Over time, the average particle dimensions increased from 18.1 to 31.5 nm, and the number of crystals per cell was from 16 to 30. After 6 h, formation of chains was complete.

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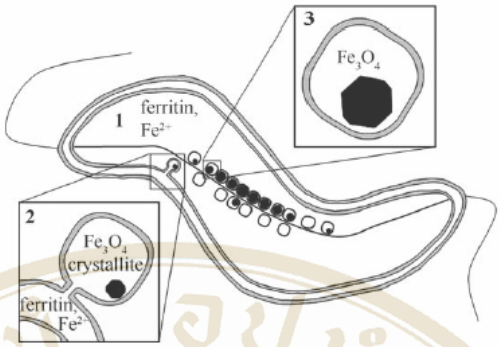

Figure 3.1 Model of iron uptake and magnetite formation mechanism. (1) A biochemical pool of iron is formed in the cells, composed of ferritin and Fe^{2+}. (2) Magnetite biomineralization proceeds first by transport of Fe^{2+} ions and ferritin into magnetosome vesicles where Fe^{2+} and Fe^{3+} ions coprecipitate. (3) Final magnetite growth occurs in magnetosomes (adapted from the reference [59])

Magnetic iron oxide nanoparticles coated with polymers have potential for applications in drug delivery, treatment of retinal detachment as magnetic resonance imaging (MRI) contrast agents, and in bio-catalysis, bio-separations, and high-gradient magnetic field separations. These colloidal dispersions, or ferrofluids, contain iron oxide nanoparticles coated with polymers to impart desirable surface properties. The high surface-to-volume ratio and good dispersibility of magnetic nanoparticles can also facilitate magnetic separations. For example, the nanoparticles were coated with triblock copolymers containing poly(ethylene oxide) (PEO) end blocks, and then the complexes were dispersed in water [60]. Adsorption of these hydrophilic triblock copolymers occurred via carboxylate anchor groups, and this led to the formation of a brush layer that sterically stabilized the magnetite particles in water (as shown in Fig. 3.2). Recently, Zhang and coworkers [61] reported that magnetite nanoparticle dispersions have been stabilized and complexed with carboxylate-functional block copolymers (see Fig. 3.3). The magnetite-copolymer complexes were near 10 nm in diameter, and the particles were superparamagnetic.

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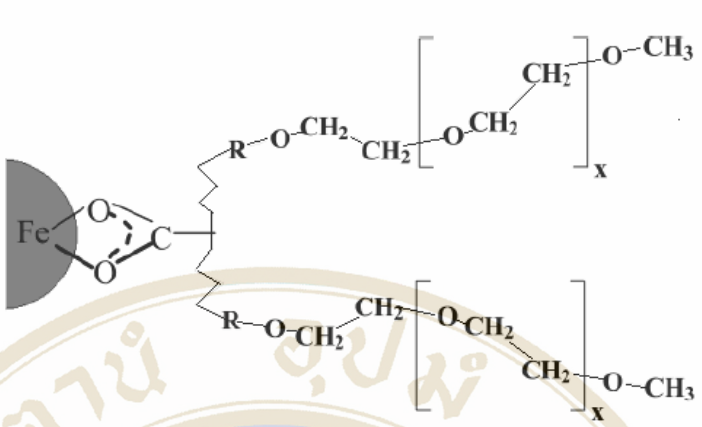

Figure 3.2 Central segment of the stabilizer anchors to the magnetite surface via the carboxylic acid groups while the hydrophilic tail blocks extend into water (adapted from the reference [60])

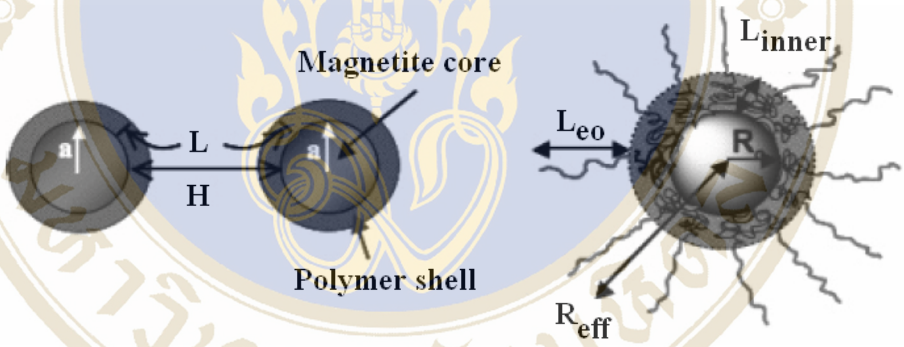

Figure 3.3 Schematic of two stabilized nanoparticles. The radius of the magnetite core utilized in the model was *R*c = 5 nm derived from TEM micrographs, the core surface-to-surface distance is *H*, and the polymer layer thickness is *L* = *L*inner + *L*eo (adapted from the reference [61])

An alternative method is that thermal decomposition has been extended to synthesize the highly-crystalline iron oxide nanoparticles. Preliminary to many possible applications is the development of techniques for organizing monodisperse particles on suitable substances. Among the materials investigated as a solid support for the layer of particles, many stabilizing agents with a long chain structure [62, 63] have been required to produce magnetite (Fe_3O_4) nanoparticles with the desired size.

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For example, a simple organic-phase synthesis can serve for the preparation of magnetite nanoparticles with sizes variable from 3 to 20 nm in diameter. Organic solution-phase decomposition of the iron precursor at high temperatures has been widely used in iron oxide nanoparticle synthesis. The synthesis has demonstrated that direct decomposition of $FeCup_3$, or decomposition of $Fe(CO)_5$ followed by oxidation, can lead to high quality monodisperse magnetic iron oxide nanoparticles. However the synthesis of monodisperse Fe_3O_4 nanoparticles with sizes below 20 nm has not yet been reported. Sun and Zeng [64] demonstrated that high-temperature (265 °C) reaction of iron (III) acetylacetonate, $Fe(acac)_3$, in phenyl ether in the presence of alcohol, oleic acid, and oleylamine can be used to make monodisperse magnetite nanoparticles (see Fig. 3.4).

Figure 3.4 Schematic representation of size-controlled synthesis of magnetite nanoparticles (adapted from the reference [64])

Sun and coworkers [63] recently reported that Fe_3O_4 nanoparticles and related MFe_2O_4 nanoparticles (with M = Co and Mn) with size tunable from 3 to 20 nm in diameter can be synthesized in the presence of 1,2-hexadecanediol, oleic acid, and oleylamine by varying reaction temperature or changing metal precursors. The nanoparticles can be dispersed into nonpolar or weakly polar hydrocarbon solvents, such as *n*-hexane or toluene (see Fig. 3.5). However, these compounds are highly reactive and potential environmental risks during the synthetic processes.

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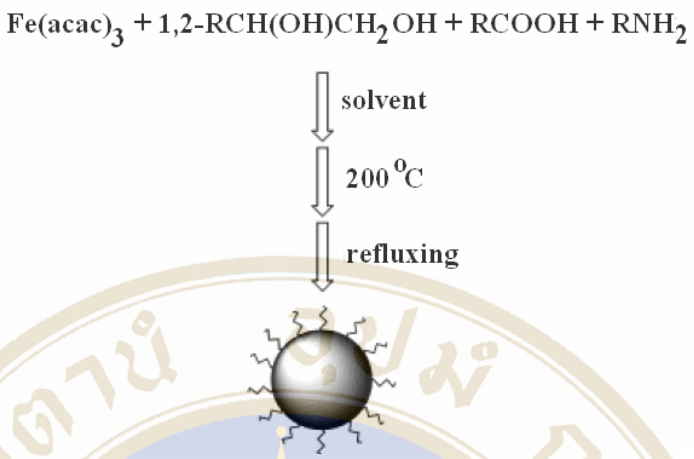

Figure 3.5 Schematic representation of monodisperse nanoparticles (adapted from the reference [63])

It has come to be recognized in past years that the science of chemistry is central to addressing the problems facing the environment. Therefore, there has an increased emphasis on the green chemistry. In the late 1980s and early 1990s, there was a considerable amount of commitment to bring the concepts of waste reduction or elimination into the design of chemical processes and chemical productions. Green and sustainable chemistry [19] concerns the development of processes that result in more efficient chemical reactions that generate little waste and fewer environmental emissions than traditional chemical reactions do. The importance of green chemistry has been enhanced into the chemistry laboratory as a part of the current and future development. Therefore, the innovative approach to the experimental design of both clean chemical reactions and processes has been much considerable from many researchers applying twelve green chemistry principles to a laboratory [18].

To make it friendly to environment, the water-soluble biomaterials have been used to be the stabilizing agent, such as phospholipids [65], dextran [66], and chitosan [67], in experimental procedures. Magnetic nanoparticles can be used in biomedical applications, where they facilitate laboratory diagnostics or in medical drug targeting. Moreover, they may be used for in *vivo* applications such as contrast agent for magnetic resonance imaging (MRI). For example, Giri and co-workers reported

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recently the preparation of uniform sized magnetite (Fe_3O_4) nanoparticles stabilized by using phospholipids [65]. The presence of phosphatidylcholine (PC) molecules controls the particle morphology and size of magnetite particles and their distribution by inhibiting growth. The presence of increasing concentration of PC during precipitation decreases the particle size as well as narrows down the particle size distribution. These surface-modified-magnetite nanoparticles within the liposomes may be suitable for hyperthermia applications.

The development of new biomaterials is still considerable a challenge for *vivo* applications. As known, starch is one of the most widely used carbohydrate polymers. For example, the magnetic particles should have a narrow size distribution and surface modified with biocompatible materials. Chitosan [68] and starch [69] polymer can be used the functional biocompatiblepolymer. For example, chitosan is a polyaminosaccharide with many significant biological (biodegradable, biocompatible, bioactive) and chemical properties (polycationic, hydrogel, contains reactive groups such as -OH and $-NH_2$).

Our interest here is that mung bean vermicelli starch used as template may bring advantageous possibilities to stabilize magnetic iron oxide nanoparticles, because vermicelli surface of starch which is present of a plenty of polyhydroxylated functional groups [69] may assist to form the precipitation of the iron oxide nanoparticles on the surface of vermicelli template, because starch is composed of repeating glucopyranosyl units: amylose and amylopectin. Moreover, the starch presents the strong hydrophilic and biodegradable behavior.

3.2 Gold and silver nanoparticles

Nanoscale-sized materials deal with extremely small structure materials in which their dimension of matters is at the range from nanometer to several hundred nanometers. The properties of the metal are different from that of bulk materials from the same atoms. Nanostructures of metals have been intensively studied in the past years using different chemical and physical methods by scientific and technological

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interest, due to their novel properties which are strongly influenced by size, shape, surface composition, spatial ordering, and interactions with surrounding environments [17, 70-74]. Among noble metallic nanoparticles, gold nanoparticles (AuNPs) [17, 74] and silver nanoparticles (AgNPs) [71, 72, 74] have been much of considerable interest because of their novel properties and potential applications. The traditional approach reported for the synthesis of metal nanoparticles is the reduction of metal ions from metal salts with a large excess of reductants, such as ascorbic acid, sodium borohydride or sodium citrate. This method described has been extremely successful in yielding noble metals.

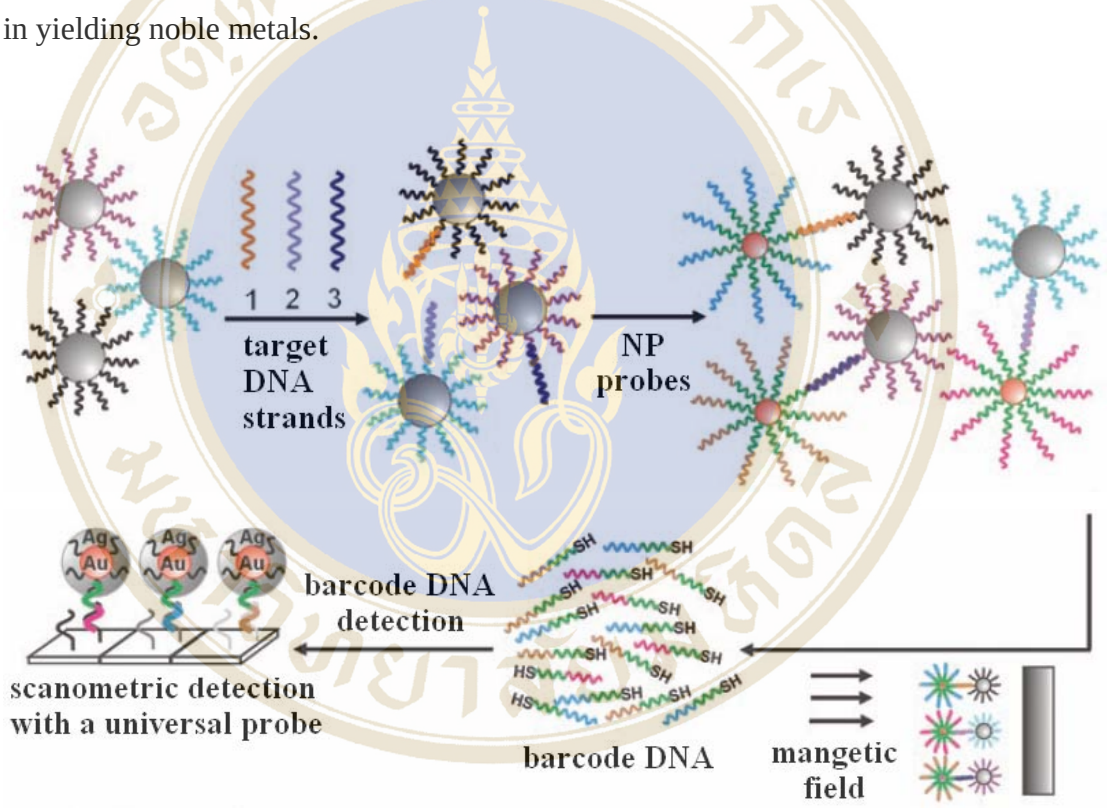

Figure 3.6 The multiplexed biobarcode assay for DNA detection of gold nanoparticles (adapted from the reference [75])

The uniform labeling and detection methodologies has led to the development of alternative approaches such as fluorophore-labeled dendrimers, fluorophore-encoded microspheres, optically encoded porous silicon photonic crystals, multimetal microrods, nanowires, and metal and semiconductor nanoparticle labels. The oligonucleotide-functionalized gold nanoparticles (AuNPs) have a

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demonstrated advantage in terms of sensitivity and selectivity over conventional probes in a variety of biodetection, such as DNA [76-79], polypeptides [80], and virus [81] for many applications in nanoelectronics, drug delivery, and catalysis. The biobarcode assay is based on AuNPs functionalized with a large number of oligonucleotide strands (the barcodes) and a corresponding recognition agent (an antibody in the case of protein detection and a portion of the barcode strand that can recognize the target of interest in the case of nucleic acid detection).

It has come to be recognized in several past years that the science of chemistry is central to addressing the problems facing the environment. Therefore, there has an increased emphasis on the green chemistry to bring the concepts of waste reduction or elimination into the design of chemical processes and chemical productions. Green and sustainable chemistry [18] concerns the development of processes that result in more efficient chemical reactions that generate little waste and fewer environmental emissions than traditional chemical reactions. Even though it is cheap and most widely used, it obtains less attention for the development of the construction of nanoarchitectures on the surface and embedded in the structure of starch. Several environmental friendly approaches have been shown by using polyaminosaccharide (i.e., chitosan) and polysaccharides (i.e., soluble starch and natural starch) as templates for the nanoparticles synthesis [82-85].

Tremendous efforts have been devoted toward developing new methods of preparing metal nanoparticles with different topologies. To prevent nanoparticles from aggregating during their synthesis, small organic molecules or polymers must be added to the synthesis medium as stabilizers. Natural polymers are also used to control the rate of the reduction process and thus modulate the shapes and sizes of the resultant particles. Such bioactive molecules and biomacromolecules have been used to synthesize “green” nanoparticles. Chitosan is the (β-1,4)-linked glucosamine derivative of the polysaccharide chitin and a biodegradable and biocompatible biopolymer [86]. Recently, a novel nanocomposites gel was prepared by neutralizing a designer nanocomposites solution of chitosan encapsulated gold nanoparticles formed by reducing *in situ* tetrachloroauric acid in chitosan [87]. The methods for

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preparation of the gel and immobilization of cells were simple and “green”. The nanocomposites gel showed improved immobilization capacity for cells and good biocompatibility for preserving the activity of immobilized living cells.

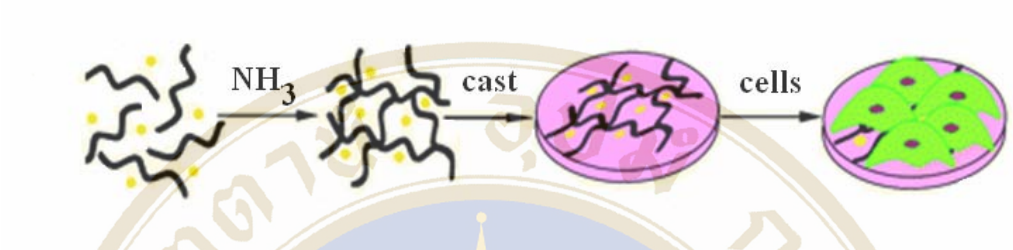

Figure 3.7 Schematic representation of gold nanoparticles-chitosan nanocomposites gel for immobilization of living cells (adapted from the reference [87])

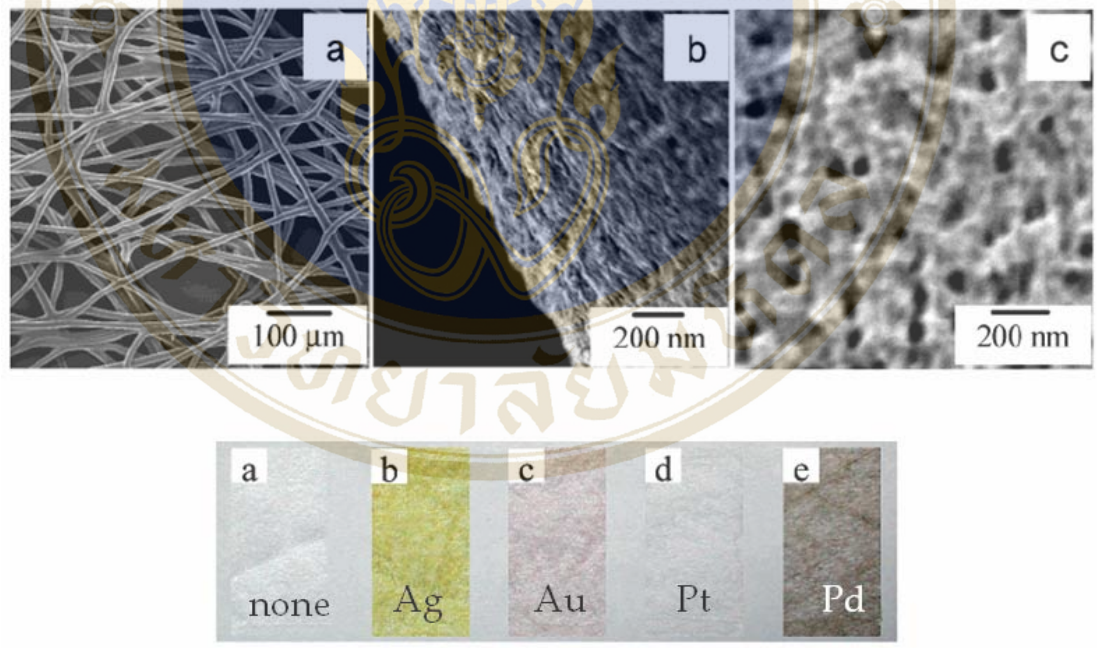

Figure 3.8 (up) FE-SEM images of the texture of cellulose (a), the edge (b) and surface (c) of a single cellulose fiber. (down) Cellulose specimens with (a) none, (b) Ag, (c) Au, (d) Pt, and (e) Pd metallic nanoparticles (adapted from the reference [71])

The stability probably arises from strong bonding interaction between the surface atoms of the nanoparticle and the surrounding oxygen atoms of noble metals.

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This effect would be expected for other oxygen-rich matrixes. For example, cellulose [71] is an oxygen-rich natural carbohydrate (polysaccharide) consisting of anhydroglucose units joined by an oxygen linkage to form a linear molecular chain. Natural cellulose fibers have a porous structure and are composed of microfibrils of 10-30-nm width that are three-dimensionally connected with each other (see Fig. 3.8). Such morphological features may provide a unique reaction vessel for synthesizing nanoparticles, allowing much enhanced access of guest molecules to catalytic centers, compared with nonporous films.

Among several biomolecules, it is noted that starch is one of the most abundant materials on earth. The knowledge on the structure of starch has been recently advanced [69]. The description of starch is well known as polymer structures containing purely amylose linear and extensively amylopectin branches. The spectroscopic and microscopic methods are now well advanced for probing the branching pattern and the branch length of starch polymers [88-91]. The structure of starch reported is packed in semicrystalline granules which can be visualized as growth rings. Amylose and amylopectin are arranged radically and aligned perpendicularly to the growth rings and to the granule surface. Starch granules can be transformed into other forms such as starch paste, starch gel, and retrograded starch depending on the cooking conditions [92-96].

The concept of green chemistry used in the synthesis of nanoparticles was first reported by Wallen and co-workers in which the synthesis of silver nanoparticles was carried out and stabilized by soluble starch and using β-D-glucose as a nontoxic reducing agent (see Fig. 3.9) [84]. The three main steps in the preparation of nanoparticles that should be evaluated from a green chemistry perspective are the choice of the solvent medium used for the synthesis, the choice of an environmentally benign reducing agent, and the choice of a nontoxic material for the stabilization of the nanoparticles. Water is utilized as the environmentally benign solvent throughout the preparation. The reducing sugar, β-D-glucose, is used as the reducing agent. With gentle heating, this system is a mild, renewable, inexpensive, and nontoxic reducing agent. Soluble starch is selected as the protecting agent for several reasons. In the

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past years, starch has been shown as a good host for many guests of inorganic nanoparticles, such as gold [17], silver [84] and iron oxides [97] to form inclusion complexes.

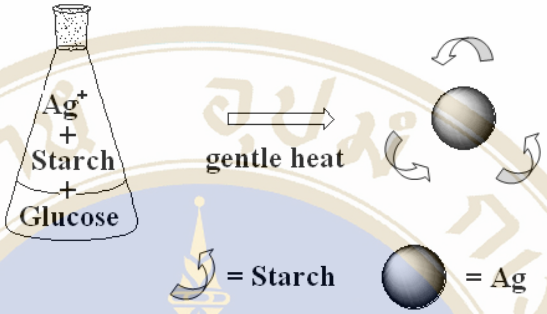

Figure 3.9 Schematic representation for green synthesis of silver nanoparticles (adapted from the reference [84])

The starch transformation provides diverse structures and properties for numerous applications. For example, starch gel can be produced from high amylose mung bean starch which is a major material for producing transparent starch vermicelli [98]. In this thesis, mung bean starch vermicelli is of our interest due to the higher percentage of amylose and its transparent structure so that the formation of color particles on the vermicelli can be readily observed. Furthermore, it would be interesting if amylose or amylopectin containing glucose units could be utilized as a template for the fabrication of nanoparticles for potential applications in biosensors, biofuels, and bioelectronics [99, 100].

3.3 Teaching and learning of the science laboratory

In the field of science education there are large numbers of researchers whose experiences of teaching and school, are confined to the primary and secondary levels. Such researchers find it more natural and easier to conduct research in a familiar context that school provides for students. On the other hand, research in science education at the tertiary level including undergraduate levels of higher education has been less extensive than at school level [101]. One reason for fewer

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studies at the higher education level may be that there is greater difficulty in gaining compliance from teachers and students.

There have been a lot of research studies in science education investigating what students actually learn from practical work and how the use of practical work can be made more effective in the teaching and learning in practical classes. There is often a tendency to think that every scientific idea needs to be illustrated through practical work. Certainly, practical work may help students to develop their practical skills. Different kinds of practical work generally are appropriate for different purposes. There is a strong temptation in the classroom to use practical work as a means of social control [102].

3.3.1 Teaching in practical classes

Most laboratory work in school science follows a familiar rubric in which students are led through the experiment or practical class following a cookbook style [26]. Students typically are presented with aims, such as hypotheses, steps for carrying out an experiment, observations, or measurements that should be recorded and questions that led them to the conclusion. Many researchers now believe this type of approach results in little meaningful learning. It is argued however that laboratory work can be an effective means for students to learn science content and that they also might learn something about the way that scientists understand the world, and how scientific knowledge is developed.

Interestingly, either cookbook or constructivist approaches often have the same stated objectives to develop student understanding about science including the dominant picture of propositional knowledge and routine procedures. However, approaches to the exploration of laboratory work have been focused on information processing and constructivist views of learning. If students are motivated to help each other either through explaining work or through contributing expertise to solve problems that arose in carrying out experiments, they come to develop an understanding of the ways in which scientists work. Hofstein and Lunetta [32] point

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out that the aims of laboratory work often are synonymous with the aims for science courses as a whole, and that it may then be more appropriate to identify particular objectives that represent the unique contribution delivered by practical work.

It is essential that the teacher and students are clear about the range of different types of practical work. There is a tendency for both the teacher and students to call any type of practical activity that they do in school laboratory an 'experiment' [31]. In fact, many of the practical activities done in school science are not experiments. They may illustrate a phenomenon, provide student with experiences or give students a 'feel' for a phenomenon. Observation of typical science teaching laboratories suggests that the following activities can be classified as practical work, such as teacher demonstrations, class practices with all learners on similar tasks working in small groups, a 'circus' experiment with small groups engaged in different activities rotating in a carousel, investigations organized in one of the above two ways, and problem-solving activities. Each type of practical work serves a different purpose: is of a different type, and has different aims [103].

A constructivist perspective has clear implications for teaching and learning in school science laboratories. Under constructivism, teaching becomes a matter of creating situations in which students actively participate in activities that enable them to make their own individual constructions. Activities, such as open discussion, group work, and problem solving, are taught to greatly enhance this individual construction [43, 104-106]. Teaching is a very personal activity, and while certain teaching styles and strategies may suit one teacher they might be deemed inappropriate by another. There is no one way to teach, because students learn in a variety of ways, meaning different approaches and strategies may be necessary. Teaching styles will vary from group to group and from individual to individual. In practical work, teaching is a continuous activity that requires creative thinking and problem-solving.

Research suggests that laboratory work can make a unique and significant contribution to the teaching and learning of science, and has the potential

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to enhance student learning with understanding [27, 32]. However, it is important to note at this point that some educators have concerns about both the effectiveness and the role of laboratory work in science education. Tobin, Tippins and Gallard [27], for example, suggest that meaningful learning is possible in the laboratory, but only if students are given opportunities to manipulate equipment and materials in an environment suitable for them to construct their knowledge of phenomena and related scientific concepts. In addition, they claim that research in general has failed to provide evidence of good learning outcomes for school science.

According to the traditional instructional strategy, teaching science in classrooms in higher levels is that the activity setting used by teachers has considerable bearing on the way student engage. Most teachers teach using whole class activities and teachers use lectures and textbooks to introduce students to science content. Therefore, teacher explanations commonly deal with surface features of content and frequently do not probe to the depth necessary for student understanding. To enhance student understanding of science, the *National Science Education Standards* (NSES) [38] suggests that science teachers should seek to assist students to learn with understanding of science concepts, and they must have theoretical and practical knowledge and abilities about science, learning, and science teaching. Science teachers can be effective guide for their students learning science if they have the opportunities to examine their own beliefs as an understanding of science.

If it is accepted that practical work should be at the heart of student learning, this begs the question as to how students can best be helped to develop and improve their learning of science in the laboratory. In regular classes, student attention might easily be diverted from the learning task. Therefore, so that laboratory work should help students a focus on their working. The most rationale for laboratory courses is that students are provided to do science as scientists do. Tamir [107] reported that there are five reasons used as a rationale for using the laboratory in school science teaching. First, science involves complex and abstract subject matters. Many students may fail to understand the concepts without the assistance and opportunities for carrying out activities in the laboratory. Second, student perceptions

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of actual investigations are an essential component of learning science as inquiry. Practical work allows students opportunities to act like a real scientist, to develop scientific attitudes such as honesty and critical assessment of results. Third, practical work experiences are essential for the development of specific student skills and strategies. Fourth, practical work is important for the identification, diagnosis and remediation of the student alternative conceptions. Finally, practical work helps students enjoy activities and become interested in science (p. 14).

In general, the laboratory in the school of science is different from each other, such as environment of the classroom. This affects the student quality in science learning of practical work. Therefore, focusing on laboratory work for achieving its goals, the aim of science education is to help students develop their understanding of the natural world: what it contains, how it works, and how students can explain and predict in a systematic way. When teaching in the science laboratory, the teacher builds up student knowledge of the world around them, by providing designed activities in which students observe or interact with real objects and materials [108]. The fundamental purpose of any laboratory work task should then be to help students to make links between two domains: the domain of real objects and observable things, and the domain of ideas. In school science, students often fail to learn the things they are intended to learn, because laboratory work tasks are carried out quickly, using basic equipment and often without sufficient care and precision [109].

As all detail described above, there is evidence to support the rationales what science educators said why science teaching can encourage students to learn science by using the practical work in science education. Considering the context of science teaching in the faculty of science at Mahidol University, the faculty of science comprises the thirteen departments [52]. The department of chemistry is one of the oldest chemistry departments in the faculty of science. In the undergraduate level, the department has also been involved in teaching activities. The staffs have often improved teaching in curriculum. The department of chemistry offers a four-year program to the Bachelor of Science degree in Chemistry (a B.Sc. program).

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In chemistry teaching, the lecture courses are fully supported by laboratory classes. In the first-year undergraduate level, the chemistry teaching in laboratory classes emphasizes students to gain general techniques of practical skills in chemistry and some experiments that would be related to lecture courses including chemical kinetics that refers to the determination of rate of a reaction, acid-base titration, and so on. In this work, we want to improve the chemistry laboratory teaching of chemical kinetics, and help students to understand the chemistry content of chemical kinetics and encourage their learning in practical classes. Therefore, one thing is important that the science teachers must concern the student learning and how to provide experiences to students.

3.3.2 Learning in practical classes

The *National Science Education Standards* (NSES) [38] reported that science in school levels must be equity and excellence for all students. All students should have the opportunity to learn high levels of scientific literacy. Science learning is something students do, not something that is done to them (p. 20). Learning is essentially an interactive process between the teacher and students. The action of the teacher and students affects the quality of the learning process. Therefore, one should consider the role of students in the learning process, and the activities and interactions associated with that learning. In practical work students need to gain meaning and understanding from teaching; understanding cannot be transmitted by direct instruction. Student understanding is gained through active involvement in learning activities [110].

The common approach to student learning in laboratories is now that based on constructivist views of learning. The main aim of science learning of activities in laboratories then is to encourage students to explain their ideas and to try and understand the thinking of others in the laboratory environment [111]. There is evidence from contemporary perspectives on learning science that students generally have difficulty in understanding what they have been taught in terms of the concepts of science. An important task then in designing instruction is to give students the

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opportunity to engage in constructing their own knowledge. Research in science education suggests that knowledge construction is particularly difficult for students of science, because they need to develop both conceptual and procedural understanding with appropriate activities [112].

In the science laboratory, students are taught with the learning situation as constructivist view of learning. A constructivist model currently serves as a theoretical organizer for many science educators who are trying to understand cognition in science [105, 106]. Students can construct their own ideas and understanding on the basis of series of personal experiences. These experiences in the school science laboratory may provide students with opportunities in which they can construct scientific concepts within a community in their classroom. Students are provided with opportunities for metacognitive activities: involving elaboration and application of one's learning which can result in enhanced understanding [43].

In laboratory work, one thing is students can construct much more knowledge building on student experiences. Knowledge can be generated as a result of student-student or student-teacher interactions. Nicholls [110] suggests that it is important that the teacher presents information, ideas, concepts and knowledge in a way students can learn, since the process of teaching can influence the learning outcomes of teaching and learning situations. This occurs he says, because students learn in different ways and it is important to understand the elements that constitute the basic learning process. Furthermore, the environment significantly affects learning as well as the relationships created in classroom.

One thing of interest in the research in science education is that the influence of gender between boys and girls might or might not affect student learning. Much attention has been paid in the area of gender differences. There is an increase in articles concerned with gender differences. Many researchers attempt to describe or explain differences gender on learning. Interestingly, it seems gender does not influence student learning experiences in the laboratory. Lock [113] used an experimental design in which the same students were tested for the same skills and

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tasks, with assessments made by direct observation, questioning, and examination of written records. When boys and girls are matched for ability and curriculum background, no gender differences are obvious in their observation, reporting, or planning skills. Similarly, Whitelegg [114] reported gender effects in science classroom and its influence on science learning at the primary and secondary level. The findings show that it is interesting similarities in gender attitudes in science learning.

Science learning in the laboratory at the undergraduate level is more intensive than for school level. To improve student cognitive skills in the laboratory work at higher education levels, learning in the laboratory has also been subject to critique. For example, laboratories classes are traditionally treated as a separate component to the lecture course. Many practical classes do not try to make much connection between the laboratory and lecture. Some researchers have attempted to enhance the effectiveness of laboratory classes in terms of cognitive and manipulative learning, and enjoyment of laboratories. Rollnick, Zwane, Staskun, Lotz and Green [115] used an action research approach to investigate how different modes of pre-laboratory preparation might contribute towards laboratory experiences for first year students. It seems student ability to prepare for their laboratory classes depends on their conceptual and procedural understanding of the laboratory. They further suggest that for students to function at a higher cognitive level, students should be required to generate their own laboratory procedures. Such a strategy is probably only feasible when students have considerable understanding of the practical skills needed in the laboratory. Strategies suggested include pictures showing new equipment, illustrations of the construction of apparatus and correct procedures. Furthermore, Davidowitz and Rollnick [116] suggest a flow diagram as a method of schematic representations of written instructions, and to require students to use this process the text of their practical manual. The use of a flow diagram like this allows teachers to examine student understanding of their practical manual. Flow diagrams also can be used for assessment of practical work.

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As the student learning described above, there is evidence to support how students should learn in practical classes at both secondary and tertiary levels. Most the student learning in both secondary and tertiary levels is very traditional outlook. The practical work can help students to correctly perform laboratories as scientists do; this means that students successfully complete the practical skills, but students might or might not understand what, and why they do the experiments. At the first-year university level in the faculty of science at Mahidol University, students often just do and have to follow the laboratory direction given in the experiments in which the experiments do not usually help students to engage in thinking about the experiments [52]. In fact, students can learn much more than just practical science skills in the laboratory. Therefore, if we want students to learn more skills in scientific thinking skills in practical classes, we need a different type of experiments. One thing is to develop student thinking that the laboratory would be more authentic, fore example more like real science, in which students do things in a way more like what scientists do, and have the opportunities to think as scientists think in the laboratory.

3.3.3 The development of scientific thinking in practical classes

As noted above, practical work is now widely used in the teaching of science at all educational levels. Even if some authors have reservations about the reported benefits of practical classes, others say there are some things (other than practical science skills) that can be learnt only in the laboratory. The rationale here is that students should learn to do science as scientists [28, 29]. But others note there is no easy answer to organizing an appropriate experiment for schools of science. One consideration is “What is a scientist? and Who forms the ‘scientific community’?”. Thus, the different ways in which different scientists work on different types of task need to be considered. This might include science technicians who require some specific scientific skills, the more stereotypical scientists seeking the truth, and applied scientists involved in solving pragmatic problems.

Laboratory activities traditionally have been used in an attempt to achieve variety of cognitive, practical, and affective goals, particularly deep learning

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[28, 29]. For the cognitive goals, this might be the promotion of intellectual development, enhancement of the learning of scientific concepts, development of problem-solving skills, development of creative thinking, and increased understanding of the methods of science. Practical goals might be the development of skills in performing science investigations, in analyzing investigative data, in communicating, and in working with others. Affective goals might be to enhance attitude-towards-science and positive perceptions of practical ability.

One might expect that student can learn the basic skills in the laboratory work from teacher demonstration. The practical class is one way available for demonstrating to students some of the concepts dealt with in the lectures, and indeed according to some authors this is a valuable benefit of practical work [43, 117]. However, in school science the activities in the laboratory vary and may include: teachers carrying out practical investigations and demonstrations for their students; students learning to do things hands-on; students observing, measuring and perhaps smelling! The physical set up of the laboratory also varies; when students come to the laboratory, they usually will find sinks, charts and pictures, various kinds of scientific equipment, and perhaps living organisms. Usually, laboratory classes are more popular with students than lectures because students are introduced to new apparatus and scientific equipment. However, the characteristics of the laboratory classroom environment also are dependent on the curriculum materials used [26].

Practical work in the school science laboratory does not have to be a truly authentic example of what real science is like. A purpose in laboratory work in which has often argued is that laboratory work provides students about the process of science. This implication is that students can learn and develop their scientific process of science for improving their knowledge. But thinking about practical work is an important exercise for any science teacher. Millar [108] suggests that practical work in science is very broad. Different kinds of practical work are for different things. Perhaps, the commonest aims of practical tasks in science education are: to help students learn some science content knowledge, to know something about the nature of the world, to understand a relationship or to receive an explanation of a

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phenomenon. Some practical tasks focus on teaching students on how to use a different kind of equipment, or follow standard procedures. Furthermore, others aim help students think about how to design and carry out an investigation. Millar [108] further suggests that it may be more useful for students to look at a few examples of practical tasks that aim to teach some science knowledge for school science practical work, such as cooling and insulation, green leaves, and metal and acid.

One factor which might improve student thinking is scientific content knowledge. In the context of higher education, students become engaged in laboratory work where they can extent knowledge. Practical work is seen as the means by which teachers can develop student conceptual understanding and practical ability; it helps students to understand science theory [32]. Most authors agree that practical or laboratory work at the tertiary level is an integral component of the teaching and learning of science. The influence of practical work became a part of the teaching and learning of science at secondary and university level in the nineteenth century, and since then educators have come to the view that practical work where students could experience with the physical world is central to tertiary science courses [43, 118].

Science educators are increasingly seeking to develop how laboratory work might influence students on scientific thinking. They suggest that laboratory work plays important role to the student mind [119]. As noted above, the role or purpose of practical work is different to other classroom or lecture-room activities. Indeed the distinction of practical work from lecture/classroom work in many countries suggests that there is a clear role or set of objectives for laboratory work. These objectives contain a number of common elements including procedural and affective skills.

Some science educators began to question the role for laboratory teaching and learning achieved in practices. To answer this question, Hofstein and Lunetta [32] suggest that science laboratories can provide students with opportunities not available in the classroom; they can, for example, help develop skills in logical

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thinking and organization. However, Hofstein and Lunetta believe that although laboratory work can be a more effective approach to science teaching, many research studies that failed to support the educational effectiveness of the laboratory instruction did so because of weaknesses in design. For example, some educational studies fail to maintain adequate selection and control of variables; others fail to take into account the effect of different group size, instrumentation, teaching behavior, or the use of documents such as laboratory manuals.

One thing is argued that student and teacher behavior in the science laboratory affect significantly the nature of actual laboratories activities. Curriculum materials and instructional strategies should allow students to interact physically with instructional materials as much as possible including handing, operating, and practicing equipment [27]. Teacher might to ease classroom management problems associated with such learning approaches and enhance science learning by giving students opportunities to learn independently. If students do pursue solutions to problems independently of the teacher, the teacher is free to become a facilitator of learning and focus on students who are in need of assistance. However, it obviously requires a lot of effort by the teacher to provide materials and instructions that give reality and concreteness to scientific concepts, and to vary instructional strategies, materials and classroom procedure with the aim of increasing the effectiveness of their teaching [120].

According to science teaching and learning in practical classes described above, the effective teaching is at the heart of science education. Science teachers at all levels should know and will be able to encourage science students with the standards. The *National Science Education Standards* (NSES) [38] suggest that students can develop their understanding of scientific knowledge when engaged with the inquiry-based teaching and learning (p. 23).

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3.3.4 Inquiry-based science teaching

The Standards produced by the National Research Council (NRC) [38] define 'inquiry' as:

> A multifaceted activity that involves making observations, posing questions, examining books and other sources of information to see what is already known, planning investigations, reviewing what is already known in the light of experimental evidence, using tools to gather, analyze and interpret data, proposing answers, explanations and predictions, and communicating the results. Inquiry requires the identification of assumptions, use of critical and logical thinking, and consideration of alternative explanations (p. 23).

Recently, there was a major symposium held, in which science educators strived to obtain an international perspective of inquiry; inquiry is seen as the *means* (i.e., inquiry as an instructional approach) and as *ends* (i.e., inquiry as a learning outcome) [121]. The participants at the symposium emphasized the significance of inquiry in science education, and distinguished between inquiry as means and as ends.

> Inquiry as means (or inquiry in science) refers to inquiry as an instructional approach intended to help students develop understandings of science content, i.e. content serves as end or instructional outcome. Inquiry as ends (or inquiry about science) refers to inquiry as an instructional outcome: Students learn to do inquiry in the context of science content and develop epistemological understandings about the nature of science (NOS) and the development of scientific knowledge, as well as relevant inquiry skills, e.g. identifying problems, generating research questions, designing and

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> conducting investigations, and formulating, communicating, and defending hypotheses, models, and explanations (p. 398).

In general, research in science education indicates that inquiry-based science teaching is manageable in science teaching. However, many science teachers have expressed a need for specific instructional materials that will help them to implement inquiry-based instructional strategies, and give students opportunities to develop the abilities and understanding of scientific inquiry (as indicated in the Standards).

Science teaching is a complicated activity, and it is not immediately obvious how science teachers can help students to understand science and how scientists themselves do experiments. However, the literature suggests that the experiments would be more *authentic* if they are inquiry-based. The National Research Council (NRC) [122] seeks to defines inquiry-based teaching, and provide the Standards that it believes science teachers at all levels should strive to achieve.

> Inquiry-based teaching refers to that inquiry into authentic questions generated from student experiences is the central strategy for teaching science. Inquiry is a characteristic of both a desired form of teaching and particular kinds of classroom activities (p. 173).

Thus teachers should provide their students with science facts and with technical words to describe these facts. They then should strive to create a situation in the laboratory classroom where students are asked to construct their own ideas, explain their opinions, and find things out for themselves. When planning and guiding inquiry, teachers have to determine how to encourage student learning, using their own knowledge of students, as well as their knowledge of science. Science teachers in an inquiry classroom should know how to initiate inquiry questions for whole class discussion. Although, students should be provided every opportunity to construct the relevant knowledge, they typically cannot discover scientific concepts,

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principles, and theories on their own. This, some authors argue means that the techniques of the effective science teaching are the same as used for effective scientific investigation [123, 124]. Thus a crucial decision for teachers is how much guidance supports student learning in inquiry classes; meaning teachers typically have to be more heavily involved in science teaching by inquiry. To illustrate, Woolnough [29] suggested that, when implementing inquiry-based laboratory tasks, teacher observe what students actually do, and should attempt to assess what they actually learn.

The teaching of science based on inquiry thus has wide support in the literature. Equally, there is strong support suggesting science learning is enhanced when inquiry-based.

3.3.5 Inquiry-based learning

The National Research Council (NRC) [122] argues that students do not come to understand inquiry simply by learning *words*, such as 'hypothesis', or by memorizing procedures, such as steps of the so-called 'scientific method'. Developing students' ability to understand and engage in inquiry-based learning activities then requires *direct* experience and continued practice with the processes of inquiry. The literature suggests teachers need to attempt to introduce students to the methods of science, and in particular to see how to design experimental investigations. In order to develop their understanding on inquiry, *students must experience inquiry directly* to gain a deep understanding of scientific characteristics, because experience and understanding go together [125]. The science teachers have to assist students to understand the inquiry processes in which they are engaged.

Research suggests that inquiry-type learning experiences are more effective if conducted in the context of the conceptual development of the topic taught. Thus it is argued that students should be presented with inquiry-based learning experiences in the subject of interest. Hofstein and Walberg [120] say that in inquiry-type practical classes, students should be involved in the process of

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scientific inquiry for seeking understanding of science phenomena, and that this process should be integrated with other complementary activities, such as the development of scientific concepts, related scientific skills and experiences. Moreover, Hofstein and Lunetta [43] suggested that laboratory activities offer important experiences in science teaching and learning; further commenting that teachers should provide students with experiences in methods of scientific enquiry and reasoning, and in the application of scientific knowledge with everyday life.

Science teaching is however a complex task that involves both classroom and laboratory activities, and in which students interact with the teacher and other students. To emphasize clearly science learning what students should do in the laboratory, the Standards [122] defines:

> Inquiry-based learning refers to learning processes. It is also an active learning process-something that students do, not something is done to them (p. 174). The Standards imply inquiry-based learning both to scientific inquiry and to studies of human learning.

As pointed out in the Standards, a characteristic of inquiry is that learning science is an *active* process. An active process here refers to the teaching and learning strategy that the teacher use inquiry to enable students to understand scientific concepts through an active learning method and students as inquirers or seekers of information are encouraged to ask questions, generate hypotheses, analyze data, solve problem, and draw conclusions [38]. Students should be able to design and conduct scientific investigations in practical classes. Therefore, because students have to learn the procedural skills of scientists, and understand the nature of science and they could understand and retain the concepts that they have learned [122]. For example, students should be have opportunities to conduct an investigation; for example, changing a single variable that might affect the rate of a reaction.

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Therefore, inquiry teaching and learning pose a challenge to science teachers and their students to understand scientific inquiry in practical classes.

3.3.6 Scientific inquiry and practical classes

There have been many benefits from engaging students in science laboratory activities. Most importantly, many authors argue inquiry-based laboratory work has the potential to enhance student constructive learning, conceptual understanding, and understanding of the nature of science [32, 126]. Science educators believe that the all science subjects have succeeded in building understanding largely because of inquiry which can be used to test ideas. Scientists have generally used the processes of inquiry to build knowledge about natural phenomena. Klopfer [127] identifies scientific inquiry with respect to science education into student science skills, including the full range of the scientific process, such as observing and measuring, seeing and seeking solutions to problems, interpreting data, generalizing, and building, testing, and revising theoretical models (p. 97). Most discourse regarding scientific inquiry in science education becomes to center in engaging students in inquiry-based practical classes.

Engage in inquiry-typed experiments in the chemistry laboratory, students can improve their ability to ask high-level and better questions, to hypothesize for further experimental investigations. The student written documentation can be used for promoting student learning in science, because writing, traditionally considered, is one of formal modes in the assessment of student understanding. For example, Keys, Hand, Prain and Collins [128] determined an important role in engaging student cognitive skills, such as scientific thinking and scientific skills, in investigating personal construction of meaning in the science laboratory. Keys [129] investigated the thinking processes used by students during the composition of a written laboratory report and explored the possibility that writing about investigation can contribute directly to science learning in students. Students have to generate and synthesize new ideas in the context of the experiment. The findings show that some students can generate new knowledge and explanations

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from their activity of writing and some cannot. Most students can develop their cognitive process.

Many science educators believe that inquiry-based laboratory can provide students to experience authentic scientific inquiry. For example, Hofstein & Lunetta [43] believe that inquiry laboratories have potential to enhance student constructive learning, conceptual understanding and understanding of the nature of science. These laboratories involve that students are provide experiences to formulate hypothesis, design experiments, gather and analyze data, and draw conclusions about scientific problems or natural phenomena, with the development of scientific concepts and processes. Inquiry-based experiences are especially effective if conducted in the context of the conceptual development of the topic taught. It is noted that some experiences provide students with opportunities to develop independent thinking, making senses of chemical concepts, enjoy a group learning environment [130] and some provide students with opportunities to learn in an authentic environment in which they can construct their knowledge regarding chemistry phenomena by conducting inquiry skills, such as asking questions, hypothesizing, and suggesting a question for further investigations [131].

Science educators suggest that practical work is essential in enabling students to gain the first-hand experience of phenomena [29, 109]. It can stimulate students to find explanations for events and give them an insight into the nature of scientific inquiry and, their own investigative work. Students can apply new experiences to construct their conceptual understanding in new contexts. Wellington [28] argued that laboratory work should be a wide variety of science tasks, designing to promote different kinds of learning, and intended to illustrate a scientific idea, or to simulate some aspects of real scientific inquiry. Hodson [119] emphasized that the principal focus of laboratory activities should not be limited to learning specific scientific methods or particular laboratory techniques. Students in the laboratory should gain methods and procedures of science to investigate phenomena, solve problems, and pursue inquiry and interest and the laboratory learning environment should shift from teacher-directed learning to student-directed learning.

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Scientific inquiry is a complex skill in science and becomes an important element of the classroom. Science teachers believe that students can be able to understand the nature of scientific inquiry by engaging in inquiry. Scientific inquiry can help students to develop the conceptions of the nature of science. In addition to knowledge of the scientific inquiry and the nature of science, the science teachers have to understand the processes that scientific knowledge is created to incorporate inquiry-based laboratory. Student knowledge about scientific inquiry and the nature of science does not occur by accident. Students do not develop an understanding simply through experiencing inquiry any more, so science teachers should allow students to come to the laboratory and to learn with their own experiences [132].

The emphasis on the inquiry-bases laboratories often seems like the use of the process of discussing and arguing with other students in order to plan and carry out an inquiry. Argument is also seen as another means in the process of carrying out an inquiry-typed experiment. The arguments can be seen to take place as an individual activity, through thinking and writing, or as a social activity to take place within a group, a negotiated social act within a specific community. Science teaching and learning takes place in a social environment; it is a social process in which the science teachers have to introduce students to the culture practices of science. There are many science educators who determine student understanding by using argumentation in practical classes [133, 134]. The method used is the social processes of learning in the classroom. The findings show that the processes of argumentation can encourage students to increase their scientific literacy and help them to understand the nature of scientific inquiry.

The trend in education reform around the world is to provide choices and alternative approaches to learning where teachers have the knowledge and skills to decide which approach to adopt when and for what reasons. It is understandable why the reform leaders in Thailand adopted student-centered learning as the main focus of change in the teaching and learning approach.

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3.3.7 Group learning in science education

A fundamental aspect of science education is a desire that students can communicate, explain, and justify their understanding, argue from data, and defend their conclusions. According to the *National Science Education Standards* (NSES) [122], a community of science students is one in which students can develop a sense of the purpose and ability to assume responsibility for their learning. Working with others not only enhances the understanding of science, but also fosters the practice of many skills. Science teachers should then provide many activities for learning science that require group work, not simply as an exercise, but as essential to the inquiry and to teach students the skills that are needed to work together.

Here, the rationale of implementing group learning in science education is that we want students to understand the nature of science and how scientists actually work. It also is useful to give students experiences that are more like scientists doing real science. In fact, scientists seldom work alone, they usually work cooperatively.

Classroom discourse is increasingly becoming a prominent area of research in education, particularly in science education. *Team* or *group work* is an important teaching strategy in designing the classroom environment. There is evidence about the process of student-centered learning situation where students have the possibility to bring their information and explanation for negotiation with others while working in a group. Research suggests we may overlook opportunities to use groups to enhance student learning and increase their own success [120, 135].

Group learning is an important component of modern educational approaches. The value of group work for learning is that, in contrast to competitive learning environments, if students value the success of the group, they will encourage and help one another to achieve [135-137]. A group is a natural social act in which students talk among themselves, and learning occurs through this talking. Students working in group learning situations are provided with the challenge of

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actively engaging with their peers to synthesize knowledge and information [138]. Students can learn from other students both by giving and receiving help. The groups thus give the opportunity for students to help each other learn. It is important that science teachers who try to develop group work consider ways of helping students to understand the social and procedural rules that students begin to appreciate with the new way of working in the classroom.

Group learning is widely used as a teaching strategy that can encourage a social science learning situation for students. Much research in science education acknowledges that knowledge is socially constructed. Students in group learning situations may be more willing to share information with other students in the groups, than in a competitive environment [42]. Group learning in science education involves the instructional setting that engages students in the authentic practice of science that is, performing and interpreting experiments for the specific purpose of verifying or revising theories and explanations, and communicating and negotiating them among the group members [139].

At the tertiary level, small-group learning in inquiry-centered laboratories has the potential to encourage student meaningful learning, their conceptual understanding, and their understanding of the nature of science [122]. Kaartinen and Kumpulainen [140] investigated the context of the learning situation derived from kitchen chemistry to develop student conceptions of solubility. When 18 university students worked in self-selected small groups, the average size of mixed-gender groups was four students, and the students negotiated the meaning of solubility. The research points to a need to construct student explanation in small-group learning of science. Small-group learning seemed to develop positive academic classroom community, compared with no group work, and a traditional learning style. Towns, Kreke and Fields [141] likewise used an action research methodology in an attempt to improve classroom practices in the chemistry subject; to determine how the activities can be improved via student suggestion, and to learn how student perception of small-group learning. In this work, undergraduates enrolled in first-semester physical chemistry at a large university in the Midwestern United States, had to

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observe what happened, evaluate it, and reflect on how to conclude data from the observation. Small-group learning seems to encourage these students to build a feeling of community in the classroom and produce positive learning outcomes.

Small groups may be formed from self-selected volunteers, or they may be constructed by teachers based on different kinds of experiences or skills in students. Group learning is an open-ended process and can be unpredictable. For example, in science laboratories, the use of small group learning is dependant on the type of the experiments and how they are constructed by the teachers. The literature suggest that teachers as facilitators, should organize mixed-gender groups and teachers should combine the different cultural backgrounds of the students where possible [142].

In traditional teaching of science, including in the Thai education system, students in the classroom are passive recipients of information. The role of the teacher is that of an information giver. The students have to wait to be ‘filled’ with information from the teacher during both the lecture and laboratory. Classes are driven by teacher talk, and are highly dependent on the structure of the course. A teacher-centered approach like this has played important role in educational system in Thailand [49].

The inability of Thai educators to simplify learning of theories and demonstrate practical applications is a major concern to the Ministry. Science teachers and educators in Thai education often teach theories rather than practical skills. The effort to change the teacher-centered approach to student-centered learning is currently discussed in terms of how to change the teaching and learning approaches and strategies in order to facilitate the acquisition of the new types of knowledge. Students, according to the Ministry, should be assisted and encouraged to expand their skills and their understanding of different types of knowledge with the new approaches to teaching and learning [143].

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In the chemistry subject, chemical kinetics is difficult to comprehend for the first-year undergraduate students. Here we attempt to enhance the student understanding in the chemistry concept of chemical kinetics for the laboratory classes.

3.3.8 Review of chemical kinetics in science education

It is surprising to many students that all everyday processes involve kinetics. One reason for this may be that some reactions are so slow that they may take thousands of years to produce a measurable amount of product; however, others are very fast. For example, acid rain is a problem in many large cities worldwide, and is caused by emission of sulfur dioxide and similar chemicals from car exhausts. In recent years this has become a serious environmental problem. It can severely damage buildings in a city, and it is obviously of interest to scientists and citizens alike to know how quickly such destructive reactions might happen: this is an example where students can come to understand the importance of basic kinetics concepts.

Chemistry is a science that is often concerned with making new substances from other substances. There are a number of definitions for the term ‘chemical kinetics’ in the literature. The branch of physical chemistry called chemical kinetics is concerned with the measurement and interpretation of the rates of chemical reactions [144]. The study of the reaction rate leads to an understanding of the mechanism of a reaction, its analysis into a sequence of elementary steps. Chemical kinetics addresses the question: How fast do reactions go? How rapidly are reactants consumed and products formed? It could be said that chemical reactions are the core of chemistry. At the molecular level, chemical kinetics seeks to describe the behaviour of molecules as they collide and are transformed into new compounds, because the basic concept underlying chemical kinetics is that molecules must collide with each other in order to react. In many reactions, the rate of a reaction changes as the reaction progresses. Initially, the rate of a reaction is large, and as the reaction

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progresses the rate decreases, eventually becoming zero when the reaction is completed.

As noted above, the study of chemical kinetics is often concerned with finding out how fast chemical reactions go? Studies of chemical kinetics can be classified into two groups [145]. The first group investigates the construction of models to describe and explain the observed relationship between the rate of a reaction and variables that affect the reaction rate dealing with the use of models in calculating reaction rate. The other group also seeks to provide critical support for the mechanism proposed for any chemistry reaction. This group investigates chemical kinetics by collecting data on changes in variables that affect the rate at which reactants are converted into products. The experimental data are mathematically integrated giving the rate law dealing with any proposed mechanism. Justi and Gilbert [146] note with surprise that there are very few research reports about chemical kinetics from the point of view of either the teaching or the learning aspects. This is true of the subject at both secondary and tertiary levels.

In general, in most countries, the teaching of chemical kinetics at the school level emphasizes the qualitative aspects of the topic. Science teachers have to introduce their students to an understanding of the rate of different examples and to investigate the influence of other variables (i.e., temperature, concentration and catalyst) on the rate of a reaction. At this level, the concept of the rate determining step, rate equation and order of reaction are introduced. Parkash and Kumar [147] report research into teaching and learning of a variety of chemical kinetics experiments involving gaseous carbon dioxide formed from the reaction between ethanoic acid and sodium hydrogencarbonate at different time intervals. They claim that chemical kinetics is typically taught as a part of theory and it is seldom illustrated practically, and that their improved experiment help students see the dependence of the rate of a reaction on the concentration of the reactants, and in determining the order of a reaction with respect to each reactant, the overall order of a reaction, the rate constant of a reaction, and the half life. Justi and Gilbert [146] likewise investigated the teaching of chemical kinetics using models with Brazilian chemistry

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students (15-16 years old). It seems this model provide explanations that are comprehensive and simple for the teaching of chemical kinetics, and contributes to the development of student views of scientific knowledge that are more authentic.

In many schools classrooms teaching approaches used in encouraging student conceptual understanding seem to often involve teacher demonstration of experiments, because such techniques are simple to use. Use of a datalogger (i.e., a computer interfaced to one or more sensors) in biology, chemistry and physics laboratories are becoming popular [148]. The major reported advantage of a datalogger is that students can, in real time, simultaneously monitor various variables, such as temperature, electric current, electric potential, pressure, and pH. But such facilities are probably not available in less well resources countries.

Chemical kinetics is an extremely important concept for university level introductory chemistry courses. At university level, the concepts taught are more complex than at the school level [145]. The literature suggests that chemical kinetics is difficult for students to comprehend, because it involves complex mathematic equations for both the rate equations and variables that affect the rate of a reaction. Here, some science teachers believe that chemical kinetics is critical, that knowledge from doing experiments can help students to understand the reaction mechanism; they should also be able to predict and determine information on the rate of the reaction using kinetics. Experiments can be effective in promoting the development of student perception and higher-order cognitive skills, if students are given an opportunity to think, learn and inquire about things by themselves. To make it possible to construct conceptual understanding, the teaching of chemical kinetics should arise from daily experiences for university students.

Chemical kinetics also often involves understanding about catalysis. Most general and physical chemistry textbooks, at tertiary level, explain that a catalyst significantly speeds up a chemical reaction by lowering the activation energy of the reaction. Students can use the initial rate to observe the role of a catalyst in a chemical reaction via activation energy calculations. Copper and Koubek [149] reported that

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kinetic experiments that demonstrate the role of a catalyst (the so-called iodine clock reaction) are accessible in almost any teaching situation since it involves minimal and equipment. It seems students find visually dramatic reactions like this interesting and that they help them to see the effect of a catalyst upon the kinetics of a reaction.

To improve the teaching of chemical kinetics, Justi [145] suggests that the main problem is the use of the teaching method in classrooms. If teachers make heavy use of mathematical treatments for kinetic equations, such as a general approach based on a matrix formulation of the differential equations for chemical kinetics, means students can learn how to derive zero-, first-, and higher-order kinetics for a variety of chemical equations. However, some authors believe that students can learn new or difficult information effectively when the classroom environment is compatible with their learning style preferences [150]. There also is increasing interest in using more creative pedagogies including the simultaneous use of a computer [148], with programs that incorporate spreadsheets used to do the calculations from experimental data [151], and analogies [152].

To help students to develop conceptual understanding of chemical kinetics is one of the most common objectives for the teaching of physical chemistry in laboratory classes. Research suggests that when chemical kinetics is taught for the first time, teachers should seek to identify student prior knowledge and difficulties for a given scientific concept. For example, it may help students to understand chemical kinetics, if there is a feeling that other students in the class have difficulties and problems in the learning of chemical kinetics. Teachers also should start teaching the fundamental concepts before moving on to more complex concepts [150].

The literature suggests students can do experiments either at home or in the chemistry laboratory, because the chemicals and equipment required are often common household items [145]. In either case, it is important for students to investigate, interpret and discuss a chemical reaction by applying related everyday processes for learning chemical kinetics in practical classes. One is that classroom activity for students to learn factors affecting the rate of a chemical reaction.

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Various variables are investigated by changing each separately, with all others constant, i.e. the reaction of acid-base between hydrochloric acid (HCl) and sodium hydrogencarbonate ($NaHCO_3$). In this activity, students have to discuss of how or why each variable affects the rate of a reaction. Many teachers believe that students can learn new or difficult information effectively when the classroom environment is compatible with their learning style preferences.

To understand what students have learned chemical kinetics, it is important to probe student perceptions of their practical classes.

3.3.9 Student perceptions of practical classes

It seems one important factor for learning in the laboratory is students' perceptions of learning experiences in the laboratory. The literature suggests students generally expect science to be a practical subject. Science laboratory classes are seen as being more popular than lectures because the teacher provides students with more exciting physical activities, such as hypothesizing experiment, making predictions, collecting observational data, and using modern apparatus and equipment [26]. Continuity and progression also are problems for student shifting from practical science in primary and secondary school. The role of the science teachers is important in science teaching. Teachers often use the experiment demonstration for improving student perceptions in practical classes. For school in country, students usually do not have the opportunity of making with their hands themselves because of lack of adequate chemicals and insufficient instruments and equipment [153].

However, literature argues that students find difficulty in continuity when they transfer from the secondary school to higher education environment, particularly in the case of science education. At tertiary education, teachers allow students to learn largely instruction-driven experiments, which are cookbook in nature and some more difficult and advanced experiments. Students have to build up knowledge with their own learning and make arguments. Teachers must take the advantage of any opportunity for their student learning. Student evaluations have

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become routine at most colleges and universities. It is noted that students often find it difficult to link ideas that are presented in lectures and laboratories [118]. In any science teaching situation, students should gain better knowledge and understanding of concepts, that is, if students do not learn from the way teachers currently teach, teachers should make every possible effort to teach the way students learn. Some educators suggest that information about student perceptions should be used to improve chemistry laboratory classroom environments and teacher-student interactions for successful students [154].

There is some evidence to suggest that the student perception is widely determined to become a positive or negative attitude to what they have learned in practical work. In science education, many researchers have examined student perceptions to understand student responses, using a combination of multi-methods approach of both qualitative and quantitative methods as a means of triangulation, such as test, survey and interview (TSI) and questionnaire on teacher interaction (QTI) [125, 155].

One reason in engaging students with the chemistry laboratory courses is that the influence of time (i.e., beginning and end of semester, or from first semester to second semester) might be a significant factor that affects students' attitude to enroll in second-year chemistry [156]. It could be said that the influence of practical experiences is an essential part of student perceptions in science education. There is some evidence to suggest that student learning experiences might develop their interest in chemistry and attitude toward chemistry. Therefore, it would be helpful both to teachers in making decisions about the structure of teaching and students in gaining a deeper understanding and developing about their perceptions of science learning.

3.3.10 Approach to inquiry-based learning in Thailand

A teacher-centered approach had been intended to play important role in educational system in Thailand [49]. The inability of Thai educators to

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simplify learning of theories and demonstrate practical applications is a major concern. Science Teachers and educators in Thai education often put an emphasis on theories rather than practical skills. The effort to change the teacher-centered approach to learning is currently discussed how to change the teaching and learning approaches and strategies in order to facilitate the acquisition of the new types of knowledge. Students should be assisted and encouraged to expand their skills and their understanding of different types of knowledge with the new approaches to teaching and learning. This is guidance for the development of new innovation practices.

The responsibility for educational planning is shared by central government agencies. The administration of primary and secondary schools is responsible of the Ministry of Education (MOE). At the higher education level, the Ministry of University Affairs (MUA) is responsible for the administration of tertiary education. In planning science teaching for all education levels, The Institute for the Promotion of Teaching Science and Technology (IPST) is a major department that plays important role in the teaching of science education in Thailand. The IPST has incorporated the inquiry approach in science learning. However, there are limitations, such as class sizes, lack of science equipment and shortage of qualified teachers that affect the learning outcomes. The IPST emphasizes on the aspects for quality science teaching that should be:

1. Inquiry-based teaching and learning process
2. Higher-order thinking process
3. Scientific process
4. Communication and decision
5. Project-based skills
6. Using Information Technology (IT) for teaching and learning
7. Learning how to learn [50].

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CHAPTER IV
METHODOLOGY

This chapter provides a description of the experiment for the preparation of nanostructures and the methodology used in science education which is relevant to a means of an inquiry-based chemistry practical class. In science part, it begins with materials, experimental procedures, and general characterizations. In science education part, it starts with theories of learning in science education and the methods and methodological approaches used in this thesis, such as probing student understanding, semi-structured interview protocol and student documentation, and data analysis used in this study, e. g. analysis of quantitative and qualitative data.

4.1. Magnetic iron oxide nanostructures

4.1.1 Materials

The starting materials used in this experiment were commercially analytical reagent grade. Transparent starch vermicelli of mung bean (100 %, Kaset brand) used as a stabilizing material was purchased from the local supermarket in Thailand (see the Fig. A.1), and also can be purchased from many countries which import the Thai production. All glassware used in this work were cleaned with 10 % nitric (HNO_3) solution and repeatedly washed with de-ionized (DI) water obtained from Nanopure® Analytical Deionaization Water with an electronic resistance ≥18.2 MΩ-cm, and dried before use. All of materials were prepared by deionized (DI) water.

4.1.2 Chemicals

Chemicals used in this work also are provided as follows.

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Table 4.1 List of chemicals for the fabrication of magnetic iron oxide nanostructures

Chemicals	Suppliers
Ferrous chloride tertrahydrate ($FeCl_2 \bullet 4H_2O$, ≥ 99.0%)	Fluka, Switzerland
Hydrochloric acid (HCl, 37%)	LabScan Asia, Thailand
Nitric acid (HNO_3, 70%)	Merck, Germany
Ammonium hydroxide (NH_4OH, 25%)	LabSacn Asia, Thailand

4.1.3 Instrumentals

The instruments used are listed in table as shown below.

Table 4.2 List of instruments for the synthesis of magnetic iron oxide nanostructures

Instruments	Details
Analytical balance	Denver instrument Company, TC-254
Hot plate and stirrer	Heildolph, Mr-3001
Furnace	Lindberg, 59256-P-Com
Thermal gravimetric analysis and differential scanning calorimetric analysis, TGA/ DSC	TA instrument-2960 SDT simultaneous analyzer
Field emission scanning electron microscope, FE-SEM	JEOL JSM-6340F, 5.0 kV
Transmission electron microscope, TEM	Philip Tecnia G-2 sphera, 80.0 kV
X-ray powder diffractometer, XRD	D-8 Advance Bruker
Vibrating sample magnetometer, VSM	Lakeshore-4500
UV-Vis scanning spectrophotometer	HP-8453 Hewlett Packard

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4.1.4 Chemical preparation

- 0.1 M $FeCl_2$/50 mL

Weigh 0.994 g of $FeCl_2 \bullet 4H_2O$ and then dissolve with a small amount of DI water in a beaker. Transfer the solution into the 50-mL volumetric flask and then adjust the volume to an exactly 50 mL with DI water. Shake for making a homogeneous solution. The solution is freshly prepared before use.

4.1.5 Preparation of magnetic iron oxide nanoclusters

The method described for synthesizing of magnetite (Fe_3O_4) nanoparticles was based on the literature [13]. The controlled size of nanostructures was prepared using mung bean vermicelli template as a stabilizing agent by the following procedures. Typically, a required amount of 1.0 g transparent mung bean vermicelli was placed into a 100-mL round bottom flask containing 50 mL of a 0.1 M $FeCl_2$ solution. Then, the mixture solution was stirred vigorously under the presence of air and also heated at 85-90°C by placing the thermometer for controlling temperature in an oil bath. To produce Fe_3O_4 nanoparticles, the pH of the mixture solution was adjusted to 12 by a diluted NH_4OH solution. The color of the reaction solution changed immediately to black, indicating the formation of Fe_3O_4 nanoparticles. The reaction system was maintained under magnetically stirring for 30 min before cooling to RT. The magnetic behavior of the precipitate was observed and removed from the solution with the magnet by applying a magnetic field from the bottom of the reaction flask, and the black precipitate powder was settled down (see Fig. A.2). After discarding the supernatant solution, the mung bean vermicelli coated with magnetic iron oxide nanoparticles was washed several times with a diluted HCl solution and DI water, respectively, in order to remove impurity ions. After washing, the solution was tested with the pH paper until the pH was neutral (usually pH~7). To avoid the phase transformation of magnetic iron oxide nanoclusters, the sample was dried by incubating in an oven at 40°C for 48 h [14].

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4.1.6 Carbonization

In order to perform the carbonization, a crucible alumina cup containing the as-prepared sample of magnetic iron oxide nanoclusters stabilized by starch vermicelli was placed in the central region of a furnace. The dry sample was carbonized with a constant heating of 5°C-min^{-1} from RT to 200°C. The system was maintained at this temperature for 12 h, and subsequently cooled down to RT at the decreasing rate of 5°C-min^{-1}. Then, the black precipitate powder was collected for further characterization.

4.1.7 General characterization

Thermal properties of the dry sample were measured by using thermal gravimetric analysis and differential scanning calorimetric analysis (TGA/DSC) with the TA instrument-2960 SDT simultaneous analyzer. The sample was placed into an alumina crucible for TGA/DSC measurement and heated from RT to 700°C with increasing temperature rate of 10°C-min^{-1} under a flowing of argon gas at 20 cm^{3}-min^{-1}.

Scanning electron microscopy (SEM) observation was carried out by employing a JEOL JSM-6340F field emission scanning electron microscope (FESEM) by accelerating at a voltage of 5.0 kV. For the SEM preparation, the sample was mounted on a Cu grid for SEM measurement and then coated with gold to minimize charging.

Transmission electron microscopy (TEM) observation was performed by using a Philip Tecnia G-2 sphera transmission electron microscope with an acceleration voltage of 80.0 kV. The sample was dispersed in a solution by using an ultrasonic bath (ca. 5 min). For the TEM measurement, the sample preparations were prepared by dispersing a drop of the sample solution on the formvar-coated Cu grids.

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The crystalline phase of the samples was characterized by X-ray powder diffraction (XRD). A D-8 Advance Bruker Analytic X-ray diffractometer was operated with the CuKα radiation at the wavelength of 0.15650 nm using 40 mA and 40 kV. The diffraction pattern over the range of 30°-70° 2θ was recorded with the scan speed of 2 min/step and the scan rate of 0.015 2θ/s.

The coercivities (Hc), the remanent magnetizations (Mr) and the saturation magnetizations (Ms) of the as-prepared magnetic iron oxide nanoparticles were performed by using the vibrating sample magnetometer (VSM). The magnetic parameters (Hc, Mr and Ms) of each sample were determined from the hysteresis loops produced. The saturation magnetization was reached at an applied field of 10 kOe. The magnetization data as a function of the applied field were plotted.

4.2 Gold nanoparticles (AuNPs)

4.2.1 Materials

The starting materials used in this experiment were commercially analytical reagent grade. Transparent starch vermicelli of mung bean (100 %, Kaset brand) used as a stabilizing material was purchased from the local supermarket in Thailand (see the Fig. A.1), and also can be purchased from many countries which import the Thai production. All glassware used in this work were cleaned with 10 % nitric (HNO_3) solution and repeatedly washed with de-ionized (DI) water obtained from Nanopure® Analytical Deionaization Water with an electronic resistance ≥18.2 MΩ-cm, and dried before use. All of materials were prepared by deionized (DI) water.

4.2.2 Chemicals

All chemicals of this experiment were shown in the list as follows.

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Table 4.3 List of chemicals for fabrication of AuNPs

Chemicals	Suppliers
Hydrogen tetrachloroaurate(III) trihydrate ($HAuCl_4 \bullet 3H_2O$, ≥ 99.9+%)	Sigma-Aldrich
Hydrochloric acid (HCl, 37%)	LabScan Asia, Thailand
Nitric acid (HNO_3, 70%)	Merck, Germany

4.2.3 Instrumentals

Instruments for the preparation of AuNPs were listed as follows.

Table 4.4 List of instruments for the preparation of AuNPs

Instruments	Details
Analytical balance	Denver instrument Company, TC-254
DI water system, R ≥ 18.2 MΩ-cm	Nanopure® Analytical Deionaization Water System
Laser pointer	Wavelength 650 nm, <5mW
House hold microwave	M-181 GN Samsung, 100-850 W, 7-power levels and 35 min dual-speed timer
Transmission electron microscope, TEM	Philip Tecnia G-2 sphera, 80.0 kV
X-ray powder diffractometer, XRD	D-8 Advance Bruker
UV-Vis scanning spectrophotometer	HP-8453 Hewlett Packard

4.2.4 Chemical preparation

- 0.1% (w/v) $HAuCl_4$/100 mL

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Weigh 0.1 g of $HAuCl_4 \bullet 3H_2O$ and then dissolve with a small amount of DI water in a beaker. Transfer the solution into a 100-mL volumetric flask and then adjust the volume to an exactly 100 mL with DI water.

- 0.01% (w/v) $HAuCl_4$/100 mL

Pipette a 10 mL of $HAuCl_4 \bullet 3H_2O$ as prepared above and then adjust the volume to an exactly 100 mL with DI water using a 100-mL volumetric flask.

- 0.01 M HCl/250 mL

Pipette 200 μL of a concentrated HCl and then dissolve in a 250 mL of DI water in a 250-mL volumetric flask.

4.2.5 Preparation of gold nanoparticles (AuNPs)

Approximately 0.1 g transparent mung bean vermicelli was added into a 50-mL Erlenmeyer Pirex flask containing a 30 mL DI water. Then, the mixture was placed in the microwave cavity and also heated by using the microwave power at 300 W under the presence of air for 1 min. A 3 mL of 0.01% (w/v) $HAuCl_4$ and 100 μL of 0.01 M HCl were added into the mixture solution, respectively. Then, solution was heated in the microwave oven under the same condition for 5 min, and reheated again for an additional 5 min. After heating process, the color of starch vermicelli obviously changed from the colorless to pale red, indicating the formation of starched AuNPs (see figure B.1). The reaction solution was cooled to RT. To further investigate the formation of AuNPs, other different experiments were also carried out by increasing the concentration from 0.01% to 0.1%.

4.2.6 General characterization

Transmission electron microscopy (TEM) observation was performed by using a Philip Tecnia G-2 sphera transmission electron microscope with

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an acceleration voltage of 80.0 kV. The sample was dispersed in a solution by using an ultrasonic bath (ca. 5 min). For the TEM measurement, the sample preparations were prepared by dispersing a drop of the sample solution on the formvar-coated Cu grids.

The crystalline phase of the samples was characterized by X-ray powder diffraction (XRD). A D-8 Advance Bruker Analytic X-ray diffractometer was operated with the CuKα radiation at the wavelength of 0.15650 nm using 40 mA and 40 kV. The diffraction pattern over the range of 30°-70° 2θ was recorded with the scan speed of 2 min/step and the scan rate of 0.015 2θ/s.

The UV-Vis absorption spectra of colloidal metal nanoparticles were performed by using a HP-8453 Hewlett Packard UV-Vis spectrophotometer with the path length of the quartz cell at 10 mm using DI water as a reference, over the wavelength ranging from 250 to 900 nm.

4.3 Silver nanoparticles (AgNPs)

4.3.1 Materials

The starting materials used in this experiment were commercially analytical reagent grade. Transparent starch vermicelli of mung bean (100 %, Kaset brand) used as a stabilizing material was purchased from the local supermarket in Thailand. All glassware used in this work were cleaned with 10 % nitric (HNO3) solution and repeatedly washed with de-ionized (DI) water obtained from Nanopure® Analytical Deionaization Water with an electronic resistance ≥18.2 MΩ-cm, and dried before use. All of materials were prepared by deionized (DI) water.

4.3.2 Chemicals

All chemicals of this experiment were shown in the list as follows.

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Table 4.5 List of chemicals for fabrication of AgNPs

Chemicals	Suppliers
Silver nitrate ($AgNO_3$, ≥99.8+%)	VWR international Ltd, England
β-D-Glucose anhydrous ($C_6H1_2O_6$, 98%)	Ajax Finechem, Australia
NaCl (NaCl, 99.5%)	LabScan Asia, Thailand
Nitric acid (HNO_3, 70%)	Merck, Germany

4.3.3 Instrumentals

Instruments for the preparation of AgNPs were listed as follows.

Table 4.6 List of instruments for the preparation of AgNPs

Instruments	Details
Analytical balance	Denver instrument Company, TC-254
DI water system, R ≥ 18.2 MΩ-cm	Nanopure® Analytical Deionaization Water System
Laser pointer	Wavelength 650 nm, <5mW
House hold microwave	M-181 GN Samsung, 100-850 W, 7-power levels and 35 min dual-speed timer
Transmission electron microscope, TEM	Philip Tecnia G-2 sphera, 80.0 kV
X-ray powder diffractometer, XRD	D-8 Advance Bruker
UV-Vis scanning spectrophotometer	HP-8453 Hewlett Packard

4.3.4 Chemical preparation

- **0.1 M $AgNO_3$/50 mL**

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Weigh 84.9 mg of $AgNO_3$ and then dissolve with a small amount of DI water in a beaker. Transfer the solution into a 50-mL volumetric flask and then adjust the volume to an exactly 50 mL with DI water.

- 0.01 M $AgNO_3$/50 mL

Pipette a 5 mL of 0.1 M $AgNO_3$ as prepared above and then adjust the volume to an exactly 50 mL with DI water using a 50-mL volumetric flask.

- 0.1 M β-D-Glucose/100 mL

Weigh 1.8x g of β-D-Glucose and then dissolve with a small amount of DI water in a beaker. Transfer the solution into a 100-mL volumetric flask and then adjust the volume to an exactly 100 mL with DI water. Shake for making a homogeneous solution. The solution is freshly prepared before use.

- 0.01 M β-D-Glucose/100 mL

Pipette a 10 mL of 0.1 M β-D-Glucose as prepared above and then adjust the volume to an exactly 100 mL with DI water using a 100-mL volumetric flask.

- 1 mM NaCl/500 mL

Weigh 5.8 mg of NaCl and then dissolve with a small amount of DI water in a beaker. Transfer the solution into a 500-mL volumetric flask and then adjust the volume to an exactly 500 mL with DI water.

4.3.5 Preparation of silver nanoparticles (AgNPs)

The experimental procedure is quite similar to that for the preparation of AuNPs. Approximately 0.1 g transparent mung bean vermicelli was

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added into a 50-mL Erlenmeyer Pirex flask containing a 30 mL of DI water. Then, the mixture was placed in the microwave cavity and also heated by using the microwave power at 300 W under the presence of air for 1 min. A 5 mL of 0.01 $AgNO_3$ was added into the mixture solution. Then, a 7.5 mL of 0.01 M β-D-Glucose solution was added into the mixture solution. The reaction flask was heated in the microwave oven under the same condition for 5 min, and reheated again for an additional 5 min. After heating process, the color of starch vermicelli obviously changed from the colorless to yellow, indicating the formation of starched AgNPs. Finally, the reaction solution was cooled to RT. To further investigate the formation of AgNPs, other different experiments were carried out by increasing the concentration from 0.01 M to 0.1 M.

4.3.6 Investigation the original of two colors of AgNPs

For the preparation of yellow AgNPs, a 50 mL of DI water was added into a 250-mL beaker containing a 0.5 g of mung bean starch vermicelli. Then, a 5 mL of 0.1 M of $AgNO_3$ was added into the beaker. The reaction solution was heated by a microwave oven at 100 W under the presence of air for 5 min. The mixture was transferred from a beaker into a 100-mL Schlenk flask. A 7.5 mL of a 0.1 M of β-D-glucose was added into the reaction flask. The reaction was heated at 60°C and stirred continuously under argon gas for 6 h (see figure B.4). The color of vermicelli was observed with a yellow color. The mixture solution was cooled to RT. Mung bean starch vermicelli pieces were then collected from the reaction, dried by incubating in an oven at 60°C before the further analysis or next procedure.

For the preparation of purple AgNPs, a 50 mL of DI water was added into a 250-mL beaker containing a 0.5 g of mung bean starch vermicelli. Then a 5 mL of 0.1 M of $AgNO_3$ was added into the beaker. The reaction solution was heated by a microwave oven at 100 W under the presence of air for 5 min. Then, only starch vermicelli was washed with *a diluted solution of NaCl*. The mixture was transferred into a 100-mL Schlenk flask. A 7.5 mL of 0.1 M of β-D-glucose was added into the reaction flask. The reaction was heated at 60°C and stirred under argon

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gas for 6 h (see figure B.4). The color of vermicelli was observed with a purple color. Then, the mixture solution was cooled to RT. Mung bean starch vermicelli pieces were then collected from the reaction, dried in an oven at 60°C before the further analysis.

4.3.7 Carbonization

In order to perform the carbonization, a crucible alumina cup containing the as-prepared sample of two colors of AgNPs stabilized by starch vermicelli was placed in the central region of a furnace, and then heated in at 200°C and 300°C in the ambient atmosphere for 24 h, respectively, at a heating rate of 5°C min^{-1}. The black precipitate obtained was collected and then characterized by XRD.

4.3.8 General characterization

Transmission electron microscopy (TEM) observation was performed by using a Philip Tecnia G-2 sphera transmission electron microscope with an acceleration voltage of 80.0 kV. The sample was dispersed in a solution by using an ultrasonic bath (ca. 5 min). For the TEM measurement, the sample preparations were prepared by dispersing a drop of the sample solution on the formvar-coated Cu grids.

The crystalline phase of the samples was characterized by X-ray powder diffraction (XRD). A D-8 Advance Bruker Analytic X-ray diffractometer was operated with the CuKα radiation at the wavelength of 0.15650 nm using 40 mA and 40 kV. The diffraction pattern over the range of 30°-70° 2θ was recorded with the scan speed of 2 min/step and the scan rate of 0.015 2θ/s.

The UV-Vis absorption spectra of colloidal metal nanoparticles were performed by using a HP-8453 Hewlett Packard UV-Vis spectrophotometer with

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the path length of the quartz cell at 10 mm using DI water as a reference, over the wavelength ranging from 250 to 900 nm.

4.4 Chemical kinetics for chemistry education

4.4.1 Theory of learning

In education, particularly science education, there are many different theories for how people learn, e.g. behaviourism, constructivism, social constructivism, and sociocultural views of learning. Many schools and universities have focused on changes for improving the teaching and learning of science in classrooms. Some models of theories of learning might be relevant to the particular classroom, but some might not. There is a need to think about the particular theory that is relevant to support a means of an inquiry-based practical chemistry class. Equally, the approach to learning in this study is based on the constructivist learning and social constructivist learning theory. The essential elements of constructivism and social constructivism are described as the following sections.

4.4.2 Approach to constructivism and learning

Although constructivism is not a theory of teaching, it suggests an approach to an instruction used in most schools and universities. Its use has become increasingly popular as a reference for research studies in past decades. The term constructivism designates a contemporary educational and philosophical theory based on the work of Jean Piaget, and has been used to refer to views about knowledge and learning. The key idea of constructivism, apart from other theories of cognition, is that knowledge cannot be transmitted directly from one knower to another, but learners have to actively construct their own knowledge rather than receive preformed information transmitted by others [157].

Constructivism is accepted as the particular theory to conceptualize both personal knowledge and scientific knowledge. How do learners come to

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understand science which involves the learning of scientific concepts? Educators believe that all knowledge can be constructed. A basic principle of constructivism is the personal construction and cognitive development bridging into learning with understanding. Knowledge construction exists only in mental activity [27, 157]. Constructivism is a form of realism in the sense that the existence of a reality is acknowledged from the outset. Learners' constructions of knowledge are constrained by experiences, which comprise subjective interactions with the real world as they have constructed it. Science does not exist as a body of knowledge separate from the knower [158].

In science classrooms, the term constructivism comprises a wide variety of theoretical positions and has been widely used to refer to views about learning, teaching, curriculum and curriculum development. Hodson and Hodson [159] provided some clear features in science education towards teaching strategies that might assist students in the task of the conceptual reconstruction. These are to:

- Identify students' ideas and views;
- Create opportunities for students to explore their ideas and test their robustness in explaining phenomena, accounting for events, and making predictions;
- Provide stimuli for students to develop, modify and change their ideas and views; and
- Support their attempts to rethink and reconstruct their ideas and views (p. 34).

In the learning of science, Tobin and Tippins [27] suggested that constructivism has been increasingly used to maximize the student's learning in a classroom, that is, the teacher takes an account of what existing knowledge students bring to the classroom. From a constructivist perspective, teachers can facilitate learning in the ways already mentioned and by constraining experiences, so that students have the opportunities to build on an extant knowledge. Students should be facilitated to learn and the role of teachers in classroom should be a facilitator for

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changing the student views. Teachers need to facilitate learning, be aware of the student capabilities, what is likely to be meaningful and interesting, and what the learner already knows. It should begin with teachers understanding the rationale for implementing the curriculum as they do. Then, teachers should negotiate with their students, i.e. asking questions to think about it, arranging students to gather so they can argue toward consensus, and pointing the way to additional learning resources (p. 47) [27].

Science teachers have to help shape students' views towards the inclusion of scientific notions in their personal frameworks of understanding, that is, they also have to intervene. It necessarily involves the provision of new experiences and the introduction of new ways of inquiring and new ways of arguing [159]. The teachers' role is to mediate the learning of students. It would be important for teachers to interact with students to a greater extent, to ascertain what they knew and what they are thinking in classrooms. As mediators, the teachers have to ensure that their students are given opportunities for quality learning experiences with understanding.

However, according to constructivist perspective, there are some limitations of constructivism in the teaching and learning of science. If knowledge construction is seen solely as an individual process, then this is similar to what has traditionally been identified as discovery learning. If, however, learners are to be given access of knowledge systems of science, the process of knowledge construction must go beyond personal empirical inquiry. Learners need to be given access not only to physical experiences, but also to the concepts and models of conventional science (p. 7) [157].

Understanding the nature of constructivist teaching is still difficult. It could be argued that teaching, which is an act of persuasion from an individual to a group, is an aspect of minority influence in group discussion-making. It is neither the simplest nor the most common kind of social constructivism. Likewise, Hodson & Hodson [159] noted that learning science is not simply a matter of making sense of

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the world in whatever terms and for whatever reasons satisfy the learners, but learning science involves introduction into the world of concepts, ideas, understanding and theories that scientists have developed. Because scientific knowledge is more than personal beliefs reinforced by personally gathered observational confirmation, it is an attempt to explain the real nature of the physical universe (p. 35).

4.4.3 Approaches to social constructivism and learning

Social constructivist approaches draw on phenomenological ideas and the work of Vygotsky [160]. The social aspects of understanding and learning are increasingly important because the knowledge construction requires an active process of an interpretation with collaborative groups of learners. All cognitive functions originate in social interactions and that learning is not simply the assimilation and accommodation of new knowledge by learners; it is the process by which learners are integrated into a knowledge community.

The social aspects of understanding and learning are increasingly important because knowledge construction requires an active process of interpretation with collaborative groups of learners, in the other word, knowledge an individual constructs with society. Language and culture play essential roles both in human intellectual development and in how humans perceive the worlds. Language and culture are the frameworks through which humans experience, communicate, and understand reality [160].

According to constructivism, it is simply to think about knowledge in both individual and social components. Tobin and Tippins [158] noted that language is a tool that facilitates communication between learners in society. When a learner thinks in terms of language, the thinking is a social process even though it occurs within the mind of a single individual. It is sometimes useful to think of knowledge as an individual construct and at other time as a social construct (p. 6). Likewise, Tobin and Tippins [158] further noted that the individual and social

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components beings parts of a dialectical relationship where knowing is always dualistically as both individual and social, never one alone, but always both (p. 20).

Science is viewed as a set of socially negotiated understanding of the events and phenomena that comprise the experience universe. Knowledge is accepted by the scientific community as viable because of its coherent with other understanding and its fit with experiences. Learners have to interact with objects and events through their senses. The knowledge creation is influenced by the prior experiences and social environment of the learners.

Likewise, learning in constructivism is that individual learners have to construct their own understandings based on their experiences. The implication for learning is that students come to class with prior knowledge. Knowledge is not personally constructed for learners through their own experiences, but also socially mediated. Learning is a social process of making sense of experiences in terms of extant knowledge [158]. Learners have to interact with objects and events through their senses associated with extant knowledge that includes beliefs and images, because knowledge is the process that exists only in the minds of cognizing beings. However, social and cultural phenomena are personal constructions through their interactions with the living and nonliving components. Therefore, knowledge cannot be seen as constructed by an individual acting alone, because it is not possible for an individual to separate individual thought from the sociocultural components of what is known.

Learning means change from the socio-cultural everyday context to a new, science context. The ideas of situated cognition; authentic learning, dominated by open-inquiry activities; cognitive apprenticeship, the novices are introduces into the new culture as the expert. Learners construct knowledge through social interaction. Talk is important as learning science also involves learning the language of science. Group situations can contribute to the development of objectivity in thinking. The student-directed experimentation is particularly important in science, because it can demonstrate the principles instead of facts [157]. Learning occurs in

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work groups, in a group discussion, or in a learner's interaction with a group. Learning is collaborative with meaning negotiated from multiple perspectives. There is the collaboration in negotiated meaning because the learner is trying to interpret the other perspective and compare it to their own [135].

4.4.4 Methodological approaches in science education

Methodology is best understood as the overall strategy for resolving the complete choices available to the inquirer. Research in science education has been dominated by the use of a quantitative methodological approach, which is based on the scientific attempts to use mathematical analysis to establish general laws. Due to the difference from scientific research, educational research leads to increasing interest in a qualitative methodological approach, which is based on data gathering tools, such as unobtrusive observation, participant observation, and semi-structured interviews [155].

Quantitative and qualitative methodological approaches for inquiry each have been used in science education depended on the situation [155]. A qualitative method is suitable for evaluation researchers to study the selected issues in dept and details. Meanwhile, a quantitative method involves the use of a standardized approach in which experiences of inquirers are limited in use. The advantage of quantitative approach is that it is possible to measure the reactions of many subjects to a limited set of questions, facilitating the comparison of statistical data. Much educational research has multiple purposes being interested in both processes and outcomes. Thus, the analysis of educational data often represents a broad range of tasks by the combination of methods. For example, a statistical analysis is used to fit the data, interpreted the output results, and generalized the findings of research. A qualitative data may be used to prove value and to support the statistically significant differences to be more meaningful. Thus, some questions can be readily answered using qualitative means, or other quantitative, and some are best addressed using a combination of the two.

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4.4.5 Research design

This research reported here is a case study conducted within an interpretive paradigm as described by Merriam [161]. Consistent with a case study approach, the inquiry employed a variety of data collection tools including the use of both quantitative and qualitative instruments of inquiry as described below:

- Investigation of the student views about their experiment through semi-structured interviews;
- Investigation of the pedagogies employed in the chemistry laboratory class through via the observation of the experiments used in the teaching laboratory;
- Development of student-centered laboratory experiments based on the use of a framework presented by Hofstein and Walberg [120]; and
- Evaluation of student views about this new experiment through a purpose-designed questionnaire, face-to-face interviews and the content analysis of the relevant documentation.

4.4.6 Teaching strategy

At the first year university level at Mahidol University, in the past chemistry experiments were very traditional in outlook. By this, we mean they followed a detailed set of instructions, in what is sometimes called a ‘cookbook style’ of guidance. The experiments can help students to perform the experiment correctly and develop their technical or practical skills, purely because the instructions are detailed and clear. This means students can often successfully complete the practical part of the experiment. However, such experiments do not usually help students to engage in thinking about the experiment, or necessarily help them to understand science and how scientists themselves do experiments.

In science education, students can learn much more than just practical science skills in the laboratory. However, if we want students to learn more

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skills we need a different type of experiment, one that is more authentic (i.e., more like real science), in which students do things in a way more like what scientists do. These sorts of experiments tend to be more ‘open’ in nature; by this we mean there may be more than one way to actually conduct the experiment, or that students have some input into the design and conduct of the experiment. Such experiments are often called ‘inquiry-based learning’ because they involve students conducting an inquiry or investigation.

In this new experiment provided here, we have designed an inquiry-based experiment based on some topical issues in modern Thai, to illustrate the importance of chemical kinetics-an important part of physical chemistry. We intend to use the inquiry-based learning and group work including other important aspects of modern science (see Appendix C.1.1).

4.4.7 Probing student understanding

In order to probe student understanding, the technique of *Prediction Observation Explanation* (POE) is based on the classic model of science education research where a hypothesis is stated and reasons are given for why this may be true, relevant data are collected, and results are discussed. This method was first reported at the University of Pittsburgh where it was initially labeled a DOE (Demonstration, Observation, and Explanation). A POE task involves students predicting the results of a demonstration or an experiment, and discussing reasons for their predictions, observing a demonstration or an experiment, and finally explaining any discrepancies between their predictions and observations. The procedure is generally used to be in observable, real-time events as stimuli to prove student thinking about concepts [162].

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4.4.8 Development of inquiry-based and student-centered chemistry practical classes

A new experimental design was developed for this inquiry, and this sought to develop student-centered learning processes based on inquiry-based learning. A key feature was to a need to move students from passive to active learning. White and Gunstone [162] proposed that the technique of *Prediction Observation Explanation* (POE) is a strongly child-centered strategy that aims to arouse student curiosity, and this was adopted as part of this inquiry. The practical classes focused on the concept of chemical kinetics in practical chemistry classes using four POE tasks. In a whole class setting, students were asked to predict the result of some events and justify the reasons used to support their prediction. Next students were asked to describe what they see when a reaction occurs while doing the experiment, students were asked to write down their observations, and lastly they were required to reconcile any conflict between what they predicted and observed. Here we used the POE activities to develop and practice a student-centered approach, in this laboratory class, in a combination with other techniques, such as small-group learning [42, 135, 138] and the negotiation or argument (Pinnell, 1984) in order to develop the student conceptual understanding (see Table 4.7).

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Table 4.7 Framework for designing and conducting teaching strategies in student centered chemistry laboratory classes (after Hofstein and Walberg [120])

Items	Sample Items
Laboratory activities	Students learn a different type of the experiment, i.e., more like 'real' inquiry-based science experiments.
Probing student understanding	Students are asked to make the prediction-observation-explanation (POE).
Inquiry techniques	Students learn inquiry-based work how to develop relevant inquiry skills, i.e., identifying problems, generating questions, conducting investigations and defending hypotheses.
Small-group discussion	Students work cooperatively on learning activities in a small group based on their group performances. Students were seen to take place as an individual activity through thinking and writing, or as a social activity to take place within a group.
The use of technology	The experimental data from the experiment can be analyzed using computer, i.e., plotting the relationships between the productions of carbon dioxide over time.

This sort of experiment tends to be more 'open' in nature, and the intention was that the students would be doing things in a way more like what scientists do. It should be noted that the students here do the practical work in a more authentic manner, than just following instructions in the laboratory manual. That is, the students had to design the experiment procedure themselves, and this was intended to help them gain an understanding of the process of scientific inquiry. So in summary in these practical chemistry classes, the learning consisted of students developing the methodology to investigate the influence of some variables on rates of reaction, and specifically to use the POE strategy in small groups to predict and think

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about the expected results and write them down on answer sheets. Their ideas were discussed in small groups. During classes they completed four POE tasks in the form of paper-based drawings on the sheets provided. Finally, students in each group reported their prediction, observations and explanations about the chemical kinetics in a whole class setting.

In summary in these practical chemistry classes, the learning consisted of students developing the methodology to investigate the influence of some variables on rates of reaction, and specifically to use the POE strategy in small groups to predict and think about the expected results and write them down on answer sheets. Their ideas were discussed in small groups. During classes they completed four POE tasks in the form of paper-based drawings on sheets provided. Finally, students in each group reported their prediction, observations and explanations about the chemical kinetics in a whole-class setting.

4.4.9 Sample description

The sample chosen for this study involved the first-year undergraduate science students (age range 18-19 years) from the Faculty of Science, at Mahidol University in Thailand. These students enrolled in general chemistry laboratory course, SCCH 108 in the first semester. The course comprises 12 practical classes in which the chemical kinetics of acid-base reactions is one session. In general, the laboratory classes consist of practical experiments that complement the lecture material and the lectures are done before the experiment. The practical classes overall try to teach basic practical chemistry techniques, including the use of pipette and burette, concentration preparation, and redox chemistry. All of the students in classes here were familiar with cooperative group work and POE tasks, and completed the experiment in a three-hour period. Here, for the student interactions in group learning, the optimum size for small-group learning in most situations used was four to five students in each group as recommended in the literature [140].

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4.4. 10 Data collection

The researchers began by collecting and conducting an in-depth analysis of materials produced in the laboratory when the students engaged in the POE inquiry-based learning practical classes mentioned above (see Appendix C.1.2). Most of the groups of students completed the pre-test and post-test laboratory questions (see Appendix C.1.3 and C.1.4). Students in groups also completed their laboratory documentation in Thai, but some completed their documentation in English (many Thai students are bilingual in Thai and English). The students then completed a short questionnaire that sought their views on their experiences of doing these new practical classes. Out of a class of 413, some 389 students consisting of 129 male and 260 female completed the questionnaire (see Appendix C.1.5). The participant students were enrolled in the general chemistry laboratory course, SCCH 108. Students' responses about their enjoyment of the experiment used a Linkert scale (SD = Strongly Disagree, D = Disagree, N = Neutral, A = Agree and SA = Strongly Agree) (Merriam, 1988). Selections of students were interviewed in pairs in order to ascertain their views on the 'new' practical classes in more depth. In order to obtain more understand, students were interviewed by semi-structure interview protocol. Interviews were audio-taped and transcribed. Any translations were checked for veracity by independent bilingual advisors. Claims were also supported the findings from interviews after completing the experiment.

4.4.11 Data analysis

The researchers intended to analyze the data by using the triangulation, which consists of the laboratory documentation, the pre- and post-test questions including the POE task, and questionnaire. The first item is based on the laboratory documentation. The students are given an opportunity to think of steps or ways to approach the problems presented to them. Scientists generally follow a number of logical steps when they attempt to solve problems such as that posed to the participants in this inquiry; in what is often termed 'the scientific method'. The participant-validated transcripts here were inspected for statements that revealed

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the students' views for each of variable which affect the rate of a reaction. These statements were collected with students' drawing in the laboratory documentation about their conceptual understanding of chemical kinetics. When students identified the independent variable, they had to provide what are the dependent variables. Their views about the concepts were compared with conceptualization by reference to the textbook in this appropriate level.

The second item for analyzing students' data is based on the pre- and post-test questions including their drawing on the paper sheet of the POE tasks during instruction. These questions, available from the researchers, were similar in nature and contained questions deemed by a panel of experts to be suitable for this educational level. Students were provided with a balanced equation for the reaction of acid with carbonate and asked questions such as how particle size, temperature or other variables affect the rate of reaction. They also were asked to provide explanations for their answers. For the POE tasks, their description resulted in forming hypotheses to be tested by collecting data, and conducting 'scientific' experiments, in which they controlled all but one variable. The researchers intended to analyze key worlds of all activities, in which students provided the outcome of their results in advance, and explained the expected results.

The last item for analyzing the student data is the use of the questionnaire. After complete the experiment, the students provided what happened in the classroom based on their perception of such the teacher role, POE technique, and group learning in the laboratory of chemical kinetics. The statistical results obtained were analyzed by the SPSS software, which is widely used and very helpful software in science education. In general, students' responses were summarized into in-dept analysis of research findings.

By interpreting all data, it was possible to gain the perspective of the students' views about the chemical kinetics. These views were organized into a series of interventions used.

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4.4.12 Validity and reliability in research

In science education, triangulation is the most effective means enhancing the validity and reliability in research findings. Triangulation [163] involves the gathering information from a number of sources in research. By comparing and contrasting one source of information with another, the researcher is able to produce a better comprehensive in responses to balance the research questions. Triangulation not only provides more information about the validity of a study, but also aids in the interpretation in which it is reasonable to support and solve the problem of the study.

There are three forms of triangulation used in practices: (i) data triangulation, (ii) investigation triangulation, and (iii) methodological triangulation.

Data triangulation involves the use of difference data sources for the study, and this can take a variety of types: time, space (i.e., situation), person, and levels: aggregate (i.e., individual person), interactive (i.e., the individuals' immediate neighbors in the social setting) and collectivity (the larger group i.e., a company or institute in the individuals' social setting).

Investigation triangulation involves the use of the different investigative staff during the data collection. This is common in quantitative inquiries that frequently involve the research assistants.

Methodological triangulation comes into two forms: (i) within-method triangulation using the same method (i.e., survey) and employing multiple strategies, and (ii) across-method (or between-method) triangulation applying different methods (e.g., observation, interviews, and questionnaire) to the same research question. In general, the researchers hold the across-method triangulation that attempts to relate different sources of data in a way to be as possible validity of a study.

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CHAPTER V
RESULTS AND DISCUSSION

This chapter comprises a description of results and discussion from the research findings. It begins with the data collection of magnetic iron oxide nanoclusters and then follows by gold nanoparticles, and silver nanoparticles. All experiments were carried out using the mung bean starch vermicelli as a template for the synthesis of nanostructures. Finally, it involves the results collected from the experiment of eggshell kinetics followed by the qualitative and quantitative analysis.

5.1 Magnetic iron oxide nanoclusters

5.1.1 Thermal analysis

In order to determine the heating process, the commonly used methods for thermal analysis of the iron oxides are thermal gravimetric-differential scanning calorimetric analysis (TGA/DSC). In general, The TGA technique provides information about the temperature-dependent weight loss of the sample. Meanwhile, the DSC method involves recording phase-specific changes or transformations of the sample during heating. To obtain these, a required amount (11.89 mg) of the as-prepared dry sample was also examined by TGA and DSC by continuously heating from room temperature (~30°C) to 700°C with an increase of a constant temperature rate of 10°C min^{-1} under a flowing of nitrogen atmosphere, and the weight loss of the sample was measured using a balance (see Fig. 5.1).

As shown in a result, a fine-grained material contains an appreciable amount of absorbed water which leads to a weight loss over a wide temperature range of between 100 and 200 °C, and this can usually be identified of the bonding water in the sample [164]. In addition, the major transition due to the weight loss occurs over

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a narrow temperature range from 282.4°C to 315.8°C. The weight loss of the sample (8.02 mg) was calculated on the basis of the TGA data and also found to be 67.50% from an initial amount. Meanwhile, the DSC scan shows two endothermic peaks around 58.9°C and 283.3°C, respectively. The first peak is due to the release of the absorbed water of dehydration. Even, the second one is obtained from a phase transition of the iron oxide change. The main endothermic effect results from the combustion process or consume of energy of the as-prepared iron oxide consuming energy at 47.8 J g^{-1}, and this may involve the oxidation in the presence of oxygen at high temperature in the TGA experiment. For example, it is well known that magnetite (Fe_3O_4) can be easily oxidized to maghemite (γ-Fe_2O_3), which can be further transformed into hematite (α-Fe_2O_3) at high temperature [13]. This was found to be the same as results reported by Mikhaylova [165] and Wang [166]. Moreover, above temperature 400°C, there was no weight change observed.

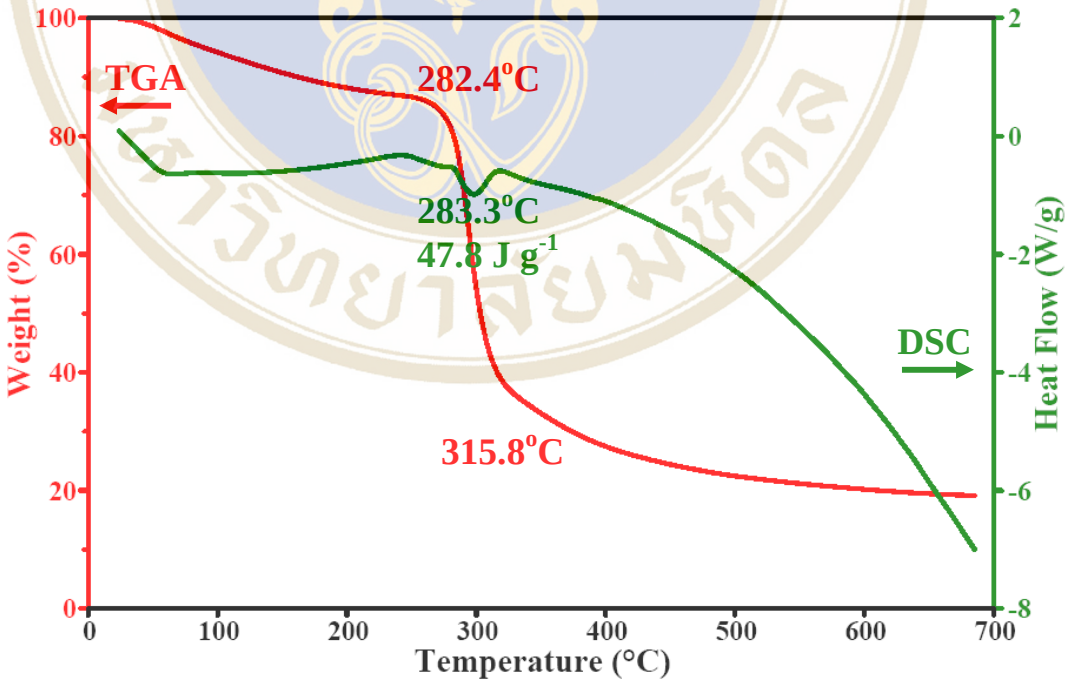

Figure 5.1 Thermal analysis by using thermal gravimetric-differential scanning calorimetric (TGA-DSC) analysis of mung bean vermicelli deposited with the as-prepared magnetite (Fe_3O_4) nanoparticles

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5.1.2 Electron microscopy (EM) analysis

Owning their small particles size, the surface morphology of the as-prepared magnetic iron oxide crystals stabilized by mung bean starch vermicelli template were commonly examined by using scanning electron microscopy (SEM). The as-synthesized Fe_3O_4 nanoparticles were prepared by using ammonia solution for the precipitation of the solid powder, and the black precipitated product was also obtained and collected by applying a magnet. After magnetically stirring, the representative field emission scanning electron microscopy (FE-SEM) micrograph shows a large quantity of the formation of the nanoscale iron oxide particles of spherical nanoclusters in shape (see Fig. 5.2). It was found clearly that spherical nanoclusters with the diameter about 200-300 nm were formed by the assembly of many small iron oxide nanoparticles.

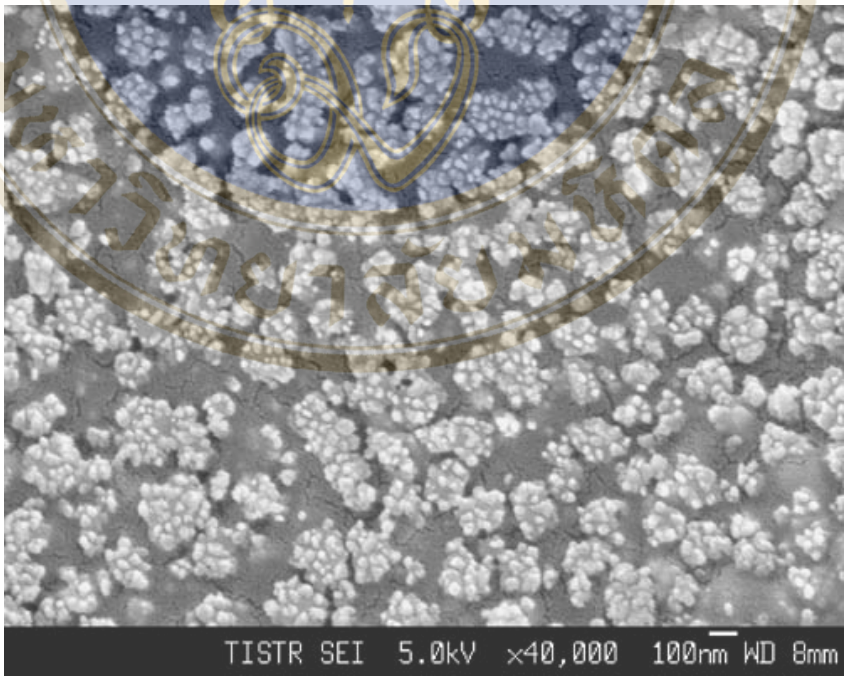

Figure 5.2 The representative FE-SEM image of the as-synthesized magnetic iron oxide nanoclusters stabilized by mung bean starch vermicelli used as a template

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To confirm the more information of the crystals of the as-prepared sample, the transmission electron microscopy (TEM) was further used to examine the morphology (see Fig. 5.3). After broken up by using an ultrasonic treatment, the representative TEM micrographs exhibit a region of magnetic iron oxide nanoclusters, which was found to be similar to the FE-SEM result. Interestingly, it can be seen that the some nanowires with several hundred nanometers in distance were organized from the connection of small magnetic iron oxide nanoparticles.

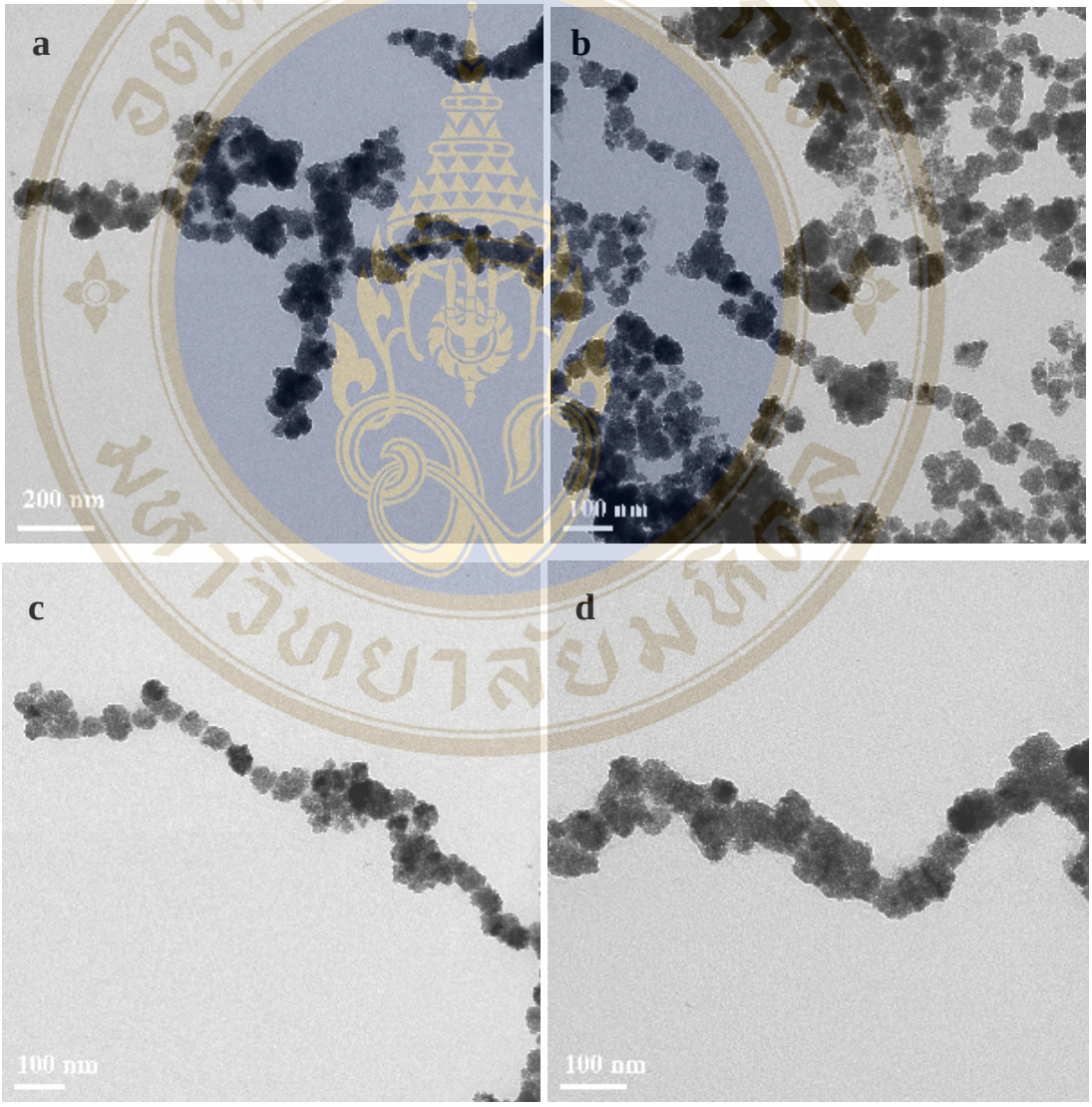

Figure 5.3 Representative TEM images showing a region of a self-assembled chain of the as-prepared Fe_3O_4 nanoparticles. The bars of (a), (b), (c) and (d) correspond to 200, 100, 100 and 100 nm, respectively

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5.1.3 X-ray powder diffraction (XRD)

The characterization of the crystalline nature of magnetic iron oxide nanostructures was carried out by using X-ray powder diffraction (XRD) analysis, which is essential for identification of the product [13, 167]. The crystal structure and phase composition of the as-prepared samples was achieved by investigating the XRD patterns before and after carbonization (see Fig. 5.4). Before carbonization process, the position and relative intensity of diffraction peaks from the XRD patterns of the as-prepared samples closely matches that of Fe_3O_4 powder diffraction data rather than similarly the structured γ-Fe_2O_3 [13, 167]. During heating process, the mung bean starch vermicelli template was carbonized under the presence of air. All the peaks of the final products can be indexed closely to the cubic Fe_3O_4 nanoparticles, which are consistent with the data in the standard card (the JCPDS file no. 19-629).

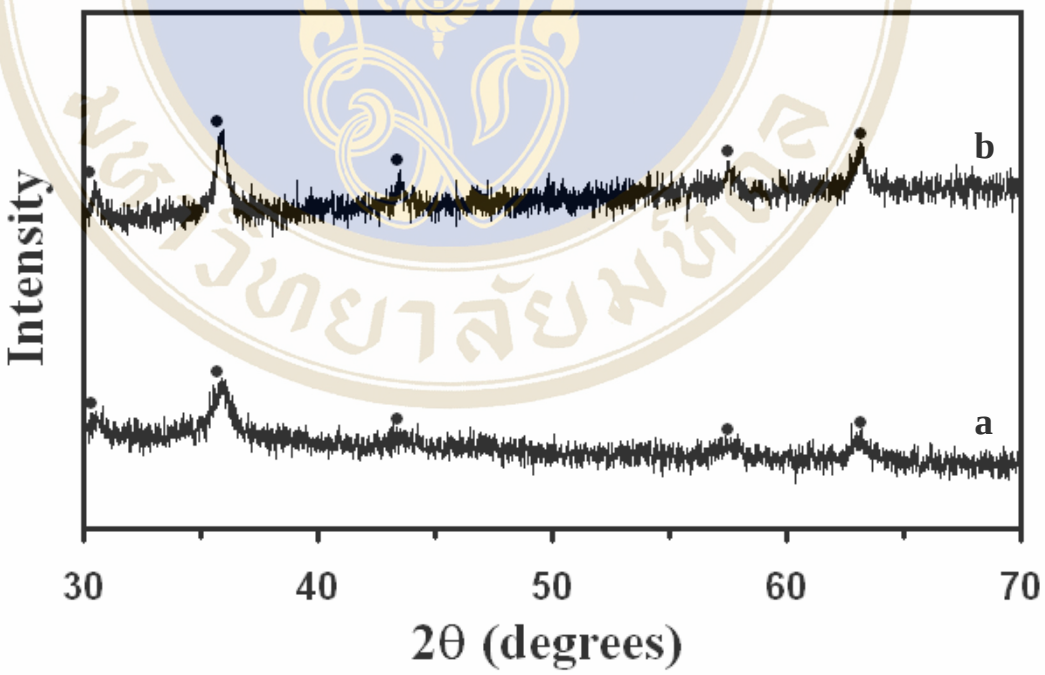

Figure 5.4 XRD patterns of as-prepared iron oxide nanoparticles (a) before and (b) after carbonization. The XRD peak positions for Fe_3O_4 were drawn from the JCPDS file no. 19-629.

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In addition, XRD results provide information concerning the crystal size of the as-prepared sample. The average size of the as-prepared magnetic iron oxide nanoparticles can be calculated from the width of the representative peaks of the XRD patterns by using the Scherer's equation [168]:

$$d = \frac{0.9\lambda}{B_\theta \cos\theta_B}$$

where B_θ is the width of the peak at half height and λ is the wavelength of the X-rays. θ_B is the Bragg angle. From the XRD results, after carbonization the average of the final Fe_3O_4 nanoparticles is about 14.8 nm. It is similar to Sun [63] who reported that high-temperature reaction of the iron oxide can be used to make the small Fe_3O_4 nanoparticles from the larger ones.

5.1.4 Vibrating sample magnetometry (VSM) analysis

The saturation magnetizations of the as-prepared samples were derived from the VSM measurement at room temperature by cycling the magnetic field between -10 kOe to 10 kOe (see Fig. 5.5). The saturation magnetization of the as-prepared magnetic iron oxide derived from pH 12 before the carbonization was 34.8 emu g^{-1}, which is quite similar to the result reported by Wang [166]. In addition, the saturation magnetization of the final powder after the carbonization was decreased to 5.1 emu g^{-1}. The saturation magnetization decreases when the crystallite size of nanoparticles is reduced as illustrated in the XRD analysis. In this case, the heating process may result the major cracking of iron oxide nanoclusters to iron oxide nanoparticles [169, 170]. The magnetic iron oxide nanoparticles stabilized by the organic structure of starch vermicelli template was carbonized in air into black carbon which could form the magnetic iron oxide nanoparticles/carbon composites.

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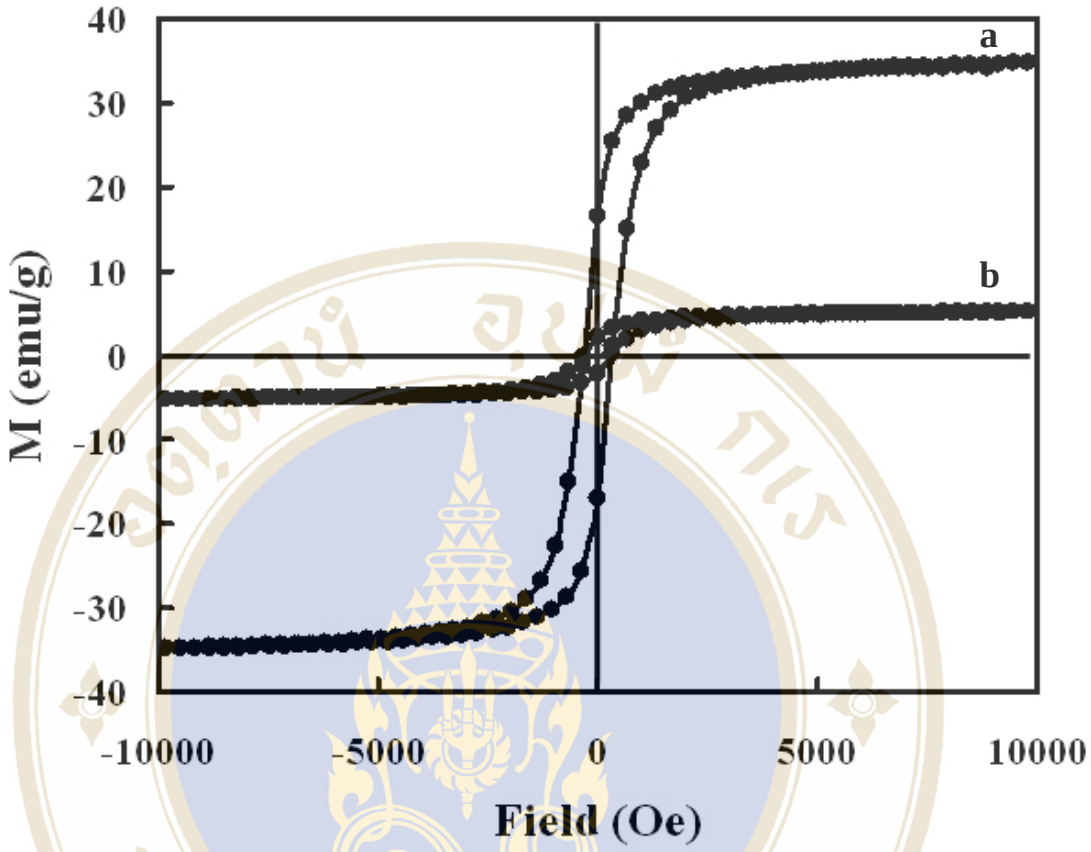

Figure 5.5 Hysteresis loops at room temperature for the as-prepared magnetic iron oxide nanoparticles (a) before and (b) after carbonization.

As the hyteresis loops of the VSM, this can be also inferred that the final product after thermal decomposition was magnetically softer. The results obtained here were found to be similar to Xiong and co-workers who recently reported the magnetic properties of iron oxide nanoparticles/carbon composites through the formation of polymer with magnetic iron oxide nanoparticles by heated at different temperatures to carbonize the carbon matrix [171]. They suggested that the carbonization of magnetic iron oxide nanoparticles stabilized by carbon matrix to form iron oxide nanoparticles/carbon composites could result in the reduction of the magnetic properties of iron oxide nanoparticles. Therefore, this is commonly employed for the reduction of magnetic properties of magnetic iron oxide nanoparticles to obtain various magnetic nanoparticles/carbon composites by the carbonization process or pyrolysis method. In addition, the coercivity of the sample

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after the carbonization remains unchanged at 298 Oe. The research findings here indicated that the carbonization can be attributed to make nanoclusters to nanoparticles. This may be used as component vehicles for the magnetic field-directed drug delivery of biomagnetic applications, since the carbonization process can reduce the strength of the magnetic field required to obtain the magnetic behavior.

5.1.5 UV-Vis spectrophotometry analysis

Another measurement of evidence supporting the formation of the colloidal iron oxide nanoparticles was monitored by using the UV-Vis performance, which can be used to provide the surface plasmon resonance band. After carbonization, the black precipitate powder was broken up by using an ultrasonic treatment (ca. 5 min). The typical UV-Vis spectrum of the as-prepared magnetite iron oxide nanoparticles was dispersed in water (see Fig. 5.6). The characteristic of the optical absorption for colloidal magnetite (Fe_3O_4) nanoparticles was not found the feature of absorption bands in the visible region, which is quite similar to the result reported by Wang [166]. This can be used as one piece of evidence for supporting the formation of the magnetite (Fe_3O_4) nanoparticles.

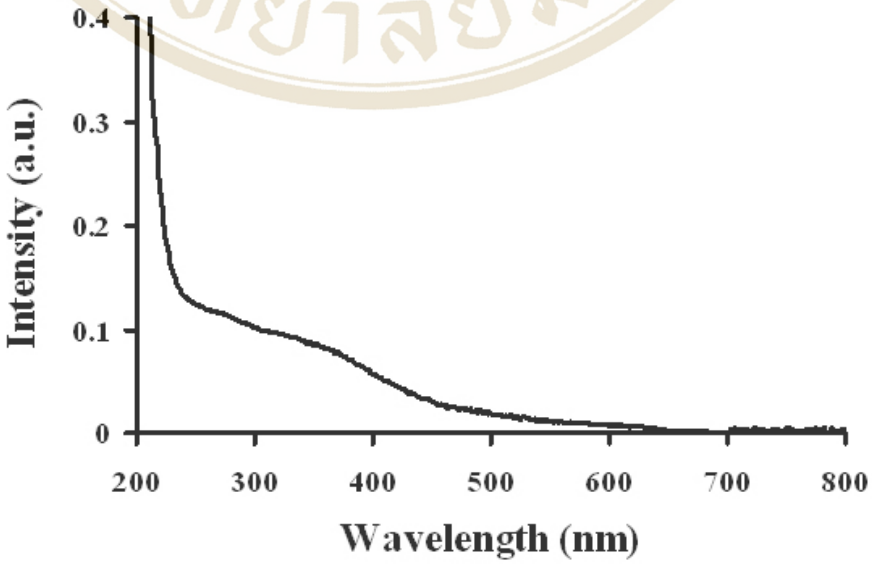

Figure 5.6 UV-Vis spectrum of the colloidal magnetite (Fe_3O_4) nanoparticles

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5.2 Gold nanoparticles (AuNPs)

5.2.1 Microwave irradiation

Microwave chemistry has experienced an exponential growth rate in both of industries and academia. The microwave irradiation becomes an increased attention in many fields of the scientific research including nanotechnology. In fact, microwave heating method has been widely used in chemistry over past decades [172, 173]. Due to the rapid, simple and selective heating of the power, the use of microwave has been also developed to enhance the reaction rates in synthetic methodology. In this work, an interest area to us is the use of the controlled microwave heating for accelerating the reaction. In this work, a household microwave oven was used to enhance both rapid heating rates and high temperature to drive the chemical reaction for the preparation of metal nanostructures. The selective heating of microwave allows both rapid heating and high temperature to be reached which drive chemical reactions. In addition, it is possible to perform reactions quickly for the synthesis of nanoparticles.

In the field of nanotechnology, it is still now a challenge to synthetically controlling the size and shape of metal nanoparticles. As known, microwave chemistry has experienced the increasing rate of use in both industrial and academic areas, and microwave-based synthesis of nanometer-scale materials has been also widely use and developed [173]. Microwave heating involves direct absorption of energy by functional groups that bear ionic conductivity or a dipole rotational effect, and this energy is then released into the surrounding solution. In our study, a microwave household oven was used to synthesize gold nanoparticles with a desired size in a substantial quantity. Compared with the traditionally conventional heat treatment; microwave-based synthesis is now accepted that it is an efficiently effective method of many strategies. The absorption of microwave energy causes the functional groups involved to have higher reactivity with surrounding reactants than when they are simply incubated at the same temperature. Moreover, there are many advantages of using microwave irradiation.

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For example, it is simple, fast, and energy efficient synthesis and can provide a uniform heating during the formation of nanoparticles. This is the great advantage of using microwave method over the conventional chemical method.

5.2.2 Transmission electron microscopy (TEM)

To further investigate morphologies and sizes of nanostructures, the transmission electron microscope (TEM) was used to examine the particle size of the as-synthesized nanoparticles. In most nanotechnology laboratory teaching, the TEM instrument is common machine to investigate nanoscale-sized particles. A randomly selective sample of colloidal gold from a fresh synthesis was prepared by dispersing a small drop of a clear sample solution onto a formvar-coated 300-mesh copper grid and allowing the samples to dry in the presence of air. The TEM micrographs exhibited clearly a well-dispersed region of nanoparticles (see Fig. 5.7).

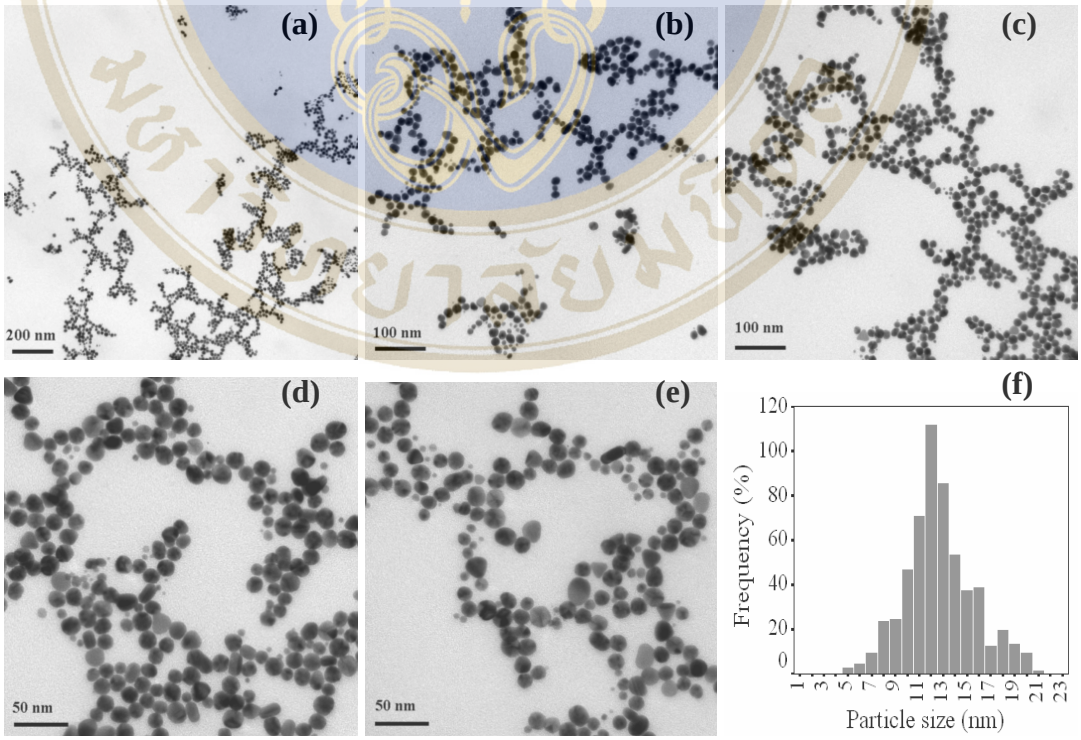

Figure 5.7 TEM images of as-synthesized gold nanoparticles corresponding to a histogram of the size distribution from TEM micrographs (average particle size = 12.7

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± 2.8 nm). The bars of (a), (b), (c), (d) and (e) are 200, 100, 100, 50 and 50 nm, respectively. The total number of single particles counted for a histogram is 573.

The resulting products mainly consist of an extremely uniform in size of spherical nanostructures of gold. The average particle size of the as-prepared gold nanoparticles with diameters was 12.7 ± 2.8 nm (n = 573), with a maximum of 21 nm in size distribution. A particle size of gold nanoparticles is quite nearly to the literature [174], but average size distribution is smaller than in size.

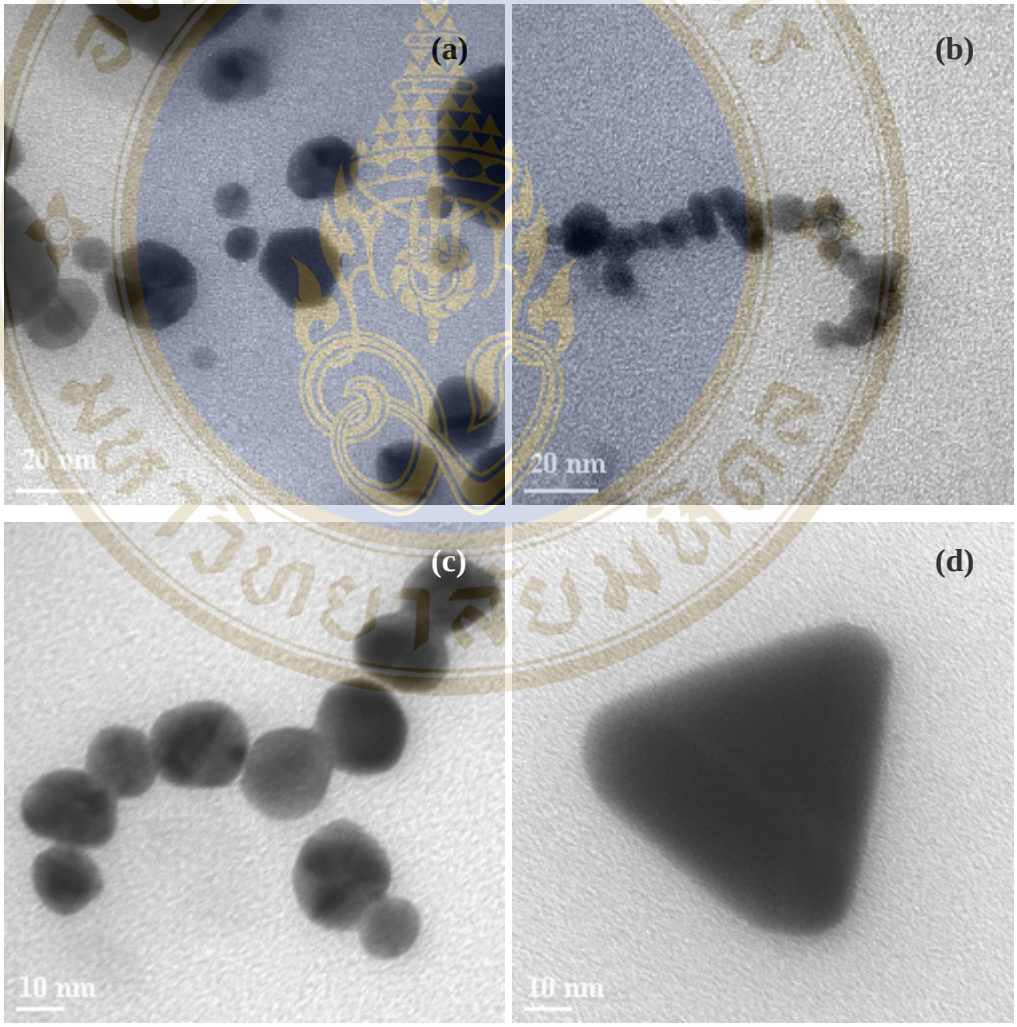

Figure 5.8 HRTEM images of as-synthesized gold nanoparticles after microwave heating process. The bars of (a), (b), (c) and (d) correspond to 20, 20, 10, 10 nm, respectively.

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Interestingly, few different morphologies of the as-prepared gold product were found in the same sample. To obtain a clear picture, the regular structure of these particles was further examined using high-resolution transmission electron microscopy (HR-TEM) observation. As shown in figure 5.8, a representative sample of the resulting product of various shapes (e.g. polygonal, hexagonal and triangular shape) of gold nanoparticles was obtained when increasing of the reaction time for the microwave heating process. As shown in figure 5.8b and 5.8c, gold nanowires with the approximate distance of 100 nm were observed in the same sample. The formation of nanowires jointed by gold nanoparticles was facilitated by the helical molecular structure of starch acting as nanoreactors.

5.2.3 X-ray powder diffraction (XRD)

In order to understand the crystalline structure of gold nanoparticle formation, the X-ray measurement is one of the premiere tools to investigate the atomic structure of small nanoparticles. It has been used as a common tool to examine the crystal structure of the samples [168].

Since gold nanoparticles was embedded within mung bean starch vermicelli acting as stabilizing agent, the broad reflection was observed over a 2θ range due to the low crystallinity of starch. After incubating, the dried powder product obtained was ground, and further characterized by using a D8 Advance Bruker analytic XRD apparatus. There existed four diffraction peaks assigned 2θ at around 38.4, 44.5, 64.7 and 77.6, respectively, for the face-centered cubic (fcc) metallic gold (JCPDS file no.04-0784). Moreover, these positions of the diffraction peaks of the as-prepared gold nanoparticles showed a match with the reported results in the literature [17, 85, 166].

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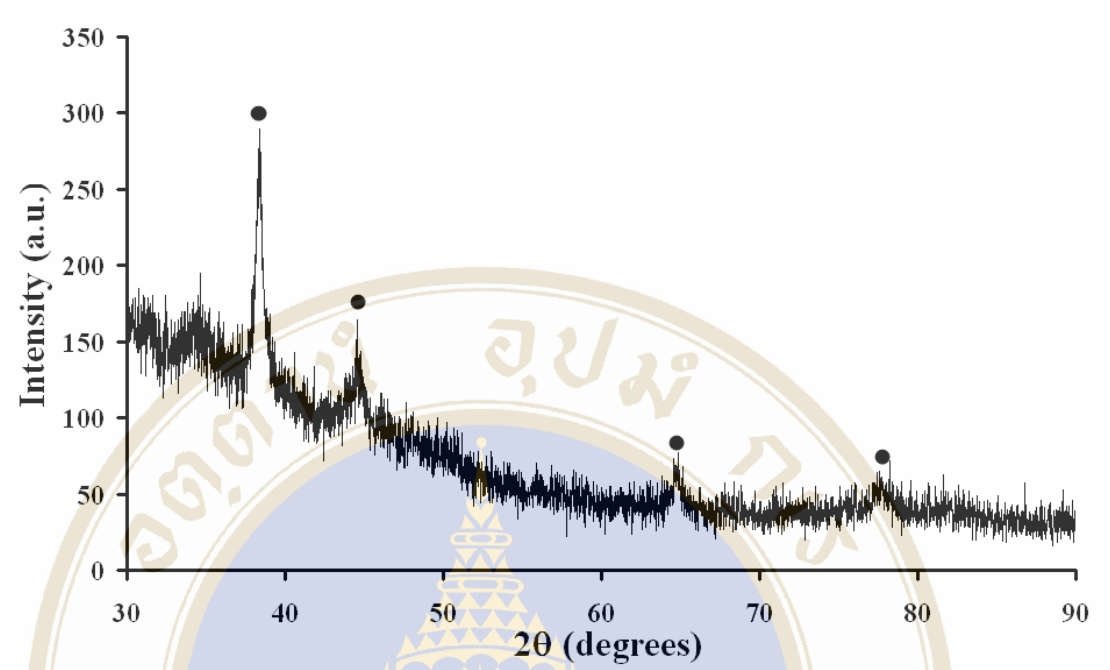

Figure 5.9 The XRD patterns of the as-prepared gold product. The XRD peaks for gold nanoparticles were drawn from the JCPDS file no. 04-0784.

5.2.4 UV-Vis spectrophotometry

The UV-Vis absorption spectra of the colloidal gold solution, according to the surface plasmon absorption of gold nanoparticles, were investigated by using a UV-Vis spectrophotometer. The experiments were carried out to study the formation of gold nanoparticles from $HAuCl_4$ solution by varying time over 5 to 35 min, and the concentration of gold ions in the aqueous solution was controlled. It was found clearly that the intensity of the surface plasmon absorption of colloidal gold nanoparticles increased with time. It is well known that the colloidal gold nanoparticles in an aqueous solution exhibit a broad absorption band in the visible region at wavelength from 517 nm to 575 nm due to Mie theory [17, 166].

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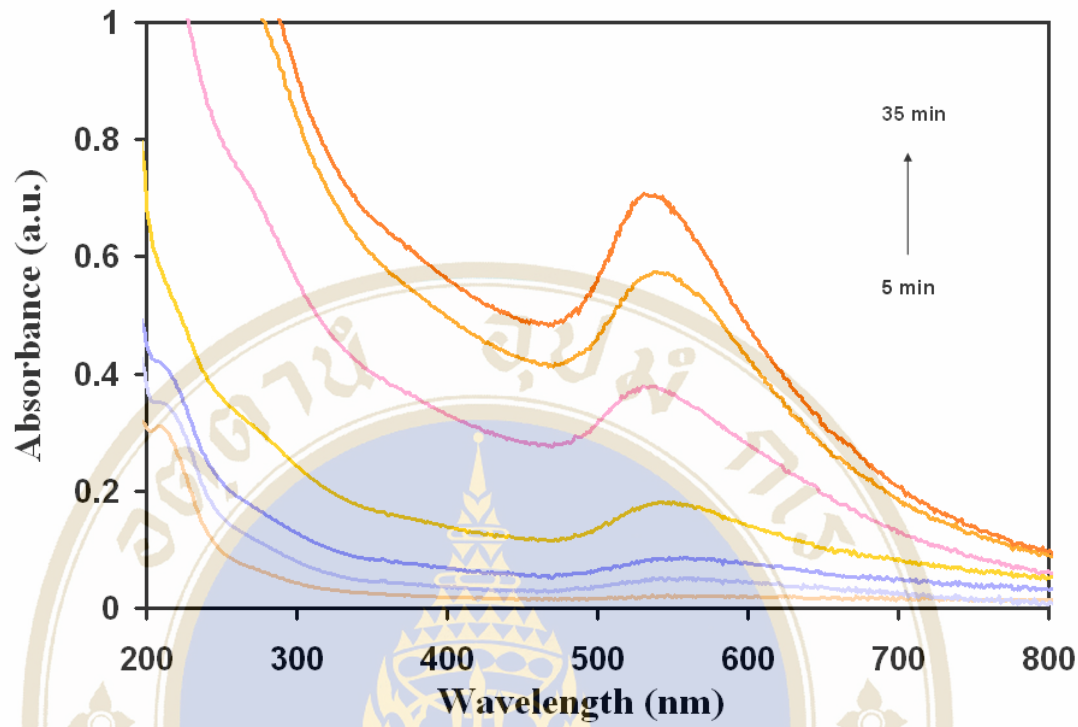

Figure 5.10 UV-Vis absorption spectra showing the time evolution of the formation of gold nanoparticles at the different time from 5-35 min

As can be shown in figure 5.10, the results showed clearly that the strong absorption peaks were around 525 nm for the as-prepared gold nanoparticles, and showed a match with the reported results in the literature [17]. These absorption values may not include the protecting agent of starch template, because the Mie scattering responds only to the nanoscale gold particles. Also, the plasmon band was symmetric, indicating that the colloidal solutions had no aggregation of particles. The strong intensity of an absorption peak became increase, indicating that the reduction reaction of gold nanoparticles has greatly proceeded. In addition, the tendency of the absorption peak also slightly shifted to red, implying a decrease in the particle size for the formation of gold nanoparticles.

5.2.5 Starch utilization in the synthesis of nanostructures

It is well know that polymers can be used for the synthesis and stabilization of nanoparticles. Many different kinds of natural polymers have been

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used as stabilizing agent for a good host embedding many guests of noble inorganic nanoparticles. However, some toxic reducing agents (e.g., sodium borohydride and dimethyl formamide), which are highly reactive and potential environmental risks, are used in the nanoparticle synthesis. In past few years, increasing awareness about the environment has become to focus on developing a “green” chemistry by using nontoxic chemicals acting as the stabilizing agent. The concept of green chemistry used in the synthesis of metal nanoparticles was first reported by soluble starch in which the synthesis of silver nanoparticles was carried out and stabilized and using the β-D-glucose as a nontoxic reducing agent [84].

Among several biomolecules, starch is one of the most abundant materials on earth. The knowledge on the structure of starch has been recently advanced [69]. As known, the simple description of starch is well known as a simple polymer of glucose. It is now accepted that there is a continuum of starch polymer structures containing amylose linear and amylopectin branches. The spectroscopic and microscopic methods are well advanced for probing the branching pattern and branch length of starch polymer. The structure of starch is packed in semicrystalline granules containing two types of α-D-glucose homopolymers, linear amylose and branched amylopectin. Amylose and amylopectin are arranged radically and aligned perpendicularly to the growth rings and to the granule surface [92]. The starch transformation can be also provided into various forms depending on different conditions. For example, transparent vermicelli can be produced from mung bean starch, which is one of the most high amylose components. In a past decade, starch has been shown as a good host for many guests of metal nanoparticles. However, there has less attention for the development of synthesizing and embedding nanostructures onto the surface and into the structure of starch.

Starch is polyhydroxylated macromolecules consisting of glucose units connected by an oxygen linkage to form amylose chains. It presents an interesting dynamic supramolecular associations facilitated by inter- and intra-molecular hydrogen bonding resulting helical structure, which can act as nanoreactors for the growth of gold nanoparticles. To avoid the use of toxic reducing agent,

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the knowledge on acid-catalyzed hydrolysis of structures of starch has been advanced for our study [175]. As far as we know, no report has been published about synthesizing gold nanoparticles with mung bean starch vermicelli as capping agent. The structure of solid mung bean starch vermicelli placed in a diluted hydrochloric acid was hydrolyzed by using microwave irradiation. The expected product from hydrolysis of starch was found to be glucose and short amylose chain containing an aldehyde terminal group.

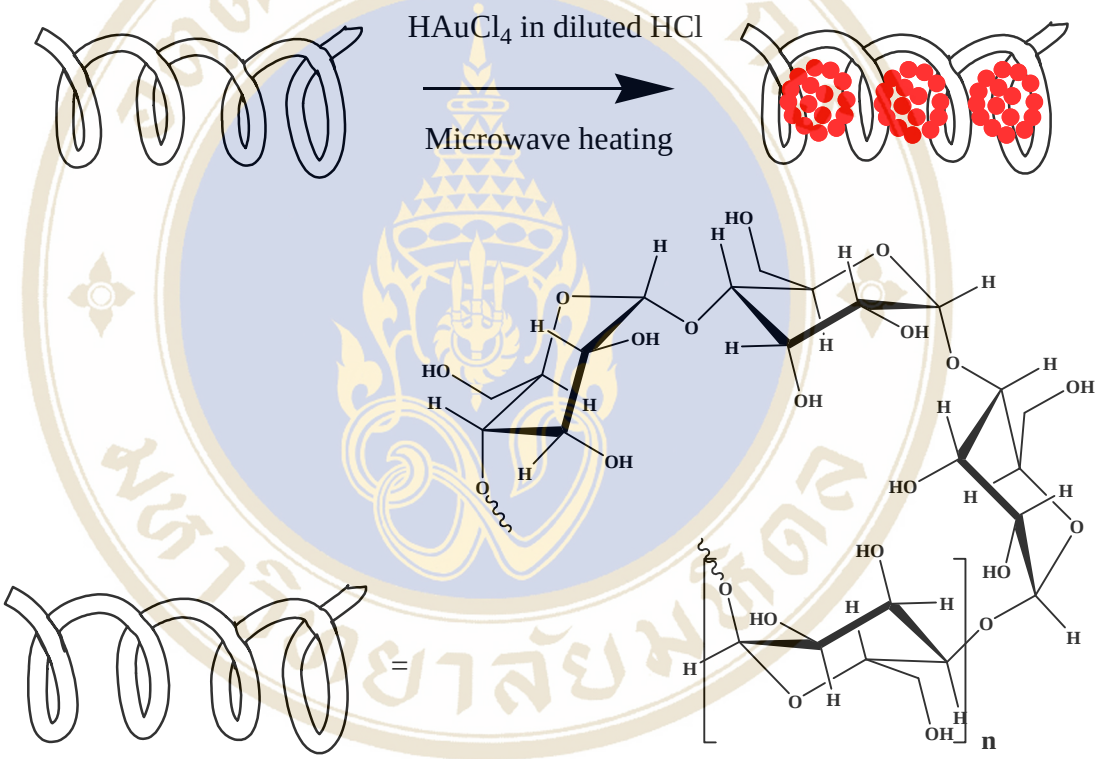

Figure 5.11 Illustration of the helical structure of starch template-assisted synthesis of size-controlled gold nanoparticles

The above results and discussion suggested the following mechanism for the formation of gold nanoparticles capped with mung bean vermicelli. During microwave heating process, microwave was able to introduce gold ions into starch template. The hydroxyl groups of starch can facilitate the gold ions by electrostatic binding in the helical structure of polysaccharide. The aldehyde terminal from the hydrolysis of starch could reduce gold ions to metallic gold nanoparticles,

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while the mung bean starch vermicelli itself was used as template to stabilize the as-prepared gold nanoparticles (as shown in Fig. 5.11 above).

Figure 5.12 Photographs of vials containing: (a) only starch template of mung bean vermicelli without the reduction of gold nanoparticles (left) and starch capping gold nanoparticles inside templates (right): (b), and (c) high magnification of starch capping gold nanoparticles after the period time of three days and three months of storage at room temperature, respectively

The color of mung bean starch vermicelli turned to pale red after microwave irradiation (see Fig. 5.12). Interestingly, starch capping gold nanoparticles was found to be stable in the vermicelli template over the period time of three months of storage at room temperature, while there was no sign of aggregation of gold nanoparticles (see Fig. 5.12c). This fact indicated that the mung bean starch vermicelli template could be used to prevent the particles from their aggregation.

5.3 Silver nanoparticles (AgNPs)

5.3.1 Transmission electron microscopy (TEM)

With the microwave heating, a yellow color was observed at the mung bean starch vermicelli and more intense yellow color was dispersed into the solution as time passed. The TEM images of the yellow silver suspension were monitored by a Philip Tecnia-G2 Sphera transmission electron microscope (TEM). The TEM images of the typical silver nanoparticles obtained from a 0.01 M $AgNO_3$ solution are shown at the different TEM magnifications (see Fig. 5.13). It is found

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that silver nanoparticles are clearly formed as a spherical shape and well dispersed of particles. The particle size distribution is shown in a histogram. The average particle size of silver nanoparticles with diameters also is about 20.0 nm.

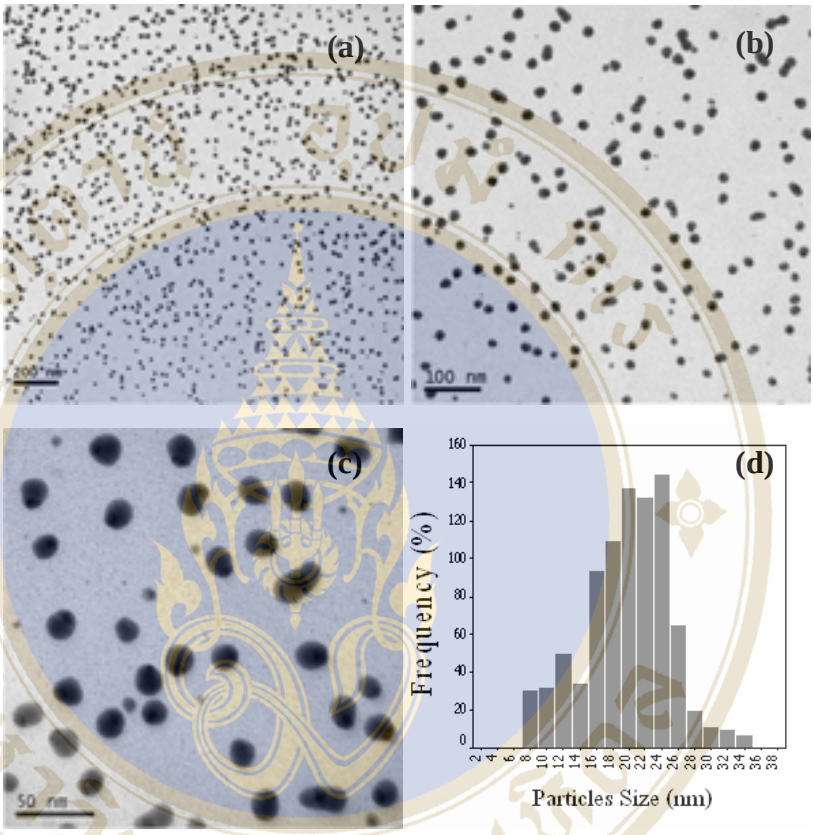

Figure 5.13 TEM images of as-synthesized silver nanoparticles corresponding to a histogram of the size distribution from TEM micrographs (average particle size = 20.0 nm, δ = 5.14 nm). The bars of (a), (b) and (c) correspond to 200, 100 and 50 nm, respectively. The total number of single particles counted for a histogram is 879.

It is noted that the concentration of $AgNO_3$ played a significant role in the formation of silver nanoparticles. In order to study further an evidence for increasing the concentration of an aqueous silver nitrate solution, the experiments were carried out to prepare silver nanoparticles from using a 0.1 M $AgNO_3$ solution while doing the same experimental conditions. As a result, the TEM images of a typical silver nanostructure are shown at a different TEM magnification. It is clear

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that most of the reduced silver nanostructures are a dendrite shape, and also there are some single silver nanoparticles dispersed between the branch/branch. The average particle size with diameters is around 12.6 nm.

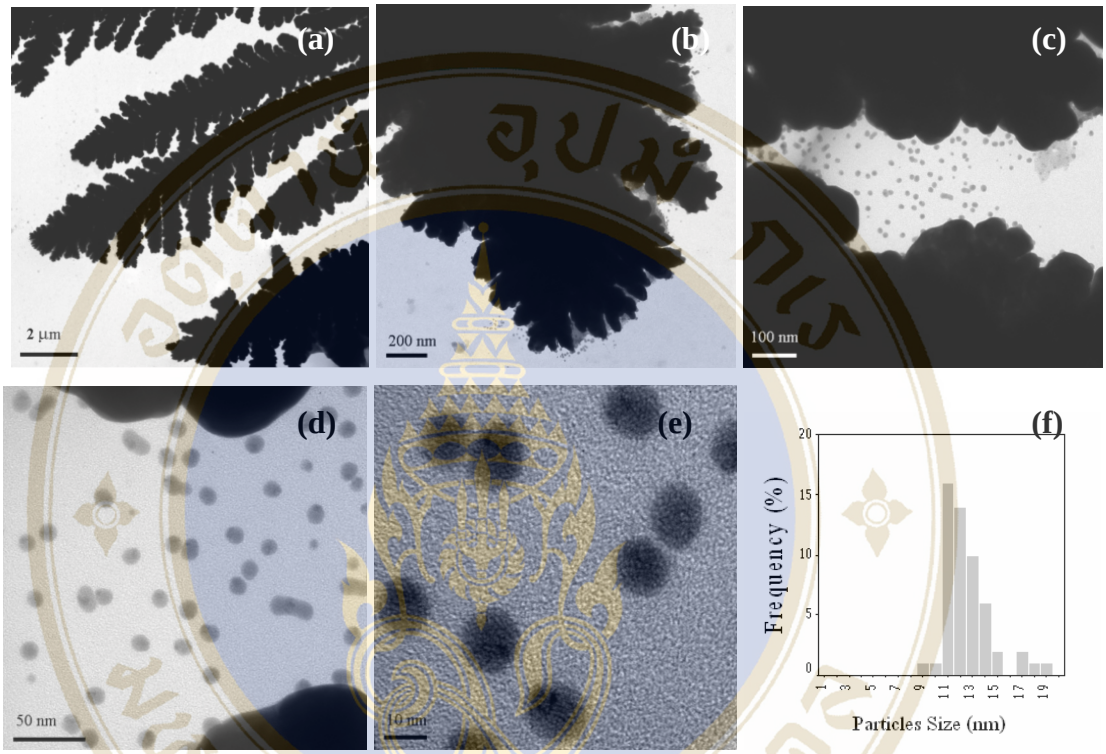

Figure 5.14 TEM images of as-synthesized dendritic silver nanostructures and single silver nanoparticles corresponding to a histogram of the size distribution from TEM micrographs (average particle size = 12.60 nm, δ = 1.84 nm). The bars are 2 µm, 200, 100, 50, and 20 nm, respectively. The total number of single particles counted for a histogram is 54.

The model for the explanation of the growth of treelike structures of silver nanoparticles has been known as DLA (diffusion-limited aggregate model) introduced by He and coworkers [176] who provided much useful insight into fractal growth in which other particles diffuse for the formation of treelike by microwave method. The concentration of $AgNO_3$ plays a significant role in the formation of the silver nanodendrites. The silver nanodendrites can be investigated when increasing the concentration of $AgNO_3$ in the experiments. It showed clearly that higher

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concentration of $AgNO_3$ was obviously favorable to aggregate nanodendrites. The silver nanoparticles aggregated and formed as silver dendritic nanoparticles, in which other particles also diffused for the growth of treelike structures.

5.3.2 X-ray powder diffraction (XRD)

The characterization of the crystalline nature of nanostructures was carried out by using X-ray powder diffraction (XRD) analysis, which is essential for identification of the as-prepared product. The crystal structure and phase composition of the as-prepared samples was achieved by investigating the XRD patterns. The diffraction patterns showed strong diffraction peaks assigned 2θ around 38.2°, 44.3° and 64.5°, which can be indexed of silver in a cubic structure (the JCPDS file no. 04-0783). The diffraction pattern does not show the presence of contaminant, indicating that silver oxide was not formed (see Fig. 5.15).

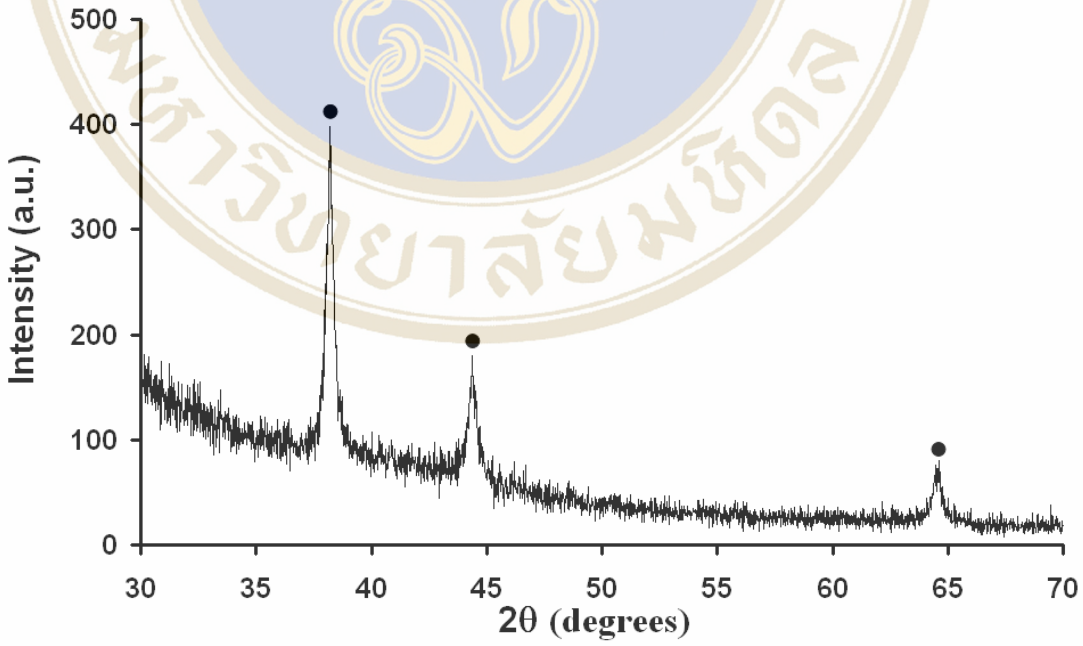

Figure 5.15 The XRD patterns of the as-prepared silver product. The XRD peaks for gold nanoparticles were drawn from the JCPDS file no. 04-0783.

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To further investigate the origin of colors of nanoparticles, two separated experiments were carried out by first incorporating Ag^+ into the inside of vermicelli by microwave radiation, then reducing Ag^+ to Ag nanoparticles by β-D-glucose under an inert atmosphere with a conventional heating. The two experiments were different only that vermicelli of one experiment was washed with a solution of NaCl before heating with β-D-glucose. Two colors of silver nanoparticles were successfully synthesized (see Fig. 5.16).

Figure 5.16 Pictures of (a) yellow and (b) purple vermicelli fabricated with silver nanoparticles

The phase of silver nanoparticle formation was further examined by the carbonization of nanoparticles embedded in vermicelli at 200°C and 300°C and then characterized by XRD. After the carbonization of yellow and purple silver nanoparticles stabilized by vermicelli templates in a furnace at 200°C and 300°C for 24 h, the black precipitate powder was also obtained in a crucible cup. The XRD was used to examine the phase of the obtaining products. The diffraction patterns of two colors of yellow and purple vermicelli demonstrated different patterns (see Fig. 5.17). From all diffraction patterns, the presence of metal oxides (e.g. silver oxide) was also not detected.

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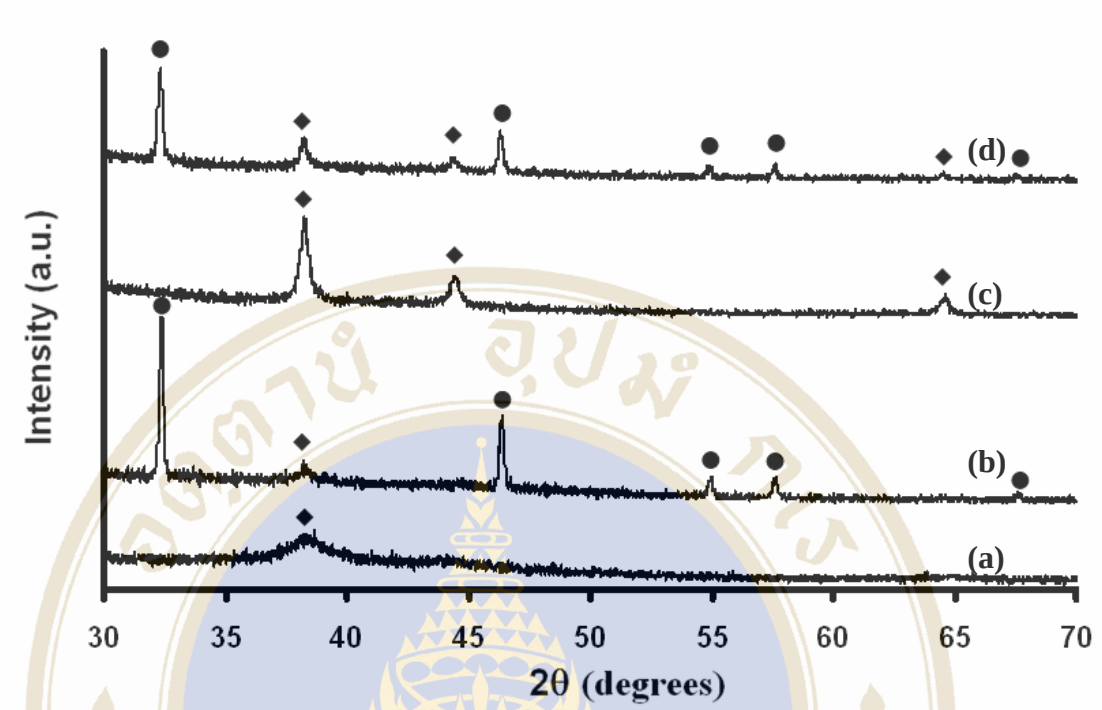

Figure 5.17 XRD diffraction pattern of yellow vermicelli (a and c) and purple vermicelli (b and d) at 200°C and 300°C, respectively. The XRD peaks for (◆) silver nanoparticles were drawn from the JCPDS file no. 04-0783, while XRD peaks for (●) silver chloride nanoparticles were drawn from the JCPDS file no 06-0480.

For the yellow mung bean starch vermicelli, the diffraction patterns (see Fig. 5.17a and 5.17c) showed strong diffraction peaks assigned 2θ around 38.2°, 44.4° and 64.5°, which can be indexed to the face-centered cubic silver (the JCPDS file no. 04-0783). However, the diffraction pattern of yellow silver nanoparticles showed broad peaks on the diffractogram. Some diffraction peaks were not observed from the XRD observation. It was noted that the absence of any different peaks was hidden most likely due to black carbon precipitate powder. This could possibly form the silver/C composites due to the inclusion of silver nanoparticles in the vermicelli templates [177]. The results from diffractogram indicated clearly that the origin of yellow vermicelli was demonstrated from the presence of silver nanoparticles.

Several 2θ positions of the diffraction pattern were observed for the sample obtaining from the carbonized purple vermicelli powder (see Fig. 5.17b and

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30d). In addition to the XRD pattern of Ag nanoparticles (peaks at 38.2°, 44.4°,and 64.6°), 5 other peaks are 32.3°, 46.4°, 54.9°, 57.6°, and 67.7° corresponding to the face-centered cubic AgCl (the JCPDS file no. 06-0480). The XRD indicated clearly the presence of silver chloride in the powder after the carbonization of purple vermicelli at 200 and 300 °C. The AgCl nanoparticles have been reported as nanocomposites with other materials. Recently, Li and Zhu [178] investigated the formation of Ag nanoparticles toward HCl. The white precipitate product was formed immediately when the HCl solution was added into the Ag colloidal solution**.** Li and Zhu also indicated that chloride ions could react with colloidal silver nanoparticles; however, the purple silver nanoparticles were not reported. Interestingly, the purple silver nanoparticles were produced in our experiment. The vermicelli before and after washing with a diluted NaCl solution was still transparent. The white precipitate of silver chloride was not observed. After the reaction of silver ions with β-D-glucose using as a reducing agent, the color of transparent vermicelli turned to purple. It might be possible that during the nanoparticles formation process some silver nanoparticles may react with chloride ions to form silver chloride.

5.3.3 UV-Vis spectrophotometry

Another measurement of evidence supporting the formation of the colloidal silver nanoparticles was monitored by using the UV-Vis performance, which can be used to provide the surface plasmon resonance band. The UV-vis spectra of purple and yellow silver nanoparticle solutions are shown in figure 5.18. During the conventional heating for 6 h, a purple color washed with diluted NaCl solution and yellow color without washing with NaCl solution were observed at the mung bean starch vermicelli and more intense color was dispersed into the solution as time passed, respectively. The purple silver nanoparticle solution was observed the strong, medium and weak UV-Vis absorption bands at 300, 400 and 600 nm, respectively (see Fig. 5.18a) which was similar to recently reported silver nanoparticles in our previous group research. The yellow silver nanoparticle solution was monitored only

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single UV-vis absorption band with λ_{max} at 420 nm (see Fig. 5.18b) which was similar to recently reported silver nanoparticles in the literature [179].

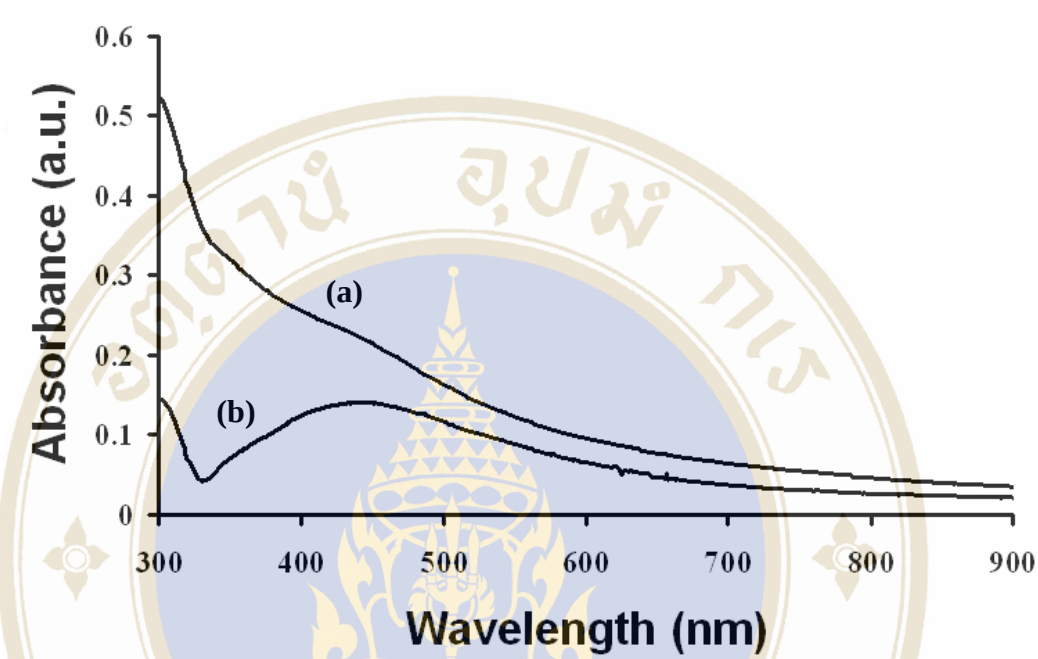

Figure 5.18 UV-Vis spectra of (a) purple and (b) yellow colloidal silver nanoparticle solutions, respectively

5.4 Chemical kinetics for chemistry education

The students were required to conduct an experiment in which they tried to examine aspects of reaction kinetics for the reaction between egg shells (mostly calcium carbonate) and acids (hydrochloric acid and vinegar). The experiment was conducted by 413 first-year undergraduate science students (age range 18-19 years) in the Faculty of Science, Mahidol University in Thailand.

The research findings for this inquiry are summaries under the following themes which emerged from the data: student laboratory performance; the role of the teacher; probing student understanding; and, student conceptual understanding of chemical kinetics. The themes were used to provide the discussion of research findings, and each of these themes is described below.

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5.4.1 Student laboratory performance

Scientists generally follow certain steps in their attempt to solve problems such as that posed to the participants in this inquiry. These steps are often referred to the scientific method (see Carin, Bass and Contant, 2005; Hassard, 2004). The researchers propose that students can learn to behave more like as scientists if they are given the opportunity to think steps by themselves. That is, here we had an expectation that they should approach the experiment done in this practical in a logical and systematic manner. So it was expected that the groups have to form hypotheses that can be tested by collecting data, conduct scientific experiments that control all but one variable, predict the outcome of the results, collect and record data accurately, and finally explain the obtained data using graphs and tables.

Here, we did see that some students provided good steps for their experimental design which could test their hypotheses. The experimental procedure was clear and simple and the students identified three groups of variables (i.e., independent variable, dependent variable and controlled variable) for investigation in the experiment (see Table 5.1).

Table 5.1 Student results providing variables and their description

Items	Description
Problem -	Influence of surface of egg shells to the rate of a reaction
Hypothesis -	The different surface of egg shells gives a different rate of a reaction
Independent variable -	Influence of surface of egg shells
Dependent variable -	The rate of a reaction
Controlled variable -	Size of Erlenmeyer flask, type of acid used, concentration of acid, reaction temperature, source of egg shells, laboratory environment

In this study, the experiment was more open than they usually encountered, and the students were allowed to design their own experimental procedure after they decided the experimental procedure to solve the problem as

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shown in figure D.1 and D.2. The students reported their experimental procedure in their laboratory documentation.

> Weigh egg shells 0.1 g and then pour them into the flask… Pour HCl (aq) 4.0 mL into the vial and then place it into the Erlenmeyer flask (be careful not do not mix HCl with egg shells)…fill the water into the 25.0 mL burette…Connect the burette and the flask with the U-tube and the rubber stopper…Shake gently the flask, mixing HCl and egg shells together …Observe the volume of CO_2 in the burette and record the results at time…Change the type of acid used from HCl to vinegar and then repeat the experimental procedure.

Because, at both secondary and tertiary levels, the chemical kinetics is difficult to comprehend for students, it is crucial for students to communicate and negotiate the results with others after completing the experiments. Students made a discussion and argumentation of the results with their peers. In the whole class setting, discussion and argumentation can help students to gain a better understanding of the concept of chemical kinetics.

At the advanced level of science teaching and learning, good experiments involve students conducting an inquiry, in a sense that simulates real scientific activity. Because this experiment was the eighth in the general chemistry laboratory course the researchers expected that students would have learned basic skills of how to conduct an experiment. Hence, in this study, students were required to design the experiment themselves, and do things in a way more like what scientists do. The role of teachers and teaching assistants is as facilitator to help their students in the laboratory.

For any laboratory, the starting point for many scientific methods of analysis is sample preparation. The sample preparation in this experiment involved a sample of egg shells and is seen here an essential stage, since it is a key step to

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successful completion of the experiment. In this experiment, there is no single method of sample preparation for the solid reactant (egg shells), meaning students have to decide how to prepare the solid sample themselves (see Fig. 5.19). Here, the egg shells were collected and their surfaces are mechanically cleaned. From analysis of students' laboratory documents it seems they felt the particle size of egg shells should be consistent, and thus they ground the shells to obtain a fine powder with uniform (homogeneous) particle size.

Figure 5.19 Students' experimental design for the sample preparation of egg shells

It seems likely many students had learned how to conduct some aspects of this experiment when they engaged in previous practical classes. For example, some students understood readily the preparation of solid sample (and acids) before doing the experiment and they thus provided detailed instructions of how this should be done, as seen below in the excerpt below.

> *Sample preparation of egg shells* – In this part, the particle sizes of the solid reactant should be made at least into three sizes: big, medium and small (like sands) size. The white layer which covers on the egg shells should be removed before grinding.

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Preparation of the concentration of acid – The concentration of acid used in this experiment is 0.5, 1.0, 1.5 and 2.0 M HCl. We then diluted these concentrations from 2.0 M HCl.

Here in figure 32 we see some clear planning in the three samples of varying particle size, and the diagram which illustrates the experimental set up to measure the amount of gases produced over time (collection by downward displacement).

Interestingly, for the dilution of the solution, students showed a good understanding of the preparation of chemical solutions, and they were able to solve problems, albeit algorithmically. The formula, $C_1V_1 = C_2V_2$, was generally used to calculate the acquired concentration of hydrochloric acid or vinegar. However, some students did not give any units of concentration when doing their calculations.

5.4.2 Student conceptual understanding of chemical kinetics

One common issue in the teaching and learning of chemical kinetics is that students need to be able to explain the basic concept of rates of reaction; that is, how variables (e.g., surface of the solid reactant, concentration, temperature and type of acid used) affect the rate of a chemical reaction. It is important not only to find out the students' conceptual understanding of chemical kinetics, but also to investigate their alternative conceptions of these ideas. The findings of this study come from analysis of participants' laboratory documentation with respect to their understanding of chemical kinetics.

In this study, students are introduced to an understanding of the rates of reaction for different examples, and investigating the influence of variables (e.g., surface of solid reactant, concentration of acid, temperature of a reaction and type of acid) on the rate of a reaction. The concept here then is first-order chemical kinetics, and the experiment involves students measuring the amount of gaseous carbon dioxide formed from the reaction of an acid with carbonate (hydrochloric acid

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or vinegar and calcium carbonate in egg shells) at different time intervals. The first stage of reporting the experiment is to present data using graphs or tables. In this way, when a variable is changed in an experiment, the data presented includes the changes in the independent and dependent variables. This reflects scientific investigation, acting in the way scientists do.

In general, here the students carried out the investigation of the influence of surface of egg shells for two different particle sizes (there is no standard size for the preparation of the solid sample, see above). Students in each group varied the particle size of egg shells themselves, and to explain the influence of surface area of the solid reactant and the rate of reaction, the students commonly reasoned that the change of the rate of a reaction is due to changes in physical dimensions of the solid reactant (see the Fig.5.20). A typical student responses and here was that they concluded that:

> The rate of a reaction is dependent on the surface of the solid reactant (egg shells). If the size of egg shells is big, the rate of a reaction is slow. On the other hand, if the size of egg shells is small, the rate of a reaction is fast.

Interestingly, some students drew upon analogy of a cube to explain the increase of the surface area of solid reactants. This was perhaps because it was simple for students to understand in their mind as is important feature of the student-generated analogy [180]:

> If we put four cubes together, the surface area is only 16 cm^2. However, if we separate all four cubes away from each other, the surface area is 24 cm^2.

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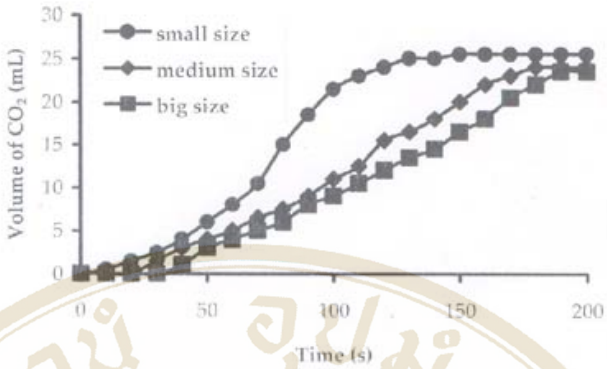

Figure 5.20 Students' laboratory data – Surface of solid reactant (egg shells) and the volume of gaseous carbon dioxide formed

In a given experiment, the students generally carried out the reaction at four different concentrations of acid. In general, the students prepared the concentration of both hydrochloric acid and vinegar at 0.5, 1.0, 1.5 and 2.0 M. To explain the increase of the rate of a reaction at higher concentrations, most said the effect of concentration of acid used was due to the number of particles in the solution. For example, some students carried out the experiment between 400 mg of egg shells, and 5 mL acid at 303 K (see Fig. 5.21) and explained their observations thus:

> If the acid used is higher concentrations, the rate of a reaction increases, because higher concentrations have many molecules that can react more than low concentrations.

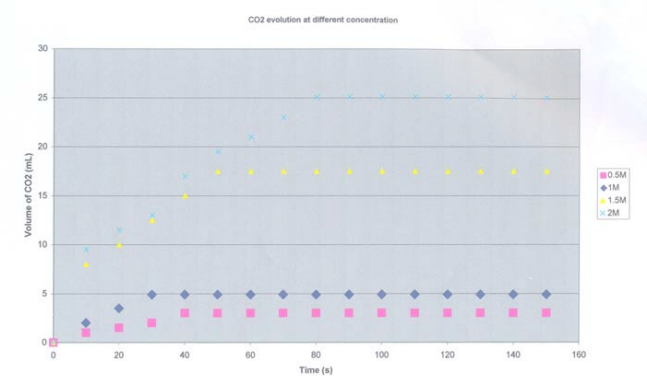

Figure 5.21 Students' laboratory documentation – Concentration of hydrochloric acid (HCl) and the volume of gaseous carbon dioxide formed

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To investigate the influence of temperature, the students carried out the reaction at least two different temperatures. They started doing a reaction at low temperature and then moved on to higher temperatures, although some .started at higher temperatures and moved to lower temperatures. The students that changed from room temperature to higher temperature reasoned pragmatically that it was easier to carry change the temperature using water bath. To explain the influence of different temperatures on the rate of a reaction, students observed that "the rate of a reaction increases at higher temperatures". This they reasoned was due to increased kinetic energy of reacting particles, in accord with the scientific view. For instance, some students carried out an experiment with 100 mg large surface area egg shells and 1.0 M HCl at three different temperatures (see the Fig. 5.22):

> From the experiment, when increasing the temperature for a reaction, the kinetics energy of reactant molecules increases. So, molecules move faster and more collision. The rate of a reaction increases and then the reaction occurs quickly.

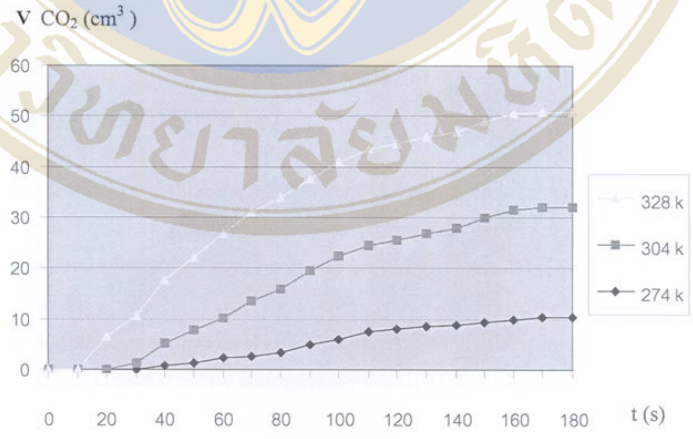

Figure 5.22 Students' laboratory data – Temperature of a reaction and the volume of gaseous carbon dioxide formed (note: the reaction has not at this early stage of the reaction, i.e., 180s, gone to completion – at completion the reaction produces the same amount of gas)

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It is somewhat surprising that some students provided considerable detail in their descriptions, as seen below:

> The rate of a reaction is dependent on the temperature. So, when you also give the temperature, if the reaction is endothermic, the rate of a reaction increases. In contrast, if the reaction is exothermic, the rate of a reaction decreases.

Finally, students investigated the influence of different types of acid - hydrochloric and vinegar on rates of reaction. Here, the students retained the same conditions, other than the acid. To explain changes in the rate of a reaction from because of the different type of acid used, most students explained in the term of the 'strength' of the acids; again in accordance with the scientific view. For instance, some students carried out the experiment between 100 mg large surface of egg shells and 0.5 M HCl and then changes the HCl replacing it with vinegar (acetic acid, see the Fig. 5.23).

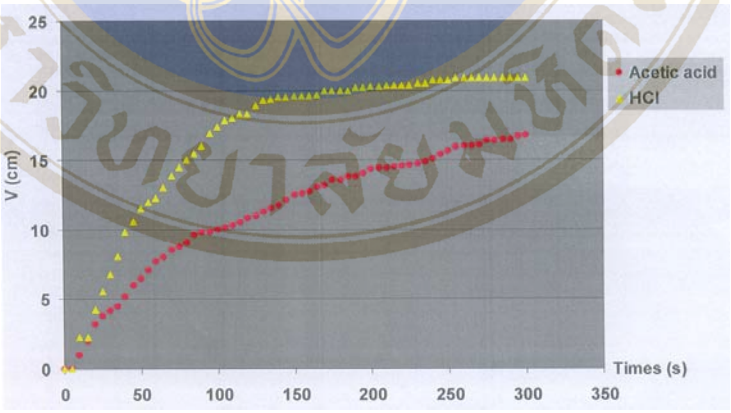

Figure 5.23 Students' laboratory documentation – Type of acid used and the volume of gaseous carbon dioxide formed

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It is surprising that some students again provided quite detailed responses, and they concluded that:

> The rate of a reaction increases when using the strong acid [i.e., HCl]. On the other hand, the rate of a reaction decreases when using the weak acid. HCl is more active than vinegar. Hydrochloric, a strong acid, dissociates more completely when it dissolves in water and it is a good H^+ donor. On the other hand, vinegar, a weak acid, dissociates only slightly when it dissolves in water and it is also a poor H^+ donor.

After completing the experiment, in whole class discussion, the experimental data from investigations of kinetics were analyzed by the class to compare the rates of reaction by plotting the relationships between the amounts of carbon dioxide over time using standard computer software (i.e., Solver Parameters in Microsoft Excel). Students typically explained their findings thus: “At the beginning, the slope of graphs is very sharp, because the rate of a reaction is large. As the reaction progresses, the reaction becomes slower. The rate of a reaction decreases, eventually to zero when the reaction is completed”. Students were able to perform the calculations of the rate of a reaction correctly. However, a few students still had problems with the calculations, because it was the first time that they had done such calculations by themselves.

5.4. 3 Findings from the survey

In this study, all first-year undergraduate science students were here familiar with the teaching and learning of chemical kinetics in general chemistry laboratory course. Before allowing students to do the experiment, students (n =129) in groups were randomly selected to perform a pre-test survey about variables which affect the rate of a reaction. After completing the experiment, students (n =103) were then selected to complete a post-test survey. These tests, available from the authors, were similar in nature and contained questions deemed by a panel of experts to be

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suitable for this educational level (see Appendix C.1.4 and C.1.5). Students were provided with a balanced equation for the reaction of acid with carbonate and asked questions such as how particle size, temperature or other variables affect the rate of reaction. They also were asked to provide explanations for their answers. The comparison of pre-test and post-test survey is provided in figure 5.24. The findings from the pre-test survey showed that 99 (76.7%), 55 (42.6%), 60 (46.5%) and 97 (75.2%) of the participating students provided a correct answer corresponds to surface of solid reactant, concentration of acid, temperature of a reaction and type of acid, respectively. Of the four variables, it was noted that students held poor conceptual understanding of the effect of concentration and temperature on reaction rate (see Fig. 37). In order to investigate the student understanding, the participating students again in groups completed a post-test survey after doing the experiment. From the post-test survey, scores of 95 (92.2%), 76 (73.8%), 91 (88.3%) and 89 (86.4%) correct responses were obtained for the surface of solid reactant, concentration of acid, temperature of a reaction and type of acid, respectively.

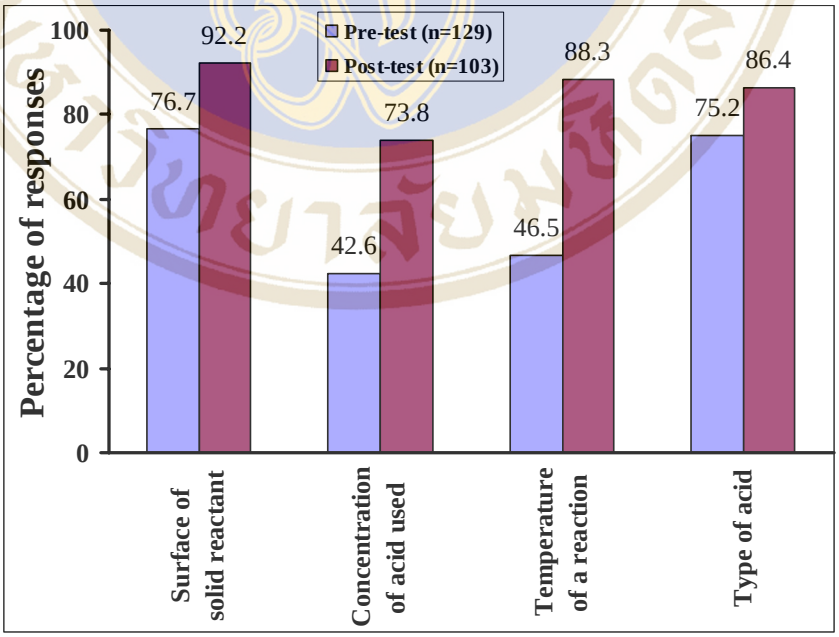

Figure 5.24 Comparison of pre-test and post-test survey from students' responses of chemical kinetics laboratory before and after doing an inquiry-base practical chemistry class.

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5.4.4 Student perceptions of the role of the teacher or TAs

The Thai National Science Education Standards [38] stated that active engagement of students in learning science is an importance aspect of effective science teaching. In this study, the role of the teacher was manifestly different to the norm, and it was of interest to see how this was perceived by the students. Here it was intended that the teachers would act as a facilitator of learning – consistent with constructivism (see above). Hence, they had to pose problems to their students during in the experiment, and to encourage the significant discussion between students about the problems (see Table 5.2). In order to understand the role of teachers, students also provided their reflections on the role of the teacher in practical classes.

Table 5.2 Students' responses to items relating to the reflection on the role of teacher or TAs (n = 389)

Items	SD	D	N	A	SA
3. In this experiment, the teacher is friendly to me.	0	8	56	184	141
4. In this experiment, the teacher is interested in answering my questions.	1	5	59	183	141
5. In this experiment, the teacher helped me when I had trouble in the class.	2	11	64	205	107
10. In this experiment, the teacher encouraged me to discuss my ideas with other students.	2	19	126	197	45

Key: SD = strongly disagree; D = disagree; N = Neutral; A = agree; SA = strongly agree

The students' responses to Item 3, 4 and 5, are interesting in that the majority of students showed that the teachers in classes were friendly and attempted to help students when they found difficulties in class. This suggests the student

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enjoyed a different role to the teacher than they were used to. This is somewhat at odds with the normal situation in Thai classrooms (Ministry of Education [MOE], [50]). However, some caution is needed the interpretation of this finding. As well as the fact that here the activities were likely perceived as more interesting and more exciting for teachers and students alike, the positive response note in Table 5.2 also may be a result of the low ratio of the teachers, including teaching assistants (TAs), to students of around 1: 20 in classes – much less than is normal in Thai schools as shown in the fingure D.3. Such a ratio would obviously allow for more student teacher interaction and probably less stress and thus more enjoyment on the part of the teachers.

5.4.5 Student perceptions of the POE technique

White and Gunstone [162] proposed that the *Prediction-Observation-Explanation* (POE) technique is a good teaching strategy in science education. In support of this proposition, the POE strategy has been reported successful in a number of studies (see, e.g., Kearney, Treagust, Yeo and Zadnik [181], and Kearney [182]). Here, in a given experiment about chemical kinetics of acid-base reactions (i.e., hydrochloric acid or vinegar and calcium carbonate in egg shells), students were first asked to discuss their ideas and make predictions of the actual outcome, and to give their reasons as shown in the figure D.4. Then, they performed the observations and explanations of the evolution of carbon dioxide gas (CO_2) produced from the experiment, and finally sought reconciliation of any discrepancies between their predictions and observations after completing the task (see Table 5.3). Students' responses showed that the students enjoyed using the POE technique during the experiment sessions.

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Table 5.3 Students' responses to items relating to the reflection on four POE tasks (n = 389)

Items	SD	D	N	A	SA
13. In this experiment, I felt that the POE was a useful way to predict.	2	13	112	211	51
14. In this experiment, I felt that the POE is a useful way to explain.	2	9	106	211	61
17. In this experiment, I learned how to use data to support my conclusion for chemical kinetics.	1	11	105	228	44
20. In this experiment, I felt that I learned to do things in a way more like scientists do	0	41	84	193	93

Key: SD = strongly disagree; D = disagree; N = Neutral; A = agree; SA = strongly agree

Student responses to Item 13 and 14 suggest that students felt that the POE was a useful strategy to predict the actual outcome. The responses to the other items suggest that the participants felt the POE approach helped them make to connections between what they predicted and observed in the experiment of chemical kinetics, and that it helped them understand the basic concept of chemical kinetics (Item 17), and how to do things in ways scientists do (Item 20).

5.4.6 Student perception of group learning

As noted above, the science education literature suggests group learning can be an important component of modern teaching approaches. A fundamental outcome of effective group learning is for students to be able to communicate, explain, and justify their understanding, argue from data, and defend

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their conclusions within their groups [122]. Here, the rationale of implementing group learning in the laboratory was so that students could come to understand more about the nature of science, and how scientists actually worked, that is, the fact that scientists seldom work alone. Students' responses about working in groups are provided in table 5.4 below.

Table 5.4 Students' responses to items relating to the reflection on their partner in groups (n = 389)

Items	SD	D	N	A	SA
21. I worked cooperatively in this experiment.	2	5	34	191	157
23. I shared ideas with other students while doing this experiment.	1	5	72	196	115
25. I felt that working in a group stimulated my thinking.	0	11	53	207	118
28. I learned ideas from other students in my group.	0	8	69	224	88
30. I felt that the group discussion helped me to understand the concept of chemical kinetics.	2	13	83	219	71

Key: SD = strongly disagree; D = disagree; N = Neutral; A = agree; SA = strongly agree

The responses to suggest that the majority of students enjoyed learning in groups (item 21). The working in group situations seems to provide ways of helping students to understand the social and procedural rules in the experiment. Furthermore, it seemed that students appreciated this new way of working in their practical classes, and that the majority of students attempted to share their ideas among others in group members (items 23, 25 & 28). Group activities can be used to

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engage students communicating and negotiating in classes. Finally, most students believed that the group discussion among group members helped them a lot to understand the basic concept of chemical kinetics (item 30).

5.4.7 Summary of research findings

The research findings of this study suggest that engaging students in inquiry-based practical chemistry classes produces sound learning outcomes in terms of conceptual understanding of chemical kinetics, the ability to design and conduct experiments in chemical kinetics, and the nature of scientific inquiry. The students also were able to explain the changes of the rate of a chemical reaction based on kinetic theory and drew upon energy and particle theory to explain changes in rates of reaction. They seemed to be quiet clear on how to conduct experiments, and the notion of investigating variables by changing each separately, while maintaining the others constant. Hence, overall analysis of students' laboratory documentation, suggests that most students had a better conceptual understanding of chemical kinetics.

As noted above, inquiry-based practical classes in Thailand represent a very different teaching approach. However, in addition to sound conceptual understanding of chemical kinetics evidenced in their laboratory reports, the students' responses to the survey instrument suggests that they enjoyed this 'new' way of learning, they were comfortable about the very different role the teacher assumed, and they enjoyed working in groups. The new experiment here was conducted in order to allow science students to develop student-centred learning processes based on inquiry-based learning. Students had done things in the way like what scientists do. As noted the in findings, the designed the experimental for procedure themselves, and this seemed to help them gain a better understanding of the process of scientific inquiry. This can be seen from analysis of their procedures which consisted of the following steps for experimental design: state the problem, make hypotheses, make predictions, make observations, provide an explanation, and the construction of knowledge of their own understanding from group discussion after completing the experiment. This is in marked contrast to the 'normal' situation for

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teaching of chemical kinetics in Thailand, which more often simply involves following laboratory instruction or teacher demonstrations.

5.4.9 Discussion

Consistent with a case study approach, the data interpretation of this research is conducted within the interpretive paradigm described by Merriam [161] and so the transferability of our findings is best judged by the reader. Here we attempt to interpret our findings in relation to other reports of research in the literature. The authors here attempted to provide insights into the learning of chemical kinetics for first year science students in Thailand when using inquiry-based learning incorporating a series of POE activities. A number of themes have emerged from this research. The research findings reported here have some similarities with work by Hofstein and Lunetta [43], Wellington [28], and Woolnough [29], all of whom investigated laboratory science education at the tertiary level). The new experiment here placed high value on 'hands-on' activities, and students were deeply engaged in the experiment in a form of student-centred learning as suggested in these reports (see also Tamir [107]).

The new experiment developed in this work was similar in nature to that of Choi and Wong [148], who focused on the acid-base reactions which demonstrated first-order kinetics, but involving the application of a datalogger (i.e., a computer interfaced to one or more sensors, see above). However, datalogger-type experiments are expensive to set up, and are not generally suitable in the Thailand educational context, where teachers typically do not have access to such electronic instruments. One intention of this new experiment was to make the laboratory class related to the daily life, since the chemicals used in the experiments, sometimes, were not those purchased from the chemical company. This also introduced an element of student choice, with respect to research design and the conduct of the experiment - in a way that might not have been the case if they were given, says a laboratory sample of calcium carbonate, of uniform particle size. Another factor was that the solid reactant, calcium carbonate, was in a form (i.e., egg

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shells) that could be easily changed by grinding to different particle sizes. Moreover, students also were provided with opportunities to investigate other factors that might influence to the rate of a reaction, such as concentration of the acid used, temperature and the type of acid used.

As noted in the research findings, most of these Thai students seemed to have a better conceptual understanding of chemical kinetics after having done the experiments. A key feature was the move from passive to active learning, since the new experimental design was developed to enhance student-centred learning processes based on inquiry-based learning. Interestingly, one of the differences from other reported work is that, in these practical chemistry classes, the learning also comprised the development of the methodology to investigate the influence of some variables on rates of reaction, and specifically to use the POE strategy in small groups. The authors found that the POE tasks in the form of paper-based drawings on the sheets seemed to help enhance student learning of chemical kinetics.

Finally, the research findings reported here suggest the students developed an understanding of inquiry that went beyond pure simply ‘fair testing’. Hume and Coll [183] note that many so-called inquiry-based experiments, are not much different in principle from conventional ‘cookbook’ style experiments, in that the former also are often rather formular in nature. However, the inquiry-based experiment as used here did provide these Thai students with some insights into how scientists conduct research. The students had to identify the problem, design an appropriate method, decide how to collect and record data, and perhaps most importantly, by drawing on the POE activities, predict the results and offer explanations. The use of interactive groups and whole-class discussion also sought to reinforce the socially-negotiated nature of scientific knowledge; more consistent with more holistic views of the nature of science and genuine inquiry-based learning.

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CHAPTER VI
CONCLUSIONS

This chapter comprises a summary of the research findings. It begins with starch vermicelli template for synthesis of magnetic iron oxide nanoclusters followed by starch vermicelli template-assisted synthesis of size- and shape-controlled nanoparticles, and enhancing Thai students' learning of chemical kinetics, respectively. The conclusions on the conceptual ideas of each work are summarized in the following topics.

6.1 Starch vermicelli template for synthesis of magnetic iron oxide nanoclusters

In summary, we have demonstrated a novel method for synthesizing magnetic nanoparticles of iron oxide by using transparent vermicelli template as a new stabilizing agent. The magnetite nanoparticles can be synthesized using the precipitation of magnetic iron oxide nanoparticles by a wet chemical method. In this case, the morphology of the as-prepared magnetic iron oxide deposited on the surface of vermicelli was spherical nanoclusters in form with a diameter about 200-300 nm. Meanwhile, the hysteresis loops exhibit that both before and after carbonization of the final products were superparamagnetic behavior of magnetic materials. The saturation magnetization of the iron oxide at room temperature (300 K) was 34.8 emu/g and 5.1 emu/g, corresponding to the before and after carbonization, respectively. Consequently, the difference of magnetic behavior of the final products can be appropriate for a variety of biomedical applications.

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6.2 Starch vermicelli template-assisted synthesis of size- and shape-controlled nanoparticles

In summary, we have successfully developed a novel rout to synthesize size-controlled gold nanoparticles, using mung bean starch vermicelli acting as stabilizing agent. To avoid the use of toxic reducing agent, microwave irradiation on hydrochloric acid-catalyzed hydrolysis of starch was useful to this study, because starch consists of several glucose unites as a reducing agent. The crystallinity of the as-prepared gold nanoparticles was investigated using X-ray power diffraction (XRD). The particle size of gold nanoparticles was measured by using transmission electron microscope (TEM) with narrow diameters around 12.7±2.8 nm, and UV-Vis absorption spectrophotometer was used to investigate the colloidal gold solution, according to the surface plasmon absorption of gold nanoparticles. Starch capped gold nanoparticles were found to be stable in the template over the period time of three months of storage at room temperature, while there was no sign of aggregation of gold nanoparticles in solution. The novel approach to synthesize size-controlled gold nanoparticles was very simple, safe and easily accessible to any research. It could be said that mung bean starch vermicelli serving as the raw material makes it possible to use for large-scale synthesis of size-controlled gold nanoparticles. This offers a great potential for chemical and biological applications.

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6.3 Enhancing Thai students' learning of chemical kinetics

Chemical kinetics is an important concept in introductory chemistry courses. From other reported work, instruction in chemical kinetics is typically teacher-dominated at both the secondary school and tertiary levels, and this also is the case in Thailand-the context for this inquiry. The research reported here seeks to shift undergraduate science students from passive learning to active learning, and involved some 413 first-year in Mahidol University in Thailand, of which 389 consisting of 129 male and 260 female completed a questionnaire about the experiment. A key feature needed to move students from passive to active learning was the use of the POE strategy along with small group and whole-class negotiation. Students who participated were asked to design the experimental procedure themselves, in order to investigate the reaction of acids and bases. A number of variables here were investigated by changing each separately, while maintaining all others constant, including solid surface, concentration, temperature and type of acid. Overall, the research findings suggest that most students had better understanding of chemical kinetics, were able to explain the changes of the rate of a chemical reaction, and also developed better conceptual understanding of chemical kinetics both qualitatively and quantitatively.

Hence, the authors suggest here that science teachers may wish to consider teaching chemical kinetics using simple chemical reactions and materials related to everyday processes, and based on inquiry-learning. An additional advantage of the experiment used here is that it is not particularly hazardous, and so is convenient for science teachers to apply this experiment in classes.

One feature of teaching chemical kinetics is that the calculations, particularly at the university level, the concepts are more complex, and complex equations are often used to describe the kinetics of a reaction, such as differential equations. The authors observed that some of the first-year students in this work did not know how the equations are derived, or how to use the equations for calculating the rate of a reaction. Therefore, in this research, the authors recommend the use of computers,

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and for example here, the experimental data from the experiments was easily analyzed using Solver Parameters in the tool function of Microsoft Excel which enables students to plot the relationships between the production of carbon dioxide and time. As shown in the findings, the students were able to see how the rate of a reaction changed for different experiments, and how investigate the influence of variables (e.g., solid surface, concentration, temperature and type of acid) on the rate of a reaction.

Finally, the authors suggest that science teachers modify experiments to suit their own needs and circumstances using say other variables, such as catalysts or a size of the reaction flask, for students to investigate how the rate of a reaction differs from others. Students here did not investigate the influence of catalysts because of limited time for carrying out the experiments.

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APPENDIX

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APPENDIX A

A.1 Magnetic iron oxide nanoparticles

Figure A.1 Starch vermicelli (100%, Kaset brand Thai bean thread)

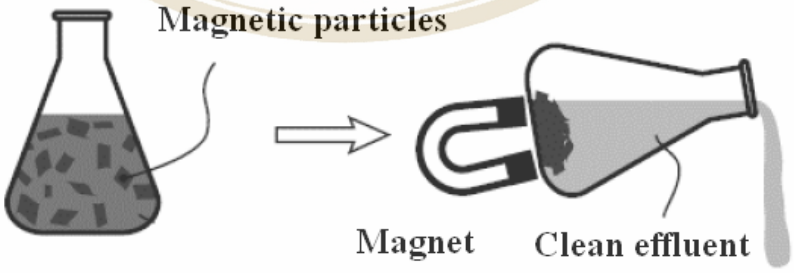

Figure A.2 Schematic representation of the magnetic composite separation

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APPENDIX B

B.1 Gold and silver nanoparticles

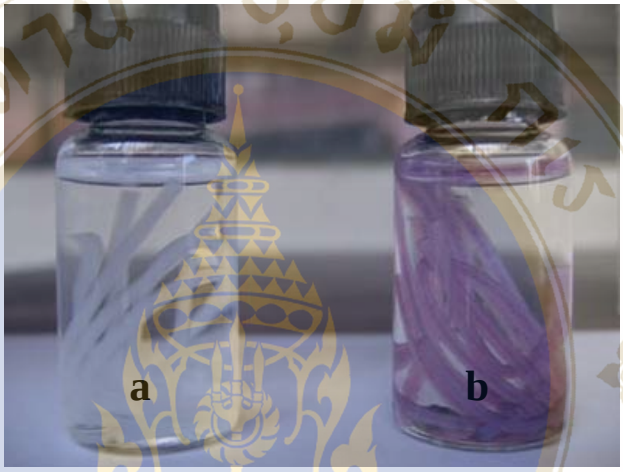

Figure B.1 A photo shows vials containing (a) only starch vermicelli template and (b) starch vermicelli template capped gold nanoparticles

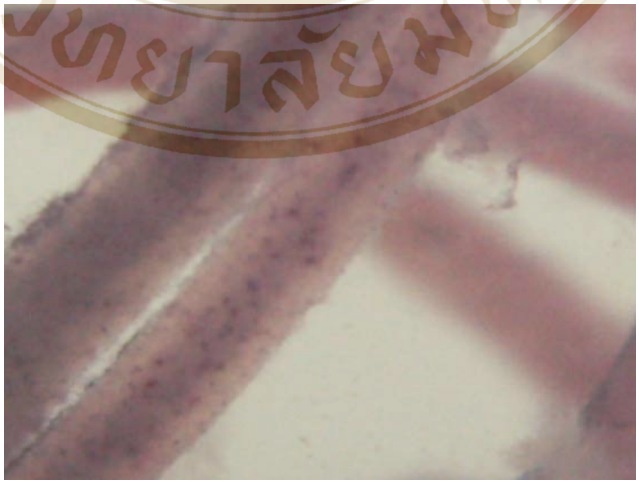

Figure B.2 A photo at high magnification shows starch vermicelli template capped gold nanoparticles

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Figure B.3 A photo shows the light scattering of (a) silver nanoparticles and (b) gold nanoparticles filled in the vial. This is a simple method showing the colloidal nanoparticle solution

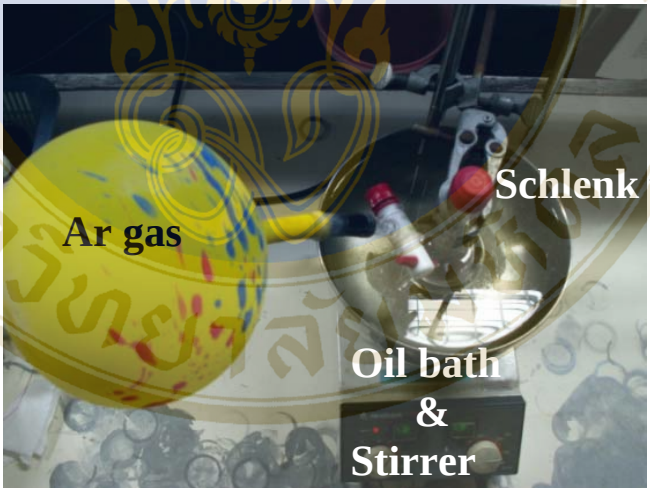

Figure B.4 Experimental setup for synthesizing two colors of silver nanoparticles

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APPENDIX C

C.1 Enhancing Thai students for learning chemical kinetics

C.1.1 Laboratory direction for students

For many first year science students enrolled the laboratory SCCH108 kinetics is difficult to comprehend. The overall purpose of this laboratory is to teach students an understanding of chemical kinetics, including: the rate of a reaction, rate law, and determination of the rate law. As noted above, we want to do this in an environment that is reasonably authentic, in which students investigate a scientific problem in a way similar to that used by scientists.

Chemical kinetics refers to the time dependence of chemical reactions. It may surprise you to find that pretty well all 'everyday processes' involve kinetics in some way. For example, the so-called 'acid rain' is a problem in many large cities worldwide, including Bangkok. Acid rain is caused by emission of sulfur dioxide and similar species from car exhausts, and in recent years this has become a serious environmental problem in Bangkok. Breathing acidic fumes causes health problems, but probably the most visible evidence for acid rain is in the damage it does to buildings. It can severely damage buildings in a city, and it is obviously of interest to scientists and citizens alike to know how quickly such destructive reactions might take: this is the sort of information we get from the study of kinetics.

This experiment focuses on the kinetics of acid-base reactions. The concept of chemical kinetics of this reaction is often taught in secondary or tertiary education levels. Whilst concrete which buildings are made of is chemically different to calcium carbonate, the overall idea is similar in that acids destroy carbonates. This experiment uses materials that are a bit easier for us to handle in the laboratory

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class. Acids such as hydrochloric acid (HCl) will react quickly with calcium carbonate to product a salt, water and release gaseous carbon dioxide. Other acids such as the acids present in vinegar also react with carbonates. The reaction is:

$$CaCO_3\,(s) + 2HCl\,(aq) \rightarrow CaCl_2\,(aq) + H_2O\,(l) + CO_2\,(g)$$

In the reaction above, how the acid and carbonate react may depend on a number of factors which we want you to investigate. Things you can consider are: the concentration of the acid, the particle size of the carbonate, the temperature of a reaction and any other factors you can think of. This chemical equation can be applied to determine the rate of a reaction by plotting the relationships between the production of carbon dioxide over time. The experiment is first-order in its kinetics with respect to calcium carbonate and acid. The experimental data from kinetics investigations can be analyzed using Microsoft Excel Solver.

Chemistry is the science that concerns with making new substances from other substances, and chemical kinetics is the branch of chemistry which addresses the question how fast reactions go. It could be said that chemical reactions are the core of chemistry. In many reactions, the rate of a reaction changes while the reaction progresses. Initially, the rate of a reaction is large, and as the reaction progresses the rate of a reaction decreases, eventually to zero when we say the reaction is complete. The rate law for any given chemical reaction is the mathematical equation which describes the progress of that reaction. Often we use a differential rate law, which relates how the rate of a reaction varies with the concentration of various species in the system. Each rate law contains a constant, *k*, called the rate constant. The units of the rate constant depend on the rate of a reaction and the concentration. It is important to realize that we cannot infer the rate of any reaction from the equation. In particular, the stoichiometric coefficients only tell us about the ratio with which reactants combine to produce products. The rate and rate law (i.e., how the rate depends on the concentrations of any of the species) for any reaction must be determined from experiments. To illustrate, consider the reaction above using HCl as the acid, the rate of this reaction, *r*, is expressed as (note that the

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rate does not depend on the stoichiometric coefficients, but the rate at which reactants disappears and products are made are related to stoiciometric coefficients):

$$r = -\frac{d[CaCO_3]}{dt} = -\frac{1}{2}\frac{d[HCl]}{dt} = \frac{d[CO_2]}{dt}$$

The quantity of hydrochloric acid is present in large excess; therefore the rate of a reaction will depend on only the concentration of calcium carbonate.

$$-\frac{d[CaCO_3]}{dt} = k\,[CaCO_3]$$

The integrated form of the rate law can then be written as:

$$[CaCO_3]_t = [CaCO_3]_o\, e^{-kt}$$

where $[CaCO_3]_o$ is the initial concentration of calcium carbonate, and $[CaCO_3]_t$ is the concentration of calcium carbonate at time *t*. We can measure the volume of carbon dioxide, V_{CO2}, generated at any point in time (see the experimental set up in Figure 1). The volume of carbon dioxide produced is thus related to the concentration of calcium carbonate reacted. The concentration of carbon dioxide generated at time *t*, $[CO_2]_t$, is equal to the initial concentration of calcium carbonate, $[CaCO_3]_o$, minus the concentration of calcium carbonate reacted, $[CaCO_3]_t$. Therefore, we can write:

$$[CO_2]_t = [CaCO_3]_o\,(1-e^{-kt})$$

Since the volume of carbon dioxide is related to the concentration of carbon dioxide, the final volume of carbon dioxide, V_f, is equal to the initial concentration of calcium carbonate, $[CaCO_3]_o$. The integrated form of the rate law for this reaction is thus written as:

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$$V_{CO2} = V_f (1-e^{-kt})$$

In this particular experiment we do not want you to determine the rate law (this has been done here above). What we want you to do is experiment with different species and reaction conditions to see how the rate of this reaction varies depending on reaction conditions. This is what we mean by inquiry-based learning, where you the student have some choices and decisions to make about the way the experiment is conducted.

In laboratory activities, students are assigned to design the experiment for studying how variables affect the rate of a reaction. The variables are:

1. Surface of solid reactant, calcium carbonate to egg shells;
2. Concentration of hydrochloric acid or vinegar;
3. Temperature of the reaction; and
4. Type of acid used in the reaction.

C.1.2 Prediction, Observation and Explanation (POE) activities

In this experiment we want to help you focus on your explanations of what you see. Scientists do much more than observe, they predict and explain their observations. If their predictions turn out to be different to what they expected, they re-examine their prediction and make new predictions for new experiments.

Here we want to mimic this process to again help you think like a scientist. We use a research tool called *Prediction-Observation-Explanation* (POE). In POE students are asked to first predict the outcome of an experiment, along with their explanation. They then conduct the experiment and see if their prediction was correct and that their explanation is reasonable (and most importantly, agrees with their prediction!). Here we want you to predict and explain a described phenomenon in this experiment of chemical kinetics using acid-base reactions.

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Tasks: Tick the appropriate box in the tasks below.

1. Predict how the surface of solid reactant, calcium carbonate, might affect the rate of a reaction, when we change the particle size from either

□ small particle sizes to larger particle sizes

or

□ large particle sizes to smaller particle sizes.

When reacting with the same concentration of acid at the same temperature:

□ the rate of a reaction increases

□ the rate of a reaction decreases

□ the rate of a reaction does not change

Explanation for Prediction:

Observation:

Reconciliation of Prediction and Observation:

2. Predict how the influence of the concentration of acid affects the rate of a reaction, when you change the concentration of acid from either;

□ high concentration to lower concentration

or

□ low concentration to higher concentration

When reacting with the same particle sizes at the room temperature;

□ the rate of a reaction increases

□ the rate of a reaction decreases

□ the rate of a reaction does not change

Explanation for Prediction:

Observation:

Reconciliation of Prediction and Observation:

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3. Predict how the temperature of a reaction might affect the rate of a reaction, when you change the temperature from either:

□ high temperature to lower temperature

or

□ low temperature to higher temperature

During a reaction at the same particle size and the same concentration of acid:

□ the rate of a reaction increases

□ the rate of a reaction decreases

□ the rate of a reaction does not change

Explanation for Prediction:

Observation:

Reconciliation of Prediction and Observation:

4. Predict how the type of acid used in the reaction might affect the rate of a reaction, when you change the type of acid from either:

□ hydrochloric acid to vinegar

or

□ vinegar to hydrochloric acid

When you maintain the temperature of a reaction and the same particle size at the same concentration of acid:

□ the rate of a reaction increases

□ the rate of a reaction decreases

□ the rate of a reaction always equals

Explanation for Prediction:

Observation:

Reconciliation of Prediction and Observation:

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C.1.3 Pre-test questions

Pre-laboratory questions seek to know what students understand the reaction of acid-base after doing the experiment, and to help student conceptual understanding from prior knowledge and from lecture class.

$$CaCO_3\ (s) + 2HCl\ (aq) \rightarrow CaCl_2\ (aq) + H_2O\ (l) + CO_2\ (g)$$

For the reaction as shown above, calcium carbonate reacts hydrochloric acid or vinegar while carbonate is converted to carbon dioxide.

1. The surface of solid reactant, calcium carbonate to egg shells: small particles sizes → bigger particle sizes

Explanation for your reason:

2. The concentration of hydrochloric acid or vinegar: low concentration → higher concentration

Explanation for your reason:

3. The temperature of a reaction: low concentration → higher concentration

Explanation for your reason:

4. The type of acid used in the reaction: week acid → strong acid

Explanation for your reason:

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C.1.4 Post-test questions

Post-laboratory questions seek to know what students understand the reaction of acid-base after doing the experiment, and to help student conceptual understanding from prior knowledge and from lecture class.

$$CaCO_3\ (s) + 2HCl\ (aq) \rightarrow CaCl_2\ (aq) + H_2O\ (l) + CO_2\ (g)$$

For the reaction as shown above, calcium carbonate reacts hydrochloric acid or vinegar while carbonate is converted to carbon dioxide.

After doing the experiment, please describe the rate of a reaction for chemical kinetics when changing variables:

1. The surface of solid reactant, calcium carbonate to egg shells: small particles sizes → bigger particle sizes

Explanation for your reason:

2. The concentration of hydrochloric acid or vinegar: low concentration → higher concentration

Explanation for your reason:

3. The temperature of a reaction: low concentration → higher concentration

Explanation for your reason:

4. The type of acid used in the reaction: week acid → strong acid

Explanation for your reason:

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C.1.5 Questionnaire

What actually happened in this practical class?

After doing the experiment

Directions for students

These questionnaires contain statements about the interactions you had with your teacher and other students and about how you found the new this practical class of chemical kinetics in a general chemistry laboratory SCCH 108.

Please indicate your honest views by circling what you think actually happened in this class. For each statement, circle only one number corresponding to your answer. Your answers will enable us to improve future science teaching in this experiment.

There are no 'right' or 'wrong' answers. Your opinion is what is wanted. Think about how well each statement describes what this practical class was like for you.

Draw a circle around:

1 if the practical takes place **Strongly Disagree**
2 if the practical takes place **Disagree**
3 if the practical takes place **Neutral**
4 if the practical takes place **Agree**
5 if the practical takes place **Strongly Agree**

Suppose that you felt the statement: 'This experiment helped me understand the concept of chemical kinetics.' helps you a lot to learn in chemical kinetics. You would circle **Strongly Agree**, circle the 5.

Section D	**Strongly Disagree**	**Disagree**	**Neutral**	**Agree**	**Strongly Agree**
This experiment helped me understand the concept of chemical kinetics.	1	2	3	4	(5)

Be sure that you gave an answer for all questions.

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Suppose that you felt the statement: 'In this experiment, the teacher asked for my opinion while doing the experiment. ' is almost always true, you would circle 1. However, if your teacher almost always asks your opinion, you would circle 5, or you can circle the number 2, 3, and 4 if one of these seems like a more accurate answer.

Section A	**Strongly Disagree**		**Neutral**		**Agree Strongly Agree**
In this experiment, the teacher asked for my opinion while doing the experiment.	(1)	2	3	4	5

If you change your mind about an answer, cross it out and circle a new number.

Section A	**Strongly Disagree**		**Neutral**		**Agree Strongly Agree**
In this experiment, the teacher asked for my opinion while doing the experiment.	~~1~~	2	3	4	(5)

Some statements in this questionnaire are similar to other statements. Do not worry about this. Simply give your opinion about all statements.

Tasks: Tick the appropriate box in the tasks below.

Your Student Identification Number: ……

Gender: □ **Male** □ **Female**

Age (write your age): ……

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Section A	**Strongly Disagree**		**Neutral**	**Agree**	**Strongly Agree**
1. Before the laboratory session, the teacher lectured problems clearly to me.	1	2	3	4	5
2. In this experiment, the teacher encouraged me to understand what I was doing in the experiment.	1	2	3	4	5
3. In this experiment, the teacher is friendly to me.	1	2	3	4	5
4. In this experiment, the teacher is interested to answer my questions.	1	2	3	4	5
5. In this experiment, the teacher helped me when I had trouble in the class.	1	2	3	4	5
6. In this experiment, the teacher asked for my opinion while doing the experiment.	1	2	3	4	5
7. In this experiment, the teacher's questions helped me to understand my problems.	1	2	3	4	5
8. In this experiment, the teacher encouraged me to discuss my ideas with my group.	1	2	3	4	5
9. In this experiment, the teacher asked for my opinion during group discussion.	1	2	3	4	5
10. In this experiment, the teacher encouraged me to discuss my ideas with other students.	1	2	3	4	5
Section B	**Strongly Disagree**		**Neutral**	**Agree**	**Strongly Agree**
11. In this experiment, I enjoyed the use of Predict-Observe-Explain (POE).	1	2	3	4	5
12. In this experiment, I understood how to use POE.	1	2	3	4	5
13. In this experiment, I felt that the POE was a useful way to predict.	1	2	3	4	5
14. In this experiment, I felt that the POE is a useful way to explain.	1	2	3	4	5
15. In this experiment, I attempted to make connections between my prediction and my observation.	1	2	3	4	5

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Section C	Strongly Disagree	Disagree	Neutral	Agree	Strongly Agree
16. In this experiment, I learned how to design the experimental procedure for chemical kinetics.	1	2	3	4	5
17. In this experiment, I learned how to use data to support my conclusion for chemical kinetics.	1	2	3	4	5
18. In this experiment, I felt that learning science stimulated my thinking to solve my problem.	1	2	3	4	5
19. In this experiment, I felt that I developed practical skills in doing experiments.	1	2	3	4	5
20. In this experiment, I felt that I learned to do things in a way more like scientists do.	1	2	3	4	5
21. I worked cooperatively in this experiment.	1	2	3	4	5
22. I learned to work in a team in this experiment.	1	2	3	4	5
23. I shared ideas with other students while doing this experiment.	1	2	3	4	5
24. I discussed ideas and methods with other students in my group before doing this experiment.	1	2	3	4	5
25. I felt that working in a group stimulated my thinking.	1	2	3	4	5
26. I participated in my group during the discussion.	1	2	3	4	5
27. I explained my ideas to other students in my group.	1	2	3	4	5
28. I learned ideas from other students in my group.	1	2	3	4	5
29. I talked with other students about how to investigate my problem.	1	2	3	4	5
30. I felt that the group discussion helped me to understand the concept of chemical kinetics.	1	2	3	4	5

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Section D	**Strongly Disagree**	**Disagree**	**Neutral**	**Agree**	**Strongly Agree**
31. This experiment was interesting.	1	2	3	4	5
32. The POE was useful in designing my experiment.	1	2	3	4	5
33. This experiment helped me to understand the concept of chemical kinetics.	1	2	3	4	5
34. This experiment helped me to understand what variables affect the rate of a reaction.	1	2	3	4	5
35. This experiment helped me to understand how variables affect the rate of a reaction.	1	2	3	4	5
36. In this experiment, I learned how to control each variable that affects the rate of a reaction.	1	2	3	4	5
37. In this experiment, I learned how to construct the graph of the relationship between the volume of carbon dioxide and time.	1	2	3	4	5
38. In this experiment, I learned how to calculate the rate of a reaction.	1	2	3	4	5
39. In this experiment, I learned more about chemical kinetics.	1	2	3	4	5
40. This practical chemistry laboratory class made me want to continue in science.	1	2	3	4	5

Thank you

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APPENDIX D

D.1 Student performance in the laboratory

Figure D.1 Students vary particle sizes of egg shells

Figure D.2 Experiment was carried out by students

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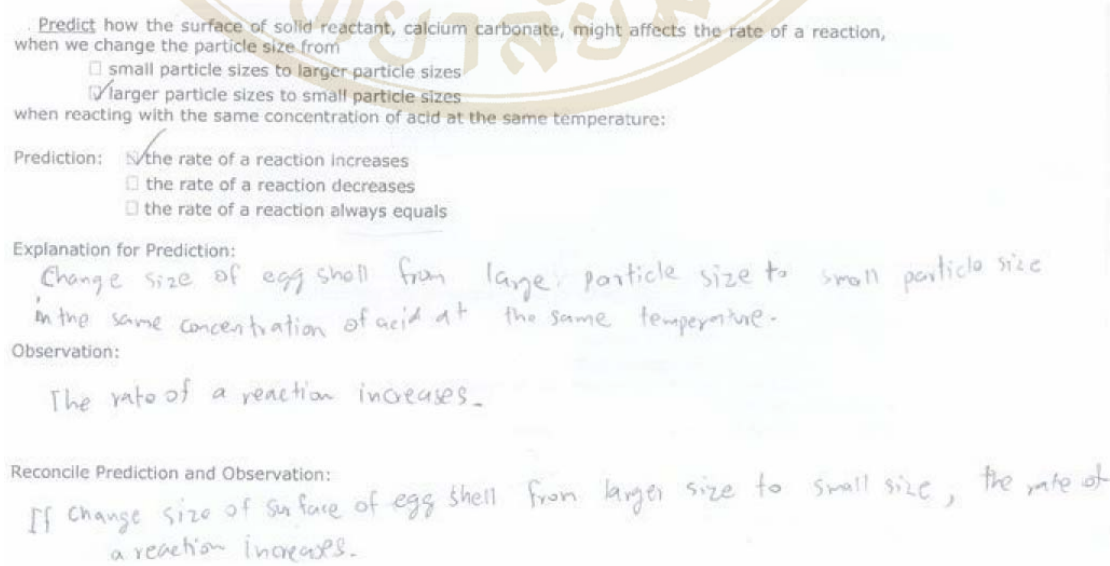
Figure D.3 The interaction between students in groups with TAs' (a) Tukta, (b) P'Peng, (c) P'Beer, and (d) Attipol during the laboratory



Predict how the surface of solid reactant, calcium carbonate, might affects the rate of a reaction, when we change the particle size from

- ☐ small particle sizes to larger particle sizes
- ☑ larger particle sizes to small particle sizes

when reacting with the same concentration of acid at the same temperature:

Prediction:
- ☑ the rate of a reaction increases
- ☐ the rate of a reaction decreases
- ☐ the rate of a reaction always equals

Explanation for Prediction:
Change size of egg shell from large particle size to small particle size in the same concentration of acid at the same temperature.

Observation:
The rate of a reaction increases.

Reconcile Prediction and Observation:
If change size of surface of egg shell from larger size to small size, the rate of a reaction increases.

Figure D.4 Students' POE Activity for chemical kinetics as used in the study

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APPENDIX E

E.1 Mean ($\bar{x}$) and standard deviation (Std. Dev.)

E.1.1 Mean ($\bar{x}$)

Sometime it is also called average, obtained by dividing the sum of replicate measurements by the number of measurements in the test,

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N} \quad \text{(B1.1)}$$

where N = number of replicate measurement, x_i = represents the individual values of x.

E.1.2 Standard deviation (Std. Dev.)

Sometime it is called Std. Dev. Which is a measure of the precision of a sample of data, the calculation is given by,

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N-1}} \quad \text{(B1.2)}$$

where N = number of replicate measurement, x_i = represents the individual values of x.

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E.1.3 Statistical analysis

Table E.1.2 Estimated Mean and Standard Deviation of the items obtained from students' responses (n = 389)

Items	Estimated Values	
	Mean	Std. Dev.
1. Before the laboratory session, the teacher lectured problems clearly to me.	3.663	0.754
2. In this experiment, the teacher encouraged me to understand what I was doing in the experiment.	3.599	0.727
3. In this experiment, the teacher is friendly to me.	4.177	0.747
4. In this experiment, the teacher is interested to answer my questions.	4.177	0.747
5. In this experiment, the teacher helped me when I had trouble in the class.	4.038	0.774
6. In this experiment, the teacher asked for my opinion while doing the experiment.	3.617	0.736
7. In this experiment, the teacher's questions helped me to understand my problems.	3.599	0.759
8. In this experiment, the teacher encouraged me to discuss my ideas with my group.	3.627	0.755
9. In this experiment, the teacher asked for my opinion during group discussion.	3.609	0.754
10. In this experiment, the teacher encouraged me to discuss my ideas with other students.	3.678	0.761
11. In this experiment, I enjoyed the use of Predict-Observe-Explain (POE).	3.591	0.856
12. In this experiment, I understood how to use POE.	3.555	0.750

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Table E.1.2 (Conts.)

Items	Estimated Values	
	Mean	Std. Dev.
13. In this experiment, I felt that the POE was a useful way to predict.	3.760	0.737
14. In this experiment, I felt that the POE is a useful way to explain.	3.822	0.734
15. In this experiment, I attempted to make connections between my prediction and my observation	3.789	0.740
16. In this experiment, I learned how to design the experimental procedure for chemical kinetics.	3.622	0.749
17. In this experiment, I learned how to use data to support my conclusion for chemical kinetics.	3.779	0.687
18. In this experiment, I felt that learning science stimulated my thinking to solve my problem.	3.951	0.773
19. In this experiment, I felt that I developed practical skills in doing experiments.	3.882	0.751
20. In this experiment, I felt that I learned to do things in a way more like scientists do.	3.730	0.807
21. I worked cooperatively in this experiment.	4.275	0.717
22. I learned to work in a team in this experiment.	4.229	0.751
23. I shared ideas with other students while doing this experiment.	4.077	0.742
24. I discussed ideas and methods with other students in my group before doing this experiment.	3.902	0.777
25. I felt that working in a group stimulated my thinking.	4.111	0.736
26. I participated in my group during the discussion.	4.273	0.664
27. I explained my ideas to other students in my group.	3.964	0.749
28. I learned ideas from other students in my group.	4.008	0.698

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Table E.1.2 (Conts.)

Items	Estimated Values	
	Mean	Std. Dev.
29. I talked with other students about how to investigate my problem.	3.977	0.686
30. I felt that the group discussion helped me to understand the concept of chemical kinetics.	3.884	0.753
31. This experiment was interesting.	3.738	0.830
32. The POE was useful in designing my experiment.	3.735	0.714
33. This experiment helped me to understand the concept of chemical kinetics.	3.668	0.708
34. This experiment helped me to understand what variables affect the rate of a reaction.	3.830	0.712
35. This experiment helped me to understand how variables affect the rate of a reaction.	3.905	0.713
36. In this experiment, I learned how to control each variable that affects the rate of a reaction.	3.810	0.687
37. In this experiment, I learned how to construct the graph of the relationship between the volume of carbon dioxide and time.	3.668	0.750
38. In this experiment, I learned how to calculate the rate of a reaction.	3.748	0.759
39. In this experiment, I learned more about chemical kinetics.	3.717	0.730
40. This practical chemistry laboratory class made me wants to continue in science.	3.836	0.833

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BIOGRAPHY

NAME	Mr. Sanoe Chairam
DATE OF BIRTH	February, 14 1980
PLACE OF BIRTH	Buri ram, Thailand
INSTITUTIONS ATTENDED	Ubon Ratchathani University, 2001: Bachelor of Science (Chemistry)
	Mahidol University, 2008: Doctor of Philosophy (Science and Technology Education)
RESEARCH GRANT	The University Development Program on Higher Education through the Department of Chemistry, Faculty of Science, Ubon Rajathanee University, Ubon Rajathanee, Thailand, 2003-2008
HOME ADDRESS	31 Moo 11 Tambol Sagae Sam, Amphur Maung, Buri ram 31000, Thailand

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