



**BETTER (AND HAPPY) LIFE  
THROUGH HETEROGENEOUS  
CATALYSIS RESEARCH**

Professorial Inaugural Lecture Series

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PROFESSORIAL INAUGURAL LECTURE SERIES

19 October 2011

*by*

Professor Dr. Hadi Nur

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Professor Dr. Hadi Nur

## **BETTER (AND HAPPY) LIFE THROUGH HETEROGENEOUS CATALYSIS RESEARCH**

The purpose of this lecture is to introduce several designs of heterogeneous catalytic systems. The aim is to assist researchers in the field of catalytic chemistry in gaining an understanding of the catalytic phenomena through the design and physicochemical properties of the catalysts. This is an inaugural professorial lecture which will be useful for several purposes. The cohesive aspect of certain advances is shown by bringing together researches published in different journals, including, in a few cases, journals of limited circulation. This should aid historical studies of heterogeneous catalysis researches at Universiti Teknologi Malaysia (UTM). It will give the reader a feeling of the general nature of research. This lecture was compiled based on classification from the catalytic processes that comprised of examples from researches that were carried out by the author together with his colleagues and students from 1996 to 2011 at UTM. Another message to be conveyed in this lecture is that scientific research is enjoyable. Hopefully, this lecture can give an inspiration on how the design of the catalyst can be related to the physicochemical properties and the catalytic action for the chemical reactions, and may assist in the further search for novel approaches to heterogeneous catalysis. Developing programmable systems and self-organizational approaches in heterogeneous catalysis are among of the most challenging design tasks for current heterogeneous catalysis.

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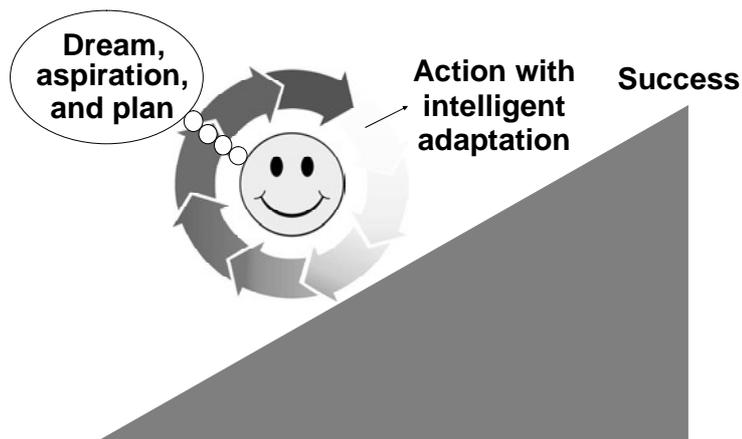
**INTRODUCTION**

Every person has his or her own dream and therefore, is immersed in a journey towards the accomplishment of his or her dream. Twenty-four years ago, when I entered the university, I dreamed to become a professor by writing the title of Prof. Dr. in front of my name on the front page of laboratory manual. Sixteen years ago I came to UTM as a Ph.D. chemistry student, and two and a half years later, I was entitled to put the title of Dr. in front of my name. Now, I deserve to put the title of Professor. The dream comes true. The laboratory manual is now a witness to my dream.

Goal setting is an important part of career planning and needs to be set in every process, so we have a commitment to achieve great results. This needs to be done with optimism and joy, in which is referred to as the joy of goal setting. Likewise, this means that it is more fun to chase the target, rather than being chased.

The message is clear that everyone should have ambitions; hence, we should determine the direction of our life. There may be people who do not take care about ambitions, but someday that person must make decisions to determine their future. So, everyone is required to understand his or her strengths and weaknesses, including his or her ability to generate enthusiasm and motivation. This is what should be sought by every student on campus, what is referred to as self-discovery. Everyone should have goals, even though Allah SWT knows what is best for His servants.

In addition to goal setting and the joy of goal setting, in my opinion, another factor that



**Figure 1** The way to achieve success with joy of goal setting and intelligent adaptation.

determines a person's success is the ability to adapt. This principle must be applied if you want to survive. Something that we think is right, maybe not necessarily right for others. Intelligent adaptation is maybe the right term to describe this situation. The goal setting circle with happiness shown in Figure 1 can be used as driving force to achieve a success.

I believe that *goal setting*, *the joy of goal setting* and *intelligent adaptation* are essential in our life. Based on these principles, *Alhamdulillah* (all praise is due to Allah), I am now a full professor and standing in front all of you to deliver my inaugural professorial lecture.

## SCIENTIFIC JOURNEY

I grew up in a university campus in West Sumatra, Indonesia. My father, Nur Anas Djamil, was a Professor in Islamology at Institute of Teacher Training and Education (now State University of Padang) in Padang. I first became interested in chemistry one day in 1984 when I was a senior high school student. I attended an additional course in chemistry subject. This course was taught by Drs. Tahasnim Tamin, a senior lecturer at Institute of Teacher Training and Education in Padang. I was interested with the beautiful concept of chemistry that he was taught with laboratory demonstrations. One of the experiments conducted in the classroom was the reaction between sodium with water where a very small piece of sodium was dropped into water inside a beaker. The violent reaction of sodium with water caused sodium to ignite on the surface of the water. Amazing reaction! This is one of the examples how the chemistry experiment leads the students become interested in science. This lesson also of course could not be learned without a dedicated teacher that one encounters along the way.

The next important lesson came as I became an undergraduate in chemistry at the Institut Teknologi Bandung, Indonesia. In my final year project, I had chosen to do research for my final project with Dr. Harjoto Djojotubroto, Director of Nuclear Engineering Research Center, National Nuclear Energy Agency, in Bandung. He was interested in Neutron Activation Analysis (NAA) and had received his Ph.D. in hot atom chemistry. We planned a comparative study on the determination of selenium in human blood by neutron activation analysis which took me the next two years to do. He also inspired me to pursue postgraduate studies. The most important event happened at this time was the meeting with Terry Terikoh, the woman, who became my wife. My goals in academia always get support from Terry, and this is the biggest factor that leads to the success I have had in my professional career.

In September of 1993, I enrolled a new master program in materials science and engineering at Institut Teknologi Bandung and was fortunate enough to work with Dr. Bambang Ariwahjoedi who was a creative lecturer and had many great ideas. Creativity was very important at that time due to lack of scientific instruments in our department. I completed a project in the topic of synthesis of hydroxylapatite bioceramics by means of precipitation and its characterization. I received M.Eng. with *cum laude* in 1995.

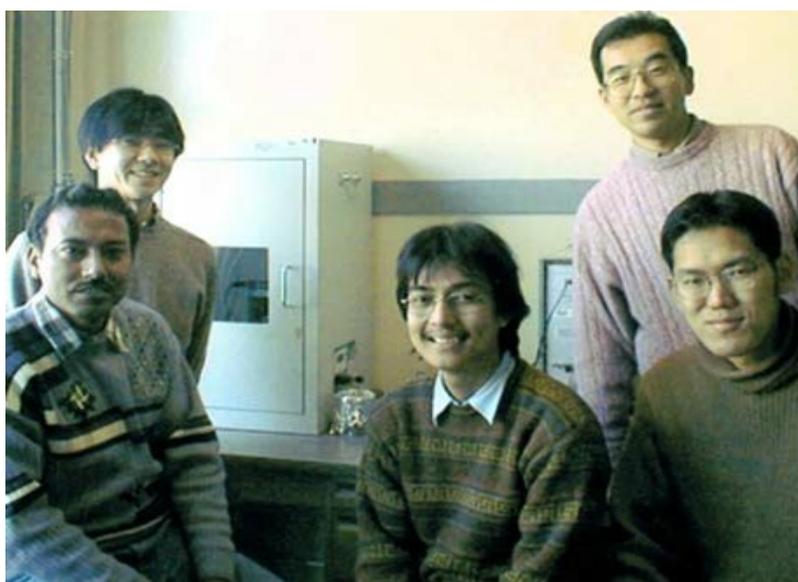
Having batted zero for my professional career up to this point, I was ready for Ph.D. research. I continued my postgraduate studies in zeolite chemistry at Universiti Teknologi Malaysia under supervision of Prof. Halimatun Hamdan. I tackled a project on the synthesis, characterization and catalytic activity of ultra-large pore of aluminophosphate molecular sieves, VPI-5, just recently successfully synthesized by Prof. Mark Davis and his co-workers [1]. After six months experimental work, I failed to synthesize VPI-5. Everything in my thesis comes from the last twelve months in the laboratory. Nonetheless, since we had another project on synthesis of zeolite from rice husk ash that both worked at once, I ended up with three international publications together on the study of the structure, physicochemical properties and catalytic activity of metal-substituted  $\text{AlPO}_4\text{-5}$ . I also proposed and successful in synthesizing NaA zeolite directly from rice husk and carbonaceous rice husk ash. This was an incredibly intense twelve months work. I finally found out what independently doing good science. It was an exhilarating period. I finished my Ph.D. project in two and half years in 1998. I continued working as a postdoctoral fellow for one year at Universiti Teknologi Malaysia. Figure 2 shows a photograph of members of Zeolite and Porous Material Research Group led by Prof. Halimatun Hamdan where I was a postdoctoral researcher in this group. I would like to acknowledge Prof. Halimatun who have supported and encouraged me throughout my entire scientific career.

In 1999 I was fortunate enough to land a postdoctoral position with Prof. Bunsho Ohtani at Catalysis Research Center, Hokkaido University, Japan (see Figure 3). Prof. Ohtani has a great contribution to my career in heterogeneous catalysis. Prof. Ohtani is a truly inspirational scientist with good personality. He had endless capacity to produce great ideas. In two years as JSPS (Japan Society for the Promotion of Science) Postdoctoral

Fellow and continued stay as COE (Center of Excellent) visiting researcher at Catalysis Research Center for half year, we published a seminal paper on a new concept in heterogeneous catalysis termed “Phase-boundary catalysis”. I finished my postdoctoral stay, which was due to the supports of Dr. Shigeru Ikeda in the Ohtani group. I will always be indebted to Prof. Ohtani for showing me how to do science in a right way. It is a debt that I can never repay, but showing new researchers the joy of chemical research not to mention a lot of fun.



**Figure 2** Members of Zeolite and Porus Material Research Group led by Prof. Halimatun Hamdan (1998).



**Figure 3** Members of Prof. Bunsho Ohtani's Laboratory, Catalysis Research Center, Hokkaido University, Japan (1999).

In May 2002, for one year, I joined the Ibnu Sina Institute for Fundamental Science Studies, Universiti Teknologi Malaysia as a research officer and in 2003 I accepted my first academic position as lecturer at this university. I am very grateful for having the opportunity to work with helpful and pleasant collaboration, especially Prof. Dr. Halimatun Hamdan, Assoc. Prof. Dr. Zainab Ramli, Prof. Dr. Salasiah Endud and Assoc. Prof. Mohd Nazlan Mohd Muhid. My research ideas on single center catalyst, hydrophobic-hydrophilic catalytic systems, bifunctional catalyst, photocatalyst, multi reaction center catalyst and chiral catalyst were published in international refereed journals. In the future we will examine heterogeneous catalyst designs for other important reactions in organic synthesis.

Lastly, I would like to say thank you to all my teachers and mentors, I am nothing without all of you.

### **SCIENTIFIC RESEARCH IS ENJOYABLE**

Eleven years ago (2000), when I was a postdoctoral fellow in Prof. Bunsho Ohtani laboratory at Hokkaido University, I read the introduction of the book authored by Prof. Akira Fujishima, a very famous scientist in the field of photocatalysis about his view and experience on enjoyable scientific research. Until now, I am very impressed with the sentences written in the introduction of the book. Prof. Fujishima has written in his book as follows:

*“Scientific research is enjoyable. Of course it seems difficult when progress is slow and there are no results of our efforts. Serious mistakes can be made through lack of consideration. However, the joy that comes from making a new, unexpected discovery cannot be compared with any other. It is like climbing along, steep mountain path: as you finally reach the top, the clouds part, and you are greatly moved by the spectacular landscape that appears before you. Furthermore, the suffering of not being able to explain the results of an experiment on the basis of past experience and the joy of finally being able to solve the problem by coming up with a new idea are just like mountain climbing.” [2]*

So, the question is, if you are a scientist, have you seen the beautiful panorama from the top of the mountain which we struggle to climb?

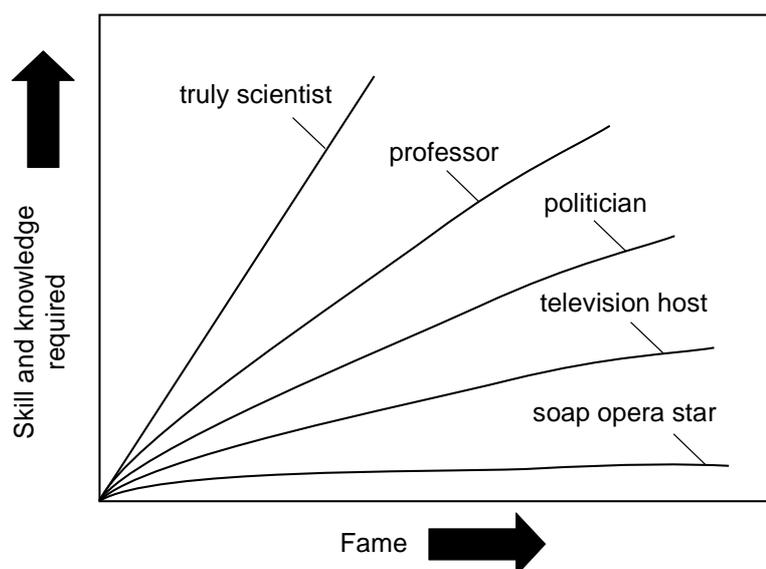
## THE BEAUTY OF A UNIVERSITY AND SCIENTIFIC INTEGRITY

Prof. Richard Feynman, a Physics Nobel laureate once said that, as a scientist, he enjoys the beauty of flowers with more in-depth than an artist. Artist or ordinary people just enjoy and appreciate only the external aesthetic of flower. As a scientist he was more to enjoy the beauty of flowers – not just from the external beauty of flower – but much deeper, such as molecular structure, metabolism, biophysics and the other scientific aspects [3].

Based on the above view, as a lecturer and researcher, I can analogize the university as “the flower of Richard Feynman”. The university is not only viewed from just the physical facilities, but much deeper – namely the process of education and research in it. This process must be based on the scientific culture. Scientific culture would not exist if there is no scientific integrity, and scientific integrity will not be formed if there is no good research practice. There are several principles in good research practices:

- Good research practice requires proper supervision and training.
- Good research practice encourages openness and dissemination of results.
- Good research practice requires proper maintaining and storing of records.
- Good research practice requires high quality outputs and good publication practice.

Based on these considerations, one argues that it is indeed very difficult to be a famous scientist compared with other jobs such as television host and soap opera star (Figure 4).



**Figure 4** It is indeed very difficult to be a famous scientist.

In order to excel in scientific research, the following fundamental premises need to be considered seriously. First, excellence in research is one of the ultimate roots of all academic excellence, in both undergraduate and postgraduate education. Second, in science, no matter how spectacular the results are, the work is not completed until the results are published. These things will not be achieved easily because of fundamental research requires critical mass. It will provide critique exchange of ideas, promotes competition, and foster humility. Thus, one suggests that the research teams are incubators of idea-multipliers. Based on these principles, let's create research superstars at UTM!

## **PHILOSOPHY OF TEACHING**

I once read somewhere, that teaching and research are multiplicative, and not additive, factors in one's stature as a scientist. This means that you are a zero as a scientist if you cannot teach. Of course, it also means that you are a zero as a scientist if you do not do research. While working as postdoctoral researcher, I took every opportunity to teach in informal "training" sessions. I find no greater job satisfaction than in assisting others to learn. My motivation to teach comes from a desire to help others and see students' excitement as they learn new concepts. I believe teaching is my way to make a positive effect to students.

As a lecturer, I recognize that I have a lot to learn about good pedagogy. In fact, I hope to be a lifelong learner on how to be a better lecturer. I began this process by being a participant in "Teaching Certificate for Higher Education" courses at Universiti Teknologi Malaysia in 2004. In my point of view, teaching is finding ways to present the material so as to engage the very best students. But teaching is also finding ways to reach the less talented students. One of the wonderful things about Universiti Teknologi Malaysia is the wonderful diversity of its students. This certainly applies to race and religion.

I believe that students must have an interest to learn a subject well and a primary source for that interest comes from the enthusiasm of the lecturer. I believe that a lecturer's passion for the subject is contagious if it is channeled in the right way. Enthusiasm can provide students the motivation to learn the material. Since I worked at Universiti Teknologi Malaysia as a lecturer, I am supervising or have supervised several postgraduate and undergraduate students. One of my B.Sc. students, graduated with 1<sup>st</sup> class honour. His thesis was awarded as the best thesis of the year 2004 by *Persatuan Sains Analisis Malaysia* (ANALIS). Part of his work was published in prestigious journal in the field. Based on this experience, it is realized that, clearly, teaching is a lot more than the time spend formally standing in front of a classroom.

## RESEARCH IN HETEROGENEOUS CATALYSIS

The following are examples of my researches which were compiled based on classification from the catalytic processes that contained examples from research that was carried out by me together with my colleagues and students from 1996 to 2011. I hope that these researches can give an inspiration for readers how the design of the catalyst can be related to the physicochemical properties and the catalytic action for the chemical reactions, and may assist in the further search for novel approaches to catalysis. Developing programmable systems and self-organizational approaches in catalysis are among of the most challenging design tasks for current catalysis.

The name 'catalysis' was coined by Berzelius in 1836 [4]. Berzelius defined a catalyst as a substance which by its mere presence evokes chemical actions which would not have taken place in its absence. This definition simply describes the observation of the phenomenon, 'catalysis', without making any attempt to interpret or explain its nature. The word was formed from a combination of two Greek words,  $\kappa\alpha\tau\alpha$  (kata) = down and  $\lambda\upsilon\delta\epsilon\iota\nu$  (lysein) = to split or break. According to Berzelius, by "awaking the affinities which are asleep", a catalyst breaks down the normal forces which inhibit the reactions of molecules. In another word, a catalyst is a substance that transforms reactants into products, through an uninterrupted and repeated cycle of elementary steps, until the last step in the cycle regenerates the catalyst in its original form. More simply put, a catalyst is a substance that speeds up a chemical reaction without itself being consumed in the process. Many types of materials can serve as catalysts. These include metals, metal compounds (e.g., metal oxides, sulfides, nitrides), organometallic complexes, and enzymes.

Catalysts are classified as *homogeneous* if they are present in the same phase as the reagents. This normally means that catalysts are present as solutes in a liquid reaction mixture. Catalysts are *heterogeneous* if they are present in a different phase. Heterogeneous catalytic reaction systems, in which fluid reactants are passed over solid catalysts, are at present the most widely used catalytic processes in the manufacturing industries. Solid, heterogeneous catalysts have the advantages of ease of recovery and recycling and readily amenable to continuous processing.

The selectivity and activity of homogeneous catalysts under mild reaction conditions is unbeaten by their heterogeneous counterparts. Unfortunately, the problem of separating the single-site-catalysts from the reaction media is still an important drawback which blocks large scale applications in industry. Only a few processes are applied nowadays in industry, such as the production of adiponitrile by Dupont, acetic acid by Monsanto and butanal by Celanese (former Ruhr Chemie) [5]. In each case an individual solution was developed to solve the problem of catalyst separation and recovery. A general toolbox for this has to be

filled. In Table 1, the advantages and disadvantages of homogeneous versus heterogeneous catalysis are shown. In this way the major problem of homogeneous catalysis becomes obvious.

**Table 1.** Homogeneous versus heterogeneous catalysis.

	Homogeneous	Heterogeneous
Activity	+++	-
Selectivity	+++	+
Catalyst description	++	-
Catalyst recycling	-	+++
Turn over number	+	+++
Quantity of catalyst	++	+++

“Catalysis by chemical design” has been a dream for decades. To specify the composition and structure of matter to affect a desired catalytic transformation with desired and predicted rate and selectivity remains a monumental challenge, especially in heterogeneous catalysis. With the advent of surface science techniques in decades past, the promise was perceived of turning increased molecular level understanding of reaction mechanisms and surface sites into principles of catalyst design. Surface science alone has not proven to be sufficient for this purpose. Over the past decade the rise of powerful, computationally efficient theoretical methods have shown promise, not just for identifying catalytic intermediates and reaction pathways accessible to experiments, but of providing quantitative predictions of energetic for elementary reaction processes not easily accessed experimentally. Much of our work is aimed at the rational design of catalysts for oxidation and acid organic reactions. This chemistry remains one of the most challenging problems in heterogeneous catalysis.

### **BETTER CATALYST THROUGH CHEMICAL DESIGN**

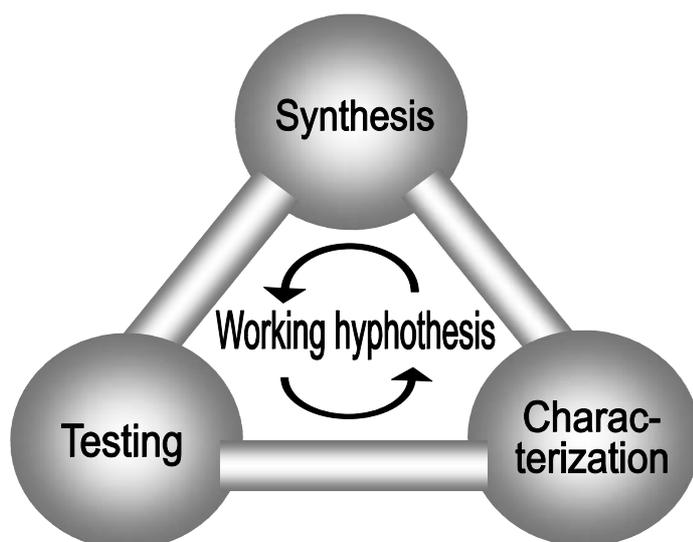
Catalysts operate at a molecular level, so study of their mechanisms falls into the realm of nanotechnology: the science of the extremely small. Most catalytic chemical reactions are heterogeneous – they involve more than one phase. Usually a gas and/or liquid phase passes over a solid catalyst that starts up the reaction – the catalytic converter that cleans

up a car's exhaust gases is a typical example. By contrast, homogeneous catalysis occurs in a single phase, for example the enzyme-modulated reactions that determine the physiology of living organisms.

Our principle research interests lie in the fields of synthesis, characterization and catalytic reaction of heterogeneous catalytic system. The development of heterogeneous catalyst may be regarded as an iterative optimization process, basically consisting of three steps, namely synthesis, characterization and testing as depicted in Figure 5.

The speed at which a progress depends on the quality of the concepts or working hypotheses used to guide this process. Historically, research in catalysis is experimentally oriented, and attempts to make a general organization of information on catalysis would be more of *correlation* than *theory*, although the concepts involved in such correlation rest on theoretical grounds. Nowadays, combination of physicochemical inspections of the catalyst characteristic with theoretical and quantum chemistry, catalyst modeling and mechanistic simulations is the more effective approach towards understanding how the catalyst is prepared (synthesis), actually 'looks' (characterization) and performs (testing). The synthesis of catalyst is divided into three steps: the preparation, reaction and isolation. The preparatory step of the catalyst is the most difficult step because many experimental parameters are involved.

In the area of catalysis it is often difficult to establish the mechanism of a reaction through experiment, even though the reactants, products and catalyst are well characterized.



**Figure 5** Schematic representation of the catalyst development cycle.

## OUR RESEARCHES

A basic feature common to all catalytic systems is that the catalytic reaction can be considered as a reaction cycle, in which catalytically active sites are initially consumed and at the end of the cycle are regenerated. The elementary rate constant for product desorption often competes with the elementary rate constant for reactant activation, leading to the Sabatier volcano curve for overall rate of reaction versus interaction strength of the intermediate reaction complexes with catalytic bonding site. There are many different catalytic systems. Of most basic mechanistic features are well understood. Here an attempt will be made to introduce several catalytic reactions in order to design a better catalyst through chemical design. The catalytic reactions are the oxidation and acid catalysis by heterogeneous catalysts. Our works are classified into six classes:

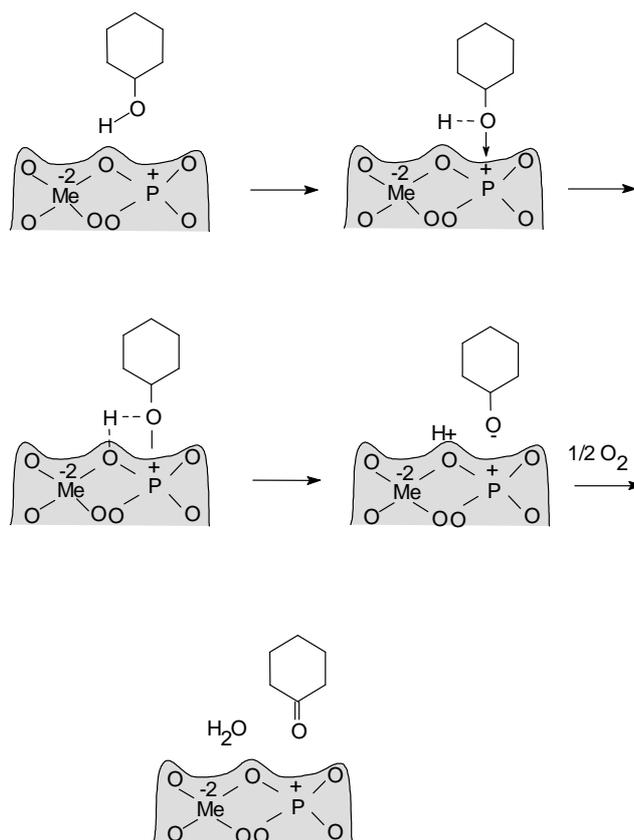
1. Single center catalyst
2. Hydrophobic-hydrophilic catalytic systems
3. Bifunctional catalyst
4. Photocatalyst
5. Multi reaction center catalyst
6. Chiral catalyst

The above classification is inspired by classification of catalytic system proposed by Prof. R. van Santen of Technische Universiteit Eindhoven [6]. The catalyst designs described below comprised of examples from researches that I carried out together with colleagues and students.

### Single center catalyst

Catalytic reactions that one could define as belonging to single center catalyst, can be considered as local events. A single metal center or a cluster of atoms is required for all of the elementary steps to occur. An example of such a catalytic reaction is dehydration and dehydrogenation of cyclohexanol by aluminophosphate molecular sieves [7-11] (see Figure 6). A large part of this works relates to metal-substituted aluminophosphates (MeAPO) molecular sieves. These materials with desired and controllable properties, be adsorptive or catalytic have been successfully synthesized and modified for the specific purposes such as dehydration and dehydrogenation of alcohols reactions. In this research, Al atom in the framework structure has been substituted with the divalent metal (Me) atoms (Me = Mn, Mg, Co and Zn) and silicon atom to generate catalytic sites. It is clearly demonstrated that the conversion of cyclohexanol to cyclohexene (as a model reaction)

involve the Me–O–P and Si–O–P sites in the framework of AlPO. This result also suggests that MeAPOs are potential catalysts for dehydrogenation of alcohols. It was demonstrated that MnAPSO-5 was the most active catalyst for dehydration and dehydrogenation reactions of alcohols. Based on our understanding on the fundamental factors in the catalytic activity of these materials, results of this research can open the innovation in applied catalysis and play role in industrial catalytic processes. This research was carried out in the period of 1996-1998 as my Ph.D. research. The other examples of researches in single center catalyst system which have been developed in our laboratory are metal complexes encapsulated in Al-MCM-41 as catalysts in oxidation reactions [12-13], enhancement of catalytic activity of TS-1 in epoxidation of alkenes [14] and Ti-OMS-2 as catalyst in oxidation of cyclohexene [15, 16], catalysis by zeolite beta [17, 18], modification of surface of titania by attachment of silica nanoparticle for the enhancement of epoxidation of alkene [19], and generation of Brönsted acidity in AIMCM-41 by sulphation method [20, 21].



**Figure 6** Proposed mechanism of dehydrogenation of cyclohexanol over a base site in MeAPO-5 [9].

### Hydrophobic-hydrophilic catalytic system

One important medium effect that has to be singled out is the hydrophobic-hydrophilic phase interplay possible in the liquid phase. Catalyst systems in which such effects play a role we propose to call hydrophobic-hydrophilic catalytic system [22-32].

In this section, a new hydrophobic-hydrophilic catalytic system termed as phase-boundary catalytic system is introduced. The catalyst has been designed in which the external part of the zeolite is hydrophobic, internally it is usually hydrophilic, notwithstanding to polar nature of some reaction centers. In this sense the medium environment in this system is close to that of an enzyme. The major difference between this system and enzyme is accessibility and flexibility. Whereas in this system to a reactant occluded in the micropore many reaction centers are available in enzymes per enzyme cleavage there is usually only one site. Another important difference, as we mentioned before, is lattice flexibility. The lattice of zeolite is rigid, whereas the enzyme is flexible.

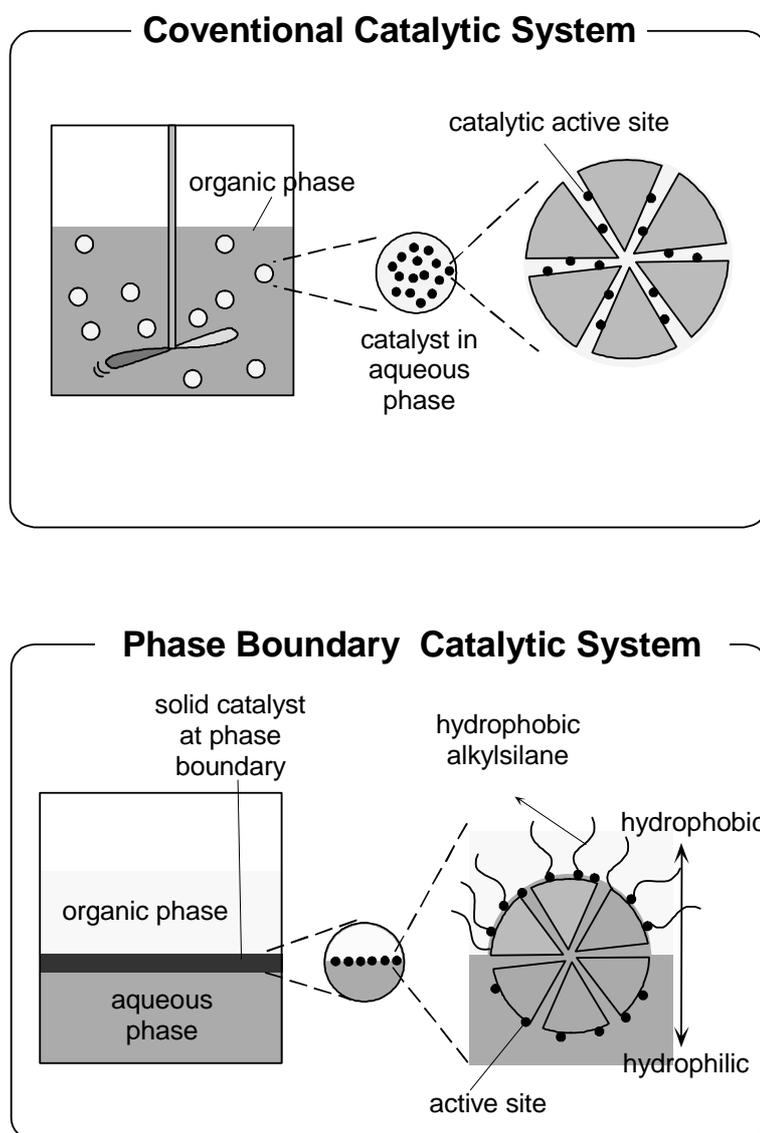
#### *Design of hydrophobic-hydrophilic catalytic system*

Figure 7 shows schematic representation of design of Phase Boundary Catalytic (PBC) system and its comparison with conventional catalytic system. The PBC is useful primarily for performing reaction at the interface of aqueous phase and organic substrate phases. PBC is needed because the immiscibility of aqueous phase and organic substrate. The name phase-boundary catalysis does what it says; the catalyst acts as a catalyst at the interphase between the aqueous and organic phases as shown in Figure 3. The reaction medium of phase-boundary catalysis system for the catalytic reaction of immiscible aqueous and organic phases consist of three phases; an organic liquid phase, containing most of the substrate, an aqueous liquid phase containing most of the substrate in aqueous phase and the solid catalyst. The two liquid phases are almost completely insoluble in one another.

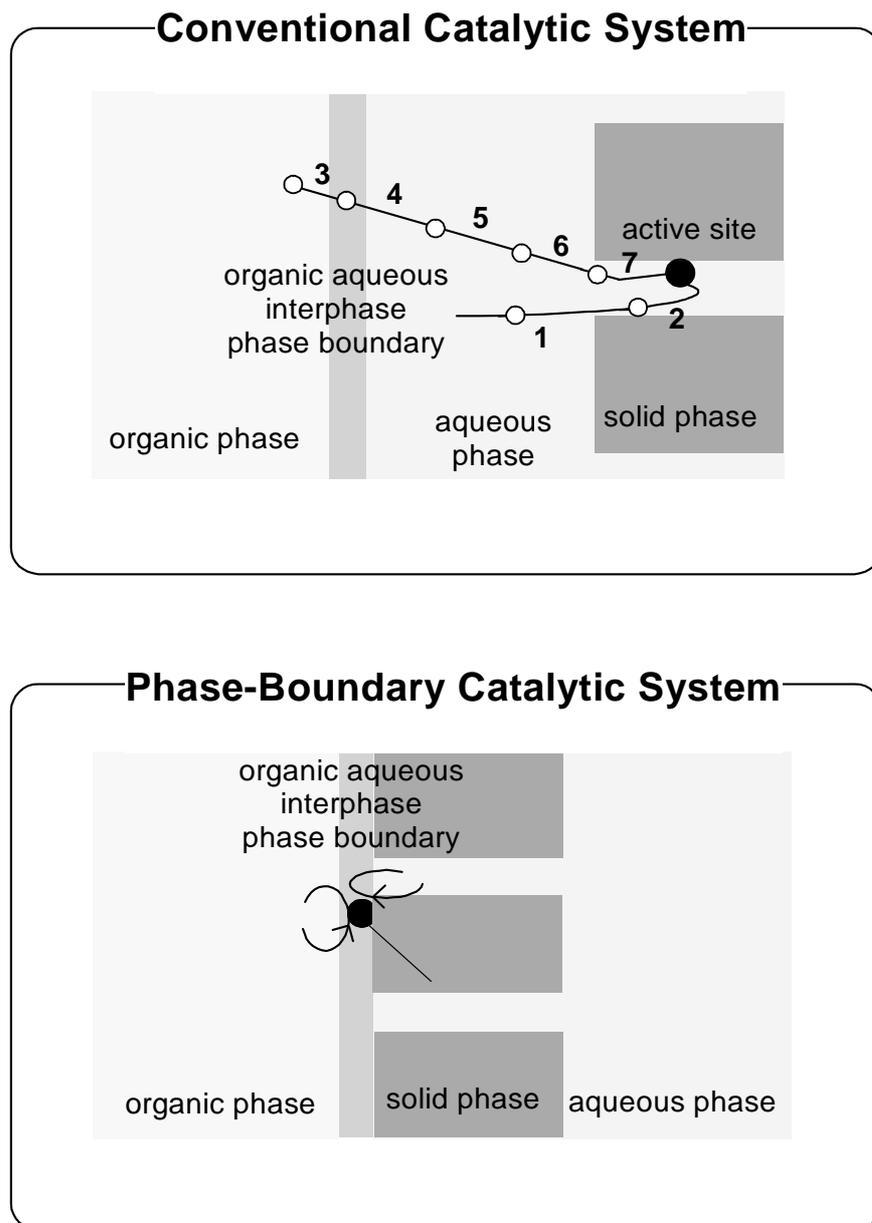
In case of conventional catalytic system (see Figures 7 and 8);

- When the reaction mixture is vigorously stirred, an apparently homogeneous emulsion is obtained, which segregates very rapidly into two liquid phases when the agitation ceases. Segregation occurs by formation of organic bubbles in the emulsion which move downwards to form the aqueous phase, indicating that emulsion consists of dispersed particles of the aqueous phase in the organic phase.
- Due to the triphasic reactions conditions, the overall reaction between aqueous phase and organic phase substrates on solid catalyst requires different transfer processes. The following steps, which are schematically represented in Figure 8 are involved: 1. transfer of aqueous phase from organic phase to the external surface of solid catalyst; 2. transfer of aqueous phase inside the pore volume of solid catalyst; 3. transfer of the substrate from aqueous phase to the interphase between aqueous and organic phases;

4. transfer of the substrate from the interphase to the aqueous phase; 5. mixing and diffusion of the substrate in the aqueous phase; 6. transfer of the substrate from the aqueous phase to the external surface of solid catalyst; 7. transfer of the substrate inside the pore volume of the solid catalyst; and 8. catalytic reaction (adsorption, chemical reaction and desorption).



**Figure 7** Schematic representation of the advantage of phase-boundary catalysis in comparison with conventional catalytic system.



**Figure 8** Schematic representation of catalytic action of phase-boundary catalysis in comparison with conventional catalytic system.

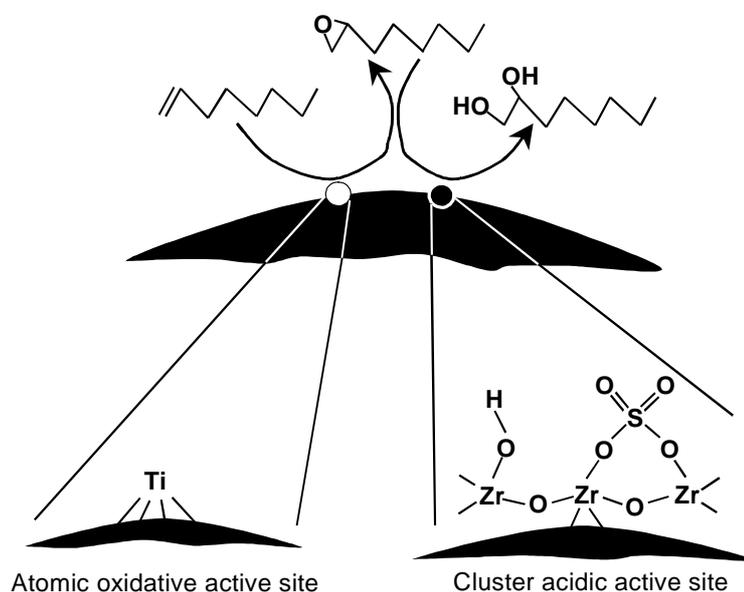
It was reported that without vigorous stirring, no reactivity of the catalyst was observed in conventional catalytic system [22-24]. As proposed in Figure 8, it is clear that stirring and mass transfer from organic to aqueous phase and vice-versa are required for conventional catalytic system. In the PBC (see Figure 8), the stirring is not required because the mass transfer is not rate determining step in this catalytic system. It is already demonstrated that this system works for alkene epoxidation without stirring or the addition of a co-solvent to drive liquid-liquid phase transfer [22-24]. The active sites located on the external surface of

the zeolite particle were dominantly effective for the observed phase boundary catalytic system [31, 32].

The work in this kind of catalytic system is based on my scientific work in the period of 1999-2002 which was devoted to the development of a novel catalytic system [22-25, 31, 32].

### Bifunctional catalyst

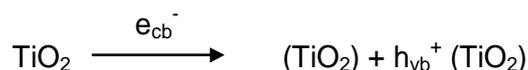
Another type catalytic system can be defined as bifunctional. The prototype catalytic system is TS-1 loaded with sulfated zirconia as bifunctional oxidative and acidic catalyst for transformation of 1-octene to 1,2-octanediol [33-40]. The catalyst concerned contains two types of reactive centers, oxidative and acidic. The titanium act as active site for the transformation 1-octene to 1,2-epoxyoctane and the protonic sites hydrolyze the epoxide. The overall reaction consists of two steps, in which an intermediate formed in one reaction olefin is consumed on the other. In heterogeneous catalysis there is usually no control over the sequence of these steps. The control that exists is basically due to differences in the reactivity of the different sites. Proposed model of bifunctional catalytic system is shown in Figure 9.



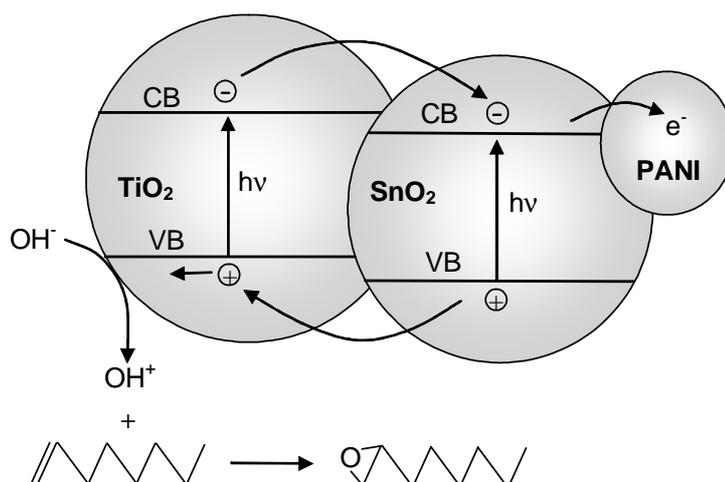
**Figure 9** Proposed model of TS-1 loaded with sulfated zirconia as bifunctional catalyst for consecutive transformation of 1-octene to 1,2-octanediol through the formation of 1,2-epoxyoctane [36].

## Photocatalyst

By definition, a photocatalyst is a substance that is able to produce, by absorption of light quanta, chemical transformations of the reaction participants, repeatedly coming with them into the intermediate chemical interactions and regenerating its chemical composition after each cycle of such interactions [41]. Titanium dioxide ( $\text{TiO}_2$ ) is one of the most popular photocatalysts. Photocatalysis over  $\text{TiO}_2$  is initiated by the absorption of a photon with energy equal to or greater than the band gap of  $\text{TiO}_2$  (3.2 eV), producing electron-hole ( $e^-/h^+$ ) pairs,



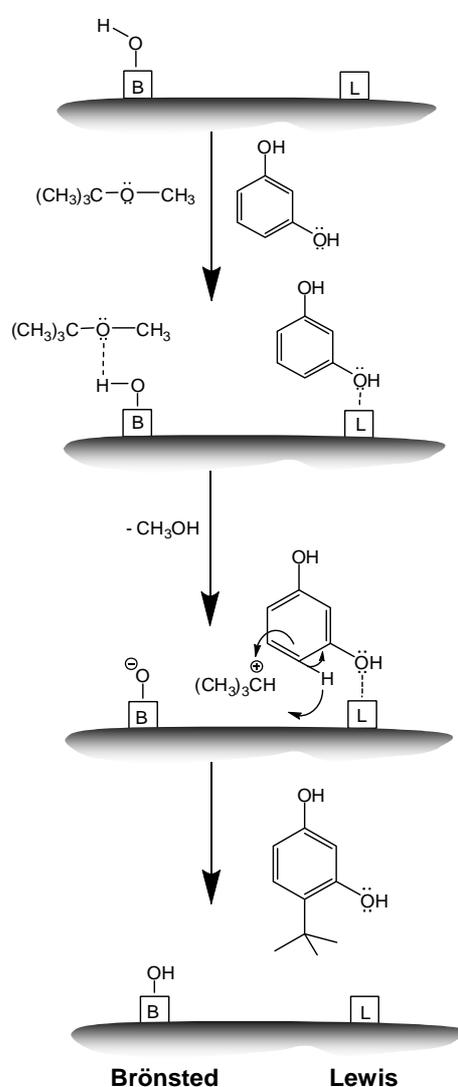
Consequently, following irradiation, the  $\text{TiO}_2$  particle can act as either an electron donor or acceptor for molecules in the surrounding media. However, the photoinduced charge separation in bare  $\text{TiO}_2$  particles has a very short lifetime because of charge recombination. Therefore, it is important to prevent electron-hole recombination before a designated chemical reaction occurs on the  $\text{TiO}_2$  surface.  $\text{TiO}_2$  and high recombination rate of the photogenerated electron-hole pairs hinder its further application in industry. Having recognized that charge separation is a major problem, here,  $\text{SnO}_2$ - $\text{TiO}_2$  coupled semiconductor photocatalyst loaded with PANI, a conducting polymer, has been studied as photocatalyst in the oxidation of 1-octene with aqueous hydrogen peroxide. We reported that the attachment of polyaniline (PANI) on the surface of  $\text{SnO}_2$ - $\text{TiO}_2$  composite will reduce the electron-hole recombination during the photocatalytic oxidation of 1-octene due to PANI's electrical conductive properties (see Figure 10) [41].



**Figure 10** The proposed mechanism of photocatalytic epoxidation of 1-octene over PANI- $\text{SnO}_2$ - $\text{TiO}_2$  [41].

### Synergetic multi reaction center catalyst

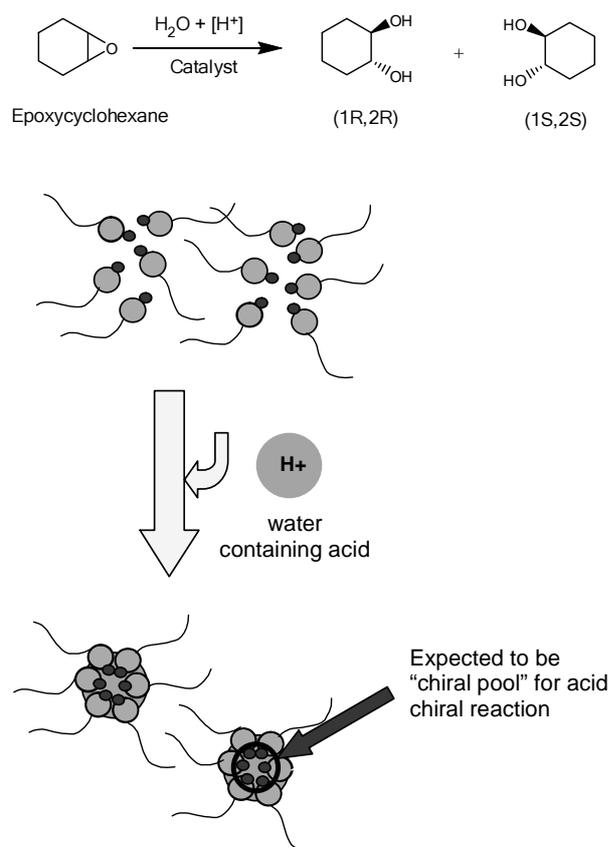
In reactions of synergetic multi reaction center catalyst, at least two different reaction centers that communicate are required. An example is synergistic role of Lewis and Brönsted acidities in Friedel-Crafts alkylation of resorcinol over gallium-zeolite beta. The role of Lewis and Brönsted acidities in alkylation of resorcinol is demonstrated through the gallium-zeolite beta by varying the amount of Lewis and Brönsted acid sites (see Figure 11). The synergism of Lewis and Brönsted acid sites take place heterogeneously in Friedel-Crafts alkylation of resorcinol with methyl tert-butyl ether to produce 4-tert-butyl resorcinol and 4,6-di-tert-butyl resorcinol as the major and minor products respectively [42].



**Figure 11** Proposed mechanism of the alkylation of resorcinol with MTBE [42].

## Chiral catalyst

The control of enantioselectivity by heterogeneous asymmetric catalysis remains a big challenge today. The main drive has been to find new, exciting routes to chiral molecules while achieving high enantiomer selectivity. Here, a new strategy to obtain active catalyst in the enantioselective hydration of epoxycyclohexane is proposed [43, 44]. The research strategy is based on the ideas that the enantioselective reactions could be induced by chiral amino acids and the use of heterogeneous catalysis for synthetic purposes will overcome practical separation problems. In order to realize these ideas, chiral amino acid needs to be attached to the hydrophilic part of hydrolyzed octadecyltrichlorosilane (OTS). Amino acids such as L-glutamic acid and L-phenylalanine have been chosen because of their water-soluble properties; hence they can be easily removed by treatment with water. It is expected that the attachment of amino acid would result in a chiral solid catalyst with bimodal hydrophobic-hydrophilic character. The schematic action of amphiphilic chiral solid catalyst is shown in Figure 12.



**Figure 12** Amphiphilic chiral solid catalyst as heterogeneous micellar catalyst in enantioselective hydration of epoxycyclohexane [43].

## **INTEGRATED RESEARCH MODEL IN HETEROGENEOUS CATALYSIS**

In this millennium, Malaysia should strive to emerge as one of the competitive nations in the world. To achieve this, several research universities have been created in Malaysia. University research is a noble intellectual activity and gives societal benefits. So, consequently, all scientific researches, including research in heterogeneous catalysis becomes more important in Malaysia and also at UTM since UTM was announced as a Research University by the Prime Minister of Malaysia on 10 June 2010. Table 2 showed the published paper indexed by Scopus in the field of catalysis in Asiatic region in the period of 1999 to 2010 [45]. Since Malaysia still lags behind Thailand, Hong Kong, Singapore and Taiwan in the field of catalysis research, one suggests that the need for total quality in education and research in this field becomes increasingly important. In this context, the strategy of the way to achieve the excellence in heterogeneous catalysis research is described in the following section where research in heterogeneous chiral catalysis is used as an integrated research model in heterogeneous catalysis.

### **Molecular and supramolecular approach to heterogeneous chiral catalysis: an example of proposed integrated research model in heterogeneous catalysis**

Catalysis is a field that is important in the chemical industry and also a key factor in world economy and for sustainable development. Substantially increased emphasis on chiral catalyst development is an important goal for chemistry-related industries. This needs to be supported by a broad and concerted effort by those who understand the opportunities and challenges that the creation of a new generation of environmentally friendly, profitable and diverse chiral catalysts will bring all groups concerned; consumers, industrialists, environmentalists and scientists, to name a few, will benefit in a very significant way.

New scientific breakthroughs through science and technology, including in the areas of catalysis, is at the core of Malaysia's vision for its development. In particular, research on heterogeneous catalysis is strategic for Malaysia, both for its economic impact and for its relevance for a sustainable future. Herewith are some key figures that help gauge the importance of the catalysis sector in the world and Malaysia [46]:

- 80% of all chemicals require at least one catalytic step for their production.
- Overall catalyst market in the world is around USD 10 billion.
- Revenue generated by catalysts in the world: between USD 1000 and 10,000 billion.
- Malaysian sector of chemicals and petroleum: expected mid-term growth > 5%.
- Catalytic processes are usually ecologically superior (viz. reduction of raw material,

**Table 2** Published paper in the field of catalysis indexed by Scopus in Asiatic region in the period of 1999 to 2010 [45].

	Country	Citable documents	Citations	Self-Citations	Citations per Document	h-index
1	China	9,855	92,302	48,584	11.15	80
2	Japan	6,887	114,911	30,334	16.59	102
3	India	3,704	41,479	12,502	13.08	60
4	South Korea	2,263	25,495	6,142	13.19	56
5	Taiwan	880	12,168	2,235	15.77	49
6	Singapore	400	5,421	763	18.52	36
7	Hong Kong	339	6,210	850	20.99	41
8	Thailand	277	2,527	610	14.88	23
9	Malaysia	213	1,420	410	7.16	18
10	Pakistan	70	303	90	9.98	9
11	Kazakhstan	42	151	7	3.05	7
12	Vietnam	31	198	9	9.86	7
13	Indonesia	27	253	16	9.56	11
14	Bangladesh	12	106	15	13.86	4
15	Philippines	8	65	1	6.43	5
16	Mongolia	10	53	11	5.44	3
17	Sri Lanka	9	236	6	31.14	6
18	Turkmenistan	2	6	0	3	1
19	Uzbekistan	2	9	2	4.5	1
20	Myanmar	1	3	0	3	1
21	Nepal	1	6	1	6	1

energy consumption, and waste production).

Therefore, the development of high performance and conceptually innovative chiral catalytic processes is crucial for chemical and pharmaceutical industries sector. Additionally, it is the key for a sustainable future for Malaysia. This is also in line with the focus of Malaysia government in prioritized investment in the 12 National Key Economic Areas (NKEAs).

The program undertaken which entitled 'Molecular and supramolecular approach to

heterogeneous chiral catalysis', and abbreviated as CHIROCAT, encompasses novel heterogeneous chiral catalysts that impart new ideas and techniques as well as methods that will eventually lead to the development of value added materials. Subsequently, 8 projects have been identified to look into the advanced organic and inorganic synthetic chemistry to support the development of heterogeneous chiral catalysts.

This program facilitates the effort to provide opportunity for the academic as well as research community to contribute towards making Malaysia a global leader in chemical and pharmaceutical industries as a whole. The outcome of this program will result in the fundamental understanding and know-how to further develop diversified heterogeneous chiral catalysts for applications targeted in the NKEAs and hence further enhance the productivity, innovation and growth of the nation. This research program will contribute at least to the 4 NKEAs, i.e. oil and gas, palm oil and related products, private healthcare, and agriculture.

Chiral compounds are important materials in the pharmaceutical and fine chemicals industries. Global sales of single-enantiomer compounds are expected to reach \$8.57 billion by the end of 2004 and \$14.94 billion by the end of 2009 growing annually by 11.4%, according to the Frost & Sullivan survey. By 2009, the share of the market realized through traditional technology would drop to 41%. The share of chemocatalysis would rise to 36% and the share of biocatalysis, to 22%, the same survey shows [47].

Although many homogeneous chiral catalyst have been developed, the difficulty of product purification and catalyst recycle is one of the obstacles of practical applications. The design and synthesis of efficient and recyclable heterogeneous chiral catalyst remain the challenges and attracted much research interests in the past decade.

### **Need for the strategic research in the field of heterogeneous chiral catalysis**

A new generation of catalysts is needed to meet the demands of society in terms of ecology and energy efficiency. At a scientific level, such a challenge is met by gaining superior control of the active site at the molecular and nanoscopic level. The existing technologies are inadequate because, except with rare exception, they do not allow control of the surface, of the nano-scale functional object and of the investigative techniques at the molecular scale.

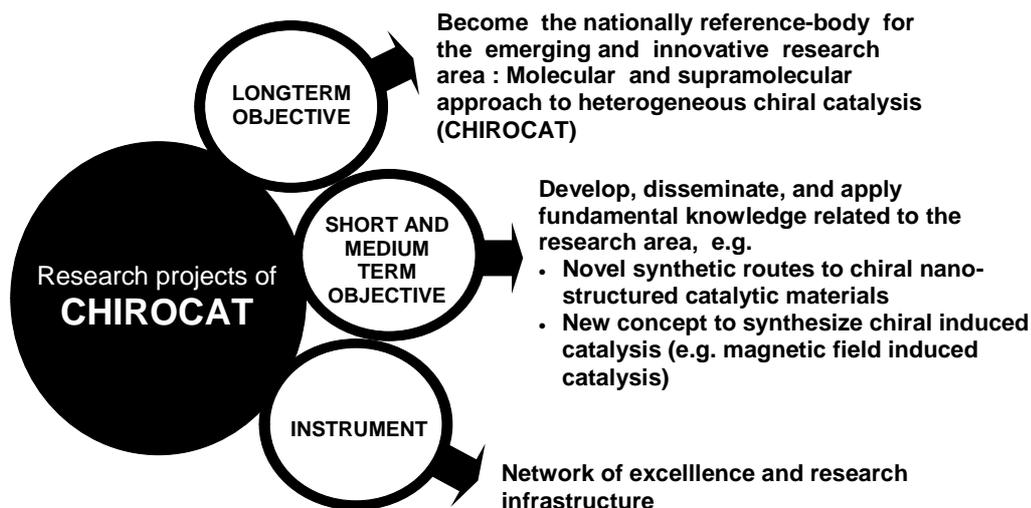
Meanwhile, in journals surveyed by Chemical Abstracts Service, the number of papers per year that are related to chiral technologies has five-fold from just over 1,300 in 1994 to more than 6,500 in 2010, for a total of more than 50,000 chiral-technology related papers published in the past 15 years. An overwhelming majority (72%) are about stereoselective or asymmetric syntheses. However, only less than 100 Scopus indexed journal papers were published by Malaysian researchers until the end of August 2011 [45].

The research program proposed here focuses on the design, synthesis, characterization and application of high performance heterogeneous chiral catalytic systems for the synthesis of fine chemicals and chiral pharmaceutical products. Along this line, new approaches to synthesize and also to separate chiral compounds are proposed in this program:

- Enantioselective heterogeneous catalysis induced by magnetic field
- Brønsted acid-assisted chiral catalyst
- Base chiral catalysis on microporous and mesoporous materials
- Photocatalyst for enantioselective catalysis
- Control rational self-assembly and recognition mechanisms for enantioselective catalysis in porous materials
- Enantioselectivity on surfaces with chiral nanostructures by molecular imprinting method
- Biomimetic design of catalysts for enantioselective catalysis
- Develop and apply novel nano-scale materials for chiral separation

The long term objective of this program is to become the nationally recognized reference networking of excellence (NoE) for an emerging and innovative multidisciplinary research field: *Molecular and supramolecular approach to heterogeneous chiral catalysis*. This novel research area spans several traditional chemical disciplines (molecular chemistry, supramolecular chemistry, theoretical modeling, heterogeneous catalysis and chemical separation), disciplines closer to physics (surface science, solid-state spectroscopies and analytical methods) and chemical engineering. The coherence of the network is due to its focus on the design, synthesis and testing of chiral nano-structured surfaces and materials displaying catalytic activity. Figure 13 shows objectives and instrument of CHIROCAT research project.

The basic principle of this research project is that a molecularly-minded approach to nano-scale catalysts, not traditionally included in the broad field of heterogeneous catalysis, will be the key to innovating in the field (i.e. transferring concepts derived from solution and supramolecular chemistry to nano-scale will lead to better performing heterogeneous catalysts). Figure 14 shows research projects involved in the CHIROCAT network and their specific research topics to networks multidisciplinary. Recent results based on a multidisciplinary approach to nano-controlled materials have shown that improvement of existing systems and breakthroughs in areas such as environment, energy, and sustainability could be achieved by improved control of catalysts at the nanoscale.



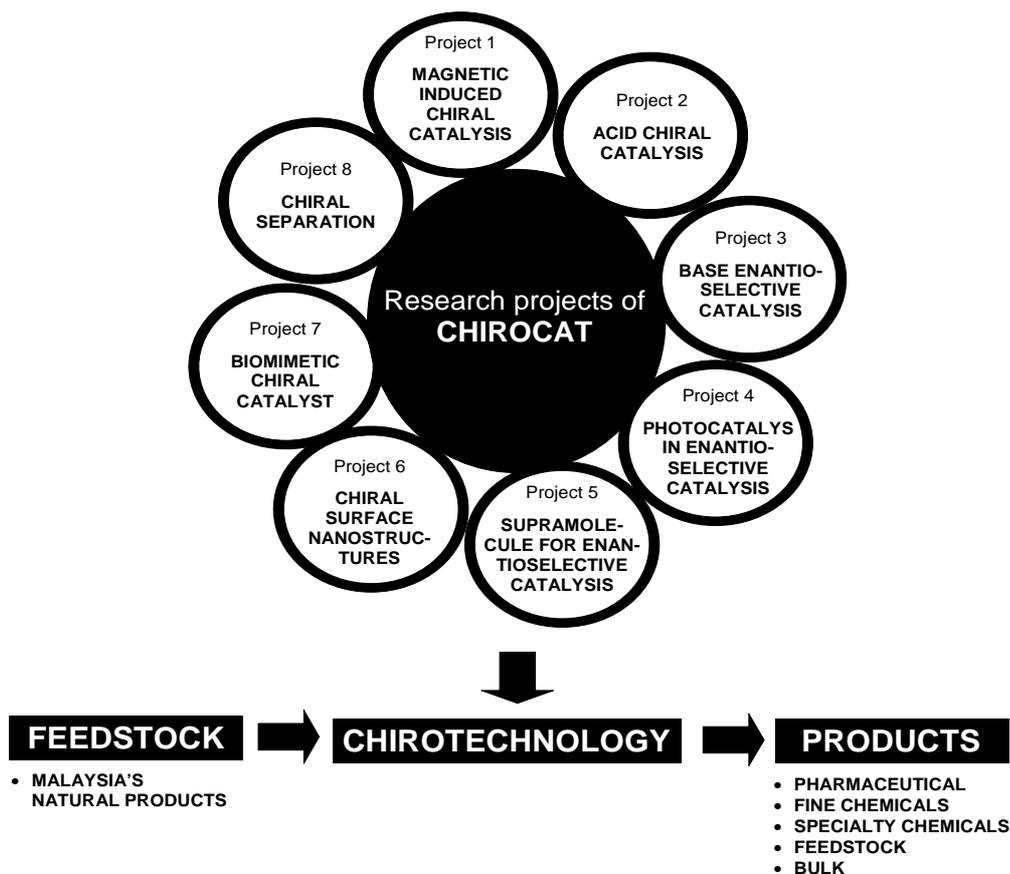
**Figure 13** Objectives and instrument of CHIROCAT research project.

## OTHER RESEARCHES

With the knowledge of chemistry, I also have researches outside the heterogeneous catalysis field. This is possible because chemistry is the mother of science. With chemistry, we can study the composition, structure and behavior of atoms and molecules that make up all kinds of material. Understanding the world at the atomic level is important for all fields of science. I also carried out researches in the field of solid-state NMR [48], analytical chemistry [49], soils chemistry [50-54], chemistry of nanoparticles [55], mesoporous materials [56-59], aerogel materials [60-62], zeolite synthesis [63], and composite materials [64-66].

## ACKNOWLEDGEMENTS

Coming to conclusion, I thank the Universiti Teknologi Malaysia for entrusting me with this professorial position. I am very grateful for having the opportunity to work with helpful and pleasant collaboration, especially Prof. Dr. Halimaton Hamdan (Universiti Teknologi Malaysia), Prof. Dr. Bunsho Ohtani (Hokkaido University), Assoc. Prof. Dr. Shigeru Ikeda (Osaka University), Assoc. Prof. Dr. Zainab Ramli (Universiti Teknologi Malaysia), Prof. Dr. Salasiah Endud (Universiti Teknologi Malaysia), Assoc. Prof. Mohd Nazlan Mohd Muhid (Universiti Teknologi Malaysia), Assoc. Prof. Dr. Sugeng Triwahyono (Universiti Teknologi Malaysia), Dr. Lee Siew Ling (Universiti Teknologi Malaysia), Dr. Leny Yuliati (Universiti Teknologi Malaysia), Dr. Hendrik Oktendy Lintang (Universiti Teknologi Malaysia), Dr. Jon



**Figure 14** Research projects involved in the CHIROCAT network and their specific research topics to networks multidisciplinary.

Efendi (Universiti Teknologi Malaysia), Dr. Dwi Gustiono (Universiti Teknologi Malaysia), Mohd Syafiq Elias (Universiti Teknologi Malaysia), Prof. Dr. Mohd Marsin Sanagi (Universiti Teknologi Malaysia), Assoc. Prof. Dr. Abdul Rahim Mohd Yusoff (Universiti Teknologi Malaysia), Dr. Mohd Arif Agam (Universiti Tun Hussein Onn Malaysia), Prof. Dr. Ismunandar (Institut Teknologi Bandung), and Dr. Rino Rakhmata Mukti (Institut Teknologi Bandung).

I would also like to thank the rest of students and research officers at Universiti Teknologi Malaysia for their support. I avoid using the feudal word 'my' in the following list. Research officers: Leiw Sook Fun, Amelia Boon Hoo, Sheela Chandren and Nur Izzati Abu Bakar. Ph.D. students: Dr. Didik Prasetyoko, Dr. Fitri Hayati, Dr. Eriawan Rismana, Dr. Amin Eisazadeh Otaghsaraei, Surya Lubis, Umar Kalmar Nizar, Nursyafreena Attan, Nur Hidayah Mohd Ran, Pratama Jujur Wibawa, Syamsi Aini, Lai Sin Yuan, Noorfatimah Yahaya, Mohammed Salisu Musa, Fatemeh Yaghoubi Doust, Shafiyah Pondi, Mita Rilyanti, and Mukhamad Nurhadi. M.Sc. students: Norizah Abdul Rahman, Helda Hamid,



**Figure 15** Members of Catalytic Science and Technology (CST) research group (2010). From left to right: Dr. Dwi Gustiono, Dr. Jon Efendi, Dr. Hendrik Oktendy Lintang, Prof. Dr. Hadi Nur, Assoc. Prof. Dr. Sugeng Triwahyono, Prof. Dr. Kanuzari Domen (Visiting professor from The University of Tokyo), Assoc. Prof. Dr. Zainab Ramli, Dr. Leny Yulianti and Dr. Lee Siew Ling.

Dr. Ng Yun Hau, Lim Kheng Wei, Gui Lee Kee, Izan Izwan Misnon, Yong Khun Fong, Sheela Chandren, Amelia Boon Hoo, Farizan Mohamad, Nor Rashidah Md Juremi, Syaiful Akhmal Saadon, and Ghani Ur Rehman. B.Sc. students: Amir Faizal Naidu Abdul Manan, Izan Izwan Misnon, Shahmeen Ismail, Nurulashikin Mohd Ariffin, Chua Yew Hean, Farah Hannan Anuar, Sim Sau Teing, Nur Hajarul Aswani Moamail, Norazlizan Abul Rashid, Noor Ayu Ismail, Jessie Jessica Anak Robin, Raazatul Aidah Abd Latif, Norshahida Zamahsari, Intan Dayana Samsuri, Ng Kar Fai, Ang Wai Leong, Salmi Fathiyah Sheikh Salim, Siti Shahidaanis Abdul Aziz, Noorulsyahidaini Golbaha, Lai Sin Yuan, Hidayati Mohamad Mukhair, Wan Faizal Wan Ahmad, Mohd Hayrie Mohd Hatta, Shazliana Mohamad @ Mat Yasin, Teoh Leong Shen, Noor Hidayah Mohd Jusof, Siti Noramalina Nazri, Lim Chai Sin and Ahmad Fahmi Abdullah.

The research would not have been possible without financial support from the Japan Society for Promotion of Science (JSPS), Ministry of Science, Technology and Innovation (MOSTI) Malaysia, Ministry of Higher Education (MOHE) Malaysia, The Academy of Sciences for the Developing World (TWAS), Trieste, Italy and Universiti Teknologi Malaysia.

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Hadi Nur was born on the 6<sup>th</sup> of May 1969. His main field in undergraduate and graduate studies was in chemistry and materials engineering at the Institut Teknologi Bandung. He obtained his B.Sc. and M.Eng. (*cum laude*) degrees in 1992 and 1995, respectively. Shortly after that, he continued his postgraduate studies in zeolite chemistry at the Universiti Teknologi Malaysia (UTM) as he received his Ph.D. degree in 1998. His postdoctoral studies started with a year as a UTM Postdoctoral Fellow and with two years as a Japan Society for Promotion of Science (JSPS) Postdoctoral Fellow at Catalysis Research Center (CRC), Hokkaido University, Japan. He continued there as a Center of Excellence (COE) Visiting Researcher at CRC for half a year. His academic career began with the appointment as a Lecturer in the Ibnu Sina Institute for Fundamental Science Studies, Universiti Teknologi Malaysia in 2003, after one year working as a Research Officer in this institute. He was promoted to the rank of Senior Lecturer in 2007, to Associate Professor in 2008 and to Professor at the Universiti Teknologi Malaysia in 2010.

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He has been invited as a speaker for the following seminar, conferences and symposium: keynote speaker at International Seminar on Environmental Science organized by Universitas Andalas (Padang, 2011), keynote speaker at The ITB Catalysis Symposium organized by Institut Teknologi Bandung (Bandung, 2011), keynote speaker at The 12<sup>th</sup> International Conference on Quality in Research organized by Universitas Indonesia (Bali, 2011), invited speaker at Nanotechnology Seminar at Universiti Tun Hussein Onn Malaysia (Batu Pahat, 2011), keynote speaker at The 3<sup>rd</sup> Junior Chemists Colloquium organized by Universiti Teknologi Malaysia (Johor Bahru, 2011), keynote speaker at The National Seminar on Engineering and Technology organized by Universitas Bung Hatta (Padang, 2010), invited lecture on Application of Solid State NMR at NMR User Meeting organized by Bruker South East Asia (Kuala Lumpur, 2010), keynote speaker at The National Seminar on Chemistry and Chemistry Education organized by Universitas Negeri Semarang (Semarang, 2009), keynote speaker at The 11<sup>th</sup> National Seminar on Chemistry organized by Institut Teknologi Sepuluh Nopember (Surabaya, 2007), and invited speaker at Graduate School of Engineering Science Seminar, Osaka University (Osaka, 2007).

He currently supervises thirteen Ph.D. and four M.Sc. students, and has previously supervised four Ph.D. and ten M.Sc. students. He has also supervised four research officers. Currently, he is a Head of Catalytic Science and Technology (CST) research group and Manager of International Affairs for Indonesia at UTM.