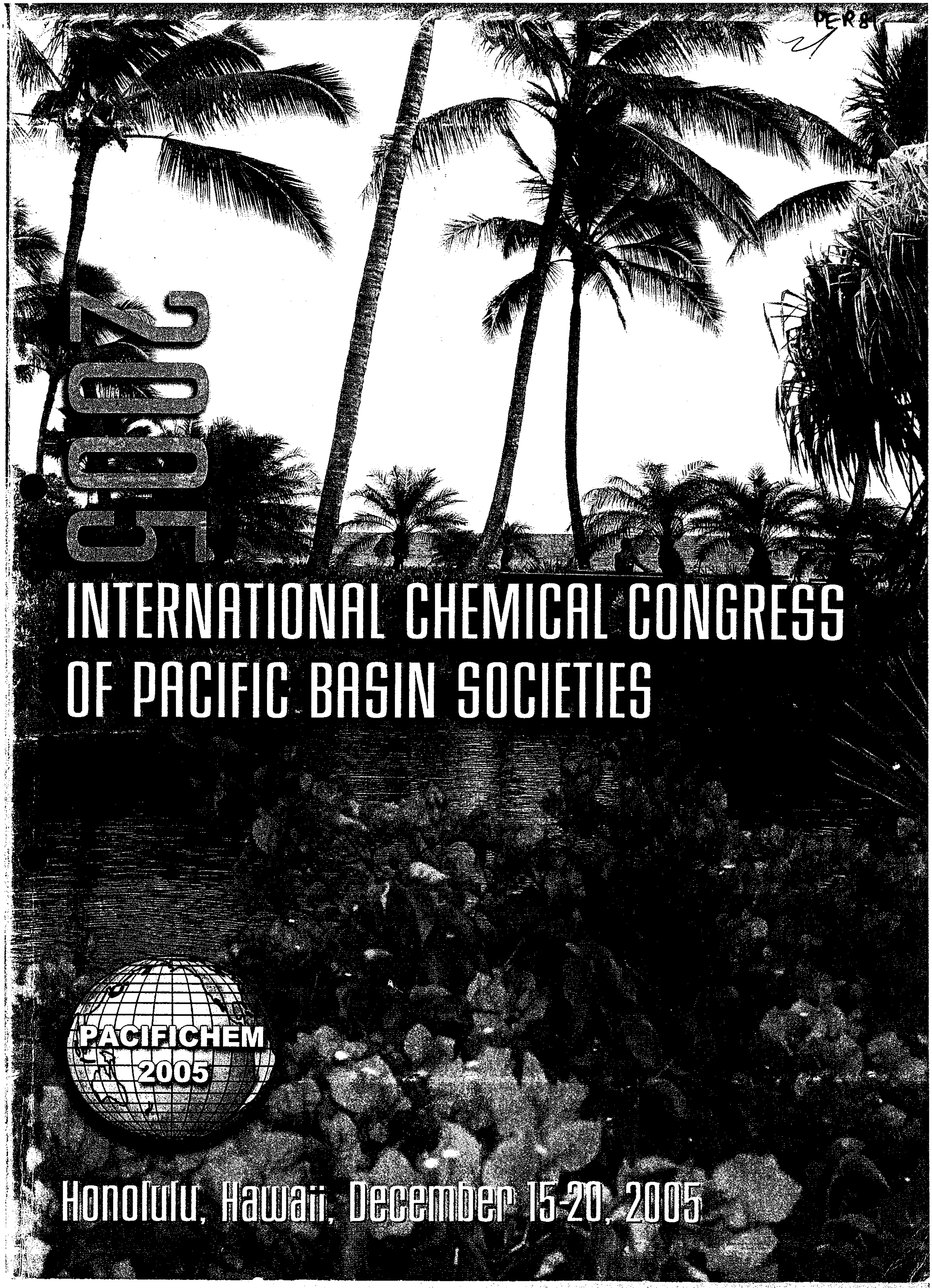

2005

INTERNATIONAL CHEMICAL CONGRESS OF PACIFIC BASIN SOCIETIES

PACIFICHEM 2005

Honolulu, Hawaii, December 15-20, 2005

Formation of Gold Microstructures by Galvanic Reduction on Carbon Surfaces

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Hamzah Darus
Foremost Excel Sdn Bhd., Malaysia

1

Introduction

- Electrodeposition of metal nanocrystals onto carbon surfaces widely investigated
  - Use as electrocatalysts
- Nanosized gold has unusual catalytic and physical-chemical properties.
- Electrochemical procedures allow variation of crystallite size
  - Control of physical and chemical properties

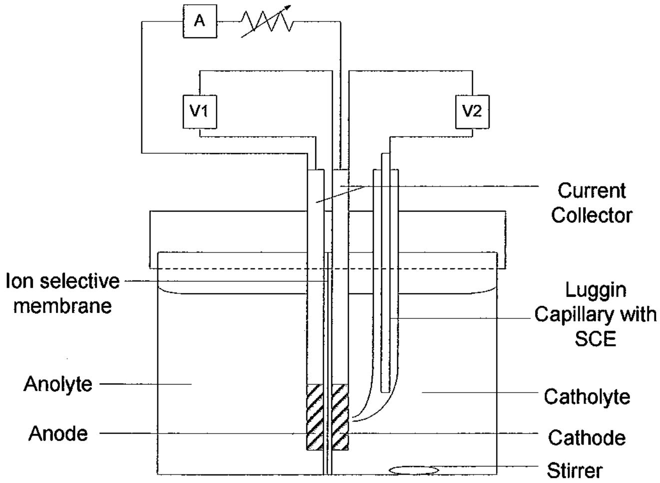
2

Gold deposition by galvanic reduction

- From gold cyanide solutions
- On graphite and reticulated vitreous carbon
- Spontaneous chemical reaction between Au/Zn couple
- No external supply of energy required

3

Static batch reactor



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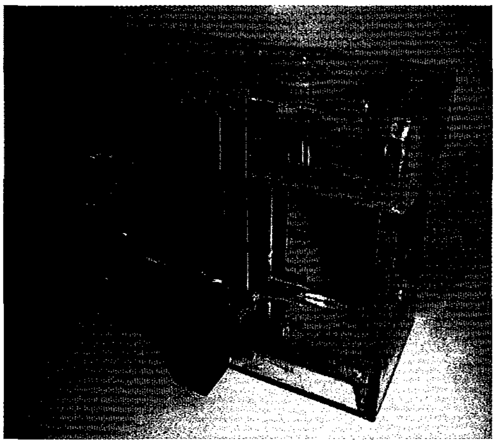
Electrogenerative process

- Static batch reactor
- Gold deposited using different combinations of anode and cathode



5

Overall Reaction

Cathode: E^0, V

$Au(CN)_2^- + e^- \rightleftharpoons Au + 2CN^-$ - 0.600

Anode:

$Zn(CN)_4^{2-} + 2e^- \rightleftharpoons Zn + 4CN^-$ -1.250

Overall reaction:

$2Au(CN)_2^- + Zn \rightleftharpoons 2Au + Zn(CN)_4^{2-}$ +0.650

- The positive overall cell potential indicates that the reaction is a spontaneous reaction.

6

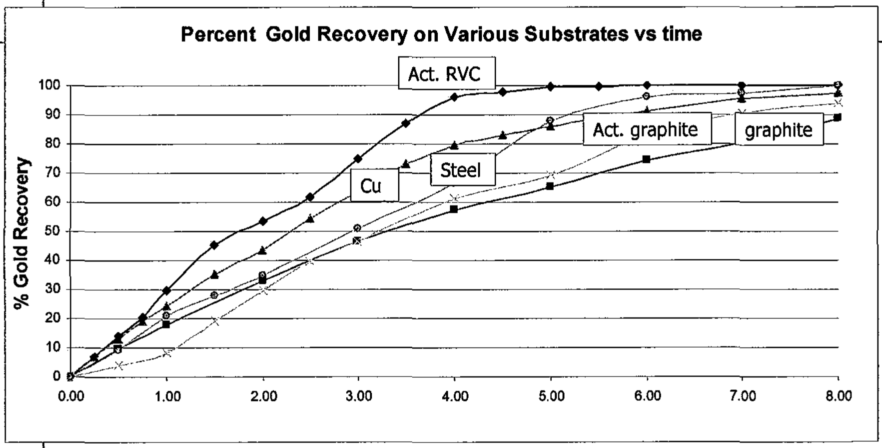
- By subjecting the system to an external load - regulating the current; different deposition rate.
- Characterization of gold particles deposited galvanically on carbon substrates- Graphite and Reticulated Vitreous Carbon (RVC).

7

Recovery of metals by galvanic reduction

- Copper (static batch and flow cells)
- Reduction of hexavalent chromium to trivalent chromium
- Gold from cyanide leach solutions

8



9

Scope of work

- Use of static batch cell for deposition of gold particles.
- Influence of external load.
- Influence of bath composition – Co, Cu.
- Effect of electrode activation .

10

Experimental conditions

- Anolyte : 0.5% sodium cyanide
- Catholyte: 500 ppm gold simulated solution in
  0.2% NaCN
  Spent Gold electroplating solutions
  (Co, Cu)
- Anode : Zinc foil 99%
- Cathode : porous graphite, 80 ppi RVC

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Experimental conditions

- Variation of external load - 0 Ω to 500 Ω .
- The graphite and RVC were activated by using tin-palladium activation solution.
- Surface composition and morphology were studied using SEM-EDX.

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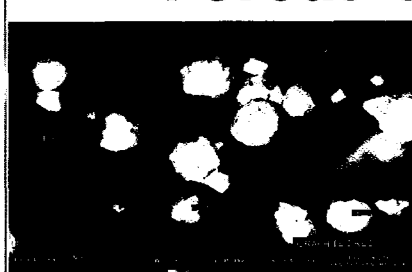
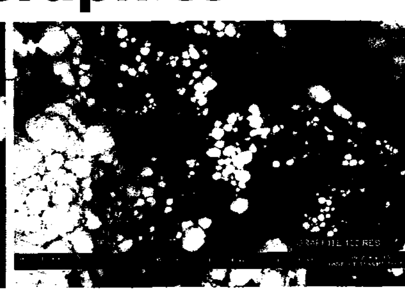
Gold Simulated Solutions

13

Porous Graphite



0 Ω

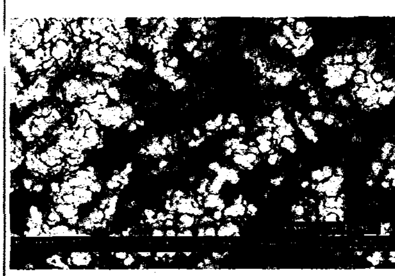


100 Ω

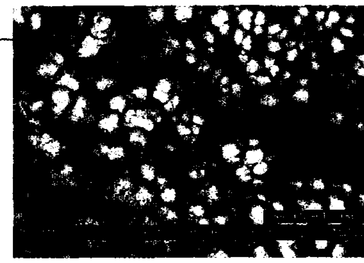
250 Ω

14

Activated Porous Graphite



0 Ω



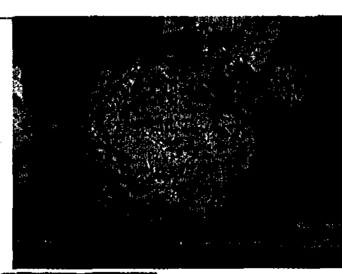
100 Ω

250 Ω

15

RVC

0 Ω

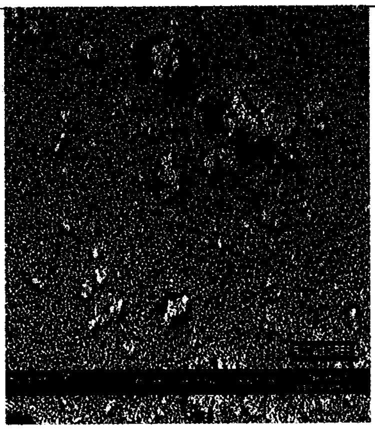


50 Ω

100 Ω

16

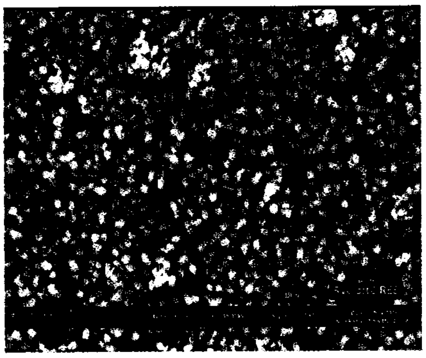
Activated RVC

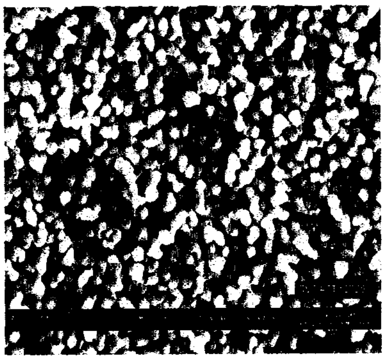


0 Ω **50 Ω**

17

Activated RVC





0 Ω **50 Ω**

18

Au solutions

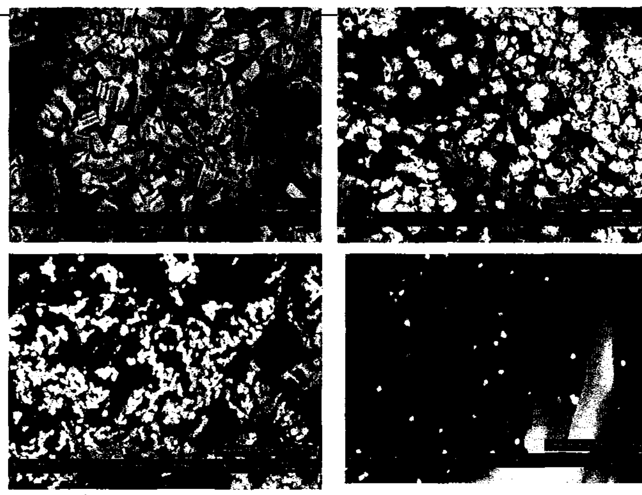
- No gold deposition on activated RVC for R - 100Ω and 250 Ω

19

Gold-Cobalt Plating Solution

20

Porous Graphite, Au-Co



0 Ω

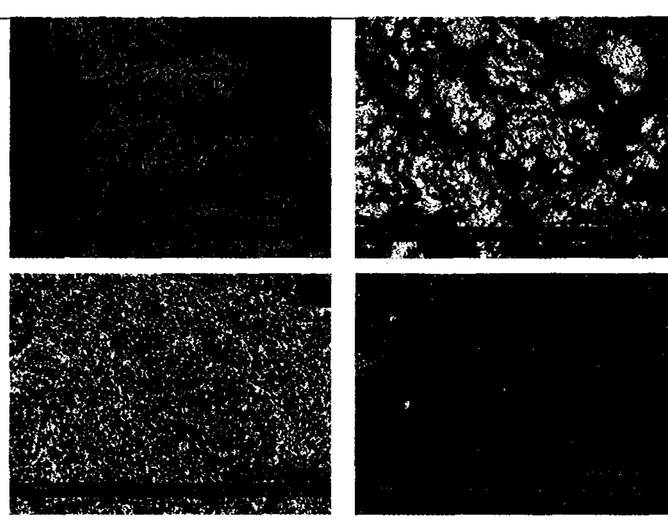
100 Ω

250 Ω

500 Ω

21

Activated Porous Graphite



0 Ω

100 Ω

250 Ω

500 Ω

22

RVC, Au-Co

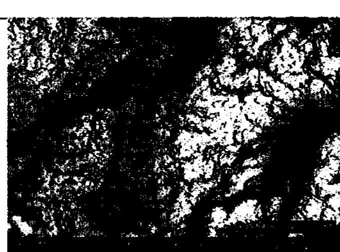
0 Ω

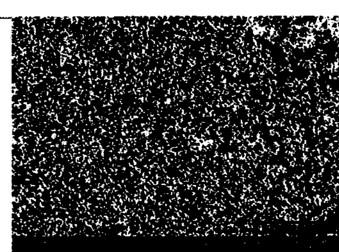
100 Ω

250 Ω

23

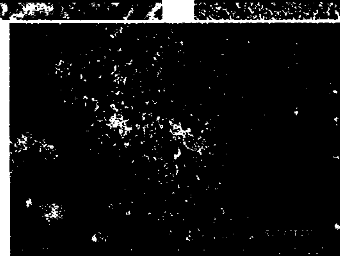
Activated RVC, Au-Co





0 Ω

100 Ω



500 Ω

24

Au-Co solutions

- No codeposition of cobalt observed
- Crystalline like deposits

25

Gold- copper plating solution

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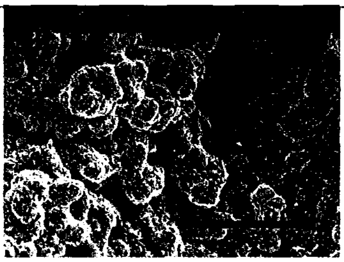
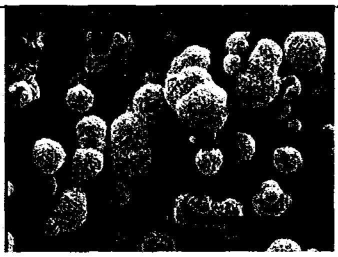
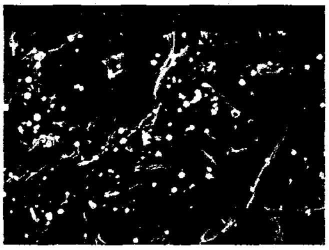
Graphite, Au-Cu

0 Ω





100 Ω

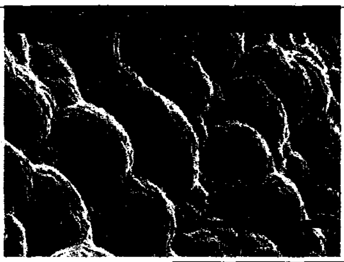


500 Ω

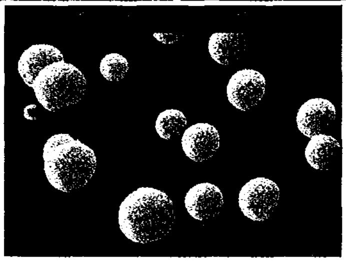
27

RVC, Au-Cu

0 Ω



100 Ω



500 Ω

28

Au-Cu solutions

- Codeposition of copper with gold
- Granular like deposits

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Conclusions

- Less gold deposition with higher external load applied (lower current densities)
- Activation of substrate increases gold deposition, produces smaller sizes
- RVC – smaller particle sizes
- Presence of Cu^{2+} and Co^{2+} affects microstructure of gold deposits

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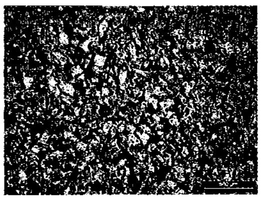
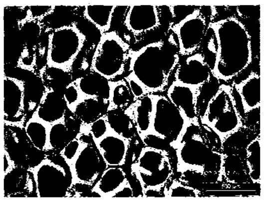
Acknowledgements

- Academy of Science Malaysia
- Nippon Sheet Glass Foundation
- Universiti Sains Malaysia



Au on graphite



Au on RVC

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The 2005 International Chemical Congress of Pacific basin Societies, 15-20 Dec. 2005, Honolulu, Hawaii

30001

DEVELOPMENT OF MICROSCALE CHEMISTRY EXPERIMENTATION FOR SECONDARY SCHOOLS IN MALAYSIA

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1

ABSTRACT

The need to produce science literate students has been recognized as an important objective for science education in Malaysia. Hands-on chemistry experiences for students are an essential component for them to acquire science process skills. However, due to various reasons which include high costs, safety, waste disposal, time constraints and teacher training, practical chemistry sometimes takes a back seat in the teaching of chemistry. One of the solutions to overcome these problems would be to implement microscale chemistry which is a laboratory based and environmentally safe approach accomplished by using miniature lab apparatus and reduced amounts of chemicals. This study involves 4 stages: development of microscale experimentation, workshops / seminars, determination of effectiveness among students and determination of feasibility among students and teachers.

2

INTRODUCTION

- Science is to be offered as a core subject to all students at the lower secondary level in Malaysia.
- At the upper secondary level, students are offered science electives (biology, chemistry, physics and additional science) in addition to the core science.
- The curriculum develops, nurtures and reinforces what has been learned at the lower primary level.
- Particular emphases on:
  - Acquisition of scientific knowledge
  - Mastery of scientific and thinking skills
  - Inculcation of moral values

3

OBJECTIVES

The main purpose of this study is to improve the quality of chemical education in upper secondary/high schools through the implementation of microscale chemistry.
More specifically, the objectives of the study are:

- To develop microscale chemistry experiments for upper secondary/high school chemistry students that correspond to traditional macroscale experiments.
- To determine if there is any significant difference in students' achievement, interests and motivation between the control group (macroscale) and the experimental group (microscale).
- To investigate students' and teachers' opinions and problems on using traditional techniques and microscale techniques

4

METHODOLOGY

The research methodology would involve a combination of qualitative and quantitative approaches involving a 4-stage process:

Stage 1: Development of Microscale Chemistry Experimentation
Stage 2: Workshops/ Seminars
Stage 3: Determination of Effectiveness among Students
Stage 4: Determination of Feasibility among Students and Teachers

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Stage 1: Development of Microscale Chemistry Experimentation

- Develop the Form Four chemistry experiments similar to the traditional macroscale experiments.
- The design is take into account less usage of toxic chemicals, reduction of chemical wastes with the use of the microscale kit and improvised apparatus whenever necessary.
- The feasibility of using the microscale chemistry kit is based on the Malaysian KBSM 2004 form four curriculum.

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 1: Introduction to chemistry

(i) Solubility of salt in water

Chapter 2 : Atomic Structure

(i) Matter is made up of particles

(ii) Determination the melting point and freezing point of naphthalene

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 3: Chemical Formulae And Equations

(i) Determination of the empirical formula of magnesium oxide

(ii) Determination of the empirical formula of copper(II) oxide

Chapter 4: Periodic Table of Elements

(i) Chemical properties of group I

(ii) Chemical properties of group 17

(iii) Chemical properties oxide of period 3 elements

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 5: Chemical Bonds

(i) Preparation of ionic compounds
(ii) Comparison between the ionic and covalent compounds

Chapter 6: Electrochemistry

(i) Classifying chemicals into electrolytes and non-electrolytes
(ii) Electrolysis of molten lead(II) bromide
(iii) Electrolysis of an aqueous solution
(iv) Chemical reaction in simple Voltaic cell and Daniel cell.
(v) Purification and electroplating of metals
(vi) Electrochemical series of metals
(vii) Metal displacement reactions

9

Chapters and experiments in KBSM form four chemistry syllabus

Chapter 7: Acid and Bases

(i) The role of water to indicate the properties of acid and alkali
(ii) Measurement the pH of solution used in daily life
(iii) The relationship between pH value and the concentration of solutions.
(iv) Preparation of a standard solution of sodium hydroxide, NaOH.
(v) Preparation of a solution with specified concentration from a standard solution through dilution.
(vi) Investigate the relationship between pH values with the molarity of diluted solutions of an acid and alkali.
(vii) Determination of the end point for neutralization reaction between the acid and alkali.

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 8: Salts

(i) Solubilities of nitrate, sulphate, carbonate and chloride salts
(ii) Preparation of soluble salts through the reaction between the acid and alkali
(iii) Preparation of soluble salts through the reaction between the acid and metallic oxide
(iv) Preparation of soluble salts through the reaction between the acid and metal
(v) Preparation of soluble salts through the reaction between the acid and metallic carbonate

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 8: Salts

(vi) Preparation of insoluble salts
(vii) To construct the balanced chemical equation through continuous variation method
(viii) Carry out chemical tests to identify gases.
(x) Effect of heat on carbonates and nitrate salts
(xi) Confirmation tests of cations in the aqueous solutions
(xii) Confirmation tests of anions in the aqueous solution

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Chapters and experiments in KBSM form four chemistry syllabus

Chapter 9: Manufactured Substances In Industry

(i) Preparation of ammonium sulphate fertilizer

(ii) The comparison of the strength and hardness of alloys with that of their pure metals.

(iii) Corrosion of steel

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DEVELOPMENT OF THE EXPERIMENTS

- **About 80% of the experiments have been worked on.**
- **Experiments that need improvised microscale techniques**

  - Preparation of insoluble salts
  - Determination the empirical formula of magnesium oxide
  - Electrolysis of molten lead(II) bromide
  - Chemical properties of group 17 elements
  - Preparation of ionic compounds
  - Preparation of a standard solution of NaOH
  - Effect of heat on carbonates & nitrate salts
  - Comparisons between an ionic and covalent compounds – melting & boiling point

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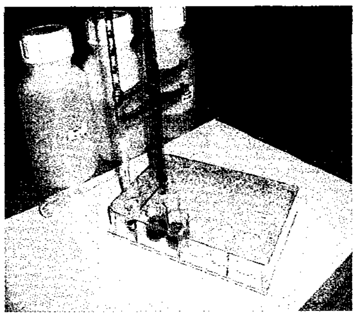
I Determination of the end point for neutralization reaction between the acid and base



Cost of Chemicals (per 30 students)/(RM)			Time Duration (Approx/ minutes)	
Chemicals	Trad.	Micro	Trad.	Micro
Sodium Hydroxide	3.60	0.18	25	12
Hydrochloric acid	0.56	0.03		

- **Data can be collected to three decimal points as compared to two decimal points using the ordinary burette.**

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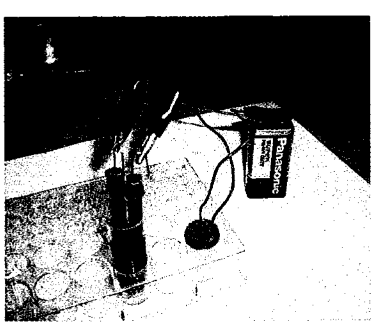
II Electrolysis of aqueous solution



Cost of Chemicals (per 30 students) copper(II)sulphate (RM)		Time Duration (Approx/ minutes)	
Trad.	Micro	Trad.	Micro
7.20	3.60	15 - 20	3 - 5

- **A higher voltage of battery must be used as compared to traditional technique.**

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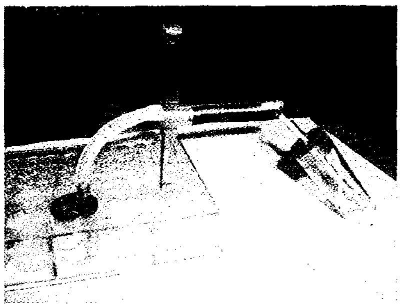
III Effect of heat on carbonate salts



Cost of Chemicals (per 30 students) calsium carbonate (RM)		Time Duration (Approx/ minutes)	
Trad.	Micro	Trad.	Micro
1.19	0.02	5 – 7 Per sample	1 – 2 Per sample

- Replace test tube with glass fusion tube
- Reaction occurs very fast and observations are similar to traditional technique.
- Problem: After the first sample is heated, it is difficult to clean the fusion tube in order to heat other samples.

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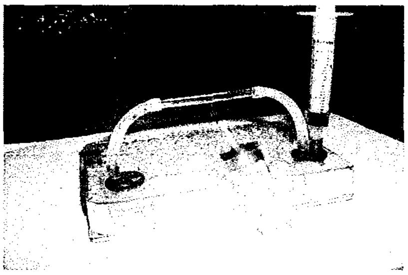
IV Construction of ionic equations through continuous variation method

Cost of Chemicals (per 30 students)/(RM)			Time Duration (Approx/ minutes)	
Chemicals	Trad.	Micro	Trad.	Micro
barium chloride	2.52	0.34	72	16
potassium chromate	3.19	0.42		

- Quantity of solution is measured in terms of drops.
- Differentiation of amount of precipitate formed can be made successfully.
- Problem: difficult to measure the height of precipitate as required by traditional technique.

,18

V Determination the empirical formula of copper(II)oxide



Cost of Chemicals (per 30 students) copper(II)oxide (RM)		Time Duration (Approx/ minutes)	
Trad.	Micro	Trad.	Micro
2.30	0.08	40	20

- done only as a demonstration for most teachers because large quantities of hydrogen produced greatly increases the potential for an explosion.
- In microscale technique, this experiment can be done safely and individually by the students.
- Can be done very quickly because only small quantities of copper(II) oxide powder used.
- reactions and observations similar to traditional technique.
- Problems:
  - accurate weight of copper oxide difficult to obtain (small quantities used)
  - Determining empirical formula of the copper(II) oxide

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Stage 2: Workshops/ Seminars

- The purpose:
  - to expose teachers to microscale experimentation in chemistry
  - to obtain teachers' feedback
- First workshop held on 5th December 2005 in conjunction with the International Conference On Science & Mathematics Education, CoSMEd.
  - Participants : Sixteen experienced teachers
  - List of experiments: electrolysis of aqueous solution, electroplating of metals, reduction of copper(II oxide and determination of the end point for neutralization reaction between the acid and alkali (acid-base titration), qualitative analysis of ions.
- The questionnaire:
  - Items based on 5 constructs : worksheet, equipments, evaluation of experiment, hands-on and feasibility of microscale experiments.
  - 5-point Likert scale used range from 5-strongly agree, 4 - agree, 3 - not sure, 2 - disagree and 1 - strongly disagree.

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RESULTS FROM THE QUESTIONNAIRE

Constructs	Average Score
Worksheet	4.43
Equipment	3.92
Evaluation of microscale experiment: Expt. 1: Electrolysis of aqueous solution Expt. 2: Electroplating of metals Expt. 3: Reduction of copper(II)oxide Expt. 4: Determination of the end point for neutralization reaction between acid and alkali	 4.75 4.80 4.77 4.52
Hands-on microscale experiment	4.49
Feasibility of microscale experiment	4.52

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TEACHERS' FEEDBACK

- Data from average score indicate that majority of the teachers agree and have a positive view of microscale chemistry.
- Teachers, opinions in term of problems that might be faced:
  - Difficult to control microburette to get the accurate end-point
  - Difficulty in cleaning the well. In order to clean the dirty one, we have to clean all of the wells.
  - Students may not have the confidence to handle the actual laboratory apparatus.

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TEACHERS' FEEDBACK

- **Teachers; suggestion:**
  - Select the experiment which is suitable for microscale or for traditional techniques

- **<u>Teachers' comments:</u>**
  - Good for future science & technology curriculum
  - Suggest more workshops /seminars
  - Very applicable to secondary schools especially when costs can be reduced greatly

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Stage 3: Determination of Effectiveness among Students

- **A quantitative design, and quasi-experimental pre test – posttest control group is proposed.**
- **independent variables : microscale chemistry and traditional laboratory approach**
- **dependent variables: students' achievement, attitudes and motivation in chemistry.**
- **Both groups will be given the pre-test before the treatment is administered.**
- **Data from the pre test and post test will be statistically analyzed to find the differences between the two groups.**

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Stage 3: Determination of Effectiveness among Students

- **The research design is illustrated in Figure 1.**

Intact	Pre-test	Treatment	Post-test
Experimental group	T1	Microscale chemistry technique	T2
Control group	T1	Traditional chemistry laboratory	T2

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Stage 4: Determination of Feasibility among Students and Teachers

- Surveys, interviews and observations will be used.
- It is proposed that 150 students from four different schools (urban and rural) will be involved in the tryout of the microscale experiments. Another 150 students will be selected to be the control or comparison group
- These last two stages are planned to be done by July 2006.

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