

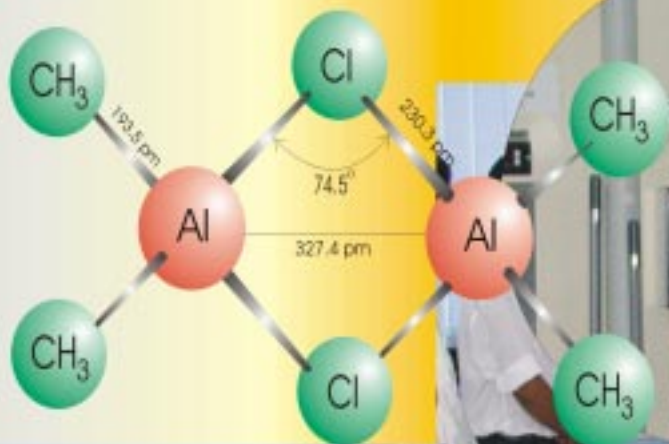


THE SCHOOL OF CHEMICAL SCIENCES UNIVERSITI SAINS MALAYSIA

BULLETIN

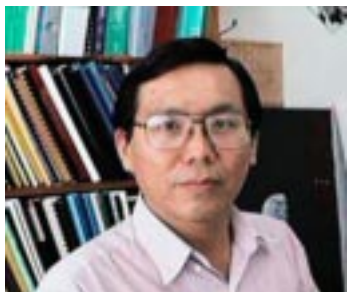
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KIMIA • USM



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From the Chief Editor



The School of Chemical Sciences, Universiti Sains Malaysia has definitely come of age. Over the years, it has built up a research culture which attracts an increasing number of post-graduate students both locally and from overseas to pursue their studies here. In terms of research productivity, more research findings were published in reputable journals. Certain research efforts have even culminated in products and inventions which have won acclaims both locally and abroad. In view of the great strides made in research over the years, the time has come for the School to have its own bulletin, namely the KIMIA.USM, to serve as a platform for staff members to share research information and ideas. In addition, this publication will provide first-hand information on the current research activities of the School. It is targeted that two issues of the Chemistry Bulletin will be published each year.

This inaugural issue has received relatively good response from the academic staff, particularly the more junior members, in contributing interesting articles. It features articles covering various research interests from computational chemistry to wastewater treatment. A special write up introducing a new

unit that offers analytical services to clients within and outside the university is also included in the current issue.

For the success of this bulletin, strong and continuous support from the academic staff of the School are absolutely essential. In this regard, it is hoped that more academic staff members will contribute articles in the forthcoming issues. We are not contented to keep the bulletin going only but to strive for excellence. Happy reading.

Prof. Lim Poh Eng

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Message from the Dean



The School of Chemical Sciences has finally succeeded in publishing the inaugural issue of its bulletin, the KIMIA.USM. The school wishes to congratulate the editorial board members for their effort and time and also extends its gratitude to the contributors of articles for their encouraging support.

The last decade has seen a substantial improvement in the basic facilities and funding for research in the school. Over the years, the school has progressed and recorded a proportionately impressive achievement in terms of the number of active researchers, postgraduate students, papers presented at national and international conferences and publications in refereed journals. New fields of interest and ideas have also emerged via researchers' expanding network and collaboration with international academic and research institutions. Multidisciplinary research is seen to be a necessity and has to be initiated at a speed that is ever increasing in the current competitive environment. In short, the school is on the move towards its goal of becoming a research status school in line with the Universiti Sains Malaysia's aspiration to become a research university.

For ideas to expand, for research findings to be further developed, and for expertise to be utilized

in converting findings to processes or products and providing solutions to problems, they must be made known and accessible to interested parties. Dissemination of ideas has to be efficient and continually improved. The KIMIA.USM is envisaged as a means to achieve some of these objectives as it provides avenue for researchers to publish their interim materials, ideas and views related to their respective field for potential students and collaborators.

This issue contains twelve articles covering different fields of research which are actively ongoing in the school. I am pleased to note the quality and a simplistic style of presentation by the respective author whom I reckon have done his/her best to maintain precision and clarity of the writing within the page-limit guidelines imposed by the editorial board. On that note suggestions are welcome as there is always room for improvement.

We wish that the KIMIA.USM can reach and be read by many people. The cost factor limits the number of copies that can be printed for distribution. Nevertheless, by having it on the school web site, hopefully this will offset the shortcoming and enable it to reach the targets.

This first issue marks another milestone in our journey towards achieving international recognition in research. It is like riding on a bumpy country road before getting onto the highway. One must not spare the effort, patience and perseverance before achieving excellence in the end.

Assoc. Prof. Jamil Ismail

Mangrove Tannin for Anti-Corrosive Coatings

**Afidah Abdul Rahim and
Assoc. Prof. Mohd Jain Noordin Mohd Kassim**



Natural tannin extracts have been employed since the turn of the last century in the leather industry. Recent development of their industrial uses as adhesives, flocculants, depressants, viscosity modifier agents and more recently as corrosion inhibitors reflect their importance as industrial raw materials relative to the synthetic phenols based on petroleum[1].

Tannins are polyphenols that occur only in vascular plants such as leaves, needles, barks, heartwood, seeds and flowers[2]. Tannins exist primarily in condensed and hydrolysable forms. Condensed tannins consist of oligimers and polymers of flavanoids while hydrolysable tannins are made up of sugars (primarily glucose) and gallic acid.

Proper surface preparation is the key in obtaining good performance by a surface coating. The major problem in preparing a steel surface is the removal of all mill scale, rust and associated contaminants from the substrate. Tannins as corrosion inhibitors are applied both in solvent and waterborne pretreatment formulations. These formulations could be

applied on partially rusted substrates thus reducing the effort normally needed for cleaning the surface by sand blasting or other methods which prove to be expensive and are not applicable in many situations. They have been called rust converters since their presence converts active rust into iron-tannates, which do not further react with corrosive agents and impede steel corrosion. The proximity of hydroxyl groups on the aromatic rings of tannin makes them able to chelate iron ions. Although other complexation products undoubtedly formed, the ferric tannate complex has been cited as the major product[3,4].

At present, Malaysia is blessed with 101,877 ha of mangrove forest. Matang mangrove forest, at 40,151 ha, is the largest single mangrove forest in Peninsular Malaysia and has been acknowledged to be the best managed mangrove forest in Malaysia. Matang mangrove has been consistently managed under a 30~year rotation since 1950. Within the 30~year rotation, two commercial thinnings are carried out when the forest crops attain the ages of 15 and 20 years, using a 1.2 m and 1.6 m stick respectively. Only two

species, specifically *Rhizophora apiculata* and *Rhizophora mucronata*, are continuously propagated in Matang either artificially or naturally.

We are currently looking into the use of the *Rhizophora apiculata* barks, waste products of the Larut Matang Charcoal Industry, for anti-corrosive coatings. The research will include the establishment of the method of extraction of hydrolysable and condensed tannin, characterization, chemical analysis, complexation of iron-tannates, formulation of anti-rust paints, inhibition mechanism studies and field tests. Future research will be expanded to include the extraction of tannin from other parts of the mangrove tree namely the leaves, fruits and the flowers as well as the complexation of other metals with mangrove tannin. We would

also incorporate computer simulation studies in this research. We have recently established a research collaboration with the Universite of Henri Poincare, Nancy, France.

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Capillary Electrophoresis- From Anions to Antibiotics Analysis

Assoc. Prof. Bahruddin Saad and Prof. Muhammad Idiris Saleh



Our association with capillary electrophoresis (CE) started in 1997 when we purchased a Waters Capillary Ion Analyzer. The unit is fitted with a UV detector and is interfaced to a Waters PC 800 Workstation. The inherent advantages of the CE such as small sample volume requirement, highly efficient separation and rapid separations have now been recognized by the scientific community. This report summarises the research activities that was conducted within our group with the CE unit, not to mention the numerous consultancy jobs that came in over the years. Details of these activities can be found in the references cited. The industrial samples were mainly for the analysis of anions in process water and organic acids in plating solutions.

Analysis of Anions and Cations in Drinking Water

We used the CE for the analysis of anions (chloride, sulphate, nitrate, fluoride) and cations (potassium, calcium, sodium and magnesium) in drinking water samples such as natural mineral water, bottled drinking water, zam-zam water and tap water [1]. Results from these determinations generally showed a low content of anions and high content of calcium and

magnesium in natural mineral water but non-detectable amounts of anions and cations in bottled drinking water. Out of the 15 mineral water samples that were analyzed, four of the brands showed anionic and cationic levels similar to that of tap water. Zam-zam water was found to be a unique natural mineral water due to its overall high levels of anions and cations.

Separation of Lanthanides

The separation of lanthanides have always posed a considerable challenge to analytical chemists due to the similar properties of these lanthanides. We have systematically investigated the feasibility of separating 15 lanthanide ions, including yttrium using the CE technique [2]. A key component of the electrolyte was the addition of complexing agent and additives to facilitate the separation. It was found that the most suitable electrolyte comprises 6.0 mM guanidine hydrochloride as UV background carrier, 2.5 mM tropolone as complexing agent at pH 4.6 and 2.0 mM dicyclo-18-crown-6 ligand as additive. By using such electrolyte and 20 kV voltage, separation of 15 lanthanides can be effectively separated in less than 15 min. The developed CE method was successfully applied for the analysis of lanthanides in a few types of minerals.

Determination of Paraquat in Biological Fluids

Paraquat is a broad-spectrum contact herbicide that is used in the agricultural sector in more than 130 countries throughout the world. Our interest is to develop a suitable analytical method for paraquat in cases of poisoning, of which 73 % are due to suicides and the remainder due to accidental and occupational exposure [3]. In such cases, speed of analysis is of immense importance, and frequently, samples for analysis are very much limited.

We have investigated the use of a few types of operating CE electrolyte and found that the glycine-HCl buffer at pH 3.0 in the presence of 40 mM NaCl and 5% methanol was the most satisfactory. Direct detection at 254 nm was done. Under such conditions and when operated at 30 kV, paraquat was eluted in about 2.2 min. There was good agreement on the assay of paraquat in formulations, urine, serum, vomitus and stomach-washout between the CE and HPLC methods.

Determination of Oxolinic Acid (Antibiotics) in Fish Feeds and Tissues

Oxolinic acid (OXA) is a common antibiotics that is used in modern agricultural practice, and was reported to be effective against gram-negative bacteria. The drug can be administered orally, mixed in the feed or, alternatively as bath treatment. As consumers and regulatory agencies likewise are interested in the residues of antibiotics, we have successfully developed a CE method that can be used routinely for the analysis of OXA in fish tissues and fish feeds [4].

Current Activities

We are currently collaborating with Prof. Zhari Ismail's group in the School of Pharmacy to investigate the viability of using CE for the standardization of herbs. A few reports have been published on the use of CE for the separation of alkaloids and flavonoids in herbs such as ginseng. We are focusing our activities on the separation of such components and others that can potentially be used as standardization procedures for herbs such as pegaga and tongkat ali to support the local indigenous herbal industry.

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Synthesis and Characterization of Noble Metal Nanoparticles and their Activities as Hydrogenation Catalysts

Prof. Liew Kong Yong



Devices fabricated from basic components in the nanometer, 10^{-9} m, size range are products of nanotechnology of the 21st century. Some nano-size materials, such as nanoparticles, as well as others, such as inorganic-polymer nanocomposites, which have superior properties as compared to the corresponding conventional materials, are already commercially available. These nanoparticles have properties quite different from that of the bulk materials due to their very high surface to bulk ratio and the quantum size effect. The nano-sized noble metals have catalytic activities orders of magnitude higher than their corresponding conventional counterparts and have received much renewed interest in recent years. However, in order to study their properties, they have to be prepared in stable forms. A number of methods, including chemical and electrochemical reduction, vapour deposition as well as microwave irradiation, all in the presence of a stabilizing matrix, have been reported.

We wish to study the effect of the particles size and shapes of nano-sized metal catalysts on their activity and selectivity for hydrogenation. We began by studying the

conditions required for the control of the particle size of Pd metal as a model. The experiments were conducted by dissolving the Pd precursor salt, PdCl_4 , in water mixed with an appropriate amount of the reducing agent and different ratios of the polymer of polyvinylpyrrolidone (PVP), which serves to provide a stabilizing matrix to prevent aggregation of the nanoparticles formed on reduction. It was found that the size of the nanoparticles stabilized in the polymer matrix can be controlled from ~2 to 10 nm using different reducing agents, the concentration of the Pd precursor, the pH and the time of reaction [1]. Further, it was found that the nano-crystals obtained have different shapes, which can also be partially controlled by varying the conditions of the reduction. For example, triangular, pentagonal, hexagonal, rhombohedral and spherical nanocrystals can be preferentially synthesized by refluxing with alcohols as reducing agents and at different stabilizing polymer to Pd precursor ratios. The morphology and structure of these nanocrystals have been characterized by high resolution transmission electron microscopy (TEM) and electron diffraction study [2]. Size control of other noble metals nanoparticles,

including Pt and Ru, stabilized by amphiphilic polymers have also been achieved [3,4]. For example, Ru nanoparticles with controlled sizes from 1 to 8 nm have been synthesized using different reducing agents and at different temperatures. In this case, polyols at their refluxing temperatures have been found to be effective. The reduction has been confirmed by x-ray photoelectron spectroscopy. The size distributions were obtained by direct measurement of the TEM images of more than 300 particles for each of the samples.

Noble metals of the Pt group in the nanometer size range are known to have activity and selectivity at ambient temperature and low pressure as hydrogenation catalysts for various small molecule substrates such as cinnamaldehyde and halonitrobenzene. The effect of boron species and metal complexes on the activity and selectivity of Ru and Pt have been studied and reported [5, 6]. We have also studied the activity of Pt nanoparticles in its colloidal form for the activity and selectivity for the hydrogenation of palm olein. It was found that the reaction occurred at 35 °C and atmospheric pressure with rates comparable to Ni catalysed reaction at high temperature and pressure. The selectivity for triene and diene to monoene was very high, 10 to 100, so that the linolenate and the linoleate can be selectively hydrogenated to the oleate [6]. Furthermore, the formation of the trans-isomer of the remaining oleate was much reduced compared with the conventional commercial Ni catalyzed reaction. The hydrogenation of soya bean oil has also been studied.

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Crystallization in Polymer Blends

Assoc. Prof. Jamil Ismail



Introduction

Polymer blending is a widely used technique in research as well as in industrial production of commercial polymer blends. The technique facilitates processing, tailors the performance properties of polymers via combination of properties of two or more polymer components, and reduces the cost of production of expensive polymers but of high potential. When two polymers are combined in blends, they either form a miscible single phase (homogeneous) or immiscible two discrete phases (heterogeneous). In cases where one of the components is semi-crystalline then a crystalline phase constitutes an additional phase. The morphology becomes even more complex when both components are semi-crystalline.

From the academic perspective, the ultimate goal of studying polymer blends is to acquire basic knowledge about the nature and strength of the physical and chemical interactions between phases. Development of multi-phases in blends depends on the regulation of interfacial chemistry and microstructure in both amorphous and crystalline phases. A precise control of the morphology, a prerequisite for a reproducible large-scale production of polymer blends, requires precise control of different phase changes such as of liquid-liquid phase separation, crystallization, and glass transition. One should also recognize that the structure of components, the blend composition, and thermal treatment are crucial factors in determining the final morphology and properties of the blends.

Our research activities, covering studies on thermal properties, phase behaviour, melting, crystallization, and morphology of polymer blends, are directed towards achieving the above goal. The polymers of interest used in our study are biodegradable and biocompatible polyalkanoates, PHA's, for examples poly(3-hydroxybutyrate), PHB, and poly(3-hydroxybutyrate-co-valerate), PHBV, all of which are semi-crystalline in nature. Since their discovery, these materials have attracted high interest especially in the material design and processing via blending because they pose a high cost of production, a narrow processing window due to relatively high melting point and exhibit inferior mechanical properties related to high crystallinity. Crystallization is unavoidable but with the knowledge and understanding of the kinetics and factors that influence melting, size, microstructure and crystallinity, there is always opportunity and means by which one can regulate a process to produce the desired and reproducible properties through blending.

Crystallization, Supermolecular Structure and Morphology

Figure 1 illustrates a simplified crystallization process of a single chain in the molten state at temperatures below its equilibrium melting point. Chain folding and assembly of the folded segments in an apparent plane termed *lamellar crystals* are the characteristic transformations signifying a crystallization process which

affects very minimally the end-to-end distance or the overall chain conformation at low crystallization temperature. The inter-lamellar regions are filled with tie segments or chain ends that form an amorphous phase.

Figure 2 illustrates a crystallization scheme involving a binary blend of polymer A and B. In a miscible blend system, molecules A and B intermingle in the molten state forming a homogeneous phase. Here, component A crystallizes in an amorphous phase comprising miscible A and B in which the ratio of A continues to decrease relative to B as crystallization proceeds. The inter-lamellar is filled with amorphous phase rich in B. In a semi-crystalline/semi-crystalline blend where component B subsequently crystallizes, the resultant crystals comprise stacks of lamella A and B interlaying in random or block sequence termed *random or block insertion*. A and B may also co-crystallize under certain conditions. The situation is very different in an immiscible system whereby the molten state forms two discrete phases comprising dispersions of minor component A in a matrix of pure B or vice versa depending on the composition of the blend. Crystallization of component A and subsequently component B, in that order as in the miscible system, results in two discrete crystalline phases of A and B. The final structure is rather complex as will be shown below.

A *spherulite* is the typical *supermolecular structure* as revealed by polarized optical microscope. Nucleation and radial growth of spherulites, both a function

of cooling rate and under-cooling, characterize a crystallization process as shown in Fig. 3. A spherulite comprises stacks of lamellar having uniform thickness and twisted plane depending on the thermal history and crystallization temperature; this gives rise to microstructural variations[1]. These variations produce, in turn, structural variations of the spherulites typically from narrow to wide ring-banded and to fibrillar patterns. Figure 4 represents a micrograph of a neat poly(3-hydroxybutyrate), PHB, showing a morphology where one can recognize the spherulites and the inter-spherulite boundaries[2]. The amorphous phase constitutes about 40% by mass, residing mainly in the inter-lamellar region while a very small fraction at inter-spherulite or boundaries. This feature accounts for the inherent brittleness of PHB.

The morphologies observed in our investigations of PHB in blend with degraded PHB and with PHBV[2], and poly(ϵ -caprolactone), PCL, in blend with poly(tetramethylene succinate), PTMS[3], are given here. The other blends studied were PHB with poly(ethylene oxide), PEO[4] and with PCL[5], and poly(ethylene terephthalate), PET, with PTMS[3]. The morphologies may be classified as follows:

- a. Miscible PHB/deg-PHB: *co-crystallization of PHB and deg-PHB in a homogeneous amorphous phase*. See Fig. 5.
- b. Miscible PHB/PHBV: *insertion (random or block) crystallization of*

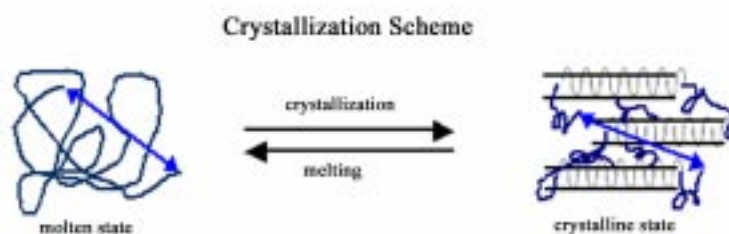


Fig. 1 : Crystallization of a single chain molecule

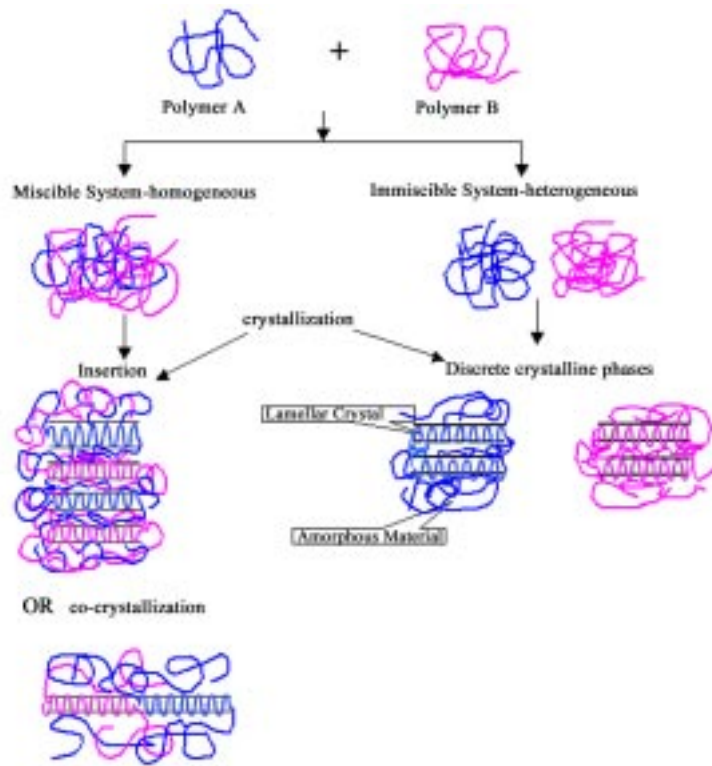


Fig. 2: Crystallization in polymer blends

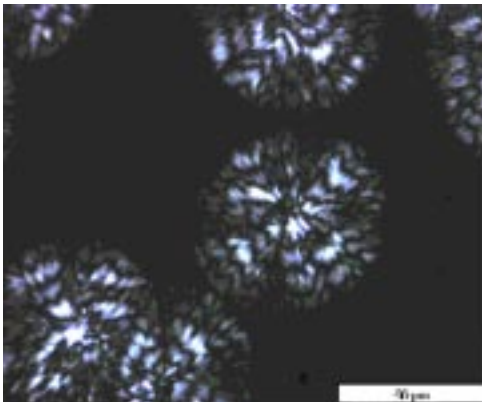


Fig. 3: Growing neat PHB spherulites isothermal crystallization at 130 °C [2]

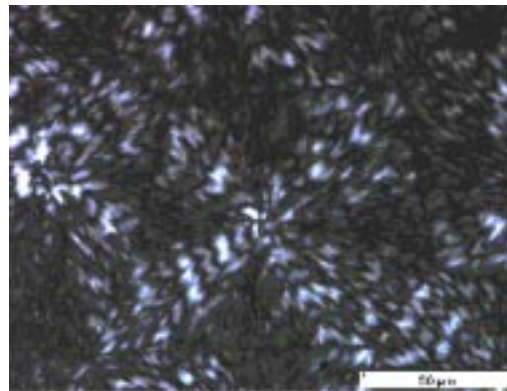


Fig. 4: Neat PHB isothermally crystallized at 130 °C [2]

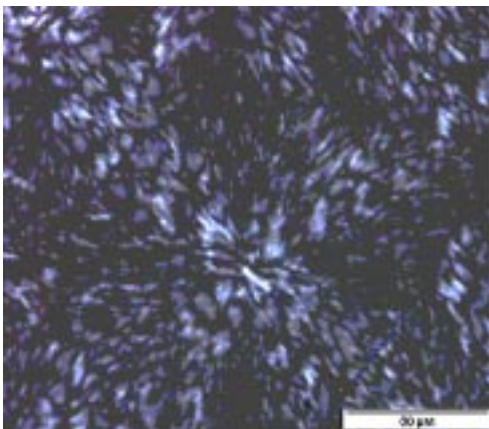


Fig. 5: PHB in blend with degraded PHB; in 80/20 PHB/deg-PHB blend isothermally crystallized at 190 °C [2]

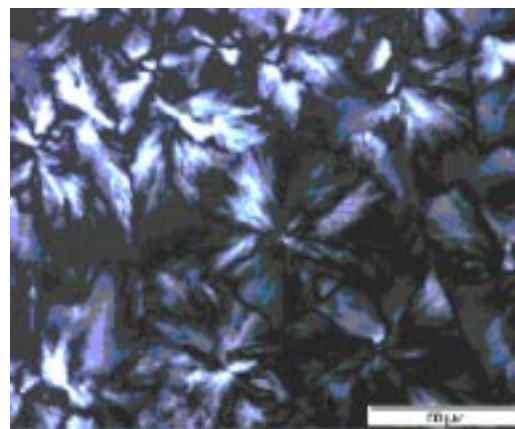


Fig. 6: PHB in blend with PHBV; in 80/20 PHB/PHBV blend isothermally crystallized at 190 °C [2]

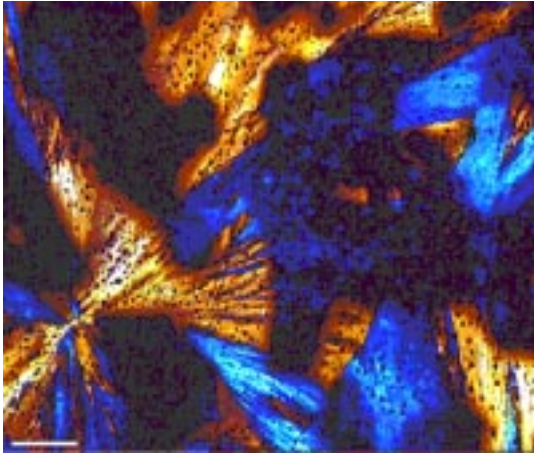


Fig. 7(a): PTMS spherulites isothermally crystallized at 90 °C in amorphous PCL melt in 50/50 PTMS/PCL blend [3]

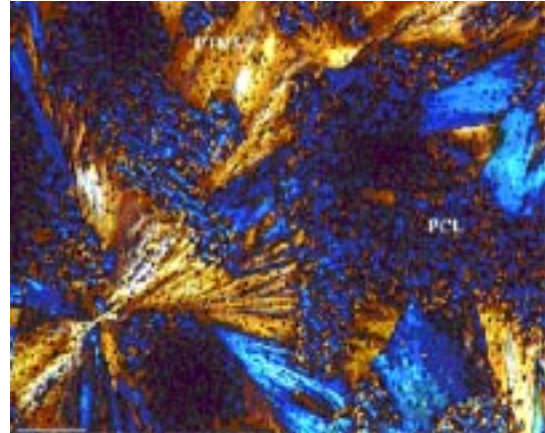


Fig. 7(b): Discrete PTMS and PCL spherulites after isothermally crystallized PCL at 40 °C in 50/50 PTMS/PCL blend [3]

PHB and PHBV in a homogeneous amorphous phase of PHB and PHBV. See Fig. 6.

- c. Immiscible PTMS/PCL: *discrete crystalline phases of PTMS and PCL in their respective discrete amorphous phases.* See Fig. 7(a) and (b).

Summary

The above features give us an idea about the extent of complexity of a crystallization process and the resultant morphology in polymer blends. The thermodynamics and kinetics interpretation based on Flory-Huggins theory, Avrami equation and linear growth becomes obviously important. One therefore needs information from other studies such as kinetics of crystallization, melting, crystallization, glass transition, and the phase behaviour of these blends to substantiate any inferences or recommendations on the prospect of biodegradable polymer blends based on PHB. In the meantime, this issue continues to motivate us.

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Synthesis of Organic-Inorganic Hybrids by Sol-gel

Assoc. Prof. Ismail Ab Rahman



The syntheses and design of silica-based materials by sol-gel chemistry have grown a lot in the last few decades. The sol-gel chemistry consists of hydrolysis of metal alkoxide, $M(OR)_n$, precursors and subsequent condensation leading to the formation of metal oxopolymers. In the interest of producing silica glass and glass-ceramic composites based on the sol-gel process, the alkoxides such as tetraethylorthosilicate (TEOS), and tetramethylorthosilicate (TMOS) are widely used.

Condensation leads to the formation of silica matrix the characteristics of which are highly dependent on reaction conditions such as alkoxide/water ratio, solvents, pH and reacting media. The process can be conveniently carried out at ambient temperature that allows incorporation of organic molecules inside the silica matrix. This incorporation leads to the formation of a new composite known as organic-inorganic hybrid.

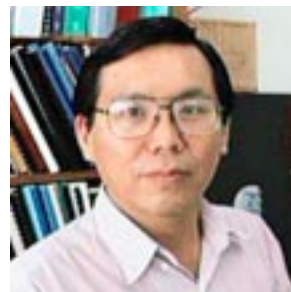
Generally, three types of interaction between incorporated organic compounds with host matrix were hypothesized. Type I deals with hybrid materials exhibiting van der

Waals forces or hydrogen bonding between organic and inorganic materials. The organic compound is dispersed in the host matrix. Type II consists of covalently bonded organic and inorganic parts resulting in the formation of homogeneous hybrid materials. Type III is hybrid material with surface modification by grafted organic group. However, detailed study on the actual interactions has not been reported. The interactions at molecular level that govern mobility and chemical reactivity of guest compounds are also poorly understood.

The present project requires the expertise of both the organic and material science groups to enable the production of a new generation of organic-inorganic hybrids. The targets of our research group are: (1) To identify the types of bonding between incorporated organic molecules and silica matrix in the system studied. (2) To correlate the relationship between mobility and reactivity of the incorporated organic molecules to the physical and chemical properties of the hybrid and (3) To produce a new hybrid that can serve as intelligent sensors or membranes.

Increase of Adsorption Capacity of Adsorbents Through Bioregeneration

Prof. Lim Poh Eng



Large quantities of rice husk are generated in Malaysia as agricultural wastes and the proper disposal of them always poses a problem. There is some urgency in searching for a better way of reutilizing the rice husk since the traditional way of burning the rice husk openly is no longer environmentally acceptable. In view of the relatively high cost of powdered activated carbon (PAC), the potential of rice husk as an alternative adsorbent to PAC has generated a lot of interest to date. Chemical modification of rice husk is normally necessary to increase the adsorbability of the materials. These include pyrolysis, acid or alkali activation and amination.

Raw rice husk was activated by pyrolysis at 600 °C for 5 hours in a nitrogen atmosphere. Using the Langmuir model, the limiting adsorption capacities of the activated rice husk (PRH) for phenol, *p*-methylphenol, *p*-ethylphenol and *p*-isopropylphenol were found to be 12, 8.1, 8.5 and 5.1 mg/g, respectively, which vary from 0.015 to 0.05 of those of PAC. Based on the equilibrium adsorption data, the potential of PRH to be

used as an alternative adsorbent to PAC in treatment process does not look promising. However, in treatment studies using simultaneous adsorption and biodegradation processes under sequencing batch reactor (SBR) operation, equivalent treatment performance was achieved with the use of PRH at dosages of only 2 to 3 times higher than those of PAC for all the phenolic wastewater studied.

The important role of bioregeneration process in renewing the adsorbent surface for further adsorption of organics during simultaneous adsorption and biodegradation processes has been well recognized. A possible explanation for the observed increase in the adsorptive capacity of PRH relative to that of PAC during the simultaneous treatment processes is that desorption of phenolic compounds from the PRH was proceeding at a faster rate than from the PAC prior to biodegradation in the bulk solution. This implies that the surface of PRH was being renewed at a much faster rate than that of PAC thus offsetting the relatively much lower adsorptive capacity of PRH as estimated from the Langmuir model.

Turning Oil Palm Lignocellulosic Wastes Into Lignin: A Mutual Benefit Solution

Dr. Mohamad Nasir Mohamad Ibrahim



A part being a major waste material in the palm oil manufacture, frond of oil palm also represents an important and cheap source of lignin left in the palm oil mill. Besides frond, large volume of sulphite liquor, which contains lignin, are discarded as wastes in pulping industry. This means that not only the lignin is lost but also rivers and lakes are contaminated. Therefore, there is a need to recycle these waste materials into a more valuable product, namely lignin.

Next to cellulose, lignin is probably the most common organic compound cycled on earth. Most plant tissues contain, in addition to carbohydrates and extractives, an amorphous polymeric (high macromolecular) material called lignin. Lignin is a phenolic polymer built up by oxidative coupling of three major C_6-C_3 (phenylpropanoid) units from enzymatic dehydrogenation followed by radical linker. They are guaiacyl alcohol (G), syringyl alcohol (S) and p-coumaryl alcohol (H).

These three types of phenylpropane are considered as a precursor mixture in different ratio, which are trans-p-coumaryl alcohol (1), trans-coniferyl alcohol (2) and trans-sinapyl alcohol (3).

Lignins can be divided into three groups or classes depending on the ratio of all the three phenylpropane units, that is, type N lignins or Guaiacyl lignins, type L lignins or Guaiacyl-syringyl lignins and Hydroxyphenyl lignins. Owing to the high macromolecular complexity of lignin, it is difficult to find a single technique to characterize their structures. Therefore, the most precise way to study this macromolecule is by using a combination of several destructive and non-destructive techniques, each providing partial but complementary information. The chemistry structure and lignin reactivity are not fully developed because all the separated lignin has gone through changes in its structure compared to the original lignin.

Lignin can be found in various usages such as dispersants for carbon black, emulsifiers for latex, metal sequestrates for industrial water, additives for drilling mud and many other applications. Although many usages of lignin have been found, it is also a known fact that research on lignin applications in certain field has not been progressed to a high achievement level.

In the pulping industry using oil palm empty fruit bunch (EFB) fibres, black liquor (waste from prepared kraft pulping) is considered as a waste and can pose a disposal problem due to its sulphite content. The main objective of this study is to transform the oil palm lignocellulosic wastes into a more valuable product, which is lignin.

Modified insoluble lignin and kraft lignin were prepared from fronds of oil palm lignocellulosic wastes and black liquor, respectively. For the insoluble lignin, the fronds were pre-extracted with cold and hot water before solvent extraction with ethanol-toluene and isolation with various concentrations of sulfuric acid were carried out. The kraft lignin was prepared by acidifying the hot black liquor until pH 2. Infrared (IR) and X-ray spectroscopic techniques were used to identify the frequencies of functional groups and linkage

types. The purity of the lignin obtained was determined from its ash content. Each of the isolated lignin showed less than 2% of ash content indicating that the purity of the isolated lignin was under control.

The yield of lignin depends on the concentration of H_2SO_4 . The study suggested that the extraction of lignin from oil palm frond using 70% H_2SO_4 was more effective than using 66% H_2SO_4 even though the yield of lignin obtained using the stronger acid was slightly lower than the latter, namely 20.39% vis-à-vis 21.55%. The extraction process needed a longer period of time to complete when 66% H_2SO_4 was used. Some further works still need to be done in order to increase the yield of the lignin.

If this work is transformed into a larger scale, it is believed that the lignin manufacturers as well as palm oil manufacturers will get the most benefit. This is because the results suggested that the lignin obtained from the oil palm fronds has a high degree of purity and has the potential for commercialization. Moreover, this work will inevitably lead to an increase in add-on value for oil palm lignocellulosic waste and at the same time reduce the pollution problem caused by this waste.

Molecular Modeling Study of the Corrosion Inhibition Properties of Ferric Tannates



Dr. Rohana binti Adnan

Molecular modeling involving the use of computers in studying chemical problems is an exciting and rapidly expanding field. Modeling techniques are finding their use in many diverse areas, from fundamental studies of molecular properties and their reactions to the design of new drugs and materials. There is much confusion over the meaning of the terms “molecular modeling”, “computational chemistry”, “quantum chemistry” or ‘theoretical chemistry’. Although they do not necessarily mean the same, they are in general related to each other. The more general meaning of molecular modeling is “a collection (computer based) of techniques for deriving, representing and manipulating the structures and those properties that are dependent on these three dimensional structures”[1].

Since Vosta and Eliasek [2] studied the corrosion inhibition efficiency from quantum chemistry aspects, the studies of this field have made considerable advancement. For instance, quantum

chemical approaches have been employed in the study of the electrochemical adsorption of organic inhibitors in order to obtain reliable information on the geometry and charge distribution of the inhibitor molecules [3,4]. More recently, Xiao Ci et al. [5] reported the inhibition mechanism and the model of the pyridine derivatives adsorption on the Al-surface by means of quantum chemical method.

The inhibition of the electrochemical corrosion of steel, undeniably, is of great scientific and economic interest. The non-toxic and biodegradable nature of tannin, on the other hand, makes the investigation of its inhibiting properties significant in the context of current priority to produce “green” or “environmentally friendly” inhibitor. Moreover, the abundant source of tannin in Malaysia has made it more attractive alternative to the present corrosion inhibitors, such as chromates.

Tannins are polyphenols (Fig. 1). The *o*-dihydroxy phenolic groups of the phenyl ring in typical flavanoid units of tannin

are capable of chelating with ferric ions giving *mono-*, *bis-* and *tris-*complexes of ferric tannate. Investigation of the inhibitive action of tannin showed a formation of passivating layer of ferric tannates on the metallic surface, which increased the steel resistance to corrosion [6]. Despite the tremendous work in this field, the controversy over the structure of the complex(es) formed and the inhibitory mechanism of ferric tannates remain [7].

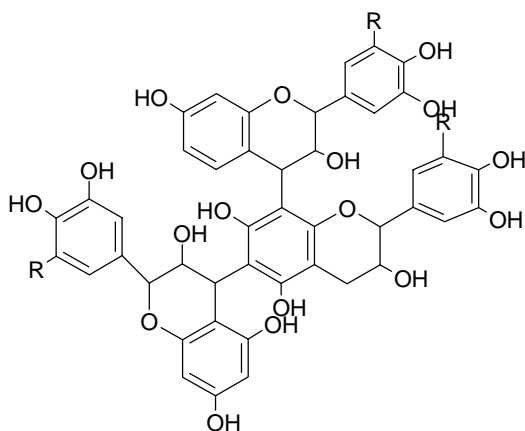


Fig.1 Polymeric structure of tannin
(R= OH or H)

The objective of the present work is to use molecular modeling approach to study the inhibition mechanism and the efficiency of ferric tannate complexes on Fe-surface in both aqueous and acidic media. Theoretically, we would like to study the relationship between the structure of the complex(es) and its quantum properties and relate these properties to the corrosion inhibition effects of ferric tannate.

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UNEP/GEF : Regionally- Based Assessment of Persistent Toxic Substances

Assoc. Prof. Md. Sani Ibrahim



This is a project initiated by United Nation Environment Programme (UNEP) chemical division through funding by the Global Environment Facility (GEF). The School of Chemical Sciences, Universiti Sains Malaysia has a direct involvement in this project through the author who is currently one of the regional team members for the Southeast Asia and South Pacific regions responsible for the collection of information, assessment and prioritization of chemicals considered to be toxic and persistent in nature. The objective of this project is to produce a global report consisting of a compilation of regional reports on the sources, environmental levels, human and toxicological effects and also the transport or transboundary movement of prioritized persistent toxic substances (PTS). The global report is expected to give a clear picture on the current status of PTS which are generally persistent organic pollutants (POPs) throughout the world. The project was commissioned in 2001 and is expected to be completed by the end of 2002.

Persistent toxic substances are chemicals that are toxic to human causing adverse health effects including cancer, reproductive disorders, and developmental deformities.

They are normally persistent in nature with half-lives ranging from 2 months to more than 10 years. Most of these PTS are organochlorine pesticides (OCPs) such as DDT which are highly lipophilic. They would accumulate in the fatty tissues of human and animals producing bioaccumulation effect which results in bioamplification through the food chain. Human at the top of the food chain would be greatly affected with high levels of these PTS found in human blood, adipose tissue and breast milk. These compounds have been released to the environment as pesticides for agricultural crops protection as well as the control of vector such as malaria mosquitoes as early as 1920's. Some of these POPs have been banned by most countries since the early 70's but they are still found in nature due to their high persistency. DDT, for example, has a half-life of between 10-15 years such that it takes more than 50 years to remove 95% of the pesticide from the environment. The problem was aggravated by the fact that some of the degradation products were more toxic and more persistent. DDT, for example, will degrade to DDD and DDE which are more toxic than DDT itself.

UNEP had gathered scientists and experts throughout the world to address these problems through symposiums, workshops and formation of steering committees to look at different issues of PTS such as the socio-economic and environmental impacts. This culminates into the Stockholm Convention with a resolution to reduce and eliminate an initial 12 compounds which pose the greatest threat to human health and the environment. These so-called “dirty dozen” include mostly OCPs such as aldrin, chlordane, DDT, dieldrin, endrin, heptachlor, mirex, toxaphene, and hexachlorobenzene as well as polychlorinated biphenyls (PCBs) and also two industrial by-products, namely dibenzodioxins and dibenzofuran. These notorious compounds had been identified to be of global concern as they may also be transported through air, water and other media to the remote parts of the world such as the Arctic and Antarctica. Other countries have reported the presence of these chemicals in their environment even though they were never imported into or used in these countries. In view of these findings, several agencies including UNEP have initiated projects to study the environmental impact of these chemicals. One of the projects is this UNEP/GEF project on Regionally-Based Assessment of Persistent Toxic Substances.

Briefly, the project involves the assessment of PTS on a regional basis with the world being arbitrarily divided into 12 regions. The project is headed by the project manager, Mr. Paul

Whyllie, based in Geneva and each region has a regional coordinator and between 3-6 regional team members. Malaysia falls into Group VIII which also includes all the Southeast Asian Countries plus South Pacific Countries such as Australia and New Zealand. The coordinator of Region VIII is Dr. Gil Jacinto from Philippines and 3 regional team members are the author from Malaysia, Prof. Des Connell from Australia and Mr. Lim Kew Leong from Singapore. The main activities of the project are to collect available data that had been reported on sources, concentration levels, human effects, ecotoxicological effects and transboundary migration of PTS. Three workshops will be organized by inviting country experts in the region to present the status report of PTS in their respective countries, deliberate on the current regional issues of PTS, identify data gaps (lack of data) and set priority for the region with respect to the management of PTS.

Data on PTS were collected by soliciting experts and other interested parties to fill in a number of questionnaires specially designed for the project. Informations were obtained mainly from published sources such as journals and conference proceedings as well as available unpublished reports. The questionnaires may be filled in directly on the web at [HTTP://www.chem.unep.ch/pts](http://www.chem.unep.ch/pts) or on printed forms available from the project manager (pwhyllie@unep.ch) or the author. Even though the project is almost

ending, we are still collecting data to fill in any data gaps for the region. The regional reports will be finalized by September 2002 before being submitted to the project manager who would then compile reports from the 12 regions to be submitted to the UNEP-Chemical. This global report will be made available after 2002.

Currently, we have collected a significantly large amount of data on the levels of PTS in the environmental media (air, water, sediment and soil) and biota (aquatic and terrestrial) from the more developed countries such as Australia, New Zealand, Malaysia, Singapore, Philippines and Thailand but other developing countries such as Vietnam, Cambodia and Laos have very limited information on PTS or POPs. Sources of PTS are difficult to identify as most countries either do not have inventories of PTS or would not be able to reveal the information. There are significant data gaps on human health and ecotoxicological effects of PTS as most of the developing countries do not have the capability to undertake such studies. We realize that very little work have been done on the transport and transboundary migration of PTS in this region. To rectify this situation, we are proposing to conduct a transport modeling study of several important PTS into, out-of and within this region.

At the time of this write-up, two workshops for Region VIII, were conducted in which representatives from each country in the region were invited. The first workshop, in which sources and environmental levels of PTS in the region were discussed, was held in Singapore in February 2002. The second workshop took place in Penang, Malaysia in April 2002 during which human and ecotoxicological effects as well as transport studies conducted in Australia were deliberated. From the two workshops, it may be concluded that there are no hot spots in the region except probably in Vietnam from 2,4-D and 2,4,5-T and thus dioxins remaining from the Vietnam War (circa 1970's) but more studies need to be carried out. Most of the OCPs are decreasing in concentrations as predicted. PCBs would be a major threat if no action is taken to supervise their proper disposal from old transformers and capacitors in developing countries. However, most country experts agreed that dioxins and furans would be the major PTS issue. Dioxins and furans are not intentionally produced. They are released to the environment from industrial processes such as pulp and paper milling, and waste treatment facilities such as incinerators. It was concluded that more concerted effort should be made both regionally and globally to reduce and eliminate PTS to ensure a cleaner and healthier environment for the future.

The Beauty of Liquid Crystals

Dr. Yeap Guan Yeow



Thousands of compounds, particularly the organic compounds and very recently some metal-containing materials, have shown mesomorphic properties. The term mesomorphism refers to the intermediate states wherein the molecules, aggregates of molecules, or macromolecules self-organize into phases in which orientational order and sometimes positional order in one or two dimensions persist over macroscopic distances. The properties of these molecules or liquid crystals rely on the chemical properties of the constituents as well as the structure of the mesophases [1-3].

One of the related researches currently being undertaken in the School of Chemical Sciences focuses mainly on the synthesis and characterization of novel or modified materials which are thermotropic in nature and show different mesophases due to the temperature change: heating or cooling.

This project which was aimed at generating liquid crystalline materials with enhanced mesomorphic behaviours was started in 1996. Since this kind of work is also widely pursued abroad, the commencement of this project has further enhanced the inter-institutional collaboration and sharing of knowledge between USM and other research institutions both within and outside Malaysia.

At present, a few projects have been targeted at obtaining liquid crystals from conventional chemical reactions, modification of existing substances, natural products especially those substances related to oil-palm, the newly synthesized monomers and the formation of polymeric liquid crystals.

One of the typical liquid crystals which has attracted considerable attention in the present laboratory is the one which exists in smectic system. Actually, smectic phases are lamellar in nature. Many structural differences of the smectic phase are known, depending upon how the rod-like molecules are arranged in the smectic lamellae, the tilt angle of the rods with respect to the lamellar planes, and the degree of correlation of structure from layer to layer [2,3].

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Nuclear Magnetic Resonance

Dr. Shafida Abdul Hamid and
Dr. Yeap Guan Yeow



Nuclear Magnetic Resonance (NMR) spectroscopy is one of the most useful tools to study the physical, chemical and biological properties of matter. As a result, it finds many applications in several areas of science. Chemists routinely use NMR spectroscopy to study chemical structure using simple 1D techniques such as ^1H and ^{13}C , while 2D techniques are used to determine more complicated molecular structure such as in proteins and other polymeric compounds. The 2D spectroscopy consists of homonuclear [^1H , ^1H] and heteronuclear [^{13}C , ^1H] correlation techniques.

The homonuclear 2D spectroscopy includes:

- (i) COSY (Correlation Spectroscopy), used to correlate the chemical shifts of ^1H nuclei, which are J-coupled to one another.
- (ii) TOCSY (Total Correlation Spectroscopy), which provides a different mechanism of coherence transfer in comparison with COSY. The cross peaks are generated between all members of a coupled spin network. The advantage is that,

it can identify the separated spin system.

- (iii) ROESY (Rotating-frame Overhauser Effects Spectroscopy) and NOESY (Nuclear Overhauser Effect Spectroscopy), the spectra of which will indicate which protons are close to each other in space.

The heteronuclear 2D spectroscopy includes:

- (i) XHCCORR [(H,X) Shift Correlation) spectroscopy, a technique that can be used to determine which ^1H of a molecule is bonded to which ^{13}C nuclei.
- (ii) HMQC (Heteronuclear Multiple Quantum Correlation) spectroscopy, which is an inverse chemical shift correlation experiment that yields exactly the same information as XHCCORR. The advantage of HMQC is that it has a higher sensitivity.
- (iii) HMBC (Heteronuclear Multiple Bond Correlation) spectroscopy, a modified version of HMQC suitable for

determining long-range ^1H - ^{13}C connectivities.

These are some of the main techniques employed in NMR spectroscopy. In actual fact, there are many other applications of NMR such as solvent suppression and gradient enhanced spectroscopy, which can help us further in determining the molecular structure.

The School of Chemical Sciences, Universiti Sains Malaysia maintains and operates two high resolution liquid FT-NMR spectrometers

(AVANCE 300 MHz and 400 MHz Bruker Spectrometers) that are capable of performing multinuclei NMR experiments. Services are provided mainly to the researchers within and outside USM. Research workers are welcomed to discuss with the School regarding the NMR experiments that they want to perform.



Multinuclei 400 MHz Bruker NMR Spectrometer

MAKMAL UNIT PERKHIMATAN ANALISIS (MUPA) **(Analytical Services Unit)**

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Advisory Panels:

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MUPA is a unit under the School of Chemical Sciences that offers analytical services to both its own staff and outside clients. The unit provides services in the form of routine analysis as well as non-routine and problem-solving type of contract research for specific industrial projects. All analysis will be based on internationally accepted standard methods or in-house methods that have been validated. A Working Committee was set up in 1998 for the preparation of MUPA Policy Manual and Standard Operating Procedures. A one-day strategic planning workshop was held on the 25th August 2001 at the Hotel Vistana to discuss and offer recommendations on the

major policies as well as to outline future directions of the MUPA set-up. Associate Professor Bahruddin Saad was formally elected as the Laboratory Director on the 3rd October 2001.

MUPA is equipped with the state-of-the-art instruments, the major ones including FTNMR (300 and 400 MHz), ICP-MS, GC-MS, GFAAS and HPLC. With the establishment of MUPA, it is hoped that consulting and testing activities of the School will become more prominent.

For Analytical Services and Consultation,
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Grafite Furnace Atomic Absorption Spectrometer (GFAAS)



The New GC-MSD



State of the art ICP-MS

School of Chemical Sciences, USM- Highlights of the First Half of 2002

Academic visitors and speakers

- 1) Mr. Peter Sprenger (Bruker, Bangkok) gave a seminar entitled "Introduction, applications and principles of FT-NMR" on 29 Jan 2002.
- 2) Dr. Thierry Sevenet of Pharmacie-A.I.H.P., Directeur de Recherche I CNRS, ICSN/CNRS gave a seminar entitled "Cytotoxic and antimetabolic compounds from tropical plants: classical methods, new strategies" on 25 June 2002.

Sanggar Sanjung 2001

The university has conferred the award of "Sanggar Sanjung" to the following academic staff from the School and their research groups in recognition of their significant contributions in research and development:

- 1) The Dean, Assoc. Prof. Jamil Ismail as a member of the Bioplastics research group,
- 2) Prof. Lim Poh Eng,
- 3) Assoc. Prof. Mas Rosemal Hakim Mas Haris and his research group,
- 4) Assoc. Prof. Mohd. Asri Mohd. Nawi and his research group.

Family Day (Hari Keluarga)

This year's family day was held on the 4th. June 2002 at Dataran Kimia. It is an annual event to enhance a good relationship among the staff and the family members. Many activities were carried out such as games, treasure hunt, drawing and colouring for the children. Dinner was served with a sumptuous spread of food and lucky numbers were drawn.

International Awards

In the 30th. International Exhibition of Inventions, New Techniques and Products held in Geneva, May 2002, two of the academic staff from the School shared the honour of winning gold medals with their respective research group;

The Dean, Assoc. Prof. Jamil Ismail is among the researchers of the Bioplastics research group which has won a gold medal for the production of liquid bioplastics, PHA (polyhydroxyalkanoate) from palm oil.

Associate Prof. Mas Rosemal Hakim Mas Haris heads the research group which has won a gold medal for developing a method to prepare a water soluble rubber as flocculant in aqueous media in a novel application.

New Academic Session 2002/2003

A total of 18 new students have registered to pursue the Master Programme in Chemical Instrumentation (mixed mode). The total number of students pursuing postgraduate degrees in chemistry by research stands at 104 of which 20 of them are foreign students from Bangladesh, Indonesia, Iran, Iraq, Myanmar, Nigeria, Sudan, Thailand and Yemen.

Second Workshop on Regionally Based Assessment of Persistent Toxic Substances.

A regional workshop on Persistent Toxic Substances was held in one of the beach hotel in Batu Feringghi from 16 - 19 April 2002. This UNEP funded workshop was attended by 18 participants from several Southeast Asian Countries and Australia. Discussions were focussed on the impact of persistent toxic substances namely organochlorine pesticides on human and the environment particularly within this Asean Region.



Dr. Beth Yam (an alumna) from Intel Technology Sdn. Bhd. giving her presentation during the Chemistry Alumni seminar.



Welcoming the new chemistry undergraduates of 2002



Participants of the Regionally Based Assessment of Persistent Toxic Substances Workshop

SKAM-15/EXPERTS 2002



Simposium Kimia Analisis Malaysia Ke 15 dan Ekspo Peralatan Sains Serantau 2002

10-12 September 2002
Bayview Beach Resort, Pulau Pinang

Anjuran Bersama:



*Universiti Sains Malaysia
Persatuan Sains Analisis Malaysia
Kerajaan Negeri Pulau Pinang
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