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The design of the cover was fashioned from two figures:

- The left of the cover, found on pp. 55 (Figure 17 — SEM micrographs) of an article entitled *A Study on the Effects of Environment on Curing Characteristics of Thixotropic and Room Temperature Cured Epoxy-based Adhesives Using DMTA* (pp. 37–58), where researchers from Universiti MARA and Universiti Putra Malaysia investigated the thermal properties of epoxy-based adhesive reinforced with nano- and micro-particles before and after exposure to the different environmental conditions. The results showed that room temperature cured epoxies were only partially cured at room temperature.

- On the right of the cover shows a three-dimensional coil structure (Figure 1, pp. 28) in the article entitled *Investigations on Physical Characteristics of Three-dimensional Coil Structure for MEMS Magnetometer* (pp. 27–36). Scientists at Universiti Kebangsaan Malaysia investigated (using FEM simulation) the physical characteristics of three-dimensional coils and its correlation with magnetic energy. The changing of the dimension size of the coil had an effect on inductance, magnetic energy and magnetic flux density.
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Creativity and innovation are recognised the world over as the key measure of the competitiveness of a nation. Within the context of K-Economy and the framework of National Innovation System (NIS), ASM will continue to spearhead efforts that will take innovation and creativity to new heights in the fields of sciences, engineering and technology and work towards making Malaysia an intellectual force to be reckoned with.
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New Formulated Hybrid Catalysts for the Synthesis of Carbon Nanotubes via Chemical Vapour Deposition Technique

B. Nor Aziah* and I. Fatiha

Transition metals play an important role in the growth of carbon nanotubes (CNTs). Series of unsupported hybrid catalysts consisting of Ni:Cu, Ni:Cr, and Ni:Mn doped with Nd catalyst, respectively were synthesized by impregnation method. The catalytic performance of the catalyst for the production of CNTs was measured in the pyrolysis process of hydrocarbon source by catalytic chemical vapour deposition method. Acetylene gas was used as the source of carbon in the pyrolysis process. The decomposition of acetylene was carried out at 700°C. The bulk properties of the catalysts were investigated by X-ray diffraction. Field emission scanning electron microscopy and thermal analysis were used to observe the morphology and thermal stability of the as-synthesized CNTs, respectively. Hybrid catalyst of Ni:Mn/Nd and Ni:Cr/Nd in 3:1 atomic ratio gave high percentage of carbon yield which was assigned for the high production of CNTs with the mass of yield 18 times greater than the initial mass of the catalyst used.

Key words: Carbon nanotubes; chemical vapour deposition; Lanthanide; acetylene; pyrolysis; impregnation method; chemical vapour deposition; X-ray diffraction; morphology; thermal stability

Carbon nanotubes (CNTs) are tubular structures that are typically of nano-size diameter and many of micrometers in length. This fascinating one dimensional material has captured the attention of researchers worldwide with their extraordinary electronic and mechanical properties. These remarkable physicochemical properties have made them useful in various potential applications such as hard composite tribological coating (Sing et al. 2009), gas storage media (Chambers et al. 1998), gas sensors (Tibbetts 2001) and semiconducting materials (Frackowiak & Beguin 2001). In order to fulfil the demands for commercial applications, large quantities of CNTs at a low cost of production are required. There are numerous established methods which have been developed to produce CNTs, including laser ablation, arc discharge and catalytic chemical vapour deposition (CCVD). Among these three methods, CCVD is the most promising because it exhibit direct control on the reaction parameters such as the reaction temperature, reaction gas composition and flow rate, reaction time and catalyst. In addition, CCVD is also an economical means of producing CNTs in larger scale (Ryu et al. 2008).

The size of the catalyst particle is controlled to the nanometer range with the development of the new formulated hybrid catalysts. In this approach, two or more metals can either be bonded to each other in a metal oxide cluster or can be a mixture of two separate oxide phases in a metal cluster. For instance, it has been observed that catalysts made by the addition of molybdenum to iron stabilized the active iron nanoparticles (Lacerda 2004). Cui et al. (2008) also found the synergy of Mo and Fe-Ni alloy is the main reason for the high yield of CNTs. The most effective and common catalysts used for the growth of CNT by means of the CCVD are Fe, Co and Ni. Co-Mo catalysts show high selectivity and activity for the synthesis of single-walled carbon nanotube (SWCNT) using ethanol as the gas precursor (Ni et al. 2006). The role of catalyst in the CCVD method for producing CNTs is to enhance the decomposition of the carbon sources such as hydrocarbon compounds, carbon monoxide and carbon dioxide by means of the pyrolysis process (Anne-Claire 2005). The introduction of lanthanide metals, ions or oxides to the formulated catalyst will contribute to the generation of interesting properties such as supports, promoters or additives (Sakata 1999).

There are three types of hybrid catalyst formulated in this work which consist of Ni, Mn, Cr doped with Nd, respectively. Investigations on the catalytic activity

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performance of the hybrid catalysts for the synthesis of CNTs were carried out using a fixed-bed chemical vapour deposition reactor. The physical and chemical properties of the hybrid catalysts were then characterized using X-ray diffraction (XRD), field emission scanning electron microscope (FESEM) and thermogravimetric analyser (TGA).

MATERIALS AND METHODS

Preparation of the Catalysts

Series of hybrid catalysts of Ni:Cu, Ni:Cr and Ni:Mn doped with Pr with the atomic ratio of 3:1:0.3 were prepared from a mixture of nickel (II) nitrate hexahydrate Ni(NO₃)₂.6H₂O, copper nitrate [Cu(NO₃)₂], manganese (II) acetate [(CH₃COO)₂Mn.4H₂O], chromium (III) nitrate (Cr(NO₃)₃.6H₂O) and praseodymium (III) nitrate [Pr(NO₃)₃.6H₂O], respectively. Optimum quantities of the respected metal salts were dispersed in a sufficient amount of water and mixed well to get a homogeneous mixture of the salt solution. The mixture was dried at 80°C for 24 h and calcined at 700°C for 5 h. The catalyst mixture was then cooled and finely ground into fine powder of hybrid catalyst.

Synthesis of Carbon Nanotubes

The synthesis was carried out using a horizontal fixed-bed CCVD reactor in atmospheric pressure. The reactor consisted of a tube furnace with a quartz tube where the decomposition of the acetylene gas took place (Figure 1). About of 200 mg of catalyst was placed at the centre (hot zone) of the tube furnace. Nitrogen as the carrier gas was simultaneously introduced into the reactor tube at the flow rate of 0.3 l/min. The mixed gases were then flown over the catalyst to allow the decomposition of the carbon precursors to occur. The reaction was left for 30 min at 700°C. The reaction chamber was then cooled to room temperature. The yield in the form of black powder was collected and the percent of carbon composition was determined from Equation 1:

\[
\text{Carbon yield (\%) = } \frac{M_1}{M_2} \tag{1}
\]

Where, \(M_1\) is the mass of carbon deposited after the reaction and \(M_2\) is the mass of catalyst used.

CHARACTERIZATION

The Hybrid Catalysts

The identification of metallic bulk crystallographic and amorphous phase in the hybrid catalysts were carried out by XRD. The analysis of the hybrid catalyst by XRD were conducted using Diffractometer D500 Siemens Crystalloflex with CuKα (\(λ = 1.54060\) Å) as the radiation source.

The As-synthesized CNTs

The as-synthesized CNTs were characterized by the JSM-6701F field emission scanning electron microscope (FESEM) to determine the morphology of the CNTs. The thermal stability and purity of the CNT was analysed using the Mettler Toledo TGA.

RESULTS AND DISCUSSION

X-ray Diffraction

The type of metal oxide in the hybrid catalysts formulation is one of the factors that influence the catalytic activity and morphology of the deposited carbon (Chai et al. 2007; Nyamori et al. 2008). XRD patterns of the hybrid catalysts were shown in Figure 2. From the diffractogram pattern, it shows that the active oxide phase, NiO present in Ni:Cr/Nd and Ni:Mn/Nd catalysts. While in Ni:Cu/Nd catalysts, Ni species tends to form the binary oxides of \(\text{Ni}_{19}\text{CuO}_{20}\) with high peaks intensity. It was identified in this work that the presence of NiO phase had contributed to the high production of CNTs with the mass of yield 18 times greater than the initial mass of the Ni:Cr/Nd and Ni:Mn/Nd catalysts used respectively, but not for Ni:Cu/Nd catalyst.

In comparison to the respective diffractograms pattern of the hybrid catalysts, it was observed that Ni:Cr/Nd was in the amorphous form, while the hybrid Ni:Mn/Nd and Ni:Cu/Nd catalysts represented the polycrystallite form. This new formulated hybrid catalysts of the respected Ni:Mn/Nd and Ni:Cu/Nd had generated two types of crystallite structures. Firstly, the spinal oxide of \(\text{NiMn}_2\text{O}_4\) which was observed in the hybrid Ni:Mn/Nd catalyst formulation, constructed from NiO and Mn₂O₃ phases and served as the active site for the excellent catalytic performance in the production of CNTs. Secondly, the ternary oxide of \(\text{Nd}_4(\text{CuNi})\text{O}_8\) which was present in the Ni:Cu/Nd catalyst and constructed from metal ions of various oxidation states of \(\text{Ni}^{2+}, \text{Cu}^{2+}\) and \(\text{Nd}^{3+}\) in the perovskite type of oxide. The ternary oxide in the hybrid catalyst of Ni:Cu/Nd existed as \(\text{Ni}_{19}\text{CuO}_{20}\) phase, instead of \(\text{Ni}_{19}\text{O}_{20}\) (or NiO phase). The molecular formula of the perovskite oxide showed that one mole of the Ni site in the NiO lattice cluster was probably replaced by one mole of \(\text{Cu}^{2+}\) ion, and resulted in the reduction of the catalytic performance of the NiO phase in the formulated Ni:Cu/Nd hybrid catalyst.

From the catalytic measurement of the Ni based hybrid catalysts, results obtained revealed that the incorporation
of the Cr, Mn and Cu, in the hybrid catalyst formulations affected the catalytic performance of the respected catalyst. It also proved that Ni was acting as the active species while Cr was acting as the dispersant to hinder the aggregation of Ni particle, so as to control the particle size of the catalyst, and it was in agreement with the work done by Chen and Wu (2005). The agglomeration and aggregation process among the Ni particles were further hindered by the addition of small amounts of Nd as the dopant, hence, it stabilized the size of the particles of catalyst in the nanometer range. In addition, with the assistance of the respected Mn and Cr in the Ni based hybrid catalysts formulation, the reduced environment during the synthesis was sustained. Thus, it contributed towards the controlled production of the specific form of CNTs.

**CHARACTERIZATION OF THE AS-SYNTHESIZED CARBON NANOTUBES**

**Micrograph of FESEM**

FESEM micrographs in Figure 3 confirmed the morphology of all carbon nanomaterials obtained by the decomposition of acetylene at 700°C over Ni:Cr/Nd, Ni:Mn/Nd and Ni:Cu/Nd hybrid catalysts are the CNTs. This proved that each of the hybrid catalysts were able to produce CNTs but they differed in term of the quality. The diameter of CNTs obtained was within the range of 10 nm to 50 nm. The smaller diameter CNTs plays an important role, specifically in the gas storage application and bio-sensors (Philippe 2009), and as for the larger diameter CNTs the applications was more to the fabrication of advanced composite materials.

The Ni:Mn/Nd catalyst gave the highest density of CNTs, whereas the Ni:Cr/Nd catalyst exhibited better quality CNTs. The Ni:Mn/Nd and Ni:Cu/Nd hybrid catalysts gave non-uniform CNTs with the mixture of different forms of carbon nanomaterials (Figure 3b and 3c). The small diameter CNTs was observed when using Ni:Cr/Nd (Figure 3a) due to the presence of small sized particles of catalyst which were assigned earlier as amorphous in the XRD analysis.

The presence of spinal NiMn2O4 compound in the Ni:Mn/Nd hybrid catalyst formulation gave the highest density of production of as-synthesized CNTs. The common active species, NiO had catalyzed the growth of CNTs in Ni:Mn/Nd and Ni:Cr/Nd hybrid catalysts. The combination of Ni:Cu/Nd hybrid catalyst resulted in the formation of ternary oxide of Nd4(CuNi)O8. This ternary compound had initiated the formation of the mixture of carbon nanomaterials with the large diameter of 50 nm (Figure 3c). The presence of the specific oxide phases in the hybrid catalysts identified from the XRD analysis had clearly explained its contribution towards the morphology of the as-synthesized CNTs observed from the FESEM.

From the micrographs of the FESEM, it was noted that the growth mechanism of MWCNT followed the based growth model, indicated by the absence of white spots at the tips of the CNTs floss (Anne-Claire 2005).
Thermal Gravimetric Analysis

Thermal gravimetric analysis (TGA) is one of the fundamental studies to differentiate the types of carbon nanomaterials. Its purity can be determined by calculating the percentage of mass loss during the heat treatment. The decomposition temperatures in Table 1 shows that the CNTs synthesized using the respected Ni:Cr/Nd, Ni:Mn/Nd and Ni:Cu/Nd hybrid catalysts gave one region of mass loss. This was an indication for the complete combustion of the specific form of CNTs with high purity, produced from the respected hybrid catalyst formulated.

It was found that, CNTs synthesized using Ni:Cr/Nd catalyst had the lowest decomposition temperature at 500°C with 90.64% of mass loss, which indicated that the diameter of the as-synthesized CNTs was the smallest compared to the other two hybrid catalysts formulated (Table 1). Small diameter CNTs usually decomposed at lower temperature compared to that of the larger diameter CNTs (Li et al. 2009). This result was in good agreement with the FESEM analysis of CNTs showed in Figure 2a. Whereas, the decomposition temperature for the as-synthesized CNTs from Ni:Mn/Nd catalyst occurred at the highest temperature of 630°C with 96.98% of mass loss. This observation as attributed to the presence of larger diameter CNTs and the presence of other form of carbon nanomaterials and was in agreement with the FESEM analysis showed in Figure 2b. The as-synthesized CNTs dominated was identified as MWCNTs and depicted high quality and purity when using Ni:Cr/Nd. However, when using Ni:Cu/Nd and Ni:Mn/Nd the CNTs produced were highly dense.

CONCLUSION

Series of new hybrid catalysts of Ni:Mn/Nd, Ni:Cr/Nd and Ni:Cu/Nd, have been successfully formulated for the production of MWCNTs with the average diameter of 30 nm. The presence of the oxide phases, identified as spinal and ternary oxides in the hybrid catalyst formulation, were the active phases which had enhanced the growth of CNTs. The metal-metal interactions in the catalyst had strongly influenced the growth of the CNTs, specifically with high purity and density. The Cr, Mn and Cu transition metals introduced in the Ni based hybrid catalysts formulation were acting as the promoter towards the formation of the active oxide phases as well as the dispersant to enhance the production of CNTs; whereas, the addition of Nd had enhanced the formation of nanoparticle catalyst with uniform size.
Figure 3. FESEM micrographs of as-synthesized CNTs over series of hybrid catalysts: (a) Ni:Cr/Nd; (b) Ni:Mn/Nd and (c) Ni:Cu/Nd.

Table 1. Percentage of mass loss of as-synthesized CNTs from TGA.

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<th>Purity of CNTs (%)</th>
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<tr>
<td>Ni:Mn/Nd 3:1</td>
<td>630°C</td>
<td>96.98</td>
</tr>
<tr>
<td>Ni:Cr/Nd 3:1</td>
<td>500°C</td>
<td>90.64</td>
</tr>
<tr>
<td>Ni:Cu/Nd 3:1</td>
<td>550°C</td>
<td>46.22</td>
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ACKNOWLEDGEMENT

We would like to express our gratitude to the Ministry of Higher Education (MOHE) for the Fundamental Research Grant Scheme (FRGS) 78543, the Ministry of Science, Technology and Innovation of Malaysia for the National Science Fellowship, and Universiti Teknologi Malaysia (UTM) for the support.

Date of submission: May 2011
Date of acceptance: September 2012

REFERENCES


With the increase in world energy demand and the depletion of petroleum crude oil, efforts have been made by researchers to seek alternative new sources of energy. Renewable energy such as solar and biomass have a high potential to cater for energy; however, with the extensive world coal reserves, proper attention needs to be considered to further upgrade and fully utilize this priceless fuel. Hence, with this in mind, studies of coal liquefaction to produce an alternative liquid fuel, as one of the upgrading coal processes, have been increasing in recent years. Intense efforts have been made to liquefy coal, with the processes such as pyrolysis, solvent extraction and catalytic hydrogenation using either batch or solvent flow reactor system have achieved some degree of success. In some countries, coal is used as a consumption material to generate steam for production of electrical power. Both gasification and liquefaction of coal produce gaseous and liquid fuels that can be easily transported (e.g. by pipeline) and conveniently stored in tanks. In the transportation and industrial sector, more than 80% of the energy consumed is provided by fossil fuel energy such as coal, petroleum and natural gas which are the main energy resources worldwide (Anon 2002).

Solvent flow reactor system was introduced into the extraction system to increase the system efficiency and enhance the extraction yield by adding fresh solvent during the extraction processes. The liquefaction experiment was carried out at various flow-rates (1, 3 and 5 ml/min), reaction times (30, 45 and 60 min) and reaction temperatures (300°C, 350°C, 400°C, 420°C and 450°C) with tetralin as solvent. Despite the ability of adding fresh solvent into the extraction process, the conversion of oil+gas was still considered to be low as there was ~25% of coal extracts left to be converted into low molecular weight compounds. One possible option to increase the oil yield is by applying catalyst that will further break up the coal extracts into small molecular weight compounds. In this study, a second reactor was introduced consisting of catalyst (NiSiO₂) assuming that the catalyst would interact more effectively with coal extracts rather than the coal itself. In the absence of catalyst, the oil yield was 55%. By introducing the Ni catalyst, the oil yield increased by 15%. Further analysis of GCMS showed that the oil from catalytic liquefaction gave out more low molecular weight compounds in comparison to the un-catalytic liquefaction oil.

Key words: Coal; liquefaction; solvent-flow; GCMS; Ni catalyst; oil conversion; molecular weight compounds; asphaltene; pre-asphaltene; tetralin

Solvent flow reactor system has been used by many researchers for its ability to investigate the coal liquefaction process rather at high temperatures. One important aspect in the solvent flow reactor system is the enabling of the removal, quenching and characterization of extracts release from the coal prior to the onset of the product degradation reactions (Begon et al. 2002). In addition, the ability to introduce fresh solvent throughout the liquefaction process will be beneficial in yielding high coal conversion. Given that the extracts will flow out to be collected, the solvent flow reactor can be manipulated by installing additional reactor to it. In this study, the second reactor filled with catalyst was installed, in order to increase the oil yield conversion. Depending on the coal rank and condition, the coal liquefaction processes are routinely being carried out at temperatures near or above 400°C at which the coal will be actively decomposed and the process is often written as:

\[
\text{Coal} + \text{H} = (\text{Pre-asphaltene} + \text{asphaltene} + \text{oil}) + \text{H}_2\text{O} + \text{gas}
\]

The role and the importance of catalyst in coal liquefaction are well known. The addition of active
catalysts improves total conversion and selectivity which will increase the yield of oils. This results in more efficient hydrogen consumption. Furthermore, the catalysts used will affect the reduction of reaction such as lower temperature, pressure and reaction time. Prasassarakich et al. (2007) studied that in the absence of catalyst, the oil yield decreased with temperatures above 410°C and the content of naphtha and kerosene increased while light gas oil and gas oil decreased with increasing temperatures. The use of an appropriate catalyst due to more severe thermolysis of the coal structure, results in an increase in the amount of the coal structure; thus, an increase in the amount of lighter and intermediate compounds at the expense of the higher molecular weight compounds.

Liquefaction of Mukah Balingian (MB) low-rank coal was carried out using semi-continuous solvent flow two-stage reactor system at temperatures above 400°C in order to study the effect of catalytic liquefaction by separating the coal and the catalyst in different reactors. Both extraction and radical reactions occurred as shown by the increase in the percentages of asphaltenes, pre-asphaltenes and oil+gas yield (Kassim et al. 2007). The second reactor filled with catalyst needed to break those asphaltenes and pre-asphaltenes. This solution will increase the oil+gas yield. In this paper, the results obtained from this process such as oil conversion and distribution of asphaltene and preasphaltenes at several parameters were discussed.

**EXPERIMENTAL**

**Sample Preparation**

The coal sample used in this study is MB Malaysian low-rank coal which originated in Sarawak, Malaysia. The procedure for coal preparation has been reported by Ishak et al. (2005). Briefly, the coal samples were ground and sieved through progressively into finer screen to obtain particle sizes of <212 μm, and dried in the oven for overnight to remove the inherent moisture. Table 1 represents the ultimate, proximate and maceral analyses of the MB coal sample. All the solvents and reagents used in this study were obtained from commercial sources and were used without further purification.

The catalyst was prepared using wet-impregnation technique (Venugopal et al. 2007). The support material used is fumed silica supplied by Aldrich Chemical and nickel nitrate (Ni(NO\textsubscript{3})\textsubscript{2}.6H\textsubscript{2}O) which is the precursor for the nickel. Briefly, the required amount of nickel nitrate is taken to give Ni loading catalyst of 5wt% with known amount of water in a 1000 ml beaker and mixed with the requisite amount of SiO\textsubscript{2} to yield the respective Ni mass percentages (i.e. 1, 3, 5 w/w%). The solution was dried and constantly stirred at 100°C, then the process of drying was continued for another 24 h. The dried sample was calcined in air at 700°C for 5 h.

**Coal Liquefaction**

The liquefaction experiments were carried out in two stainless steel tubing (40 ml and 20 ml). A schematic diagram for the semi-continuous two-stage reactor system is shown in Figure 1. For each experiment, 5 g of coal sample was loaded into the 40 ml tubing reactor. The second tubing reactor was filled with 5 g of catalyst. Nitrogen gas was charged into both reactors to remove any air left in the reactor. The solvent was pumped into the reactors first with the exit closed, to permit the build-up of the desired pressure and temperature. The heater was turned on to reach the desired temperature at the heating rate of 4°C/min. The reactor pressure was controlled by a back-pressure regulator valve to maintain a 4 MPa of pressure. Upon reaching the desire temperature, the solvent was pumped in at varied flow-rates.

The coal extracts were collected at various temperatures and various reaction times. At the end of each experiment, the furnace was withdrawn and the reactors were cooled down to room temperature. The coal residue was recovered by filtration and dried overnight in vacuum oven at 100°C. The weight loss of the coal sample after extraction on dry-ash-free (daf) basis defined the degree of coal conversion. The extracted product collected was then separated into oil (hexane-soluble), asphaltene (toluene-soluble, hexane-insoluble) and pre-asphaltenene (THF-soluble, toluene-insoluble) using a soxhlet extraction method. The hexane-soluble was put in a rotary evaporator under vacuum to remove the solvent and the oil obtained was analyzed using GCMS. The coal conversion and liquid yield were calculated using following expressions (Gozmen et al. 2002):

**Coal conversion**:

\[
\text{[wt. % daf]} = \frac{W_{\text{coal}} (1 - \alpha_{o}) - W_{\text{residue}} (1 - \alpha)}{W_{\text{coal}} (1 - \alpha)} \times 100
\]

**Product yields**:

\[
\text{[wt. % daf]} = \frac{W_{i}}{W_{\text{coal}} (1 - \alpha)} \times 100
\]

where, \(W_{\text{coal}}, W_{\text{residue}}\) and \(W_{i}\) were the weight of parent coal, residue coal and each constituent of extracts (i.e. asphaltenes, pre-asphaltenes and oil+gas); and \(\alpha_{o}\) and \(\alpha\) were ash content in parent coal and ash content in coal liquefaction residue, respectively. In this study no attempt was made to recover the tetralin in order not to remove some other volatile products as well; however, the oil+gas yield values were calculated by difference.

**Extracts and Residue Analysis**

The soxhlet extraction method was used to separate the coal extracts into oils, asphaltene, and pre-asphaltenene. Hexane, toluene and THF were used separately as solvents in this
 extraction method. The coal mixtures contained in the 1000 ml round-bottom flask was heated until a colourless solvent was observed in the soxhlet extractor (i.e. about 4 h). The hexane-soluble was then cooled down before the n-hexane was evaporated by using the rotary evaporator. Toluene was used to replace the hexane and the extraction was continued on the hexane-insoluble in the soxhlet extractor and the same procedure was applied in the extraction using THF. Figure 2 shows the schematic diagram of soxhlet extraction method.

The coal liquid was analyzed by Gas Chromatography Mass Spectroscopy (Varian Model CP-3800) according to ASTM D2887. The column used is CP8944, 30 m × 0.25 mm × 0.39 mm packed with VF-5ms. The oven temperature was raised from 60ºC to 270ºC at a constant heating of 5ºC/min. The separated compounds will immediately enter the mass spectrometer which generates the mass spectrum for the compounds. The carrier gas used was helium through a flow rate of 20 ml/min and the analysis was run in split mode at 30:70. The raw and the coal liquefaction residue were analyzed for proximate analysis using DTA/DSC TA Model SDT Q600 that follows the ASTM D2974. Carbon, hydrogen and nitrogen analyses were performed using an Elemental Analyzer (EA), model CHNS-932 series, with helium gas as the carrier and followed the ASTM D3172 for the ultimate analysis.

RESULTS AND DISCUSSION

Liquefaction Using Solvent-flow Reactor System

Effect of reaction temperature. Effects of the reaction temperature on the liquefaction of MB coal are represented in Table 2 and Figure 3. During this experiment, other parameters such as solvent flow-rate and reaction time were fixed at 1 ml/min and 30 min, respectively. It is equally important to determine the appropriate temperature for the liquefaction in order to enhance the coal conversion and oil yield. Figure 3 shows that the coal conversion increased with increasing temperature. It was also noted that the increased reaction temperature also increased the oil+gas yield. At low temperature (300ºC), the coal conversion was at the lowest and increased significantly until it reached 420ºC where the conversion began to linear up.

According to Karaca et al. (2001), the bond cleavage reactions in lignite and pre-asphaltene occurred simultaneously beyond 350ºC. Thus, it was possible that

<table>
<thead>
<tr>
<th>Exp</th>
<th>Extraction conditions</th>
<th>Effect of solvent flow-rate</th>
<th>Effect of reaction time</th>
<th>Effect of reaction temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1 30 420</td>
<td>78.9 12.4 11.9 54.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>3 30 420</td>
<td>78.1 12.0 16.5 49.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>5 30 420</td>
<td>78.4 11.4 17.3 48.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>1 30 420</td>
<td>77.9 12.4 11.9 54.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1 45 420</td>
<td>78.7 11.6 8.8 58.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1 60 420</td>
<td>79.4 11.9 7.3 60.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1 30 300</td>
<td>41.2 15.1 12.4 13.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1 30 350</td>
<td>59.5 15.6 13.5 30.4</td>
<td></td>
<td></td>
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<tr>
<td>9</td>
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<td>73.1 7.6 11.7 53.8</td>
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<tr>
<td>10</td>
<td>1 30 420</td>
<td>78.9 11.8 12.3 54.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>1 30 450</td>
<td>79.2 11.3 13.1 54.8</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

FR = Solvent flow-rate; RT = Reaction time; Temp = Reaction temperature; Total = Coal conversion; PAS = Pre-asphaltene; AS = Asphaltene; O+G = Oil+gas.
Figure 1. Schematic diagram of semi-continuous two-stage solvent flow reactor system.

Figure 2. A schematic diagram of soxlet extraction.
Figure 3. Effect of reaction temperature on product distribution yields (Nitrogen pressure — 4 MPa. See Table 2 for the other nomenclature).

Figure 4. Effect of reaction time on product distribution yields (Nitrogen pressure — 4 MPa).
during that temperature, all pre-asphaltene and asphaltene were converted into lighter molecular components of oil+gas. Therefore, it was definitive that the coal extraction tended to pre-dominate at lower liquefaction temperature, with radical reaction which started to occur at liquefaction temperature at or above 400ºC. According to Prasassarakich et al. (2007), increasing the temperature above 410ºC will cause a decrease in liquid yield. This was because when using high reaction temperature, the liquid was fragmented and formed free radicals and was stabilized by hydrogen to form the gas phase. However, further increasing of the temperature would result in more gas formation and less oil. Hence, it was suggested that 420ºC was the optimum temperature which resulted the high coal conversion and oil yield.

**Effect of reaction time.** The effect of reaction time on coal conversion and product yields of MB coal was also investigated and the results are shown in Figure 4 and Table 2. Apparently, the percentages of coal conversion at each different reaction time were relatively the same. There was a slight difference in the oil+gas yield as the reaction time increased. The data indicated that the reaction time affected only a little in enhancing the total conversion. A fair amount of coal conversion and oil+gas yield were observed with the lesser amount of reaction time which also could be improve by the introduction of catalyst in the liquefaction process. The less reaction time also meant less solvent used which is economical compared to the amount of solvent used with a longer reaction time.

**Effect of solvent flow-rate.** Another significant parameter that could improve in the development of coal liquefaction process is solvent flow-rate. In an attempt to achieve high coal conversion and oil+gas yield, it is important to ensure that enough fresh solvent is present during the extraction process. One option is by providing solvent into the reactor continuously during the liquefaction process. The effects of solvent flow-rate are shown in Figure 5 and Table 2. Generally, the coal conversions for three different flow-rates were relatively the same. The percentage of asphaltene increased while the percentage of pre-asphaltene was found to decrease with the increasing of solvent flow-rate.

These results indicated that by enabling the solvent to flow during liquefaction process, it was possible to extract more components out of the coal with high percentage of coal conversion (~80%) regardless of the different flow-rate of the solvent used. Furthermore, the presence of fresh hydrogen-donor solvent during the reaction process stabilized most of reactive radical species to form low molecular weight products such as asphaltene and pre-asphaltene, and also the light products of oil+gas. In other word, with a sufficient supply of fresh donor solvent during coal liquefaction, most of the asphaltene and pre-asphaltene were converted into oil+gas.

Similar results for the amount of solvent on coal conversion were reported by Simsek et al. (2001), where the extent of conversion of coal depends on how the radicals are being stabilized. Thus, the liquefaction yield would be high if there was sufficient hydrogen available, while the liquefaction yields would be low if there was insufficient availability of hydrogen which would cause the radicals to undergo re-polymerisation. The liquefaction performed with a high solvent flow-rate produced a low percentage of oil yield and a high percentage of asphaltene+pre-asphaltene. This might be due to the extraction during liquefaction which only dissolved, due to the less time, for the solvent to fully interact with the coal radical. In other words, less hydrogen was transferred during the liquefaction process, thus resulting in higher molecular weight compounds, which in this case is asphaltene and pre-asphaltene. Hence, more extensive formation of extractable materials was obtained at a lower solvent flow-rate.

**Liquefaction Using Two-stage Solvent Flow Reactor System**

The two-stage reactor was introduced to increase the percentage of oil yield. The second reactor was filled with NiSiO2, as the liquid tar passed through after the extraction process in the first reactor. In order to maintain the condition for both reactors, several parameters in the second reactor such as temperature, pressure and solvent flow-rate were fixed at 420ºC, 4 MPa and 1 ml/min, respectively.

The effects of Ni loading on the product distribution yields are shown in Figure 6. From Figure 6, it was clear that the oil+gas yield increased as the percentage of Ni loading increased. Higher molecular weight compounds such as pre-asphaltene and asphaltene decreased and were converted into lower molecular weights compound, that is, oil+gas. The second reactor did not affect the total conversion, thus, there was no difference in the total conversion of the catalyst.

Liquefaction was also conducted isothermally for a specific duration of time. Using the two-stage reactor system, the percentage of asphaltene and pre-asphaltene decreased and this correlated with the increased oil+gas yield. The assumption was that both asphaltene and pre-asphaltene have been converted into oil+gas. Generally, with the sufficient amount of hydrogen available, these free radical species generated from the coal cracking process combined with hydrogen to yield stable species such as asphaltene, pre-asphaltene and oil. The asphaltene and pre-asphaltene would further fragment to smaller molecular weight radical species, and then the hydrogenation process stabilized it to form oil+gas, a light molecular weight product.

The use of catalyst also increased the oil+gas yield. Both the hydrogenation reaction and hydrocracking of coal molecule occurred with the availability of catalyst.
Figure 5. Effect of solvent flow-rate on product distribution yields.

Figure 6. Effect of Ni loading on product distribution yields.
Another reason was that the low rank coals with their high concentrations of oxygen functional groups adsorbed the multi-charged metal cation through an ion exchange mechanism and this property is used for dispersing metal catalysts across a coal surface prior to extraction (Hu et al. 1998). The catalyst itself improves the reactivity of liquefaction by weakening the dependence of conversion on molecular hydrogen and hydrogen donor solvent (Hu et al. 2000). Thus, it would be beneficial in decreasing the hydrogen consumption during liquefaction and increasing the conversion of coal liquefaction.

Coal Liquid Analysis by GCMS

The aim of this section is to compare the chromatography spectra for the catalytic and un-catalytic coal liquefaction liquid. Theoretically, the reducing catalyst used would further reduce the coal extracts including coal liquid into smaller molecular compounds. By observing the difference in the chromatography spectra between those two coal liquids, the reducing effect of catalyst could be proved. Figures 7 and 8 show the results of coal liquid extract from coal liquefaction at a temperature of 420 ºC without and with catalyst by using GCMS, respectively.

For MB coal extraction, the existence of a complicated mixture and that of many compounds were identified. MB coal is a low rank coal having high carboxyl and/or sulphidic type functional groups and based on the previous study, the covalent breakage starts at temperatures between 400 ºC to 425 ºC (Begon et al. 2002). According to Prasassarakich et al. (2007), during coal liquefaction the main reactions were thermolysis, dehydrogenation, cleaving of cyclic hydrocarbon structures into an open-chain hydrocarbon and condensation reactions. Moreover, Prasassarakich et al. (2007) found that the improvement in coal liquefaction can be achieved by adding catalyst into the coal. Furthermore, he concluded that in the absence of a catalyst, the oil yield decreased and the content of naphtha and kerosene increased while the light gas oil and gas decreased. The presence of catalyst would benefit the formation of lighter components, kerosene and light gas oil.

From Figure 8, it can also be seen that there is a collection of compounds that appeared at the beginning of retention time. It showed that there are differences in compounds between these two chromatography spectra (Figure 7) which indicated that the catalyst did play its part in further reducing the molecular compounds in coal extracts. The major compounds that were identified (by mass spectrometer’s library) in coal liquid extraction for non-catalytic liquefaction were mostly alcohols and aromatic hydrocarbons. Benzene and naphthalene are two popular compounds in coal. Yagmur et al. (2008) found that most of the components in coal consist of condensed aromatic and oxygenous aromatic compounds during their study on liquefaction of Zonguldak bituminous coal using tetralin as solvent.

Furthermore, several aromatic compounds were found to decrease in intensity when the chromatography was compared for catalytic and non-catalytic coal extracts. Compounds such as 1-methyl-indan, 2-ethyl-2,3-dihydro-1H-indene, and 3,4-dihydro-1(2H)-naphthalenone have high intensity in non-catalytic coal extracts but low in

Table 3. Conversion, PAS yield, AS yield and O+G yield.

<table>
<thead>
<tr>
<th>Ni Loading (wt%)</th>
<th>Extraction conditions</th>
<th>Conversion (% daf)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>FR (ml/min)</td>
<td>RT (min)</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>30</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>30</td>
</tr>
<tr>
<td>7</td>
<td>1</td>
<td>30</td>
</tr>
</tbody>
</table>

Table 4. Proximate and ultimate analyses of raw and CLR of MB coal.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Analyses</th>
<th>Calorific value (MJ/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Proximate (wt% db)</td>
<td>Ultimate (wt% db)</td>
</tr>
<tr>
<td>Raw</td>
<td>VM</td>
<td>FC</td>
</tr>
<tr>
<td></td>
<td>44.7</td>
<td>51.1</td>
</tr>
<tr>
<td>CLR</td>
<td>27.9</td>
<td>65.0</td>
</tr>
</tbody>
</table>

VM = volatile matter; FC = fixed carbon; a = calculated by difference.
Figure 7. Chromatography of coal liquid extract from coal liquefaction without catalyst.

Figure 8. Chromatography of coal liquid extract from coal liquefaction with catalyst.
catalytic coal extracts. These correspond to the catalyst reaction in forming more lighter aliphatic components in coal liquefaction. Thus, by enabling the availability of catalyst, there would be more formation of lighter components.

**Proximate and Ultimate Analyses of Coal Residue**

In order to analyze the properties of coal before and after liquefaction, proximate and ultimate analyses were carried out on the coal liquefaction residue (CLR). The proximate and ultimate analyses of CLR of MB coal are shown in Table 4, with raw MB coal results for comparison. All liquefaction processes were carried out at the best condition (temperature of 420ºC; processing time of 30 min; solvent flow-rate of 1 ml min⁻¹; and pressure of 4 MPa).

From Table 4, it can be seen that the volatile matter of the CLR decreased when compared to the raw coal from 44.7 wt% to 27.9 wt%. However, the fixed carbon content for the CLR increased from 51.1 wt% to 65.0 wt% due to the releasing of volatile matter that occurred during liquefaction. In addition, an increase in value for the ash content was observed, i.e. from 24.6 MJ/kg to 27.6 MJ/kg.

The decrease of volatile matter corresponds to the increased fixed carbon and ash content which resulted in the increase of the heating value of the CLR. The increased value in the ash content of the CLR was due to the removal of organic matter from the coal liquefaction with mineral which is inorganic materials being concentrated in the coal residue (Ishak et al. 2005). From the results thus far, it showed that the coal underwent an upgrading process producing a CLR with higher calorific value. Thus, these results indicated that the CLR would be a good feedstock for gasification and/or combustion in coal upgrading processes as indicated by Sangon et al. (2006), Hu et al. (1998) and Williams (1981).

**CONCLUSION**

Liquefaction on MB coal was successfully carried out at three different variable parameters: temperature; reaction time; and solvent flow-rate. The first phase was liquefaction using an ordinary one-stage solvent reactor system which produced high coal conversion but there was 24% of the total conversion converted into high molecular weight compounds (asphaltene and pre-asphaltene). The second phase of this study was liquefaction using the two-stage solvent flow reactor system which consisted of NiSiO₂ as catalyst in the second reactor. It was found that an increase of 15% was achieved in the oil+gas yield from the conversion of asphaltene and pre-asphaltene. This showed that the use of catalyst could increase the oil+gas yield.

**ACKNOWLEDGEMENTS**

The authors would like to thank the Ministry of Science, Technology and Innovation, Malaysia (MOSTI) for the e-Science research grant (No: 03-01-01-SF0017) and Universiti Teknologi MARA.

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Electron-phonon Coupling Constants of Two-dimensional Copper Oxide-based High Temperature Superconductors

R. Abd-Shukor1* and W.Y. Lim1

The electron-phonon coupling constant of the copper oxide-based high temperature superconductors in the van Hove scenario was calculated using three known models and by employing various acoustic data. Three expressions for the transition temperature from the models were used to calculate the constants. All three models assumed a logarithmic singularity in the density of states near the Fermi surface. The calculated electron-phonon coupling constant ranged from 0.06 to 0.28. The constants increased with the transition temperature indicating a strong correlation between electron-phonon coupling and superconductivity in these materials. These values were smaller than the values estimated for the conventional three-dimensional BCS theory. The results were compared with previous reports on direct measurements of electron-phonon coupling constants in the copper oxide based superconductors.

Key words: Acoustic methods; electron-phonon coupling constant; van Hove scenario; transition temperature models

The understanding of the mechanism of superconductivity in the cuprates has been one of the major challenges in condensed matter physics since the discovery of this class of superconductors many years ago. Several models have been proposed throughout the years. Basically there are two opposing views on the possible mechanism. The first idea is based on the electron-phonon interaction and the other is based on the one-band Hubbard Hamiltonian. The electron-phonon interaction in the cuprates is said to be too weak to produce a high $T_c$. On the other hand, it is not easy to explain the thermodynamic properties of the cuprates with positive potential of the Hubbard model. A combination of both ideas to explain high $T_c$ superconductivity has been reported recently (Szczesniak et al. 2012).

Although the possible role of phonons in the cuprates has been abandoned earlier on, the important role of phonons in the pairing mechanism in the materials has regained attention in the past few years following a number of experimental and theoretical evidences (Lanzara et al. 2001; Reznik et al. 2006; Shimada et al. 2002). By taking into consideration the singularity in the density of states at the Fermi level for a two-dimensional system, recent works have shown that such a model can be viable for high temperature superconductivity (Newns et al. 1995).

A large effect can be produced near the van Hove singularity even for arbitrarily weak interactions (Vozmediano et al. 2002). Hence in principle, a very small electron-phonon coupling is sufficient for the formation of the Cooper pairs. It is interesting to determine the value of the electron-phonon coupling constant in the cuprate. In a previous paper we reported the electron-phonon coupling constant in the cuprates for the van Hove scenario (Abd-Shukor 2007). In this paper we calculated the electron-phonon coupling constant for the van Hove scenario derived from three expression of the transition temperature (Getino et al. 1993; Newns et al. 1992; Tsuei et al. 1990) and by using parameters determined from acoustic methods. The electron-phonon coupling constants were then compared to experimental results of direct measurements of the electron-phonon coupling constant.

Models

The density of states at the Fermi level in the van Hove scenario is given as:

$$N(E) = N_0 \ln \left| \frac{E_F}{E - E_F} \right|, \quad (1)$$

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where, $N_0$ is a constant and $E_F$ is the Fermi energy.

**Model 1.** Using the BCS expression for energy gap with the above density of states (Equation 1), and the approximations $\tanh \left[ \frac{E-E_F}{2k_BT_c} \right] = \frac{E-E_F}{2k_BT_c}$ for $|E-E_F| \leq 2k_BT_c$ and $\tanh \left[ \frac{E-E_F}{2k_BT_c} \right] = 1$ for $|E-E_F| > 2k_BT_c$, the transition temperature can be written as (Tsuei et al. 1990):

$$T_c = 1.36(10\theta_D) \exp \left\{ -\frac{2}{N_0V} \left[ \ln \left( \frac{k_B(10\theta_D)}{k_BT_c} \right)^2 + 1 - \ln(10) \right] \right\}, \quad (2)$$

where, $\theta_0$ is the Debye temperature = $T_c/10$, $N_0V = \lambda_{VH}$ and $T_F$ is the Fermi temperature. From Equation (2), the electron-phonon coupling constant can be written as:

$$\lambda_{VH1} = N_0V = \frac{2}{2coth\left( \frac{\theta_D}{2k_BT_c} \right) + \ln\left( \frac{D}{E_F^{1/2}} \right) + 1}, \quad (3)$$

**Model 2.** In another scenario Getino et al. (1993) used the energy gap equation:

$$\frac{2}{V} = \int_{E_F+\Delta_0}^{E_F+\Delta_0} \frac{dE}{\sqrt{(E-E_F)^2 + \Delta^2(T)}} = \frac{1}{N(E_F)} \times \tanh \left[ \frac{(E-E_F)^2 + \Delta^2(T)}{2k_BT_c} \right], \quad (4)$$

and the density of states as in (Equation 1) and showed that the transition temperature could be written as:

$$T_c = \frac{1}{2} T_F \exp \left\{ -\frac{1}{N_0V} + D \left( \frac{\theta_D}{2T_c} \right) \right\} \times \left[ 2coth\left( \frac{\theta_D}{2T_F} \right) + \ln\left( \frac{T_F}{E_F^{1/2} \theta_D} \right) + \frac{1}{2} \right], \quad (5)$$

From (Equation 5), the electron-phonon coupling constant could be written as:

$$\lambda_{VH2} = N_0V = \left\{ \left[ \ln^2 \left( \frac{2T_F}{E_F^{1/2}} \right) - \ln^2(10) \right] \right\}^{-1} \times 2coth\left( \frac{\theta_D}{2T_F} \right) - D \left( \frac{\theta_D}{2T_F} \right) 2T_c 2T_c, \quad (6)$$

**Model 3.** For the third model, Newns et al. (1992) derived the transition temperature:

$$T_c = 1.36 T_F \exp \left\{ -\ln^2 \left( \frac{D}{E_F^{1/2}} \right) + 1 \right\}, \quad (7)$$

by employing the conventional BCS expression together with density of states as in (Equation 1) and using the exchange of excitations with a characteristic low electronic energy scale $E^*$ given as $E^* = \frac{\sqrt{\Delta_0^2 + \Delta^2(T)}}{D}$ where $f$ is the fraction of the Brillouin zone occupied by the van Hove singularity, $Z$ is the electronic factor, $D$ is the slave boson propagator, $V_F$ is the electron coupling and $\xi$ is a constant. By using (Equation 7) and $E^* = 0.44D$ at $E = 0$ (Maximal transition temperature), $D = 1.6 \text{ eV}$, the electron-phonon coupling constant could be written as:

$$\lambda_{VH3} = 2 \left[ \ln^2 \left( \frac{T_F}{13.6 \theta_D} \right) - \ln^2 \left( \frac{D}{E_F^{1/2}} \right) + 1 \right], \quad (8)$$

In previous papers the electron-phonon coupling in a two-dimensional system where the van Hove scenario is applicable have been reported (Abd-Shukor 2002; Abd-Shukor 2007; Getino et al. 1993; Newns et al. 1992; Tsuei et al. 1990). In the weak coupling limit ($\lambda \ll 1$), $T_c$ can be written as $T_c = 2.72 T_F e^{1/\lambda}$ where, $T_F = \frac{E_F}{k_BT_c}$ (EF is the Fermi energy) Getino et al. (1992). In this paper the characteristic ratio of $T_F/\theta_D$ was given as 10 in the high-$T_c$ materials. In the high $T_c$ material, for example the La-Sr-Ca-Cu-O system, $T_F/\theta_D$ ranged from 7.4-13.9 and in the Y-Ba-Cu-O system $T_F/\theta_D$ can be as close to 11.7 (Krunakakarn et al. 1998). The ratio of $T_F/\theta_D = 10$ was the correct order of magnitude. $\theta_D$ values were obtained from our acoustic experiments (Yahya et al. 1999).

**EXPERIMENTAL DETAILS**

Samples (about 12.5 mm diameter and 2 mm thickness) were prepared via solid-state reaction method using high-purity (>99.9%) metal oxides and carbonates by sintering in air at various temperatures followed by annealing in oxygen and various gases.
The electrical resistance versus temperature measurements were carried out using the four-point probe technique with silver-paint contacts. XRD analyses using Siemens D5000 diffractometer with CuKα radiation were used to determine the phases. The Matec 7700 system which utilizes the pulse-echo-overlap technique was used to measure the sound velocity. The samples were bonded to a transducer using Nonaq stopcock grease. X-cut (longitudinal) or Y-cut (shear) quartz transducers were used. The frequency of operation was between 5 MHz and 10 MHz. The absolute longitudinal and shear velocities were evaluated at 80 K in an Oxford Instruments liquid nitrogen cryostat model DN 1711.

The sound velocity in an ideal void-free material was determined from the measured velocity, \( v_M \), using the relation, \( v_I = \frac{\rho_M}{\rho} v_M \), where \( v_I \) is the void free velocity and \( \rho_M \) is the theoretical density. The Debye temperature, \( \theta_D \), was estimated by using the standard formula,

\[
\theta_D = \left( \frac{h}{k} \right) \left( \frac{3N}{4\pi V} \right)^{\frac{1}{3}} v_m, \]

where \( \frac{3}{v_m} = \frac{1}{v_I} + \frac{2}{v_s} \), \( h \) is the Planck constant, \( k \) is the Boltzmann constant, \( N \) is the number of mass point and \( V \) is the atomic volume.

RESULTS AND DISCUSSION

The electron-phonon coupling constant \( \lambda_{\text{LVH}} \) (Abd-Shukor 2007), \( \lambda_{\text{LVH1}} \), \( \lambda_{\text{LVH2}} \) and \( \lambda_{\text{LVH3}} \) increased with the transition temperature (Figure 1). Table 1 shows the calculated electron-phonon-coupling constant of the three models which were almost similar to each other but are higher than the values reported in Abd-Shukor (2007). The electron-phonon coupling constants \( \lambda_{\text{LVH1}}, \lambda_{\text{LVH2}} \) and \( \lambda_{\text{LVH3}} \) were between 0.060 and 0.28. The electron-phonon coupling constant for the conventional BCS theory, \( \lambda_{\text{BCS}} \) is shown for comparison.

Detailed studies on the phonon and oxygen vibration modes in the CuO2 planes have been widely reported (Devereaux et al. 2004; Piekarz et al. 1999). It is interesting to note that the electron-phonon coupling constant for the breathing mode is \( \lambda = 0.02 \) (Devereaux et al. 2004), and it is of the same order of magnitude with the \( \lambda_{\text{LVH}} \) values determined in Abd-Shukor (2007) but slightly higher than the values calculated for the three models above \( (\lambda_{\text{LVH1}}, \lambda_{\text{LVH2}}, \lambda_{\text{LVH3}}) \).

The main physics in these materials lies in the CuO2 planes, where interplanar coupling is weak. The important
phonon modes in the plane are related to oxygen vibrations which are the breathing (in plane vibrations) and buckling modes (out of plane vibrations). For each mode there can be a diagonal and non-diagonal electron-phonon interaction. The non-diagonal electron-phonon interaction of the breathing mode has been shown to have attractive interaction leading to pair formation (Piekarz et al. 1999).

The three models discussed above might be somewhat similar and thus gave almost similar coupling constant. The reported experimental results of electron-phonon coupling constants for Bi based and RBa$_2$Cu$_3$O$_y$ (Devereaux et al. 1998; Friedl et al. 1990; Pattnaik & Newns 1989) was closer to the coupling constant $\lambda_{\text{VH1}}$ in Abd-Shukor (2007). Hence the model by Getino et al. (1992) used in Abd-Shukor (2007) was probably more applicable to the copper oxide-based superconductors. The electron-phonon coupling constant $\lambda_{\text{VH1}}$ was smaller than the three models ($\lambda_{\text{VH11}}, \lambda_{\text{VH12}}$ and $\lambda_{\text{VH13}}$) because in deriving the expression for $T_c$ and $\lambda_{\text{VH1}}$ the density of states were assumed as pure logarithmic singularity. The density of states in the three models ($\lambda_{\text{VH11}}, \lambda_{\text{VH12}}$ and $\lambda_{\text{VH13}}$) were modified logarithmic singularities and thus contributed to higher electron-phonon coupling constant. The electron-phonon coupling constant in this scenario was very small, which is surprising considering the high transition temperature. However, from our results on a broad range of cuprates and various reports, the small electron-phonon coupling constant might be sufficient and essential for the formation of Cooper pairs. The density of states diverged near the Fermi level and even very weak electron-phonon interactions could result in an unexpectedly large effect.

Our results also showed that the non-diagonal electron-phonon interaction of the breathing mode was important for the pair formation in these high temperature cuprate superconductors. This result might provide insights into the mechanism of superconductivity in the cuprates.

### ACKNOWLEDGMENTS

This research has been supported by a grant from the Ministry of Higher Education, Malaysia (ERGS/1/2011/STG/UKM/01/25) and Universiti Kebangsaan Malaysia (UKM-DLP-2011-018).

Date of submission: October 2012

Date of acceptance: January 2013

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Cellulolytic, Nitrogen-fixing and Phosphate-solubilizing Micro-organisms as Biological Indicators of Forest Soil Rehabilitation

A. Tang¹, S.K. Wong¹*, O.H. Ahmed² and N.M. Majid³

Deforestation often results in land degradation in the tropics. In such degraded areas, the native plant species are prone to fail in surviving because the degraded soil conditions are different from the original conditions (Kanowski et al. 2005). Rehabilitation of degraded forest is essential to prevent further degradation of the soil. Abundance of soil microbiota could serve as an essential biological indicator of soil health for rehabilitation success. An investigation was conducted to study the relationship between cellulolytic, nitrogen-fixing and phosphate-solubilizing microbial counts and age of rehabilitated forest. A random sampling design was used to obtain four replicates of five composite soil of 0–10 cm depth soil samples of 4, 9, 14 and 19-year-old rehabilitated forest. Three selective media: Congo red cellulose, nitrogen-free malate and calcium phosphate media were used for the enumerations of cellulolytic, nitrogen-fixing and phosphate-solubilizing microbes, respectively. Cellulolytic and phosphate-solubilizing microbes were counted based on the formation of clearing zones, while nitrogen-fixing microbes were based on the formation of blue halo on the respective media. There was positive linear relationship between age of the rehabilitated forest and microbial count. These findings revealed that the potentials of cellulolytic, nitrogen-fixing and phosphate-solubilizing microbial populations could be used as biological indicators of forest soil rehabilitation.

Key words: cellulolytic; phosphate solubilising; nitrogen-fixing; soil micro-organisms; forest age; forest soil rehabilitation

MATERIALS AND METHODS

Soil Sampling

This study was conducted at the rehabilitated forest areas at Universiti Putra Malaysia, Bintulu Sarawak Campus. Soil
samples (0 cm – 10 cm) were obtained from the 4, 9, 14 and 19 year-old forests through random sampling design. Each site consisted of demarcated 20 m × 20 m area. Samples were collected in 3 composites, each consisting of 5 sampling points with 4 subsamples per point. Samples were sieved using 2 mm-sieve and diluted before plating on three types of selective media. Soil moisture content was determined gravimetrically by oven drying 10 g of fresh sieved soil for at least 24 h at 105ºC. Ten g of sieved soil was added to 95 ml of sterile 0.85% saline solution and agitated at 180 r.p.m. for 10 min prior to serial dilutions for microbial enumeration purposes.

Enumeration of Nitrogen-fixing Micro-organisms

Nitrogen fixing micro-organisms were grown on nitrogen-free malate (Nfb) medium (Döbereiner & Day 1976). The Nfb plates were incubated at 30ºC for 7 days. Colonies with a blue halo were counted in triplicates and the colony forming unit (cfu) number was calculated.

Enumeration of Phosphate Solubilizing Micro-organisms (PSM)

PSM enumeration technique is based on the formation of halos around the colonies of micro-organisms capable to solubilize calcium phosphate (Nautiyal 1999; Gupta et al. 1994). The micro-organisms were grown in medium containing insoluble phosphate (Kokal 1990; Buis 1995) and incubated at 30ºC for 12 days.

Enumeration of Cellulolytic Micro-organisms

The cellulolytic micro-organisms were enumerated in triplicate by spreading plate inoculation onto cellulose-Congo red agar (Hendricks et al. 1995). Following incubation at 30ºC for 7 days, the colonies exhibiting zones of clearing were counted.

Calculations and Statistical Analysis for Enumeration of Micro-organisms

The number of colonies per plate for the dilution giving between 30 and 300 colonies was averaged (Germida & de Freitas 2008). The number of cfu/g dry soil was determined as follows:

\[
\text{cfu/g soil} = \frac{\text{(Mean plate count)} \times \text{(Dilution factor)}}{\text{Dry weight soil of initial dilution}}
\]

Where,

\[
\text{Dry weight soil} = (\text{Weight moist soil of initial dilution}) \times (1 - (\% \text{moisture, soil sample/100})]
\]

The counts of nitrogen fixing, phosphate solubilizing and cellulolytic micro-organisms detected by the respective media were analyzed statistically using trend analysis. Statistical Analysis System version 9.1 was used for the statistical analysis. The decision level for hypothesis testing was at \(p \leq 0.05\).

RESULTS

The experimental data revealed the positive linear relationship between age of the rehabilitated forest and microbial counts. The adjusted \(R^2\) was highest for the relationship between forest age and cellulolytic microbial count at a value of 0.84 (Figure 1), followed by the relationship between nitrogen fixing microbial count and age (\(R^2 = 0.83\)). The lowest degree of relationship was observed between phosphate solubilizing microbial count and forest age at \(R^2\) value of 0.78. Cellulolytic microbial count (Count_c) with forest age had a functional relationship of \(\text{Count_c} = 25.31 + 1.94\text{Age}\), while nitrogen-fixing microbial count (Count_n) with age had its functional relationship as \(\text{Count_n} = 15.23 + 1.07\text{Age}\). Functional relationship, \(\text{Count_p} = 6.74 + 1.11\text{Age}\) was used to describe phosphate-solubilizing microbial count and forest age relationship.

DISCUSSION

It was observed that there was a general trend of increasing cellulolytic, nitrogen-fixing and phosphate solubilizing microbial counts over the ages of forest (Table 1 and Figure 1). Many factors affect soil microbial population. It is well known that the interplay of varieties of factors such as physicochemical properties of soil, temperature, vegetation, carbon availability and environmental conditions greatly affect soil microbial flora in terms of its numbers and activities (Sulasih 2005; Jha et al. 1992; Setiadi 1989). Changes in the number of soil micro-organisms can affect changes in the organic matter content of soil (Odum 1965; Zak et al. 1994) and hence when observed altogether with other ecological indicators such as biomass, and species diversity measurements etc., changes in the numbers of these micro-organisms can also act as an indicator of changes in soil health and productivity (Jastrow & Miller 1991; McBride et al. 1993).

Findings of Matias et al. (2007) revealed that soil phosphate solubilizing and cellulolytic population were inhibited by soil disturbance while the total bacterial and fungal population were not changed in the degraded soils of iron-ore mined land. It was also reported that micro-organisms exhibiting nutritional variability were less negatively impacted by lack of soil nutrition rather than those that need a specific substrate for their growth (Matias et al. 2007). Matias et al. (2007) stated that microbial life in degraded lands such as mined lands grows only in collaboration with the root systems of plants. Since vegetations have varying chemical composition, physiological and nutritional properties, they would be...
drawn by specific groups or species or organisms, which may be beneficial (Katznelson et al. 1948).

The ability of micro-organisms to react quickly to changes enables their rapid adaptation to environmental conditions (Nielsen & Winding 2002). Thus, microbial analyses resulted from the adaptation allow for discrimination in soil health assessment, and resulting altered microbial populations and activities may therefore provide an excellent indicator of change in soil health (Kennedy & Papendick 1995; Pankhurst et al. 1995). While studies have indicated that free-living nitrogen-fixing micro-organisms may be suitable as biological indicators of heavy metal toxicity in soil in terms of their enzymatic activities (Martensson & Witter 1990; Brookes et al. 1986), the results obtained in this study might also prove some potentials of nitrogen-fixing micro-organisms as soil health indicator in rehabilitated forest.

**CONCLUSION**

The soil microbial counts of cellulolytic, nitrogen-fixing and phosphate solubilizing microbes increased with increasing age of the rehabilitated forest. Among the three microbial populations, cellulolytic microbes had demonstrated the highest potential which could be used as biological indicator in regards to the rehabilitation degree or soil health of rehabilitated forest.

**ACKNOWLEDGEMENTS**

This research is funded by the Fundamental Research Grant Scheme (FRGS, 07-04-10-908FR) under the Ministry of Higher Education, Malaysia.

Date of submission: May 2011
Date of acceptance: January 2013

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FES Microbiology Letters


FEMS Microbiology Letters


Investigations on Physical Characteristics of Three-dimensional Coil Structure for MEMS Magnetometer

N. Sulaiman* and B. Y. Majlis

Measurement of low magnetic field has played an important role in many electronics applications such as navigation, military, non-destructive test, traffic detection as well as medical diagnosis and treatment. The presence of magnetic field, particularly its strength and direction, can be measured using magnetometer. There are many types of magnetometers being investigated through the years and one of the prominent types is fluxgate magnetometer. The main components of fluxgate magnetometer consisting of driving coils, sensing coils and magnetic core are developed by MEMS silicon processing technology. In this paper, an investigation on physical characteristics of the three-dimensional coil structure for a micro-scaled fluxgate magnetometer is presented. The physical characteristics such as width of the coil, distance between successive coils, and gap between the top and bottom coils which would influence the magnetic energy in magnetometer is discussed. In this work, finite-element method simulations to investigate the physical characteristics of the sensing coils were carried out, where the parameter of interest is the coils’ inductance as well as the magnetic flux density. Based on the simulation results, the varying of physical characteristics of the coils had its effects particularly in coil inductance, magnetic flux density, and magnetic energy. It could also be seen that the simulated results agreed with the theoretical aspects of magnetism in a coil. From the investigations, suitable coil dimensions were proposed.

Key words: Coil; magnetic flux density; energy; MEMS device; fluxgate magnetometer; simulation

Magnetometer is a device mainly used to measure the strength and direction of magnetic fields. It has a wide range of application which includes space exploration, navigations, geology studies, geophysics analysis and medical diagnosis. Many types of magnetometer with different principle of operation have been invented. Fluxgate is a type of magnetometer in which the principle of operation is based on second harmonics detection of voltage induced in sensing coil (Ripka 2003).

In recent years, the miniaturization of fluxgate magnetometer has come into interest due to the advantage of its smaller size, less power utilization and low fabrication costs. Miniaturized fluxgate with different coil designs have been reported that includes spiral single layer planar (Yunas et al. 2010), multilayer/double coil (Atta 2004; Ripka et al. 2001), double axis type (Baschirotto et al. 2007), toroidal type (Dezuari et al. 1999), and three-dimensional solenoid type (Liakopoulos et al. 1999; Wang et al. 2006; Lei et al. 2009).

The performance of fluxgate is closely related to the coil design type. This is due to the fact that different coil design produces different inductances, and magnetic flux density which among others are the parameters that influence the performance of the device.

In this paper, we investigated the physical characteristics of three-dimensional coils and its correlation with magnetic energy. The investigation was done by means of FEM simulation. The parameters of interest in the simulation were coil inductance and magnetic flux density. Information from the simulation results could aid in proper geometry design of three-dimensional coils.

THREE-DIMENSIONAL COIL

Fluxgate coils consist of a driving coil and a sensing coil. In order to achieve three-dimensional structure, the coils are designed in such a way that it consists of three parts

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— bottom conductor, via, and top conductor. The chosen material for the coils was Cu, because of its excellent conductivity.

Illustrations of the three-dimensional coil structure are shown in Figure 1. The layer structure from bottom upwards, starts with a substrate of alumina ceramic or glass. This is followed by a metal layer comprising of a bottom conductor. Next, is the insulation layer used to insulate between the bottom and top metal layers. The three-dimensional structure is then completed with the top metal layer which made up of the top conductor. The top and bottom conductors are connected by means of vias.

SIMULATION

Investigations on the physical characteristic of the coils were executed by using FEM simulation software. The aim of the simulation was to measure the inductance and magnetic flux density of the coils to manipulate three physical characteristics of the coil i.e. width of the coil; distance between successive coils; and the gap between the top and bottom coils.

First, we modelled the geometry of three-dimensional coil by using draw function. The initial dimension of the coils' width, distance between successive coils, and gap between the top and bottom coils were 20 µm, 50 µm and 40 µm, respectively.

The thickness of the coils were set at 20 µm and the number of windings were 2. Windings were kept at a minimum to simplify the modeling geometry, model meshing and solving processes. The current passing through the coils was set at 1A for all conditions.

Figure 2 illustrates the model and defines the three physical characteristics of the coil to be manipulated by means of simulation.

After geometry modeling, the next step was to set the module and application mode for simulation process. Since the aim of the simulation was to determine the inductance and magnetic flux density at constant current, the module and application mode chosen were AC/DC module and magnetostatics.

Mesh process was done after the model Physics settings in which the subdomain and boundary of the model had been set. Consecutively, the solving process was executed after the meshing. Table 1 summarizes the simulation processes in this work.

As a preliminary study, this model did not include the magnetic core. This is to simplify the simulation process.
Figure 2. Model of the three-dimensional coils and definition of physical characteristics.
and we only focused on the behavior of certain parameters at different physical characteristics of the coil structure.

RESULTS AND DISCUSSION

The output of a fluxgate is actually induced voltage at sensing coils and given as:

\[ V_{\text{ind}} = N A \frac{dB}{dt} \]  

(1)

The complexity of Equation 1 is increased to take into account the magnetizing and demagnetizing of the magnetic core (Ripka 2001). Equation 1 then becomes:

\[ V_{\text{ind}} = N A \mu_o H_o \left(1 - D\right) \frac{d\mu(t)}{dt} \]  

(2)

where, \( H_o \) is outside of the core magnetic field while \( D \) is the demagnetizing factor.

However, in this study, magnetic core is excluded to reduce the complexity. With the exclusion, the structure design of the three-dimensional coils resembles the solenoid. Hence, basic equations related to the induced voltage of the solenoid can be applied.

Based on Faraday’s law of induction, induced voltage, \( V_{\text{ind}} \), can be determined as follows:

\[ V_{\text{ind}} = -N \frac{d\Phi}{dt} \]  

(3)

and also,

\[ V_{\text{ind}} = -L \frac{dI}{dt} \]  

(4)

where, \( L \) is the inductance, with current, \( I \) and number of turns, \( N \). Magnetic flux is denoted as \( \Phi \). It can be seen that, the induced voltage is directly proportional to the inductance, and number of turns.

Magnetic energy in a coil is given as:

\[ E_m = \frac{L I^2}{2} \]  

(5)

Physical characteristics of the three-dimensional coil structure are: width of the coil; distance between successive coils; and gap between the top and bottom coils. Simulations were done based on the three physical characteristics of the coils.

Width of Coil

In this part, only the width of the coil is varied and parameters such as inductance and magnetic flux density are determined. Table 2 summarizes simulation settings for this part.

The resulting plot between inductance, magnetic energy and magnetic flux density against varying width of coil is shown in Figure 3. It can be seen that inductance decreases as the width of the coil increases.

There is an obvious explanation. As the width of the coil increases, the cross-section of the coil also increases. This leads to higher conductivity, less resistance and inductance. Magnetic energy is half the values of inductance when

<table>
<thead>
<tr>
<th>Step</th>
<th>Process</th>
</tr>
</thead>
</table>
| Step 1 | Geometry modeling  
Thickness: 20 µm  
Initial width: 50 µm  
Initial gap: 40 µm |
| Step 2 | Module and application setting  
AC/DC module  
Magnetostatic application |
| Step 3 | Physics settings  
Sub-domain  
Boundary |
| Step 4 | Mesh |
| Step 5 | Solve |
current $I$ is kept constant, therefore it follows the same pattern as inductance plot.

Magnetic flux density is simulated at point $X$ between the top and the bottom coil. The illustration can be seen in Figure 4.

As the width of the coil increases, the magnetic flux density at point $X$ also increases. This is because the area where point $X$ is located becomes nearer to the coil, and hence increases the magnetic flux density value.

**Distance Between Successive Coil**

Distance between successive coils is varied while others are kept constant. Table 3 summarizes simulation settings. Figure 5 shows the resulting plot between inductance, magnetic energy and magnetic flux density against varying distance between successive coils.

From the plot, inductance, magnetic energy and magnetic flux density decreases with the increase of distance between successive coils.

Increase in the distance between successive coils denotes increase in the distance between turns. This results in less flux linkage between coils and causes less inductance.

Magnetic flux density as simulated at point $X$ of Figure 4, decreases due to the distance between successive coils increases. The further point $X$ from the coil, the less flux density becomes.

**Gap between the Top and Bottom Coil**

The gap between the top and bottom coil is varied to observe the effects on inductance and magnetic flux density. The simulation settings are shown in Table 4. Figure 6 shows the plot between inductance, magnetic energy and magnetic flux density against the gap between the top and bottom.

Both inductance and magnetic energy increases as the gap increases. When the gap between the top and bottom coils increases, so does the diameter of the winding. The increase in the winding diameter $A$, causes the increase in inductance based on the following basic Equation 6.

$$L = \frac{N^2 \mu A}{l} \quad (6)$$

Magnetic flux density at the same point $X$ decreases as the gap increases. This is mainly because point $X$ becomes further from the coil as the gap increases.

**CONCLUSION**

The physical characteristics of three-dimensional coils have been investigated using FEM software. The changing of the dimension size of the coil had an effect on inductance, magnetic energy and magnetic flux density.

In this work, the inductance value increased as the gap between the top and the bottom coils and the width of the coils was increased. While inductance decreased as the distance between successive coils increased. High inductance was preferable however it was not the sole condition to the performance of fluxgate magnetometer.

The increase in coil width had a positive effect in increasing the magnetic flux density but at the same time it decreased the inductance and magnetic energy. Increase in the width of the coil also meant an increase in the overall space-area of the occupied device which defeated the purpose of miniaturization of devices. With these conflicting requirements, moderation in coil design was necessary.

Based on the investigation and having moderation in mind, the following was proposed:

- The width for coils could be 30 µm – 50 µm;
- The distance between successive coils could be 40 µm – 60 µm. Since the distance contributed towards the area size of the device, moderation is significant;
- The gap between the top and bottom coils could be 40 µm – 60 µm.

### Table 2. Width of coil simulation settings.

<table>
<thead>
<tr>
<th>Physical characteristics</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width of coil</td>
<td>Varying dimensions (20, 30, 40, 50, 60, 70, 80) µm</td>
</tr>
<tr>
<td>Distance between successive coils</td>
<td>Constant at 50 µm</td>
</tr>
<tr>
<td>Gap top and bottom coil</td>
<td>Constant at 40 µm</td>
</tr>
<tr>
<td>Current</td>
<td>Constantly at 1 A</td>
</tr>
</tbody>
</table>
Figure 3. Inductance, magnetic energy and magnetic flux density against width of coil.
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Figure 4. Magnetic flux density simulated point.

Table 3. Distance between successive coil simulation settings.

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width of coil</td>
<td>Constant at 20 µm</td>
</tr>
<tr>
<td>Distance between successive coils</td>
<td>Varying dimensions (20, 30, 40, 50, 60, 70, 80) µm</td>
</tr>
<tr>
<td>Gap between top and bottom coil</td>
<td>Constant at 40 µm</td>
</tr>
<tr>
<td>Current</td>
<td>Constantly at 1 A</td>
</tr>
</tbody>
</table>

Table 4. Gap between top and bottom coil simulation settings.

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width of coil</td>
<td>Constant at 20 µm</td>
</tr>
<tr>
<td>Distance between successive coils</td>
<td>Constant at 50 µm</td>
</tr>
<tr>
<td>Gap between top and bottom coil</td>
<td>Varying dimensions (20, 30, 40, 50, 60, 70, 80) µm</td>
</tr>
<tr>
<td>Current</td>
<td>Constantly at 1 A</td>
</tr>
</tbody>
</table>
Figure 5. Inductance, magnetic energy and magnetic flux density against distance between successive coils.
Figure 6. Inductance, magnetic energy and magnetic flux density against gap between top and bottom coils.
ACKNOWLEDGEMENTS

The authors would like to thank the Institute of Microengineering and Nanoelectronics and Universiti Kebangsaan Malaysia for the support and facilities.

Date of submission: May 2011
Date of acceptance: November 2012

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In the study of adhesives and their applications, it is important to understand the concept of the glass transition temperature, \( T_g \). As the temperature rises above the \( T_g \), the adhesive becomes more rubber-like because bonds between polymer chains become weak and the polymer becomes soft. Thus, knowledge of \( T_g \) is essential in the selection of materials for various applications. The use of thermal characterization studies on thermosetting resin materials helps to determine the processing properties of the adhesives. Thermal analysis measures chemical or physical changes as a function of temperature. These measurements allow access to processing and performance information relating to adhesives and composites. Properties obtainable include gel points, glass transition temperatures, reaction rates and cure kinetics, effects of individual or combinations of components, polymer stability and material life predictions.

There are many thermal analysis techniques available in the market but most frequently used are dynamic mechanical thermal analysis (DMTA), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and thermomechanical analysis (TMA). Much information regarding thermal analysis and its applications appears in the literature (Prime 1982; Mafi 2005). DSC which measures the heat flows into and out of a sample as a function of temperature has been extensively used to study the cure kinetics of various thermosetting polymers (Montserrat 1993; Thiagarajan et al. 1990). DSC is the most common technique for evaluating changes in \( T_g \) and cure state of thermosetting polymers (Turi 1981). However, DSC does not provide information on structural changes at the molecular level. By contrast, the DMTA technique imposes a small oscillatory deformation which generates viscoelastic materials properties such as: the storage modulus \( E' \); loss modulus \( E'' \) and the mechanical loss tan \( 
\delta (E''/E') \), all of which are characteristic of the changes of the material’s structure during cure. The DMA technique involves in the measurement of storage and loss modulus in shear, tension, compression or bending for a range of temperature and loading (frequency, amplitudes) conditions. The measurement of the loss modulus provides a quantitative method for the identification of mechanical relaxation temperatures (such as \( T_g \) and sub-\( T_g \)).
addition, examination of the frequency-dependence of the relaxation temperatures allows the determination of activation energies of the associated transitions (White et al. 2002). DMTA has been used extensively to characterize the thermal and mechanical properties of cured thermosets and cured thermoset-based composites (Lesser & Crawford 1997; Crawford & Lesser 1998).

DMTA can be used to study the effect of cross-linking. When a polymer is cross-linked, covalent bonds are formed between the polymer chains, which bring them closer together. This reduces the free volume and causes an increase in the $T_g$ (Ward & Hardley 1993). Another effect of cross-linking is to cause a broadening of the loss factor peak; to the extent that very highly cross-linked materials have no glass transition at all since the motion of long segments of the chain is not possible. This can also be seen as a decrease in the amount by which the storage modulus decreases at $T_g$ (Ward & Hardley 1993).

The cross-linking of epoxy adhesives is achieved through the addition of hardener. Depending on the type of hardener used, the final properties of epoxy network are different (Pascault et al. 2002). When the curing agent is an aliphatic amine, the curing process occurs at room temperature, but it is slow and often incomplete. Following initial cure the polymer network has a low glass-transition temperature ($T_g$) and high ability to absorb water (Prolongo et al. 2006). When this type of adhesive is exposed to high temperature and high humidity, the molecular structure is altered, causing dimensional changes and reduction or increase in $T_g$ (Moy 1980).

Bonding of rods into timber members has been investigated by Broughton and Hutchinson (2001), they indicated that efficient, high strength joints can be made with epoxy adhesives due to their capability to produce thicker gluelines. There are many types of adhesives available in the market but among the studies conducted, connection made using CB10TSS adhesive showed superior performance (Harvey & Ansell 2000). However, CB10TSS has Tg of 31.7°C and is considered low for service temperature. Hence the previous work by researchers (Ahmad et al. 2006, 2009, 2010) were concentrated in modifying the CB10TSS by adding nano- and micro-particles and found that the additions of nano filler (liquid rubber) has improved the tensile strength by 36% and the addition of micro-fillers has increased the modulus of elasticity by more than 300% however, reduces the tensile strength by 23%.

Therefore this study investigated the thermal properties of epoxy-based adhesive reinforced with nano- and micro-particles before and after exposure to the different environmental conditions, namely; 20°C/95%RH, 30°C/95%RH, and 50°C/95%RH (to study the effect of temperature), 20°C/65%RH, 20°C/75%RH, 20°C/95%RH (to study the effect of humidities), soaked in water (to determine the effect of moisture alone) and placed in the oven at 50°C (to study the effects of high temperature alone) for 90 days.

**MATERIALS AND METHODS**

**Materials**

The adhesive system under investigation is a thixotropic, non-Newtonian two-part, epoxy gap-filling adhesive system. The base adhesive, CB10TSS, is a mixture of diglycidylether of bisphenol-A + (DGEBA/F) and monofunctional and di-functional reactive epoxy diluents together with treated silica fume nano-particles (3.5 weight%) which control thixotropy. The curing agent contains polyetheramines combined with a rheology modifier. The other three adhesives were formulated by modifying the standard adhesive with the addition of either liquid rubber (carboxyl-terminated butadiene and acrylonitrile (CTBN) at 2 weight%) or micro-particles (ceramics particles, a mixture of bentonite, quartz and mica) and these adhesives were designated as Albipox and Timberset, respectively. The rheology and the mechanical properties of these adhesives have been reported by various authors (Ahmad et al. 2006; 2009; 2010).

**Preparation of Specimens**

The adhesives were mixed manually and then the mixture was transferred into a plastic rectangular mould of 3 mm thick, 500 mm wide and 500 mm long (Figure 1). The mould was coated with release agent. Another glass plate was placed on top of the mould and weights were added to apply some pressure in order to achieve a flat surface.

The adhesive was left to cure for 10 days. After 10 days of curing, the samples were removed from the moulds and left to cure for further 15 days at room temperature. The adhesive plates were cut using a diamond cutter into bars with dimensions of approximately 5 mm width × 3 mm thickness × 20 mm length. The DMTA specimens were arranged on the plastic shelves inside the chamber. In order to make sure that the environmental chamber provides the heat and humidity as specified, the temperature and humidity were monitored using the built-in temperature and humidity meter and from time to time, the temperature and humidity was counter checked with thermometer, external humidity meter and humidity indicator. The specimens were kept in the chamber for 90 days.

**Test Methods**

*Viscosity measurements.* Rheological tests were carried out by employing a cone and plate method using a Bohlin CS Rheometer at various shear rates at 25°C. The angle
and radius of the measuring cone were 4° and 20 mm, respectively. The height of the tip of the cone and the plate was set at 0.15 mm.

The adhesives were prepared in accordance with the manufacturer’s instructions with due regard given to handling precautions for the adhesives. The two-part adhesive was prepared by mixing the base adhesive and the hardener using a rotary stirrer at constant speed for 5 minutes. The adhesive was left to rest for 2 min before placing a small amount of adhesive on the plate sufficient to fill the gap between the upper and lower elements. Shear stress and viscosity were recorded as a function of shear rate.

Dynamic mechanical thermal analysis. The dynamic mechanical properties of the three types of adhesives were measured using a Trritec 2000 DMTA analyzer. The sample was gripped as a cantilever beam between two clamps in the DMTA system (Figure 2).

A sinusoidally alternating force was applied to the end of the cantilever beam at the center of the equipment at a constant frequency of 1 Hz and the temperature was ramped up at a constant rate of 2°C/min from −50°C to 180°C.

Scanning electron microscopy. The broken samples before and after exposure to different environments were examined. The fracture surface of adhesives was inspected.

![Figure 1. Mould for DMTA specimens.](image1)

![Figure 2. Beam sample clamped in single cantilever mode.](image2)
RESULTS AND DISCUSSIONS

Effect of nano- and Micro-particles Additions on the Rheological Properties of the Adhesives

In order to examine the effect of micro- and nano-fillers on the viscosity of adhesive formulations, rheological measurements were carried out at 25°C in a Bohlin viscometer. The viscosity values for the CB10TSS, and Albipox are shown in Figure 3 for different rates of shear and the values recorded were confirmed as valid by the Bohlin software.

From these graphs it is evident that the viscosity of each adhesives decreases with increase in shear rate, i.e. each adhesive behaves like a non-Newtonian fluid. In order to confirm that the adhesive behaves thixotropically the adhesives need to be subjected to increasing and decreasing shear to generate a hysteresis loop.

However this was not done due to the limitations of the equipment. However by observation and during handling, the CB10TSS was found to be thixotropic in practical use. The viscosity of Timberset was not determined as it has a very high viscosity and crosslinks rapidly so damage to the equipment would have occurred. The time for the shearing to take place at a constant rate of increase in shear rate varied from one adhesive to another.

CB10TSS is much easier to shear compared to Albipox as it required less shear stress to reduce its viscosity. The Albipox matrix contains a rubbery phase and showed the least pronounced pseudoplasticity (i.e. the viscosity decreasing with increasing shear rate) and it had the highest viscosity at low shear rates, indicating physical interaction between the epoxy and the rubbery phases. It is possible that cavitation or debonding of rubber particles took place which increased the resistance of the adhesive formulation to flow under shear.

Effect of Nano- and Micro-particles Additions on the Thermal Properties of Adhesives

The effect of filler additions on the dynamic properties of based adhesive, CB10TSS was then compared. Figure 4 shows the plots of storage modulus, E’ and tan δ versus temperature for the different adhesive systems used. The storage modulus E’ represents the elastic properties and demonstrates the stiffness. The storage modulus values were not constant in glassy region but slightly decreased with temperature. As pointed out by Lewis and Nielsen (1970), the residual stress field due to different thermal expansion coefficients of matrix and filler may induce relaxations in the polymeric phase which account for the negative slope of E’ versus temperature in the glassy region. According to Mohd Ishak and Berry (1994), the relaxation peak is assigned to the breakage of hydrogen bonds between the polymer chains which induces long range segmental motion in the amorphous area. The whole curve of the dynamic storage modulus versus temperature consists of three stages. (The curve for Timberset is used as an example to explain the general characteristics).

The stages are: glassy (0°C – 55°C); highly elastic (55°C – 65°C) and viscous (55°C – 120°C). In the first stage, due to the gelation of the macromolecule movement chains (Yao et al. 2005), the modulus of Timberset is between 14 GPa to 17 GPa, which shows a high stiffness. In second stage, the molecule chains starts to move freely, and the modulus decreases rapidly to 1 GPa because of the large deformation of the material.

In the third stage, the whole molecular chain causes slippage movement and the viscous flow leads to an irreversible deformation, therefore the modulus suddenly falls to zero. From Figure 4, it can also be seen that, on the whole, the dynamic mechanical behaviour of the three adhesives is similar. The differences are in the storage modulus and the temperature of the main transition from elastic to viscoelastic behaviour, which are related to the types of nano-filler since the base adhesives are the same. The ceramic particles in Timberset, show a remarkable stiffness effect on the adhesive with the highest E’ value (Figure 4a). At 20°C, the storage modulus of all the adhesives are in the order of Timberset > Albipox > CB10TSS. This observation agrees with the values for the static elastic modulus obtained from authors’ previous work (Ahmad et al. 2010) as shown in Table 1.

The dynamic modulus was found to be higher than static modulus by a factor of 1.6 to 3.6 times higher. The modulus in the rubbery plateau region is proportional to either the number of crosslinks or the chain length between entanglements (Menard, 1999). For the three adhesives other than Timberset the storage moduli are similar and very small in the rubbery zone. The rubbery plateau modulus for Timberset is found to be higher than the other adhesives.

The addition of high modulus ceramic particle has allowed Timberset to retain a higher modulus even at a temperature above the glass transition. Timberset is again seen to have the highest E’ peak value at 58.1°C (Figure 4b) which means there is large amount of damping in the material as a result of the high fraction of filler particles.

The glass transition temperature of the adhesives were determined based on the onset E’ value from E’ versus temperature curves and the T_g values for CB10TSS is
Figure 3. Viscosity and shear stress plotted versus shear rate for (a) CB10TSS, and (b) Albipox.
Figure 4. Graphs of: (a) storage modulus, $E'$, (b) loss modulus, $E''$, and (c) $\tan \delta$ versus temperature for all adhesive (control specimens).

Table 1. Thermal and mechanical properties of adhesives.

<table>
<thead>
<tr>
<th>Adhesive types</th>
<th>$T_g^*$ ($^\circ$C)</th>
<th>$E''$* (GPa)</th>
<th>Bending strength*** Mean (%)</th>
<th>COV (GPa)</th>
<th>MOE**** Mean (%)</th>
<th>COV</th>
</tr>
</thead>
<tbody>
<tr>
<td>CB10TSS</td>
<td>31.7</td>
<td>8.8</td>
<td>62.7</td>
<td>8.67</td>
<td>2.4</td>
<td>5.37</td>
</tr>
<tr>
<td>Albipox</td>
<td>42.8</td>
<td>9.2</td>
<td>80.9</td>
<td>5.79</td>
<td>3.6</td>
<td>4.99</td>
</tr>
<tr>
<td>Timberset</td>
<td>53.8</td>
<td>15.0</td>
<td>47.1</td>
<td>5.80</td>
<td>9.3</td>
<td>10.29</td>
</tr>
</tbody>
</table>

* $T_g$ based on onset $E'$ curve.
** Storage modulus values measured at 20$^\circ$C in GPa.
*** Bending strength.
**** MOE values from static bending test (Ahmad et al. 2006)
31.7°C, Albipox is 42.8°C and Timber set is 53.8°C. The modulus in the rubbery plateau region is proportional to either the number of crosslinks or the chain length between entanglements (Menard 1999). The rubbery plateau modulus for Timberset is higher than the other adhesives which is also another prove that Timberset has higher crosslinking effect.

The tan δ curves of the adhesives are shown in Figure 4b. The tan δ peak of Albipox is seen to be much higher. This is probably due to better interfacial bonds. The tan δ peak for Timberset is lower and wider than that for the other adhesives. Its less height reflects the poor adhesion between the ceramic particles and the adhesive matrix, but its greater width points to the higher degree of crosslinking (Ward & Hadley 1993).

This observation is supported by the results of bending test for different curing times conducted by the authors (Ahmad et al. 2009) as shown in Figure 5, where the flexural strength of Timberset did not change over the period of time from 7 to 40 days, which is also indicative of fully crosslinked structure.

The polymer molecules experience reduced chain mobility as the reinforcing effect of the ceramic particles dominates. This is also evidenced from the viscosity test where the viscosity of Timberset was not determined as it has a very high viscosity and crosslinks rapidly (hardened very fast).

Figure 6a shows the TEM micrograph of nano-silica dispersion in CB10TSS. The low damping capacity of Timberset suggests that the interaction with the heavily fillers ceramic particles as shown in the Scanning Electron Microscopy (SEM) (Figure 6c) effectively lower the polymer mobility.

**Effect of High Temperature and High Humidity on the Thermal Properties of the Adhesives**

To determine the effect of temperatures and high humidity on thermal properties of adhesives, they were evaluated following conditioning at 20°C/95%RH for 90 days and the experiment was repeated for another two batches of samples at 30°C/95%RH and 50°C/95%RH. Figures 7 to 9 display the storage modulus E’, loss modulus, E” and tan δ curves for each adhesive after 90 days exposure and Table 2 summarizes the thermal properties of the adhesives. From Figure 7a, the E’ curve for CB10TSS exposed to 20°C/95%RH shifts to a lower temperature with respect to the control curve.

The adhesive aged at 30°C/95%RH shifts to a greater extent. However for the adhesive aged at 50°C/95%RH the curve shifts back close towards the control curve. This indicates that at 50°C further crosslinking has taken place but the moisture induces plasticization which reduces the T_g. The E’ and tan δ curves (Figures 7b and 7c) hardly shift but the peaks of these curves vary in magnitude compared to the control samples.

Figure 8 shows that E’ for the Albipox aged at 20°C and 30°C is initially less than E’ for the control material but the T_g based on the onset of E’ curves hardly changes. When aged at 50°C/95%RH, the Albipox E’ curve shifts to the right of the curve for control specimen, so T_g values are improved by approximately 6°C.

![Figure 5. The effect of curing time on flexural strength of all adhesives (Ahmad et al. 2009).](image_url)
Results for the Timberset adhesive as shown in Figure 9, show a slight shift to lower temperatures for the $E'$, $E''$ and tan $\delta$ curves for samples aged at 20°C/95%RH but when aged at 30°C/95%RH and 50°C/95%RH, the curves are shifted towards higher temperatures and move beyond the curves of control samples. When aged at 50°C/95%RH, Timberset did not suffer any loss in the thermal properties values as seen in Figure 9a.

In fact the $T_g$ values increases remarkably by 24°C. Therefore the exposed samples have higher $T_g$s than the control samples. Cook and Tod (1993) suggested that $T_g$ is depressed by the presence of moisture but as the temperature increases, the moisture is driven off and the adhesive samples cure further modifying the mechanical properties.

After aging for 90 days at 30°C/95%RH, again Timberset shows higher storage modulus values and $T_g$. After ageing, the $T_g$ for Timberset increases by 7°C but the $T_g$ for Albipox reduces by 3°C and the $T_g$ for CB10TSS reduces by 9°C.
When exposed to 20°C/95%RH, 30°C/95%RH and 50°C/95%RH, the $T_g$ of CB10TSS decreases as the temperature increases. In contrast, the $T_g$s for Albipox and Timberset increases as the temperature increases due to further cross-linking.

**Effect of Humidity on the Thermal Properties of Adhesives**

To determine the effect of humidity on the thermal properties of the adhesives, the adhesives were conditioned at 20°C/75%RH for 90 days and the experiment was repeated for another batch of samples at 20°C and 95%RH for 90 days. Figures 10 and 12 display the storage modulus, $E'$, loss modulus, $E''$ and tan $\delta$ curves for CB10TSS, Albipox and Timberset respectively after 90 days exposure to 20°C/75%RH and 20°C/95%RH together with control samples (approximately 20°C/65%RH).

The results for CB10TSS adhesive show a drop in dynamic modulus $E'$ with a shift to lower temperatures (Figure 10a) as RH is increased. If $T_g$ is measured based on the onset $E'$ value, then it is seen to decrease as the humidity increases. The $T_g$s are in the order of $T_{g\text{control}} > T_{g\text{75RH}} > T_{g\text{95RH}}$. The loss modulus $E''$ (Figure 10b) and the tan $\delta$ curves (Figure 10c) are shifted towards lower temperatures and the peaks also decrease as the humidity increases. This behaviour is often referred to as the plasticizing effect of moisture on the polymer (Lee & Peppas 1993).

From Figure 11a for Albipox, it can be seen that the $E'$ curve for 20°C/75%RH shifts to the right of the curve...
for the control specimen. However the graph of $E'$ curve for 20°C/95%RH is not shifted with respect to the control. The $E''$ and tan δ curves for 20°C/75%RH are also shifted to higher temperatures with no loss in the peak heights. This means that increase in relative humidity at lower temperature has no significant effect on the network structure of the Albipox although some plasticization is clearly occurring because the initial storage modulus decreases.

Timberset is unique in the sense that all three thermal parameters are shifted to the right after exposure to high humidity, Figure 12a, indicating an increase in glass transition temperature. However, the magnitude of the storage modulus $E'$ decreases with increase in RH. The $E'$, $E''$ and tan δ curves for 20°C and 75%RH showed peculiar behaviour after reaching about 80°C for CB10TSS (Figure10) and Albipox (Figure11), and for Timberset (Figure12) was after 100°C. This may be due to the moisture induces swelling and plasticization of the material to different extends in different regions which depends on the microstructure of the adhesives. This has affected the clamping pressure on the specimens in the DMTA.

**Thermal Properties of Adhesives after Exposure at 50°C**

Figure 13 shows the results of DMTA analysis on the adhesives after 90 days aged at 50°C. The trend of $E'$, $E''$ and tan δ curves after ageing at 50°C are the same as before
ageing where Timberset curves are to right of Albipox and CB10TSS and towards higher temperatures. However the curves shifted to much higher temperatures. This shows that Timberset has the highest glass transition temperature followed by Albipox and CB10TSS.

By post-curing the adhesives at 50°C, the glass transition temperature of all adhesives increased significantly with $T_g$ of Timberset increased by 34°C, Albipox by 24°C and CB10TSS by 2°C.

**Thermal Properties of Adhesives after Soaked in Water at Room Temperature**

Figure 14 compares the DMTA traces for CB10TSS, Albipox and Timberset after soaked in water. The curves of dynamic mechanical analysis present similar behaviour with differences in the magnitude of $E'$, $E''$ and tan $\delta$ values, that depend on the polymeric matrix and the moisture absorbed during conditioning. curves of $E'$, $E''$ and tan $\delta$ for CB10TSS versus temperature after soaked in water shifted to lower temperature but Albipox and Timberset shifted to higher temperature.

From Figure 14a it can be seen that the $T_g$ for CB10TSS decreased by 12.5°C but the $T_g$ for Albipox increased by 3°C and $T_g$ for Timberset increased by 6°C when compared to control specimens. It has been pointed out by Johncock (1990), that absorbed water can caused an increased in $T_g$ as a result of water induced additional cure of incompletely cured systems. The height of tan $\delta$ can be used to explain the cure characteristic of the adhesives. Figure 14c shows that
the height of tan δ is in the order of CB10TSS > Alipox > Timberset. This indicates that Timberset has high cross-link density and that might hinder water diffusion. This result is supported by other studies. In authors’ previous work on moisture uptake studies for all the adhesives (Ahmad et al. 2010, 2011), the water uptake for Timberset is less than Alipox and Alipox is less than CB10TSS as shown in Figure 15.

The change in the height of tan δ peak cannot be explained by only in terms of water as plasticizing effect. The free volume and the epoxy-water interactions could be used to explain the behaviour observed. Pethrick et al. (1996) reported that in epoxy compounds, water exists in two distinct forms; free water that fills the microcavities of the network and bound water in strong interactions with polar segments.

The rupture of the interchain hydrogen bonding by water molecule would produce an increment of the chain mobility and hence a reduction in the effective crosslink density of the material (Nogueira et al. 2000). An increment of tan δ height values and a reduction of E’ values should be expected. Thus, the stronger the water-polymer interactions, the greater the increments of tan δ and the greater the reductions in E’ and Tg values. Therefore in this case, the water-polymer interaction for CB10TSS is stronger than Alipox and Timberset which results in higher tan δ peak and lower Tg value. The superior water uptake levels for CB10TSS as seen in Figure 15 at low temperature
could be related to its highly polar structure. If the tan δ values reduces with increase in the water content and the
$T_g$ shift to lower temperature then it is often attributed to plasticization effect of water but it should also be pointed out that it could be related to the ordinary sorption of water in the free volume of the network that is, unbound water (Noguira et al. 2000). Tan δ curve after soaked in water presents noise (Figure13c) at higher temperatures and this may be because the adhesive shrink after the moisture has dried out at high temperature.

**GENERAL DISCUSSION**

Tables 2 and 3 summarizes the thermal analysis results for all adhesives before and after ageing at $20^\circ C/65\%$RH(control), $20^\circ C/75\%$RH, $20^\circ C/95\%$R, $30^\circ C/95\%$RH, $50^\circ C/95\%$RH, soaked in water at room temperature and placed in the oven at $50^\circ C$. CB10TSS already contains nano-fume silica particles (3.6% by weight) which is dispersed in close to nano-sized particles (~300 nm) (Figure 6a). Reinforcement of CB10TSS with phase-dispersed nanoruher particles (CTBN) (Figure 6b) remarkably improves glass transition temperature of the standard adhesive. This study confirmed this effect and the addition of only 2.0% CTBN raised the $T_g$ of CB10TSS by 11°C. The same trend was also observed for Timberset. The addition of microfillers to CB10TSS improves the glass transition temperature but reduces the flexural, tensile and interlaminar shear strengths and toughness (Ahmad et al. 2006, 2010). Therefore the reinforcement with nano rubber and micro-sized ceramic particles satisfied one of the key
aims of this study, to raise the glass transition temperature of CB10TSS.

This research has deduced that the high glass transition temperature of Timberset was related to its high crosslink density which was supported by the height of tan δ for Timberset (Figure 4c) that was smaller than for CB10TSS, reflecting lower molecular chain mobility.

In general, the presence of high temperature and moisture induced plasticization which reduced the \( T_g \) and the strength of the adhesives. After exposed to all conditions, the storage modulus, \( E' \) and the \( T_g \) for Albipox and Timberset were higher than base adhesive, CB10TSS, at those conditions and this showed that the addition of fillers gave less reduction in the strength value and \( T_g \). For Albipox, this was probably due to the nano-particulate rubber content where the rubber particles reacted with the epoxy forming an interphase of amorphous nature. It is known that CTBN possesses reactive-end groups which will phase-separate during cure and induce a high reactivity with the epoxide ring of the epoxy molecule (Wise et al. 2000). This might lower the intensity of the tan δ peak during plasticization and further crosslinking that has taken place.

The \( E' \) curves yielded information on the modulus of all adhesives and the values decreased as temperature and humidity increased except for CB10TSS and Albipox, where the modulus increased when exposed to 50°C. By post-curing at 50°C the storage modulus increased. This result supported other studies that room-temperature curing
epoxies do not go to complete reaction (Herzog et al. 2005), therefore post-curing was necessary in order to achieve a high degree of crosslinking. Most room-temperature cured epoxies have $T_g$s not more than 50°C – 60°C (Pocius 2002).

The $T_g$ values obtained from DMTA methods after exposure to all the environmental conditions are displayed in Figure 16. When conditions at 20/75%RH, 20°C/95%RH, 30°C/95%RH, 50°C/95%RH and soaked in water, the $T_g$ of CB10TSS was always decreased but showed a slight increase when aged at 50°C. When aged at 50°C/95%RH, Timberset did not suffer any loss in the values of thermal properties. By post-curing the adhesives at 50°C, the glass transition temperature of all adhesives increased significantly with $T_g$ of Timberset increased by 34°C, Albipox by 24°C and CB10TSS by 2°C. Timberset is a denser and more rigid polymer than Albipox and CB10TSS and it possesses a higher density of cross-links resulting in a decrease in free space, reduction in water uptake (Figure16) and increased $T_g$.

From Figure 16, it can also be seen that the $T_g$s of all adhesives reduced as humidity increases at room temperature and reduced much higher after soaking in water. The water alters the molecular structure of the adhesive by an increase in the molecule mobility which is due to the softening or plasticizing effect and this is manifested as dimensional changes and reductions in $T_g$ values (Lee 1991).

In contrast, when the temperature increases at high humidity, the $T_g$s tend to increase which proved the effect of further crosslinking. When a polymer is crosslinked, covalent bonds are formed between the polymer chains, which bring them closer together and reduce free volume
Figure 14. Graph of: (a) Storage modulus versus temperature; (b) Loss modulus versus temperature and (c) Tan δ versus temperature for all adhesives after soaked in water.

which causes an increase in glass transition temperature (Ward & Hardley 1993).

**Morphology Characterization**

Micrographs for all the unaged and aged CB10TSS specimens (control, 50°C, soaked in water, and 50°C/95%RH) are presented in Figure 17.

There are distinct micro-structural differences before and after aging. The rougher cleavage pattern for as-received CB10TSS (Figure 17a) stretched and cleaves are smoother (see Figure 17b) after exposure to a temperature of 50°C. This smoother surface might be due to continuous crack propagation and less crack branching as a result of plasticization. When soaked in water for 90 days at 20°C, the fracture surface of CB10TSS lost its roughness (Figure 17c) much more compared to aging at 50°C (Figure 17b) with no yielding of the adhesive and the development of numerous micro-cracks. When aged at 50°C/95%RH, the matrix seemed to have ‘melted’ which reflected that plasticization had taken place.

The fracture surfaces of Albipox before and after aging are shown in Figure 18. In the as-received Albipox in Figure 18a, cleaves in a petal-like form and the presence of micro-voids indicates the cavitation of rubbery particles that enhanced resistance to shear yielding and therefore increase the tensile strength of the Albipox (Ahmad *et al.* 2006).

At 50°C, the Albipox shows similar features but more packed and denser due to cross-linking. The petal formation seems to have flattened out due to plasticization
Table 2. Summary of thermal analysis results for all adhesives after ageing at 20°C/95%RH, 30°C/95%RH and 50°C/95%RH.

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>20°C and 65%RH (Control)</th>
<th>20°C/95%RH</th>
<th>30°C/95%RH</th>
<th>50°C/95%RH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$T_D$ 1 (°C)</td>
<td>$E'$ 1 (GPa)</td>
<td>$T_D$ 2 (°C)</td>
<td>$E'$ 2 (GPa)</td>
</tr>
<tr>
<td>CB10TSS</td>
<td>31.7</td>
<td>9.2</td>
<td>0.95</td>
<td>26.7</td>
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<tr>
<td>Albipox</td>
<td>42.8</td>
<td>9.3</td>
<td>1.04</td>
<td>40.0</td>
</tr>
<tr>
<td>Timberset</td>
<td>53.8</td>
<td>16.0</td>
<td>0.89</td>
<td>51.6</td>
</tr>
</tbody>
</table>

1 $T_D$ based on onset $E'$ curve.
2 Storage modulus measured at 0°C in MPa.

Table 3: Summary of thermal analysis results for all adhesives before and after ageing at 20°C/75%RH, 50°C and soaked in water.

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>20°C and 65%RH (Control)</th>
<th>20°C/95%RH</th>
<th>30°C/95%RH</th>
<th>50°C/95%RH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$T_D$ 1 (°C)</td>
<td>$E'$ 1 (GPa)</td>
<td>$T_D$ 2 (°C)</td>
<td>$E'$ 2 (GPa)</td>
</tr>
<tr>
<td>CB10TSS</td>
<td>31.7</td>
<td>9.2</td>
<td>0.95</td>
<td>28.1</td>
</tr>
<tr>
<td>Albipox</td>
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<td>9.3</td>
<td>1.04</td>
<td>50.0</td>
</tr>
<tr>
<td>Timberset</td>
<td>53.8</td>
<td>16.0</td>
<td>0.89</td>
<td>62.5</td>
</tr>
</tbody>
</table>

1 $T_D$ based on onset $E'$ curve.
2 Storage modulus values at 0°C in GPa.
Figure 15. Moisture absorption curves for adhesives soaked in water at room temperature (Ahmad et al. 2010).

Figure 16. Glass transition temperatures obtained from DMTA after the adhesives specimens exposed to different environmental conditions.
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(Figure 18b). In the fracture micrographs of Albipox tested after soaking in water for 90 days (Figure 18c), the fracture surface was rather smooth with radiating soft flex-ridges indicating plastic deformation. Micro-voids indicating loss of rubbery particles could also be seen. The presence of the ridges showed that more energy was absorbed to break the specimens. The smoothness of the matrix also demonstrated the plasticization. When exposed to 50°C/95%RH, Figure 18d, the fracture surface became smoother with finer crack propagation lines and extensive minute cracks in the matrix but still possessed plastic yielding. The deformed voids (due to cavitation of rubber particles) could also be seen which indicated weak bonding.

The fracture micrographs for Timberset are shown in Figure 19. The fracture surface of the control specimens (Figure 19a) revealed intimate bonding between the micro-filler and the matrix. However when exposed to more severe environmental conditions (Figures 19b to c) there was an indication of more debonding of the fillers together with cracking of the adhesives and the damages to the microfillers. The presence of moisture that cause the plasticization of the adhesive matrix could be seen in Figure 19c.

CONCLUSION

The thermal properties of adhesives reinforced with nano- and micro-fillers before and after exposure to different environments were investigated using DMTA techniques.

The following effects of nano- and micro-filler additions on rheology properties of adhesives were evident:

- In the measurement of viscosity during cure, CB10TSS was much easier to shear compared to Albipox. Therefore the addition of rubber nano-fillers to Albipox increases resistance to flow in shear; and
- The viscosity of Timberset was not determined as it had a very high viscosity and crosslinked rapidly.

The effect of nano- and micro-filler additions on thermal properties of adhesives were as follows:

- Timberset, containing high-modulus ceramic particles, had the highest storage modulus E', peak
Figure 18. SEM micrographs of the fracture surface of Albipox: (a) control, $\times 1500$; (b) aged at $50^\circ C$, $\times 1500$; (c) soaked in water, $\times 1200$ and (d) aged at $50^\circ C/95\%RH$, $\times 1500$.

Figure 19. SEM micrographs of the fracture surface of Timberset: (a) control, $\times 1400$; (b) aged at $50^\circ C$, $\times 1200$; (c) soaked in water, $\times 1200$ and (d) aged at $50^\circ C/95\%RH$, $\times 1400$. 
loss modulus E’’ and Tg, followed by Albipox, and CB10TSS.

- Timberset had a higher crosslink density than Albipox and CB10TSS demonstrated by a wider tan δ peak and higher storage modulus in the rubbery region. This result was supported by the results of bending tests as a function of curing time where Timberset developed crosslinks most rapidly. The smaller tan δ peak reflected the high crosslink density and possibly the poor adhesion between the ceramic particles and the matrix; and

- The addition of micro-fillers and nano-fillers increased the Tg in the order of Timberset > Albipox > CB10TSS. The presence of ceramic particles in Timberset improved the Tg by about 22°C compared to CB10TSS. The nano-rubbery phase in Albipox improved the Tg by 11°C compared to CB10TSS. There was not much of a difference in the Tg values of CB10TSS.

The effect of the environment on thermal properties of adhesives was as follows:

- The effect of various environment on the thermal properties of adhesives investigated using DMTA showed similar trends.

- When exposed to 20°C/65%RH, 20°C/75%RH and 20°C/95%RH, the Tg of CB10TSS decreases as humidity increased, \( T_{g_{\text{control}}} > T_{g_{75\% RH}} > T_{g_{95\% RH}} \). For Albipox and Timberset, the Tgs were in the order of \( T_{g_{75\% RH}} > T_{g_{control}} > T_{g_{95\% RH}} \). In all cases the measured trends were due to plasticization of adhesives.

- When exposed to 20°C/95%RH, 30°C/95%RH and 50°C/95%RH, the Tg of CB10TSS decreased as the temperature increased. For Albipox and Timberset, the Tgs increases as the temperature increases due to further crosslinking.

- Following exposure to 50°C, the Tgs of CB10TSS, Albipox and Timberset increase by 2°C, 24°C and 34°C, respectively. This shows that room temperature cured epoxies were only partially cured at room temperature.

- After soaking in water, the Tg of CB10TSS decreased by 12.5°C but Tgs for Albipox and Timberset increases by 3°C and 6°C, respectively. This was due to CB10TSS having a stronger water-polymer interaction than for Albipox and Timberset based on the analysis of the height of tan δ peak. This result corresponded well with the results following exposure to different humidities; and

- SEM micrographs showed evidence of shear yielding, plasticization, debonding and presence of voids in CB10TSS, Albipox and Timberset which explained the increase or decrease in strength and thermal properties after ageing under different environment.

ACKNOWLEDGEMENT

The work reported here was financially supported by the Ministry of Science, Technology and Innovation of the Malaysian government. The advice and support by Rotafix Ltd is gratefully acknowledged. We wish to thank the technical staff of the Materials Engineering Group at Universiti Teknologi Mara for their assistance and support.

Date of submission: October 2011
Date of acceptance: December 2012

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Towards Improved Construction Waste Management: A Review on the Issues, Challenges and the Need of the Supply Chain Management Mechanisms in Residential Housing Development in Malaysia

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Throughout the years, the construction industry has made an important contribution to the Malaysian economy. Moreover, the Ninth Malaysia Plan (2006–2010) has also played a significant role in the demands of executing major residential housing project developments where it has been observed that construction waste was one of the priority waste streams. Due to the increasing number in the population that is actively involved in economic activities, and the modernization of the country, the types of construction waste that are being produced, and identifying the source such as waste are becoming more complex. Therefore, appropriate actions and approaches are needed to be taken with respect to its effective management in handling the solid waste from construction sites. This paper is intended to review the issues and the challenges enclosed within the supply chain management mechanisms in order for improving construction waste management. Throughout this review, useful information and better understanding concerning the current issues, challenges and the supply chain management mechanisms would be made inclusive in the field to be explored. The findings would also assist in improving the quality and awareness on the construction waste management that is being practiced.

Key words: Construction waste; construction waste management; supply chain management; quality

The construction industry plays a significant role in helping the nation to achieve sustainable development. Moreover, in achieving the quality of life among residents, the ninth Malaysia Plan (RMK9) had concentrated in providing sufficient residential housing project developments. Based on RMK9 (2006–2010), the demands on the residential housing project development approximately around 709 400 units with the highest proportion is Selangor (19.2%), followed by Johor (12.9%), Sarawak (9.4%) and Perak (28.2%) (Economic Planning Unit 2006). The RMK9 target on residential housing project is less than the completed residential housing project in RMK8 (2001–2005) which is 844 043 units more than the target which was 615 000 units (Economic Planning Unit 2006).

With the demands in implementing residential housing development, it is shown that the construction sector is being expanded and developed. Due to the factors of construction stage, type of construction work and practices on site, the construction sector will produce the amount and the types of waste (Begum et al. 2007). The most quantity of waste generated is from waste materials or construction waste materials. Based on the Solid Waste and Public Cleaning Management Act 2007 (Act 672) (Solid Waste and Public Cleaning Department 2007), construction solid waste is “any types of solid waste that developments for RMK8 (2001–2005). Therefor, the completed units of residential housing was 844 043 units more than the target which was 615 000 units (Economic Planning Unit 2006).

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produced from any construction activities or demolition or including accomplishment, arrangement, renovation or modification”. Therefore, in order to construct the housing residential development, this construction waste that is generated will impact the environment. The effects on the environment includes unbalanced ecology, change of living environment, potential sewage, depletion of natural sources, energy consumption and generation of wastes (Yip 2000). After some decades of an extensive “use and throw away” philosophy, it has now been recognized that this uninhibited use of natural resources and resultant pollution levels are unsustainable (Chong et al. 2001). Therefore, it is essential to raise the awareness on construction waste management and revise previous common practices within the construction industry.

The generation of construction waste from construction activities will continuously give the negative impact on the environment. From a survey conducted in Hong Kong, housing projects had generated the highest wastage level compared with other types of projects (Shen et al. 2000). The reason may be due to the fact that the private projects normally are of non-standardized building structures of different sizes and shapes of building components such as formwork, reinforcement and brickwork (Shen et al. 2000). In addition, the generation of waste will produce negative impact on the environment, public image, resources, increases the time and cost for building processes and disposal of landfills sites (Shen et al. 2000). Normally the conventional method is practiced when the extra waste material and construction wastes are finally dispose of in landfills (Symonds 1999). Currently in Malaysia, the common practice of dealing with construction waste is by dumping at illegal dumping sites, but there are no statistics available for the volume of solid wastes dumped in this way (Ministry of Local Government 2005). Based on this situation, it is shown that Malaysia is still implementing improper methods in managing the construction waste and there is a lack in the controlling system for construction wastes.

Currently in Peninsular Malaysia, solid waste management together with the construction, operation and maintenance of waste disposal facilities are traditionally the responsibility of the state and local government authorities (Ministry of Housing and Local Government 2005). There are 101 Local Authorities in Peninsular Malaysia. Even though, there is a lack of a single legally empowered agency to undertake solid waste management for the country. The Local Government Department in the Ministry of Housing and Local Government provides the technical guidance and policy to local authorities.

Over the years, the weakness of financial aspect, operational, technical resources and infrastructure had caused an inadequate and insufficient level of various levels of the waste hierarchy. The privatization process was initiated in 1996 with the aim of achieving an efficient technology for advanced management system and enhancing the environment quality through resource recovery and waste minimization. In 2001, solid waste management was fully privatized whereby four major private waste services were provided a twenty year concessionaire period for municipal solid waste management. Additionally, the country is divided into four zones to be managed by four consortia with a 20 year concession. These are Alam Flora (Central and East Coast Region), Southern Waste Management Sdn Bhd (Southern Region), Northern Waste Sdn Bhd (Northern Region) and Eastern Waste Management (Sabah and Sarawak Region). These consortia will undertake the solid waste management services such as collection, transportation, treatment, reducing, reusing and recycling (3R), and public awareness.

In order to enhance the level of CWM, National Strategic Plan for Solid Waste Management would be introduced in the RMK9 (2006–2010) period as an initiative to highlight the strategic plan which is more focused on the principles, guide for comprehensive planning and adoption of hierarchy of waste options related to the provision of solid waste management (Economic and Planning Unit 2006).

The supply chain management (SCM) is viewed as a strategic tool (Dale et al. 1994) which is vital to be most effective solutions for managing the waste (Andrew et al. 2004). SCM can improve efficiency and productivity, and reduce overall operating costs (Lambert et al. 1998). This is the approach to improve current practices of CWM. The purpose of this paper is to conduct a review on the issues and the challenges enclosed within the supply chain management mechanisms in order to improve CWM.

Review on the Construction Waste Management (CWM)

CWM can be defined as activities including collection, transport, processing, reuse, recycling or disposal, and monitoring of construction waste materials (Coelho
2006). Salvaging construction waste during demolition and construction can assist to provide materials that can be re-used, recycled, sold or donated, thus decreasing the cost of constructing the building and creating new business opportunities among small- and medium-manufacturing industry (Coelho 2006). Otherwise, good practices of CWM is also important because it helps to protect the environment and it makes good business sense. Thus, adoption of waste minimization saves money through environmental and it makes good business sense. Thus, adoption of waste minimization saves money through avoidance of disposal costs, creates safer working conditions for employees, and protects human health and the environment (Coelho 2006). General methods of waste management are source reduction, recycling, and treatment. In Portugal, recycling is a new business, especially when it comes to construction and demolition waste, where recycling efforts are minimal, and this is gaining momentum (Coelho 2006).

There are studies showing that the good practice of CWM is important to protect the environment. Therefore, further studies on this issues and challenges need to be conducted in order to implement the mechanism of supply chain management for CWM.

The Issues and Challenges in CWM

In the developing countries, the issues and problems of CWM are of immediate importance. Based on the extensive literature review in a number of developing countries, for instances in Hong Kong, construction waste is directly disposed off at landfills without separating the waste into its constituent parts which aggravates the landfill shortage problem (Poon et al. 2001). The main reasons for the challenges are the lack of financial resources to cope with the increasing amount of waste generated, uncontrolled open dumps, the operational in efficiencies of waste services due to inefficient institutional structures of operators, inefficient organizational procedures and use of inappropriate technologies, financial and institutional constraints for adequate disposal of waste (Zurburgg & Schertenleib 1998).

A study in Indonesia had identified the other problems which affected the construction projects, included inefficiencies in using materials, equipment shortages, imbalances in organizational structure, unfair competition, limited funds, planning uncertainties and a lack of human resource development (Royat 1994). The problems of CWM in Thailand is similar to Hong Kong which have caused environment perspectives comprising insufficient budgetary allocation for CWM management and ineffective collection of service fees; no active planning on establishing common disposal facility among adjacent communities; no definite regulation and guideline for CWM hierarchy starting from source separation, collection, transportation, disposal and monitoring; lack of skilled personnel in operating an efficient waste collection and disposal practices; absence of waste recycling programme in most communities; associated existing legislation does not adequately facilitate the CWM in an effective direction; lack of public co-operation and participation; lack of government legal enforcement amongst others (Kofoworola & Gheewala 2009).

In a developing country like Malaysia, construction waste is a vital issue which recently received greater attention in the country. Due to the rapid growing population, urbanization and industrialisation, the amount of waste generated gradually increases. The challenges that occur in the Malaysian context are related to contractors not practicing source separation, source reduction, reusage and recycling in construction sites and disposal of waste into landfills. Additionally, there is still a lack of basic legislation and clear policies on CWM (Hussein et al. 2010).

In Malaysia, the important issues are related to low level of legislative enforcement and administrative inefficiencies in managing solid waste (Agamuthu et al. 2009). The general issues and problems concerning the entire CWM in developing countries could be placed into three categories including the problem of landfill development and management. The first category of problems is more or less external to the solid waste management sector, and to some extent are beyond the capability of the waste management authority to solve. The second category is partly external to the solid waste management authority and other related agencies. The third category is mostly internal to the solid waste sector (Idris et al. 2004).

Therefore, the study focussed on issues and challenges that would assist to identify the main current issues and challenges that are evident. The main issues and challenges are in construction wastes which are directly disposed in the landfills without being separated into constituent parts. There is also no recovery process or mechanism for reusing and recycling. Legal enforcement and existing legislation is inadequate to facilitate CWM effectively and there is no specific regulations and guidelines too, budgetary allocation for CWM were also insufficient. Based on the findings, a suitable mechanism could be proposed for improving the CWM.

Overview on the Supply Chain Management (SCM) Mechanisms

Supply Chain is a concept that can provide a useful framework for analyzing the construction process. It can be determined by the interconnected series of activities concerned with the planning and controlling of raw materials, components and finished products from suppliers to the final consumer (Stevens 1989). The chain is traditionally characterized by the forwarding of materials and outflow of information (Beamon 1999). The current...
interests in the manufacturing environment has been sought to extend the traditional supply chain to include “reverse logistic” which consists of the material recovery for the purposes of recycling, remanufacturing and reuse (Beamon 1999).

In construction waste, the supply chain includes all business and other organization which are involved in the process of extraction of construction waste from the construction site, the recovery process of waste including reuse and recycling, distribution of finished goods for the customers and disposal of its components (Beamon 1998).

Whilst the SCM is the interconnected series of activities or integrated manufacturing process (Stevens 1989) which is concerned with the planning and controlling of raw materials from the supplier, manufactured and until it is delivered to the customers via distribution or retailers (Beamon 1999). A study done in the United Kingdom shows that, this mechanism is the most effective solution for managing waste (Andrew et al. 2004). There are several mechanisms that have been applied in others countries in order to manage waste, for example a closed-loop supply chain mechanism can be defined as a system without waste compared to the traditional supply chain which has open ends (Figure 1); a closed-loop supply chain put all output back to the system (Solvang 2007). The case study done by Voordijk (2000), shows the supply chain and manufacturing process for building materials especially for concrete and ceramic products which are referred to as the traditional supply chain mechanism.

A study done in the United States described that the fully integrated extended supply chain mechanism contains all elements of the traditional supply chain, but extends the one way chain to construct the semi-closed loop that includes the recovery process including recycling, remanufacturing and reuse operation and follow by the re-distribution of reused products to customers (Beamon 1998). The purpose of extending the traditional supply chain is to allow consideration of the total immediate and eventual environmental affects of products and processing including the extraction of raw materials to the use of goods produced and to the final disposal of those goods (Lamming & Hampson 1996). Figure 2 illustrated the extend of supply chain mechanism which contains the recovery process or recovery activity including reuse, recycling, redistribution and remanufacturing.

Reuse is the process of collecting used materials, components or products from the field, and distributing or selling them to be used. Thus, the ultimate value of the product is also reduced from its original value and no additional processing is required (Beamon 1999; Kumar & Malegenat 2006). Whilst, recycling is the process of collecting used products, material and/or components from the field, disassembling them (when necessary), separating them into categories and processing into recycled products, components, and/or materials. In this case, the functionality and identity of the original materials are lost (Thierry et al. 1995). The success of recycling depends on whether or not there is a market for the recycled materials; and the quality of the recycled materials (since most recycling process actually reduce the value of the material from its original value, as the material itself has degraded) (Beamon 1999; Kumar & Malegenat 2006).

Remanufacturing is the process that consists of collecting a used product or component from the field, assessing its condition, and replacement when worn, broken or obsolete parts with new or refurbished parts. Then, the remanufactured product is inspected and tested with the goal of meeting or exceeding the quality standards of brand new products (Beamon 1999).

The research done in Hong Kong also proposed a comprehensive conceptual framework as a mechanism shown in (Figure 3) (Shen et al. 2005). This conceptual framework is used to co-ordinate the cross functional product logistics flows and used product reverse logistics flow in a given green supply chain which is not limited to applications for specific industries. The factor was oriented from governmental regulations for environment protection, e.g. the subsidies for used product recovery, recycle fees and return ration charged to manufacturers are considered in the proposed framework as a mechanism (Shen et al. 2005).

![Figure 1. Traditional supply chain.](image)
Figure 2. The Extended supply chain.

Figure 3. Conceptual framework for integrated logistics control for a green supply chain.
Additionally, a study done in the United States treated that closed supply chain and reverse supply chain as equivalent and suggested that collection, inspection/separation, reprocessing, disposal, distribution are five main groups of activities conducted (Kumar & Malegenat 2006). Collection is the process that consists of all activities related to used items (product, component, or material) available and physically moving them to some point for further treatment which may involve product acquisition, transportation and storage. While, undergoing inspection or separation is may involve testing, disassembly, sorting, shredding and storage; in this activity, the material or components will be split for various recovery and disposal options (Kumar & Malegenat 2006).

Re-processing is occurring in the reusable flow, the actual transformation of a used item will go through into a reusable item of some kind; based on the recovery option chosen, this covers various activities such as disassembly, shredding, repair, replacements, etc. (Kumar & Malegenat 2006). While disposal is a process where the non-reusable flows are disposed off to landfills and incinerators. Besides, the re-distribution process which is containing the reusable items directing to be marketed to new markets, and physically moving them to potential new users. This involves transportation, sales activities, and storage (Kumar & Malegenat 2006).

A study done in Canada purposed a close loop supply chain model (Figure 4) as a mechanism for manufacturing small- and medium-sized enterprises because of the increase in waste generated from manufacturing operations are attracting increased attention in industrialized countries (Talbot 2007), due in part to depleting landfills and incineration capacities (Guide et al. 2003). This mechanism involves combining traditional forward supply chain activities and reverses supply chain activities into a single system (Krikke et al. 2004) with the potential and has capabilities to raise the environmental performance of industrial operations to a new standard (Pappis et al. 2004) and to make new profit opportunities and competitive advantages for supply chain participants and contributors; this mechanism also consists of holistic product life-cycle concepts (Ferrer & Whybark 2003).

CONCLUSIONS

There are several critical issues and challenges related to CWM practices which could be described as the current situation in this area of study where the waste generated directly was disposed to landfills without being separated into constituent parts. There was also no mechanism for recovery processes, reuse and recycling. There was a lack of government legal enforcement and the existing legislation did not adequately facilitate CWM in an effective direction. Specific regulations, guidelines and sufficient budgetary allocations for CWM were not available.

A review on the supply chain showed that all the mechanisms focused on construction and manufacturing were in perspective, thus further research was required on current practices, issues and challenges in Malaysia, to assist and improve CWM.
ACKNOWLEDGEMENTS

The authors are grateful to Universiti Teknologi MARA for funding the research work. Prof Ahmad Ruslan Mohd Ridzuan and Dr Intan Rohani Endut are acknowledged for their continuous support and encouragement.

Date of submission: May 2011
Date of acceptance: February 2013

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Continuous growth of air traffic and its corresponding increase in CO₂ emissions can be offset by high-quality bio-jet fuels which meet all kerosene-related parameters. Some technologies are already certified and on the market. Greasoline is an innovative new technology which uses bio-based non-food resources and residues. It therefore broadens the raw material basis for bio-jet fuels and can supplement existing technologies.

Aviation Industry Growth and related CO₂-footprint
Aviation is amongst all transport sectors growing very strongly: annual growth rates are projected at approximately 4.5% per year throughout the next decades. The majority of this growth is expected to be linked to Southeast Asia. Technological progress in the aviation industry — mainly more energy-efficient planes — might reduce the fuel consumption a bit. Without further changes in the fuel sector, aviation-related CO₂ emissions would anyhow increase by 3% per year. The aviation industry expects to be reliant on liquid fuels for the next 30 to 50 years, since no alternatives for (bio)fuels — e.g. like batteries for cars — exist for airplanes.

This would put even more pressure on the CO₂-footprint of aviation: already today, 12% of transport CO₂-emissions and 3% of the whole man-made CO₂-emissions are due to aviation. European airlines consumed 53 million tons kerosene in 2010, the whole world even 200 million tons.

Challenges for Aviation Companies
Governmental bodies are discussing joint targets and implementing several regulative actions in this context. The European Union decided via its Renewables Energy Directive to use 10% renewable energies in the transport sector by 2020, and is targeting 2 million tons of ‘sustainable’ kerosene by 2020. Details can be found in the technical paper, A Performing Biofuels Supply Chain for EU Aviation, of DG ENER 2011.

Aviation companies agreed on a voluntary self-commitment that they will only grow in a climate-neutral way from 2020 onwards. They are however confronted by several challenges. Their cost pressure is tremendous, and fuel costs are continuously increasing: more than 30% of operating costs in aviation are due to fuel. Established biofuels for land transport like bioethanol and biodiesel cannot be used in airplanes due to their fuel properties — air transport requires biofuels which are chemically identical with fossil kerosene.

State-of-the-art Processes for Bio-jet Fuel
State-of-the-art for producing non-fossil oil-based alternative fuels is summarized in an IATA report. Basically two routes are accepted by American Society for Testing and Materials (ASTM):

First, there is the so-called Fischer-Tropsch (FT) process. In particular coal-to-liquid, gas-to-liquid and biomass-to-liquid products are addressed. Technology-wise, it implies gasification of feedstock to syngas with ash as a by-product. Syngas is converted further to a mixture of fuels containing naphtha, olefins, kerosene, diesel und waxes, which are treated in several steps by so-called hydro-processing and isomerization to yield fuels including jet fuel. Since those processing steps require very large units, typically more than 50% of fuel costs are due to investment costs.

Second, there are so-called hydrogenated vegetable oils, sometimes also called hydro-processed renewable jet (HRJ). Processing steps comprise, besides distillation, the steps hydro-treating, hydro-cracking and isomerization to yield fuels including jet fuel. Since those processing steps require very large units, typically more than 50% of fuel costs are due to investment costs.

References
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Technologies not yet considered by ASTM but lining up for testing:

- Hydro-processed 'Synthetic Paraffinic Kerosene' derived from fermented alcohols. Initially focused on isobutanol, but other variations will be considered too (Alcohol-to-jet, e.g. Lanzatech/ Swedish Biofuels)
- Synthetic biology, i.e. genetically engineered micro-organisms converting sugar to pure hydrocarbons, resulting in farnesene and other similar terpenes (Sugar-to-jet, e.g. Amyris Biotechnologies, Gevo and Cobalt)
- 'Synthesized Kerosene Aromatics' implying alkylated benzenes, a fuel component important for elastomeric seals and fuel lubricity (e.g. UOP and KIOR)
- Pyrolysis of cellulosic biomass to synthetic crude products (plus hydro-processing)
- Co-mingling petroleum and biomass in refinery hydro-processing. This is supported by refineries to optimize efficiencies, but is currently not allowed.

Greasoline Technology as a New Approach

In contrast to these approaches, Greasoline® technology is starting from bio-based fats and oils like HRJ. Greasoline however is based on a gaseous phase reaction technology and therefore can transform raw materials of significantly lower quality, because residual water and inorganic residues are separated in the evaporation step (Figure 1). The catalyst for the gaseous phase reaction also is highly tolerant to impurities. As a result, bio-based residues and side-products can be utilized instead of feedstock in the food-quality range.

Primary products are hydrocarbon chains identical with fossil diesel and kerosene fuels. Most of the diesel components can be transformed into the kerosene boiling range via isomerization. In addition to this the technology also produces bio-based alkylated benzenes which are crucial for jet fuel properties, especially as expanding agents in seals as well as for lubrication. These products cannot be obtained by hydro treating processes and therefore HRJ fuel has to be blended with fossil jet fuel.

The basic technology does not need external hydrogen, because the formation of coke as a by-product on the catalyst automatically closes the carbon-hydrogen-balance within the system. A subsequent hydrogenation step with little hydrogen consumption is optional to guarantee all product quality parameters. The catalyst itself is regenerated after the biofuel reaction in an industrially established process. The process is currently performed in a pilot plant at Oberhausen; plans for a demonstration plant are being elaborated together with partners mainly in the oil industry.

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Towards a Fully Functional Marine Mammal Stranding Response Network in Malaysia

Cheryl Rita Kaur

The string of marine mammal strandings and casualties in Malaysia have made headlines and attracted huge public outcry. Among the incidents include the case of the baby dugong being accidentally caught in a fisherman’s net in Johor in 1999, the sighting of a stray dugong calf off Terengganu in August 2006, the stranding of an injured Bryde’s whale in the shallow waters off Kota Kinabalu in December 2006, a ten-meter-long Bryde’s whale beached at Sungai Nenasi estuary, Pahang in October 2008, and more recently the death of a thirteen-meter-long baleen whale found at Pulau Mengalum, Sabah in February 2012, to name just a few. These cases not only indicate the presence of marine mammals in Malaysian waters but also point to possible threats to their survival. Incidences of marine mammal strandings in Malaysia also signal the fact that the issue is more serious than thought considering that there could be cases that were not reported.

Marine mammals are largely imperilled by traditional hunting for meat (especially in Sabah and Sarawak), degradation of natural habitats, starvation, collisions with vessels, dynamite fishing, entanglement in fishing gears, disease, pollution from coastal development and other anthropogenic threats. The slow growth and low reproductive rate of most marine mammal species further impedes their population’s recovery. Their generally high trophic levels and degree of habitat specificity make them even more vulnerable.

To address these issues, the Maritime Institute of Malaysia (MIMA) first organised a Seminar on Marine Mammals Conservation in Malaysia: Adopting Sustainable Management Strategies in April 2009 in Kuala Lumpur; highlighting pressing issues facing marine mammals, discussed conservation and management strategies towards their sustainable and effective, and prioritised efforts towards promoting effective implementation of conservation and management strategies to avoid their extinction. A major shortcoming identified was the absence of a national marine mammal stranding response network to assist in the protection of such mammals in Malaysian waters. Subsequently in 2010, MIMA collaborated with the Institute of Ocean and Earth Sciences of the University of Malaya and the Malaysian Nature Society in organising a Roundtable Discussion on Establishing a National Marine Mammal Stranding Network. This meeting emphasised in greater detail the need for and issues pertaining to marine mammal survival in the Malaysian waters. Standard procedures for rehabilitating stranded marine mammals were reviewed while exploring the expertise and facilities of relevant agencies and organisations in handling marine mammals stranding.

The recommendations from the roundtable were promptly taken up by the Turtle and Marine Ecosystem Centre, a research institute under the Department of Fisheries Malaysia (DoFM). A meeting was called involving the relevant stakeholders to move forward on the suggestion. Active participation and support from stakeholders involved was the catalyst to kick-start the network. Leading from the discussions among the agencies involved, the National Marine Mammals Stranding Response Network was established in 2011 with DoFM as the lead agency. Members of the network include Maritime Institute of Malaysia, the Department of Marine Park, Department of Veterinary Services, Department of Fisheries Sabah, Forest Department Sarawak, Sabah Parks, Malaysian Maritime Enforcement Agency, National Oceanography Directorate, Royal Malaysian Police, Fire and Rescue Department, Fisheries Associations, Academia, World Wide Fund for Nature Malaysia, Malaysian Nature Society, MareCet, and the Wildlife Trace Monitoring Network. During discussions, the main areas stressed in the handling of marine mammal strandings included the need to impart knowledge about the various aspects of marine mammals strandings, including anatomy, biology, causes of strandings, first response, rehabilitation, decision-making, and public concerns; adopting skills needed in the emergency first response of stranded marine mammals; as well as promotion of skills needed in the medical management of stranded marine mammals including hands-on activities and demonstrations.

The first meeting of the Network in 2013 was recently held in Kuala Lumpur. It detailed the standard operating procedures for the handling of stranding incidences and finalised the list of members in the task force and working group under the

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network, as well as discussed the financial requirements and resources for anticipated activities i.e., trainings for members and relevant stakeholders. The meeting also discussed the data compiled on marine mammal strandings in the country, aimed towards identifying hotpots and suitable management strategies. It was also agreed that members of the network meet two to three times annually to discuss related matters and updates on the progress of the group.

It is envisaged that a fully functional Marine Mammal Stranding Response Network with all relevant stakeholders co-operating with each other would place Malaysia on par with others that have established similar conservation programme in the region.
The thirty-two year old man, 150 cm in height, and old for his age, was hunched over a piece of flat-shaped white rock and chipping away at the edges of the rock with another hand-held piece of rock, this one of a reddish-brown colour. He was sitting along a river bank terrace surrounding a lake, just meters away from where he sat and worked. A large river flowed alongside the lake. It was growing dark and he had been at his task since day-light broke in this river valley. He heard a sound, turned around and saw his wife, bringing him some roasted meat, the remains of a monkey the hunters of his village had killed the previous day. His two children were with her. He stopped work and sat down on a boulder and took the meat from her and began cutting it with a sharp-edged stone. He grunted with satisfaction, pleased that his earlier work was good. It would fetch a good bargain, maybe a porcupine or fish or even a monitor lizard in exchange. His tools were in good demand and he was a well-known tool-maker in the area. He gave his wife and children pieces of meat and began chewing on his. He looked around at the many pieces of smooth rock around him, and pointed to a few pieces. These will make good scrapers and arrow-heads. His wife nodded in agreement. His children ran to pick up the pieces and turned them over to look at them.

It was then they heard a loud thunderous sound, coming from the direction where the sun set. He grew fearful and shouted to his family to gather up his work and they ran towards their village, a kilometre away. It must be the river, he shouted to his family, as they ran uphill away from the river. He had seen how the river would occasionally flood and wipe away his and the others’ little workshop huts on the river-bank in the past. He had not had time to pick up his finished work, some 8 pieces, a morning’s work.

Minutes later, the earth beneath their feet shook. His children tripped and fell. He paused to pick up one and his wife picked up the younger child and ran into their hut. This is not the river; it was never like this, he thought to himself. The sky suddenly darkened as he huddled with his family. He called to his neighbours if they knew the source of that sound and they all answered, maybe a herd of elephants. No, elephants would not have made that sound!

Soon, it was quiet again but something strange was happening. Hot ash began to fall and soon fall was getting heavier. He smelt burning which hurt his nose and inside his chest. He looked at his family, all with fearful looks on the faces. His hut and the others in the village began to smoulder and then catch fire. They all shouted and ran into the river and squatted. There was hissing sounds as the hot "bits" fell into the river. Some were still hot. The fall grew heavier until everything around them, the trees, the rock boulders and the ground was covered in hot, white ash-like material. The next morning, the whole village moved away; this village was not safe anymore.

What he, his family and his village did not know was that a terrestrial super-volcano had erupted in Sumatra.

Various researchers had attributed the volcano to be Toba, erupting, they reported, some 74,000 years ago. In a recent paper, to be published in the Proceedings of the National Academy of Sciences by Storey, Roberts and Saidin (<www.pnas.org/cgi/doi/10.1073/pnas.1208178109>), reported that, by using sanidine (a form of feldspars) crystals extracted from the volcanic ash (Figure 1), their astronomically calibrated \(^{40}\text{Ar}/^{39}\text{Ar}\) for the volcanic ash in the Lenggong Valley gave a determination of 73.88 ± 0.32 ka (or, thousand years), or 73,880 ± 320 years.

In a paper by Renne et al. (1994) (<http://geology.gsapubs.org/content/22/9/783.abstract>), they said that “the \(^{40}\text{Ar}/^{39}\text{Ar}\) radiogenic dating technique is one of the most precise and versatile methods available for dating events in Earth's history, but the accuracy of this method is limited by the accuracy with which the ages of neutron-fluence monitors (dating standards) are known”. They further stated that “the emerging astronomically calibrated geomagnetic polarity time scale offers a means to calibrate the ages of \(^{40}\text{Ar}/^{39}\text{Ar}\) dating standards that is independent of absolute isotopic abundance measurements”.

Paleolithic Age (Tampanian) Stone Tool Production ‘Workshops’, Kota Tampan, Perak — Was it Toba which Destroyed the Industry 70 000 Years Ago?

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The eruption of the Toba Volcano, they argued, had caused a layer of ash of up to 5 metres thick in places to be deposited in Kota Tampan. Today, Kota Tampan is an important archaeological site in Peninsular Malaysia. The volcanic ash occurs “among and above stone artefacts, manufactured by anatomically modern humans” (Storey et al.).

However, in an earlier paper by Tjia and Fatihah (2008), they quoted work by a number of researchers who had worked on samples attributed to four volcanoes in Sumatra, that is from Toba, Maninjau, Aekgadang and Tanjung Karang (Figure 2). They quoted from fission-track age dating conducted on zircons collected from the rhyolitic ash of four volcanoes in Sumatra by Nishimura (1980) (Table 1).

Nishimura (1980) reported that the Tuba tuffs from three locations gave three different fission track ages of 1.2 million years, 100 000 years and 30 000 years (the youngest eruption), none giving an age equivalent to 70 000 years. Tjia and Fatihah (2008) reported that there were at least two more Toba eruptions. An ignimbrite from the eastern lake shore of the Samosir peninsula, some 700 m below the summit surface, gave a K/Ar age of 1.9 ±0.4 Ma (Tjia & Kusnaeny 1976) and possibly represent an older and fourth eruption. A fifth catastrophic eruption at Toba was suggested by Yokoyama et al. (1980).

The only tuffs which gave fission track ages identical or close to Storey, Roberts and Saidin’s 40Ar/39Ar of 73 880 years are those from Bukittinggi (70 000 years) and Maninjau (80 000 years). The two other volcanic centres of Aekgadang and Tanjung Karang, in the south of Sumatra, gave fission track ages of 3.5 million years and 1 million years respectively, ruling out both volcanoes effectively as the source of the Kota Tampan’s youngish ash beds. Maninjau in western Sumatra is some 500 km south-south-west of Kota Tampan. Tjia and Fatihah (2008), when describing the Bukittinggi tuffs covering a large area around Maninjau, reported that the “extremely great thickness (about 200 metres) of the second tuff unit should represent a paroxysmal event whose tephra was distributed throughout a
wide region”. Is it therefore possible that it was Maninjau’s tuff, and not Toba’s, the tuff which covered parts of the Paleolithic Kota Tampan?

Stauffer et al. (1980) reported that radio-carbon dating of woody material directly below the rhyolitic tuffs in Ampang and Serdang, both localities in Selangor, gave ages of between 30 000 years to 36 000 years. This radiocarbon date ties in with Nishimura’s fission track age dating of zircons from the Tuba tuff of 30 000 years. This suggests that there were at least two episodes when erupting Sumatran volcanoes rained tephra over parts of Peninsular Malaysia.

Extensive alluvial tin mining in Selangor and Perak had "removed" much of the tuffs deposited. Kota Tampan, Perak, was not a tin mining area and thus had its tuffs essentially undisturbed (except by tropical weathering and erosion, as well as agricultural and infrastructure developments later) preserving the tool artefacts, basically in situ.

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The desire for healthier and better world in which to live our lives, and raise our children is common to all people and all generations. As we enter the 21st century, our past achievements and technological advances make us more optimistic about our future than perhaps at any stage in recent history.

More than 100 million people will die and global economic growth will be cut by 3.2% of gross domestic product (GDP) by 2030 if the world fails to tackle climate change, a report commissioned by 20 governments said recently. As global average temperatures rise due to greenhouse gas emissions, the effects on the planet, such as melting ice caps, extreme weather, drought and rising sea levels, will threaten populations and livelihoods, said the report conducted by the humanitarian organization Dara.

It is calculated that five million deaths occur each year from air pollution, hunger and diseases as a result of climate change that toll would likely rise to six million a year by 2030 if current patterns of fossil fuel usage continues. More than 90% of those deaths will occur in developing countries, said the report. The report was commissioned by the Climate Vulnerable Forum, a partnership of 20 developing countries threatened by the climate change.

Temperatures have already risen by 0.8ºC above pre-industrial times. The world’s poorest nations are the most vulnerable as they face increased risk of drought, water shortages, crop failure, poverty and disease. On average, they could see a 11% loss in GDP by 2030 due to climate change, the report said.

Many efforts have undertaken to bring health issues more into focus in climate change discussions. Evidence based discussions of climate change on health has been positively expanding in the past few years. This has allowed a greater understanding to both governments and the general public on the relationships between changes in climate and how it will affect specific areas. Yet much more work needs to be done, especially in understanding local scenarios.

Infectious tropical diseases are still the world’s biggest killer of children and young adults. For those living in developing countries, among the poorest of the poor no matter what their age, the risk of death and disability is always many times higher than those living in the developed world. Over 500 million people on earth, that is one living person in ten, suffer from one or more of the major infectious tropical diseases. While health globally has steadily improved over the years, on the other hand many people living in poorer countries have seen little, if any, improvements at all. The gaps between the health status of rich and poor are at least as wide as they are half a century ago, and are becoming wider still. Despite this, the most important pattern of progress now emerging globally is an unmistakable trend towards healthier, longer life.

In recent years we have all been affected by the increased spread of infectious diseases, be it SARS, swine flu, bird flu, H1N1, Nipah virus, dengue or monkey malaria. As humans dominate more of the world we become an even larger target for these and other diseases. A rise in the incidence of new and previously suppressed infectious diseases is being linked by scientists with dramatic climatic and environmental changes now sweeping our planet Earth. Deforestation, destruction of natural habitats for agriculture, road and irrigation, pollution of rivers and coastal waters, are promoting conditions for new and old pathogens to thrive along with climate change, the report said.

A case in point is the highly pathogenic Nipah virus in Malaysia was found in the Asian fruit bats. In the late 1990s it emerged as a fatal disease in humans. This has been linked with a combination of forest fires in Sumatra and the clearance of natural forests in Malaysia and Indonesia for palm oil plantations. Bats, searching for fruits were forced into closer contact with domestic pigs giving the virus its chance to spread to humans via people handling pigs. Almost similar but ecologically different situation was reported later from Bangladesh with Nipah virus infections. Anoher example also from Malaysia was the transmission of monkey malaria Plasmodium knowlesi to human in Kapit in the state of Sarawak. The issue of environmental degradation and a rise of many new and old infectious diseases is a complex, sometimes subtle, one that is causing increasing concern among scientists and public health specialists. Overall, it seems intact habitats and landscapes tend to keep infectious agents in check, whereas damaged and degraded environment shifts the natural balance thereby triggering the spread to people of new and existing diseases. Environmental distruption and change, and the poor handling of human and animal wastes are also to blame. International travel, technological change and globalization of trade in agriculture and other products favours the spread of diseases.
Remember the SARS outbreak a while back? Remember the chaos it created for international travel? In Asia, at least, air travel almost came to a standstill. Then, came H1N1. Again, the tourism trade suffered.

As of 2008, mankind is confronted by 346 generic infectious diseases, distributed in a seemingly haphazard fashion across 220 about countries. An average of three new diseases are described every two years and a new infecting organisms published every week. Over 1600 human pathogens have been reported, each with a specific set of phenotypic, genomic and susceptibility characteristics which must be confronted by diagnostic laboratories and clinicians.

The present global emergence of infectious diseases is clearly associated with the social and demographic changes of the past 50 years, particularly urbanization and globalization, with attendant spread of pathogens via infected human hosts and vectors. It is through human activities that the levels of greenhouse gases in the atmosphere have increased at the alarming rates. Experts now agree that the consequent warming of the world by the uncontrolled release of such greenhouse gases is now the most serious threat to the sustainability of the human race.

The changes in the environment caused by human activities are also apparent in the transformation of much of our landscape and conversion of regional systems once dominated by natural ecosystems. Factors include expansion into urban or peri-urban habitat, deforestation and the spread of intensive farming. The environment’s role in the emergence of diseases is apparent in the connections between the direct consequences of human changes to urban and rural irrigated agriculture for example, can create breeding grounds for mosquitoes and snail vectors. Likewise, the inadequate storm drainage and sewerage systems often associated with rapid urbanization not only increases the breeding habitat for disease vectors but facilitate the spread of water borne pathogens causing diseases like cholera, leptospirosis and other protozoan and bacterial infections. Overwhelming evidence points to human demographic changes and environmental degradation as major direct and indirect factors contributing to the increase in infectious diseases along with temperature increase due to climate change.

The UN GEO year book report links the emergence of many of the diseases like malaria, Japanese encephalitis, dengue and others transmitted by mosquitoes to sullage water stagnation. Increasing level of rubbish and solid wastes in developing countries — as a result of increasing consumerism, poor collection and refuse handling services, fly tipping, lack of recycling schemes and inadequate disposal sites — all aggravate the problem. Discarded plastic bags, old tin cans and car tyres offer, when filled with rain water, perfect breeding sites for disease carrying insects. Increased and unplanned urbanization, lack of proper waste-water management schemes in many developing countries and population growth are also important factors in the spread of these diseases.

Poverty, poor living conditions, including lack of proper sanitation along with lack of infrastructure for water and solid waste management, increase the opportunities for vector-borne and water-borne diseases to be transmitted from humans to humans and animals to humans. The geographic spread of Aedes aegypti and Aedes albopictus into peri-urban and urban areas throughout the world spreading dengue, chikunguya and other virus diseases is a good example of how a potential vector of viral diseases has taken advantage of man made environmental changes.

Climate change represents a potential environmental factor affecting disease emergence. Shifts in the geographic ranges of hosts and vectors, the effect of increasing temperature on reproductive development of vectors and pathogens along with climate variability on flooding and droughts have all a positive or negative influence on the incidence of infectious diseases. Thus climate change may aggravate the treat of infectious diseases in many ways.

A working group on land use and changes and infectious diseases ranked 12 environmental factors which influence the public health impact on the prevalence and transmission of diseases. They being:

<table>
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<tr>
<th>1. Agriculture development</th>
<th>7. Habitat fragmentation</th>
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<tr>
<td>2. Urbanization</td>
<td>8. Road building</td>
</tr>
<tr>
<td>3. Deforestation</td>
<td>9. Climate change</td>
</tr>
<tr>
<td>5. Introduced species or pathogens</td>
<td>11. Water and air pollution</td>
</tr>
<tr>
<td>6. Biodiversity loss</td>
<td>12. Irrigation and dam building</td>
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In conclusion, one may say that changes in global environment degradation, agriculture practices along with climatic changes are among some of the overlooked factors in the persistence, emergence and re-emergence of infectious diseases. These also interact with trends in economic development, population growth, urbanization, migration and pollution. Climate change and variability add new factors to this driving force. This is further exacerbated by the mushrooming of urban slums in many developing countries which lack proper sanitation. Will this change? How do we balance the need to open up new lands for agriculture and food production, but at the same time safeguard the habitats of life threatening microbes from spreading?

Recent investigations attribute more than 150,000 deaths per year and a global disease burden of approximately USD5 million annually to climate change. An area that has received particular attention is the potential impact of global warming on shifts in the spatio-temporal distribution of diseases. Vectors, pathogens, parasites and hosts survive and reproduce within certain optimal climatic conditions. Changes in climate will alter the transmission of vector-borne diseases in various ways. The potential impact of global warming on the transmission of neglected tropical diseases has received insufficient attention from researchers.

The resurgence of infectious diseases worldwide reflects our quick fix mentality, with poor development planning, a lack of political determination and institutional inertia. It is indeed a man-made situation which is assisted by climate change. Much can be done to reverse the current trend. As well as rebuilding the public health infrastructure for infectious disease control, there is substantial evidence on how regional planning and development, including urbanization, agriculture expansion, management and conservation of forests and ecosystems can minimize and even reduce the outbreaks of infectious diseases as well as environmental change. Basically, we need an integrated approach to pathogen control. This approach will involve integrating social and economic development programmes, environmental and natural resource management, with intervention based on disease ecology and community participation.

A few months ago, the United Nations in New York world leaders met to assess the achievements of the Millenium Development Goals. Eradicating poverty is the greatest challenge facing the world today and an indispensable requirement for sustainable development, particularly for all developing countries. 1.2 billion people are still living on less than USD1 a day and half the developing world lacks access to sanitation. Every week over 200,000 children under the age of five die of diseases and 10 000 women die giving birth. Climate change is a reminder of the fact that poor people are most likely to be the first victim and greatest sufferers of environmental degradation. The recent massive flooding in Pakistan is a testimony of this.

The world is changing. Competition is on the rise. Nations, regions, companies and individuals compete. The future global economy will be increasingly knowledge-based. Innovation is key to future global economic strength and competitiveness. Science, technology and innovation is expected to play a dominant role in economies planning to remain competitive. Resource-poor countries in the world have shown the way how technological superiority can make them powerful in the economic competition. Those which are technologically incompetent, despite being resource-rich, face difficulties creating wealth.

We had a decade or two of unprecedented scientific progress in medicine and there is great promise of more. But we cannot rest on our laurels. The infectious tropical diseases are in danger of being forgotten by a rich world that has forgotten its poor, and they will be forgotten, unless we take an aggressive and entrepreneurial approach, to grasp the scientific, political and economic opportunities that arise, and set in place good defense against the evolution of our biological enemies.

It is our task to make sure that infectious tropical diseases will not fall back into the darkness of Middle Ages.
“Instrumentation? What on Earth are you learning? Is it some kind of music? Do you mean instrumental? Will you end up as an instrumentalist who plays in orchestra?” That’s how people reacted when I answered that I am pursuing my bachelor degree in instrumentation science. Yes, instrumentation science. Again, it is SCIENCE! Do you expect me to be a musician for my future career? Think logically, if I want to be a musician, I would rather sing by the street or sign up for an audition to let people see my talent or even further my study on something to do with music. Never mind. I am well understood that this field is somehow still new in our country and rarely heard by people. Therefore, I would like to share some information about the unknown side of instrumentation science.

First of all, you have to know the definition of instrumentation science. Instrumentation is a set of instruments used to operate and control a piece of machinery. Science, on the other hand is a discipline of knowledge related to the structure and behaviour of natural and physical world, based on facts that can be proved, for example by experiments. Thus, it is clarified that instrumentation science is more concern on the process, methods of manipulation and control system with the application of the study of science. This field revolves around the role of technology implemented in a particular operating system and machinery. It is to make use the scientific knowledge in order to create, construct and maintain measurement systems. As for my course, I will be dealing with scientific tools and electronic devices — not any musical instruments, to carry out scientific research especially experimentation which involves a lot of laboratory work.

Out of twenty IPTA’s, only five institutions offered a programme with instrumentation based so far. They are Universiti Teknologi MARA, Universiti Putra Malaysia, Universiti Malaysia Pahang, Universiti Teknologi Malaysia Melaka, and Universiti Malaysia Terengganu. However, in other countries, instrumentation science has been acknowledged earlier than us. They even have a special classification for it. For example, there is the Department of Instrumentation Science in the University of Pune which offers a wide area of interdisciplinary research activities such as sensors, embedded instrumentation, analytical instrumentation using LabVIEW and virtual instrumentation, photonics and laser-based instrumentation.

Instrumentation science mostly comprises the study of physics theories. In general, physics theories, majorly the electric and electronic studies, are applied for creation, modification, and maintenance of components used in operating a system. By powering the physics concept, a person will build up a firm base of instrumentation science to further expand it to a level where it is very crucial to satisfy current needs in line with the rapid growth of technology nowadays. For instance, we have seen the revolution in kitchen utensils for cooking purposes — in the past, mortar and pestle are used to crush substances but now we have blender. Apart from that, ancient people cooked rice by boiling method. This method used firewood and charcoal as fuels to produce fire which takes a longer time for the burning process and besides the fire strength is uncontrollable. They need to look after the cooking. When the world is modernized, people start to use the gas stove. Today is even more easy and quicker with the production of induction cooker which functioned using the electrical power source. The temperature and time can be set. This is rather convenient when people, especially housewives are likely to be busy with their career and has little time to cook. This is all about revolution in instrumentation system. Can’t you see how huge the contribution of instrumentation science in our daily life?

Enough about instrumentation in the knowledge perspective. Let’s move on to see how this particular field can affect the research and development (R&D) sector. One of our challenges in Vision 2020 as stated by Tun Dr Mahathir Mohammad is to establish a scientific and progressive community. It is crucial for us to overcome the challenge in order to achieve our goal to be an advanced country. Why is that? Most of advanced countries such as Sweden, Germany, Norway and United States of America are highly develop in technologies which allow them to be giant manufacturers. This empowers the countries to be the major exporters all over the world. The achievement is encouraged by excellence in R&D because without a broad and comprehensive research in science and technology, they are unable to invent new products and also increase the efficiency of existing products.

Hence, our country needs to excel in R&D to progress further. What are we actually lacking? It is the weakness in technological resources that limits us to do well in R&D? Instrumentation science is the main key of a successful technological breakthrough because we know that technology involved a lot of scientific work with full use of instruments.
An article in *The Star* (Monday, 9 July 2012) highlighted “Call for ‘scientific power’ boost” which said that leaders in Beijing want more innovation in science and technology. What does this mean? Leaders all over the world is urging and emphasizing on technological development. Besides, we should put effort in reducing our reliance on import technology. Every year, major expense is issued for the purchase of technology from other country. If we have enough capability to produce our own technology, we don’t need to buy from foreign countries. No instrumentation, no research and when no research, no technology. Later, we will have to hire foreign workers who have already explored the field many years ago in various expertise. In addition, we must make counter offer by giving higher salary and allowances because to compete with other associations which may also need to do the same things.

We have enough human resource — the academician, scientists and researchers. They are the professionals, but they have no back up to assist them in gathering data. We are thirsty for expertise in instrumentation science to carry out research. With their presence, they can help the professionals to handle the equipment, devices and tools — a technical support. The curriculum structure of instrumentation science in universities should give the opportunity for the students to explore the technology used in other countries that have not been adopted yet in this country. This exposure is important, so they can learn about the operating mechanism of more complex machines, sophisticated devices and gadgets. They will bring back the knowledge obtained as a reference material to start a new research or to accomplish incomplete researches. There are just so many things to be discovered with instrumentation science in diverse area such as medicine, electronics, transportation, military, sports, manufacturing industry, agriculture, construction, and education. Instruments are needed widely — surgical instrument, electronic instrument, aircraft instrument, optical instrument, precision instrument, etc.

To summarize, we need a plan of strategic enhancement in instrumentation science field. We should realize that the world is changing so fast. Let the young Malaysians know that learning instrumentation science can be exciting and rewarding. I hope that people will not treat graduates in instrumentation science like second class engineers. Everybody play different important roles to lead our country in order to become a highly-developed country.
Emeritus Prof Augustine S.H. Ong — bestowed Malaysia's prestigious 2013 Merdeka Award for Health, Science and Technology

Emeritus Prof Augustine Ong Soon Hock was born in 1934, in Malacca and was raised by his paternal grandmother in a small rubber plantation in Malacca, following the death of his father when he was seven years old. Academically inclined since his early years in school, his academic pursuit began when he decided to challenge himself with chemistry, physics and mathematics during his secondary residential education at St Francis Institution in Malacca.

Excelling in school, he was accepted for pre-university education at the St Johns Institution in Kuala Lumpur. He attended University of Malaya between 1954 and 1959 and graduated with a Bachelor of Science (First Class Honors), and subsequently an MSc. His passion for chemistry showed in his excellent academic achievements, where he was awarded a Gold Medal for chemistry. “It is a way for me to understand nature,” he says. “Chemistry is a powerful discipline to explain nature”, he opined.

Dr Augustine Ong began his career as a lecturer with University of Malaya in 1959. He was a Fulbright-Hays Fellow at the Massachusetts Institute of Technology USA from 1966 to 1967 and prior to this he read for a PhD in Organic Chemistry at the University of London King’s College, 1961–1963.

In 1970, he was appointed as a senior lecturer at the University of Science Malaysia, and was promoted to the position of Professor of Physical Organic Chemistry in 1974. Seeking to expand his knowledge, Dr Augustine took a one-year sabbatical leave to attend the University of Oxford as Visiting Professor at the Dyson Perrins Laboratory in 1976. On his return to USM, he was appointed Dean of the School of Chemical Sciences at USM from 1977 to 1981. From 1981 to 1985 and 1990 to 1991, he was appointed Visiting Professor at the same university.

Between 1959 and 2011, Tan Sri Ong authored or co-authored 400 articles. “All these articles arose from ideas,” he says. “My strategy has been concentrated on the world of ideas. Ideas are top priority in getting new findings and inventions.”

Together with his colleagues, Prof Ong also co-authored two books. His first book with SH Goh and Rayson L Huang, The Chemistry of Free Radicals was published in 1974. The book has been used by the University of Oxford as a resource material. His significant research findings include the conformation of free radicals and SH2 cleavage of t-butyl peroxide. Along with his colleague, Prof Etsuo Niki, he co-authored a book on free radicals and antioxidants entitled Nutrition, Lipids, Health and Disease in 1995.

Prof Ong obtained his first patent from Britain in 1974 on lipids research of olein-stearin separation method. He later added 15 more patents to his name in palm oil research. These patents are from the US, the UK, Japan, Australia and Malaysia. His research on palm oil covered several aspects of palm oil; from its chemical composition, nutritional value, technical training and to its waste treatment. He was also a co-researcher in the isolation of tocotrienols from palm fatty acid distillate. Tocotrienols, an anti-oxidant, has beneficial effects on brain neurons, is loaded with anti-cancer properties, and lowers the levels of bad cholesterol. His research inspired other scientists in other parts of the world to conduct similar research on palm oil.

The conversion of palm oil to biodiesel was conceptualised by Prof Ong in 1981. The project began with a pilot plant study, went on to field trials and subsequently proceeded to mass production for commercialisation in Malaysia, Thailand and South Korea. Today, Malaysia is one of the world’s leading biofuel producers, with 58 plants approved for production.
He has worked tirelessly and passionately in advocating and promoting Malaysian palm oil to the world. Just shy of one month in office, as Director-General of the Palm Oil Research Institute of Malaysia (PORIM), had to deal with the Anti-palm Oil Campaign from the American Soybean Association (ASA) in March 1987. He spent two years in challenging the anti-palm oil campaign. As part of his efforts to counter the fight against palm oil, he advised the US Food and Drug Administration on the nutritional aspects of the palm oil, addressed the American media and nutritionists and established several nutrition advisory committees worldwide. Dr Ong used research and scientific evidence to convince his detractors of the health benefits of palm oil. “Although efforts made to work out peaceful arrangements with the ASA were attempted, these were turned down. We had no choice but to counter the campaign which was essentially a trade issue under the guise of health”, he says.

The ASA called for a truce in 1989, ending the Anti-Palm Oil Campaign. Today, scientists acknowledge the nutritional value of palm olein, a new source of healthy oil with nutritional values at par to extra-virgin olive oil but at a fifth of the price. “The Campaign stimulated a lot of research all over the world, some sponsored by PORIM. It was a blessing in disguise. These efforts helped us to realise that palm oil is not harmful to health and that it has almost a perfect structure for good health”, he adds.

Prof Ong continues to devote his time and energy to learning, discovering, and enhancing the chemistry and technology of palm oil. Between 2006 and 2011, he conducted continuous research on palm oil and contributed to several discoveries. Among them is the sn-2 hypothesis, proposed with his colleague Dr S.H. Goh, which showed that palm oil is less fattening than corn oil and soy bean oil, based on comparisons of their triglyceride structures. He created a new palm oil milling process which resulted in zero waste. The patented process was based on a concept that oil palm fruits are edible, including palm puree fractioned upon removing crude palm oil, thus creating a new source of healthy food for the world. The novel process identified new sources of carotenes, vitamin B complex, beneficial polyphenols and vitamin E including tocotrienols. Prof Ong also created a new 'green' product by expoxidising used cooking oil which is very beneficial to the environment.

His love for science is not only limited to research and development, but also in nurturing new scientists, technologists and inventors. He established the Malaysian Invention and Design Society (MINDS) in 1987 and remains its President today. MINDS was established to encourage people to think independently and creatively without barriers. MINDS is also designed to assist scientists and technologist to develop their ideas into inventions, and to commercialise the innovations. “Ultimately MINDS encourages every Malaysian, hopefully Asians, to believe in themselves; to think and provide solutions for development and for life. When we deal with nature where science can play a role, members of MINDS could be trained to deal with the material world and use knowledge of science to provide solutions,” he reiterated.

Tan Sri Ong has received various awards for his achievements. In 2009, Tan Sri Ong was made Honorary Fellow of The International Society of Professional Engineers in Los Angeles California. The International Federation of Inventors’ Association awarded him the Officer of The International Order of Merit of the Inventors in 2009 and in 2010 he was presented with a medal and certificate on the 20th Anniversary of the Polish Union of Association of Inventors and Rationalizators. In 2011, he was made Fellow of King’s College, London, and received the Palm Oil Industry Leadership Award.

A discussion on Malaysian palm oil rarely takes place without the mention of Prof Ong, who is considered by many to be a ‘father figure’ in the Malaysian palm oil industry. He played an important role in overturning the negative campaign against palm oil and has set a legacy that continues to strengthen the palm oil industry and its contribution to the Malaysian economy. A pioneer in Malaysia’s palm oil industry, he has paved the way for many scientists to build on his work in supporting the industry.

A father of four, septuagenarian Augustine Prof Ong remains active in the palm oil industry today, as he continues to nurture his passion for chemistry and for nature. Currently, his research is focused on environment-friendly projects that could help the world reduce its carbon footprint.

As an eminent scientist, innovator and inventor he continues to guide and nurture younger scientists in their quest to achieve world-class breakthroughs. His unerring commitment and dedication truly personifies the Spirit of Merdeka and its pursuit of excellence.
Innovation is now acknowledged as the key driver of competitiveness in the new Millenium. Countries and companies which invest heavily in innovation tend to stay ahead of competition. While those which fail to engage and embrace innovation effectively continue to struggle to deliver sustainable growth. Experts predict that the coming years will witness even more increasing significance of innovation. Many now crave for the right formula to make innovation work for them.

WIF-KL made its debut in 2012 as a global forum to discuss and exchange views on the many challenges of innovation. It attracted the participation of many international experts in the various levels of innovation, from the grassroot to industry and government. It was a good start for WIF-KL which aims to one day be a meeting of global minds on innovation and its many challenges.

WIF-KL 2013 will be held in Kuala Lumpur from 12–14 November. Judging by the turn-out in 2012, the attendance this year is expected to be even higher. We have chosen the theme, “The Future We Desire” for this year's WIF-KL 2013.

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Announcements

TRSM

Top Research Scientists Malaysia

Vision

To build a culture of excellence in scientific research

Mission

To develop a knowledge hub of top notch Malaysian Research Scientists

Objectives

- To develop a list of leading research scientists
- To recognize and showcase top Malaysian research scientists as drivers of the national STI agenda
- To identify Malaysian research scientists with pioneer mindset to move the country forward in an innovation-led economy
- To build a critical mass of leading research scientists

Eligibility

- The recognition is given to Malaysian research scientists working in Malaysia whose outstanding achievements in STI have been nationally and internationally recognised
- At the time of application, the candidate should be actively involved in research in the last 5 years with at least 10 years cumulative contribution towards the progress of STI

Application

- Online application is open throughout the year at www.mytopscientists.org

Malaysian research scientists contribute significantly to the economic growth of the nation via generation and dissemination of knowledge, wealth creation and the productive pursuit of scientific excellence for the benefit of humanity. In relation to this, the TRSM project intends to acknowledge their contributions and showcase the accomplishments of Malaysian research scientists as role models of excellence, mentors to the next generation and leaders to forge ahead with the STI agenda of the nation.

The accumulation of expertise through this initiative would simplify the process of identifying areas of need and interest to the country which subsequently allows the selection of the best candidate to pursue those areas. In addition, the TRSM list would serve as a gateway for the international scientific community in particular academia, industries and international STI organizations that wish to seek top-notch STI expertise in Malaysia. The TRSM project would also be a good reference guide to facilitate nomination of outstanding Malaysian candidates for various national and international STI awards.
Selection Criteria

Privileges

- Facilitation by ASM for attachment to world-renowned research laboratories and centres of excellence by leveraging on ASM’s international and national linkages
- International Fund Facilitation Programme (IFFP) to obtain international R&D grants
- Profiled in leading media
- Featured in STI databases as ‘Top Research Scientists Malaysia’ for promotion and positioning
- Featured in a book on ‘Top Research Scientists Malaysia’ to be published by ASM
- Preference for research funding

The Top Research Scientists Malaysia (TRSM) will be selected through an objective and standardised scoring mechanism based on the following criteria:

(i) Knowledge Generation
(ii) Knowledge Dissemination
(iii) Impact of Research Output

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