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-PRENEUR VERSUS -PRENEUR

ACHIEVEMENTS



It has been some time now that PoCResT was established and each year this center moves to fulfill its objectives and strive for excellence. In 2015, we concentrate more on the management of our center in ensuring that it is reachable by the interested researchers and related industries. The strategy is to revamp our web page (http://ios.uitm.edu.my/ pocrest/) and boost networking. By doing that for 2015, 85.7% of the total equipment usage charge comes from researchers outside Universiti Teknologi MARA and industries interested

in knowing the properties of their product. We are glad with the acceptance of the outside community on the service we provide and with continues support we plan to do better.

For the year 2016, it will be the most challenging years with the economic crisis resulting to shrunken of funding for research. We need to work within this limitation and we believed with proper networking and planning we will excel. Our main research areas for the year 2016 until 2018 are as follows:

1) Polymer matrix improvements and modifications 2) Polymer composite as implant materials 3) Hybrid reinforced polymer composite system 4) Bio-based filled polymer composites

We would like to welcome mer composites nationally our new permanent and associate members to the center and hope that everybody will work hard as to push our center to be one of the recognized center in the field of polymer and poly-

and internationally. Not to forget the contribution from the previous associate members, your contribution will always be remembered and acknowledged.

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UiTM once again successfully advocate the Invention, Innovation & Design Exposition (IIDEX) 2016 with overwhelming participation from multidisciplinary areas including small medium enterprises commercial institutions, institution of higher learning and also secondary school. The chosen theme for IIDEX 2016 was "Revving Innovation Soaring Commercialisation"

Polymer Composites Research and Technology Center (PoCRest) also does not miss the chance to participate in this event that was held from 20th - 23rd September 2016 at Dewan Agung

Tuanku Canselor (DATC) where we had sent out two team to present their projects.

The first team which led by Dr Ahmad Zafir Romli and members: Dr Siti Nur Liyana Mamauod, Muhamad Faizal Abd Halim and Noriman Selamat was presenting their project entitled "The Light Weught IBS (L-IBS)" and won bronze medal. The second team whom led by Dr Siti Nur Liyana Mamauod and members; Dr Ahmad Zafir Romli and Mohd Ismail Rifdi Rizuan was presenting their project entitled "ePO Plasticised SBR/r-NBR" and won silver medal.

http://ios.uitm.edu.my/pocrest/

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GENERAL KNOWLEDGE

RUBBER COMPOUNDING: BASIC INGREDIENTS & FORMULATION – MUHAMAD FAIZAL ABD HALIM, RASSIMI ABD GHANI & NIK NOOR IDAYU NIK IBRAHIM

Introduction

Rubber compounding is the complex, multidisciplinary science of selecting and blending the optimum combination of elastomer and other ingredients that meet the performance, processing, environmental, and cost requirements for rubber goods made and used in commerce. In theory, the blending of rubber and vulcanizing agent will produce a rubber compound. However, the combination of only those two will fabricate a weak product, which is low in mechanical properties and environmental resistance make it almost unusable. Hence, other compounding ingredients are blended into the rubber to produce the compounded rubber that has many unique properties which is not found in other materials such as dampening properties, abrasion resistance and high elasticity. There is a wide range of compounding ingredients when it comes to designing a rubber formulation such as activators, accelerators, antioxidants, plasticizers, fillers and many more. In most cases, end-product performance will be the main objective in selecting the compounding ingredients. By mixing other ingredients into the compounded rubber, not only the mechanical strength of rubber will increase, but also other characteristics such as service life performance and even create the properties that do not naturally occur.

The main objectives of rubber compounding are as follows:

- To produce a useful elastic product by means of addition of cross-linking agent.
- To produce a vulcanized rubber product that meets various physical properties such as hardness, modulus, tear strength and abrasion resistance.
- To ensure that the rubber compounds can be easily converted or processed into the shape and form of the final products by the addition of processing aids.
- Cost considerations, for example by the addition of cheap fillers.

Basic Compounding Ingredients 1. Rubber/Elastomer

The most important ingredient in the rubber compounding is the rubber or elastomer itself. Elastomers can be divided into two main categories that is natural rubber and synthetic rubber. Natural rubber (NR) is collected in a form of milky fluid or latex, which is produced from rubber trees known as *heveabrasiliensis*. The chemical structure of pure NR consists of polyisoprene (Figure 1), which can also be

produced synthetically such as in the production of synthetic Polyisoprene Rubber (IR). The chemical structure of NR and IR are similar. The difference is only on the percentage of cis-1,4 content in which NR is 100% cis-1.4 whereas IR consists of 96% to 98% depending on the type of catalyst used during polymerization. Generally, synthetic rubber is made from the polymerization of a variety of monomers including isoprene (2-methyl-1,3 butadiene), 1,3-butadiene, chloroprene (2-chloro -1,3-butadiene) and isobutylene (methylpropene) with a small percentage of isoprene for cross-linking. Some of the most important and widely used synthetic rubbers are Ethvlene Propylene Rubber (EPM), Polybutadiene Rubber (BR), Styrene Butadiene Rubber (SBR), Butyl Rubber/Isoprene Isobutvlene Rubber (IIR). Nitrile Rubber (NBR) and Polychloroprene Rubber (CR).



Figure 1: Chemical structure of natural rubber

2. Vulcanizing/Curing Agent

Vulcanization, named after Vulcan, the Roman God of Fire, is a process whereby chemical crosslinks are introduced to "tie" the molecules together to form a 3-dimensional (3D) network that prevents flow and provide the elasticity characteristic by converting the elastomeric liquid into an elastic solid. In practice, it is achieved by subjecting the ready mixed (compounded) rubber to heat and pressure. Vulcanizing agents that are normally used are sulfur, peroxides, metal oxides and resins.

<u>Sulfur</u>

Sulfur is the most common curing agent used because of its abundance, cheap and easy to mix with the rubber. It reacts chemically with the raw gum elastomer forming crosslinks between the polymer chains, resulting in a more dimensionally stable and less heat sensitive product. Its' cost is relatively low, but its' function is essential. It is available in different particle sizes (fineness) as rubbermakers sulfur, and can also have a small quantity of oil added to reduce its dust in the air during handling. Rubbermakers sulfur is sulfur suitable for vulcanizing rubber; it has a low ash content, low acidity and sufficient fineness for adequate dispersion and reaction.

Peroxides

Peroxides vulcanization can be used to cure many types of elastomers, since, unlike sulfur, they do not need the presence of double bonds or unsaturated bonds. However, peroxide vulcanization is not recommended for some elastomers such as IIR (Butyl Rubber), CIIR (Halobutyl Rubber) and ACM (Polyacrylic Rubber) because of their tendency to decompose. Since peroxide vulcanization does not require double bonds, thus they can be used to cure saturated elastomers including polyurethanes, certain fluoroelastomers, silicones and many others. Although not nearly as popular as sulfur, peroxides have a distinct place in rubber compounding. and a major curative for silicone rubber. In peroxide vulcanization, all the other ingredients that are usually needed in the sulfur vulcanization such as Zinc Oxide and Stearic Acid are unnecessary in this method which led to the production of product with excellent resistance to compression set at high temperatures (70°C to 100°C).

3. Accelerators

Accelerators are usually understood to mean an organic chemical and as the name implies, it speeds up the rate of vulcanization. There are many accelerators available to the rubber chemist, grouped into several chemical classes. Some have a built in delay time, so that when heat is applied to the compound at the beginning of the curing process, no vulcanization takes place for a specified initial period of time. They are appropriately called delayed action accelerators. An example would be the sulfenamides. This delayed action is highly beneficial if a compound takes a long time to completely fill a cavity in a heated mold. The following is a summary of some typical chemical classes of accelerator available to the rubber chemist.

- Zinc dibutyl dithiocarbamate (ZDBC)
- Tetramethylthiuram disulfide (TMTD)
- Thiazoles
- Guanidines
- Sulfenamides

4. Activator

In rubber compounds, activator used to increase the cure rate by forming the chemical complexes with the accelerator, so that it performs more effectively. The most common activators for sulfur vulcanization are Zinc Oxide and Stearic Acid. These two materials, together with sulfur and accelerators, constitute the core system for the formulation. Zinc Oxide reacts with Stearic Acid to form Zinc Stearate.

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which is soluble in the rubber thus facilitates the crosslinking process. Zinc Stearate joins together with accelerator to speed up the rate at which sulfur vulcanization occurs. With sulfur alone, the curing process might take hours. However, with the addition of activator the curing process can be reduced to minute.

5. Antioxidants & Antiozonants

Antioxidants are necessary to protect other organic materials such as most elastomers from aging. Aging can be caused by the ravages of oxygen, accelerated by heat. Antioxidants are designed to slow this process and can act as free radical scavengers. Antiozonants such as p-phenylene diamines which provide sacrificial protection against ozone. An antidegradant is a compound material used to retard the deterioration caused by oxidation, ozone light or metal combinations. Table 1 shows some example of antioxidant, antiozonant and antidegradant.

Table 1: Examples of antioxidant, antiozonant and antidegradant

Antioxidant	Amines, Phenolic
Antiozonant	Wax
Anti- degradant	Ketone-amine, Aldehyde- amine, Diphenylamine, Phenol Sulphides

6. Processing Aids & Plasticisers

The functional role of process aids and plasticizers in rubber compounds is to reduce the frictional heating of the ingredients during processing. Process aids facilitate the incorporation of fillers and allow processing at lower temperatures and power requirements. Plasticizers soften the rubber compound, reducing modulus and hardness, increase elongation, and in some cases improved low temperature flexibility. They also improve the flow in extrusion and molding by making the uncured compound less elastic and reducing the viscosity and friction. At low level, plasticizers aid in the dispersion of filler while at higher amount, they reduce the uncured compound viscosity which often lowers the overall raw material compound cost but they also reduce the stiffness of the vulcanizate. Plasticizers are ranked according to how compatible they are with polar elastomers. Generally polar groups such as acrylates, acrylonitriles, glycols, aromatic/ phenyl groups and halogens add degrees of polarity. Olefins are nonpolar. Paraffinic process oils have little to no polar characteristics while process oils with aromatic content are compatible with semipolar elastomers.

7. Fillers

Filler has a significant influence on

the rubber processing and vulcanizate properties. Fillers are normally incorporated into the rubber compound to reinforce and improve the strength, color the material, extent and dilute, lower cost and change processing characteristics. The size and shape of filler particles have a major effect on the properties of end-product. Generally, filler with a particle size larger than 10 µm can reduce the physical properties by creating localized areas of stress. Fillers with a particle size between 1 and 10 µm such as ground limestone and clay, are relatively neutral on vulcanizate properties and serve as material and cost diluents. Fillers which improve strength and modulus properties have particle sizes from 0.1 to 1 µm. Higher level of reinforcement is achieved with fillers having particle sizes of 0.01 to 0.1 µm. Most commonly used fillers in rubber compounding are carbon black, calcium carbonate (CaCO₃) and silica.

Compounding Formulation

Designing and developing the right rubber compound formulation is vital to achieve the desired product performance and to meet the processing conditions, technical specifications and cost. The amount of each ingredient that is required to be added into the rubber must be specifically prescribed prior to the mixing process. This prescription is a compilation of various ingredients into a formulation. In the formulation, the quantity of each ingredient is quoted as an amount based on a total of 100 parts of the rubber or combinations of rubbers (or masterbatches) used. This notation is generally listed as phr (parts per hundred of rubber).

Hence, if we are to compare the outcome of two or more different recipes with respect to the processing characteristics or the physical properties of the final vulcanizate, the effects of varying any particular ingredient, from one recipe to another, can be easily identified. Table 2 shows the basic rubber compounding formulation.

Table 2: Basic rubber con	pounding formulation
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Material	Phr		
Rubber	100		
Sulphur	From 0 to 4		
Zinc Oxide	5		
Stearic Acid	2		
Accelerators	From 0.5 to 3		
Antioxidant	From 1 to 3		
Filler	From 0 to 150		
Plasticizer	From 0 to 150		

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The Many Uses of Rubber

The used of rubber, in one form or another began back in thousand of years ago and the used of its widen from day to day. Due to its beneficial properties including strength, elasticity, long lasting and many more make it a perfect material for the production of tires. As the most essential material in the tire production, careful selection of rubber and filler types along with the designing the right formulation is very important steps to optimize the performance, to maximize traction in both wet and dry conditions and to achieve superior rolling resistance which is all depending on the intended use of the tire.

Beside in the production of tire, rubber also finds application in ensuring the safety of children's playing space. Rubber playground mulch is a soft mat that able to reduce the risk of injuries when children fall while playing. It is generally made from 100% recycled tires, which make it affordable. This rubber playground mulch is a definite replacement to wooden mulch which is susceptible to rot, attract bugs and the risk of splinters. The use of rubber mat not only restricted to the playground but also as a house flooring and matting. Rubber floor mats and rubber flooring that widely install in the bathroom add a safety element to the human being. It is so because the rubber flooring or rubber mats stick to most surfaces without the use of adhesives and does not slip neither does the person who walks on the mat. The soft. elastic and slip resistant nature of this rubber flooring mat minimizes the risk of broken bones as it helps to cushion the joints between bones that would normally be impacted by a harder surface such as concrete.

Rubber also can be found in human clothing especially boots. In the past, rubber boots were made for industrious people as a safety boot due to the insulating properties of rubber that can prevent an electrical shock. But nowadays, rubber boots are worn by women and men as a fashion. There are many other applications of rubber in the market nowadays including hose, medical tube, automotive part, lattice mattress and many more.

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RESEARCH IDEAS & PRELIMINARY WORK

THE PREPARATION AND CHARACTERISATION OF RECYCLE WASTE CHROME TANNED LEATHER **REINFORCED UNSATURATED POLYESTER COMPOSITE** – SATARIAH TALIB & AHMAD ZAFIR ROMLI

In leather industries, almost 80% of leather worldwide using chromium in tannerv process which requires well managed of leather waste containing chromium. Precisely most common leather waste disposal method practiced is via landfilling or incineration. The oxidation of Cr III to Cr VI by oxidants such as peroxides and hypohalide occurs with ease. Cr VI is well known as carcinogenic and mutagenic element which is very harmful. Leather waste composed of collagen fibres with intrinsic strength. It is noted that inadequate study conducted and reported for animal based fibre reinforcement composites pertaining to thermal properties respectively. The dissimilarities of this research over others are the leather waste used in this research is devoted to one type of leather which is corrected leather and specifically selected from the tanning process of chrome tanned only. This is to ensure conclusion or the outcome of this study can be concluded pertaining to constant sources of leather type and tanning process.

This research serve as alternative of utilizing waste materials into environmental friendly heat insulation concurrently benefits the ecosystem and environment. Leather waste from footwear manufacturing company is utilised as reinforcing filler in unsaturated polyester composite (leather reinforced unsaturated polyester composite-LRUPC). This leather waste will be pulverized using 0.25 mm mesh size to obtain wool-like fibre and the fibre percentage will be varied by 0.5 % each up to 3.0 % based on weight percentage of matrix. LRUPC will be fabricated via compression moulding technique. Based on the excellence thermal properties owned by both filler and matrix, it is believe that the new vielded composite can be used as low cost thermal insulation panel. The properties of physical, mechanical, electrical and morphological behaviour of the leather waste reinforced UPC will be studied. LRUPC can be categorized into semi-biodegradable biocomposite which able to aid reducing environmental issue.



Figure 1: Waste Chrome Tanned Leather

Good to KNOW about reather

- The thickness of surface coating applied to the leather can be used to describe the genuine leather. For instance the leather that has a surface coating, the mean thickness of this surface layer, however applied, has to be maximum of 0.15mm or less, and shall not exceed 30% of the overall thicknesses.
- If the tanned hide or skin is disintegrated mechanically or chemically into fibrous particles, small pieces or powder and then, with or without combination of a binding agent, is made into tiles, sheets or forms, such products are not leather!
- Leather has an exceptionally long useful life which normally lasts about 5 times longer than fabric.

eral Guide: Location of Which best leathers are cut



First grade: best value, solid closed fibres and water resisting: Highly recommended and guaranteed for best wearing-Second grade: good value recommended: - Third grade: fair or doubtful value: Loose fibres and swell in

water IV - Fourth

EFFECT OF CNTs AND NANOSILICA ON THE PHYSICO-MECHANICAL BEHAVIOURS OF THE MODIFIED DGEBA-ATBN RESIN - HAIZUM FAIQAH MUSTAFA BAKRI, SITI NORQHALIDA MOHD FAUZI & SITI NUR LIYANA MAMAUOD

Diglycidyl ether of bisphenol -A (DGEBA-A) is one of the thermosetting epoxy resins that was used extensively in many applications due to their superior mechanical, chemical and thermal properties. However, this type of thermosetting resins produces high crosslinking density and hence causing the brittleness of the cured epoxy resin. The brittleness state of epoxy tends to lower the toughening properties and limits it application. Therefore, the toughening behaviour was improved by introducing liquid amine terminated polybutadiene (ATBN). The incorporation of ATBN introduce an elastomeric part in the epoxy matrix and it acts as a rubber phase to

absorb energy.

Although ATBN shows a positive impact on the toughness part but it will lead to decreasing of the modulus and strength of the cured epoxy resin due to the presence of the rubber phase. In this preliminary study, both modulus and strength will be alter by incorporating the nanofiller (NF). In order to see the effect of nanofiller on the modulus and strength, two types of nanofillers woll be used which are the carbon nanotubes (CNTs) and nanosilica (NS). The addition of these nanofillers will leads to the enhancement in strength and modulus of the cured epoxy resin and at the same time maintaining the good toughness behav-

iour. From this study, the effectiveness of nanofiller in the epoxy system will be identified not only based on the particle size, shape of NF but also the polarity as it is one of the identified parameters affecting the compatibility between matrix, filler and liquid rubber.



Figure 1: Carbon nanotubes





Figure 2: Nanosilica

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RESEARCH IDEAS & PRELIMINARY WORK

EFFECT OF SILANE COUPLING AGENT LOADING ON THE PHYSICO-MECHANICAL PROPERTIES OF NSI/ MWCNT REINFORCED m-EP/CTBN — SITI ROEYHAN SAAIDIN, NURHASANAH NORHISAM, SITI NUR LIYANA MAMAUOD & MOHD ISMAIL RIFDI RIZUAN

Good interaction between nanofiller (NF) and matrix is one of the factors influencing the strength of composites. The interaction between both phases can be improved by using silane coupling agent (SCA). There are few types of silane coupling agents that can be used but the difference between them is the type of functional group that is located at the second end of the chain (E2). Basically, on the other end of the chain (E1) has the same number and functional group which is 3 of OCH₃. Apart from improving the interaction between nanofiller particles and m-EP/ CTBN resin, SCA also acts as a dispersing agent where it will improve the dispersion of nanofiller in m-EP/CTBN resin. By improving the dispersion of NF in modified EP resin will directly improve the strength and load transfer efficiency.

There are few possibilities of interactions that might occurs when SCA is added. The interactions of SCA with other phases are depending on the step of adding SCA with m-EP/CTBN and NF. If the SCA is added into the m-EP, the SCA might react with this resin first before interact with the nanofiller particles. The other method is whereby the NF will be added in the solution of silane where the silane will react with the functional group of NF particles. In this study, sonication method will be used to enhance the dispersion of NFs in the polymer matrix. Both of these interactions will influence the properties of composites and this interaction is categorized as a chemical interaction. The m-EP/CTBN will reinforce with hybrid NF where these NFs are bridging together and form a cosupporting network.

In this preliminary study, two types of silane coupling agents will be used which are the E2 - (3, 4-epoxycyclohexyl) trimeth-

oxysilane and (3-Glycidyloxypropyl) trimethoxysilane where different percentages of SCA will be incorporated into the m-EP/ CTBN ranging from 1-5 % in order to study the effect of SCA on the physical and mechanical properties of composites. Besides that, the effectiveness of the route chosen for the addition of SCA into the dispersion of nanofiller particles in m-EP/CTBN resin will be studied using the Transmission Electron Microscopy (TEM). Therefore, by conducting the morphological analysis, physical and mechanical properties, the synergistic toughening effect of using hybrid nanofiller in the presence of SCA will be analysed.



Figure 1: The chemical structure of Carboxyl-Terminated Acrylonitrile-Butadiene Rubber

HYBRID NANOFILLER REINFORCED CARBOXYL TERMINATED POLYBUTADIENE MODIFIED EPOXY RESIN (m-EP/CTBN) - NOOR HIDAYAH JUAHIR, SITI NUR LIYANA MAMAUOD & AHMAD ZAFIR ROMLI

Epoxy resins are thermosetting polymers that exhibit brittleness, low fracture toughness and low impact strength that will limit their application in the industrial field that requires high impact strength properties for end products. The brittleness of the cured epoxy resin occurs due to the high crosslink density. Therefore, in order to make the system become more toughened and flexible, epoxy resin will be modified by introducing liquid rubber which acts as a toughening agent. The addition of liquid rubber will improve the toughening behavior but it will reduce the strength of the epoxy. Due to the reduction of strength, hybrids nanofiller will be introduced to reinforced the modified epoxy resin and hence, increase the strength of epoxy system. MWCNT and nanosilica fillers will be used. The nanofiller fillers will acts as coreinforcing agent in this system by forming stronger network between MWCNT and m-EP/CTBN. Epoxy resins and both nanofillers are polar substances and there are possibilities for these materials to have better interaction between them. Good interaction between fiber matrix phases will improve load transfer efficiency.

In this project, sonication technique will be used to improve the dispersion of

nanofillers in the modified epoxy resin. In order to study the synergistic effect of using hybrid fillers and the optimum amount of both nanofillers in the modified epoxy composite, thermal, physical and mechanical testing will be conducted such as the differential scanning calorimetry (DSC), tensile, impact tests, and etc. This project is expected to produce a new polymeric matrix (m-EP/CTBN) reinforced with hybrid nanofillers that shows the synergistic effect of combining two fillers and make the system becomes more effective than single filler system.

The essentials of this project which can improve the properties of some product range of coating industries for instance in automotive industries whereby the epoxy toughened with liquid rubber (CTBN) reinwith nanofillers (MWCNT/ forced Nanosilica) can be coated at vehicle's bumper and significantly reduce the impact energy experienced in an event of collision as the coating can absorb some amount of the impact energy compared to normal coating. This also can be applied by the sports industries which the products required to withstand or prone to a repeated amount of impact such as baseball's bat, hockey's stick, racquet and other related sports equipment. This can dramatically increase the in-life service while maintaining the aesthetical value of the products by reducing the amount of wear and tear. There are many other applicable possibilities from this research which can further help to improve the polymer industries to consistently evolve by incorporated science and technology towards the new and improved products range for future.



Figure 1: CTBN to be used in this research

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ESTABLISHED RESEARCH WORKS

THE DMA EVALUATION OF THE DAMPING PROPERTIES OF SIOX IN EPOXY SUBSEQUENT TO BATH SONICATION – NUR ZARIFAH RAZALI & AHMAD ZAFIR ROMLI

In this research, the SiOx was mixed with the epoxy resins as to improve the impact and damping properties of the epoxy especially upon impact. One of the methods that can identify these properties is by using Dynamic Mechanical Analyzer (DMA). The impact strength can be correlated with the damping behaviour of materials. Damping (tan delta, δ) can be explained as the ability of converting the mechanical energy into heat energy when the material is subjected to an external load. Figure 1 displays the temperature dependence of tan delta, δ (loss factor) for Ep-1%SiOx, Ep-2%SiOx, Ep-3%SiOx, Ep-4% SiOx, Ep-5%SiOx and blank epoxy at a frequency of 1 hertz (Hz). Each weight percentage of nano-silicon was sonicated at 3 different times, i.e. 15, 30 and 45 minutes, but excluded for unfilled epoxy which was stirred only by using a mechanical stirrer. In order to evaluate properties of damping, the temperature range of tan $\delta > 0.3$ is commonly used.

From Figure 1, it shows the differences of peak broadness between filled epoxy and blank epoxy. For epoxy nanosilicon-filled, the curves are wider than blank epoxy. A broader or wider curve indicates the activity of internal molecular motion: a matrix viscoelasticity, the grain boundary sliding (filler/ filler) and interfacial sliding (filler/ matrix) friction. These three factors produce heat. An energy dissipation rate emitted by these frictions is related to a damping of composite of material. The addition of nano-scale powder is substantially improved and enhanced the rate of dissipating energy in which the filled epoxy generates excess heat rather than blank epoxy (which is only contributed by matrix viscoelasticity). Thus, an increased rate of dissipating energy could be observed and the composite system is said to have an improved damping behavior. Table 1 shows the broadness of the peak for each loading and sonication times.

At 30 minutes of sonicating times, epoxy filled with 1% content of nano-silicon reached the maximum value of 0.69 and the corresponding temperature range with tan $\delta > 0.3$ was about 25 °C. Once the composite system achieves the highest values of tan δ (which in this case is 0.69), it denotes that Ep-1%SiOx (sonicated in 30 minutes) has good damping properties. A sufficient time of distributing nano-silicon contributes to the increased of internal friction of composite. The efficiency of sonicating time is vital in a matter of dispersing nano-powder in the matrix. The adequacy of timing may partially or fully disrupt/ separate agglomerates to primary particles or smaller in size of agglomerates and thus distributing it throughout the viscous matrix. After the nano-silicon is homogenizely distributed, the primary particles or smaller size of agglomerate will permanently constrain in the matrix. The uniform dispersion and size of cluster aid to the efficient increased of internal friction of composite. However, as the percentage of nano-silicon ascended, the tan δ of maximum values descended and the temperature ranged became narrow. It is clearly observed at the loading of Ep-2%SiOx (30 minutes) and Ep-4%SiOx (30 minutes) where tan δ magnitude were 0.55 and 0.54 respectively and the corresponding temperature range with tan δ > 0.3 were 25.8°C and 21.2°C respectively.

	tem				
A 1.00 • Ep-1%SiOx • Ep-2%SiOx • Ep-2%SiOx	B 1.00 0.80 - + Ep-1% SiOx • Ep-2% SiOx = Ep-2% SiOx	Samples	Temperature range, °C Tan delta, δ > 0.3	Tan delta, δ	Tg, °C
× Ep-4%SiOx	* Ep-4% SiOx		15 minutes sonicate		
		Ep-1%SiOx	71.4 - 98.5	0.56	85.6
e 0.40 -		Ep-2%SiOx	72.3 - 92.3	0.46	80.4
	0.20	Ep-3%SiOx	71.2 - 102.0	0.59	83.0
0.00 70.0 110.0 150.0 190.0	0.00 0.00 10.0 150.0 190.0	Ep-4%SiOx	69.3 - 94.4	0.59	82.6
Temperature/°C	Temperature/ °C	Ep-5%SiOx	72.1 - 90.9	0.48	80.3
	D 1.00 T		30 minutes sonicate		
• Ep-2%SiOx	0.80 - • Epoxy blank	Ep-1%SiOx	68.9 - 94.2	0.69	80.6
× Ep-4%SiOx ∞ Ep-5%SiOx		Ep-2%SiOx	71.4 - 97.2	0.55	81.9
	- 00.0 j	Ep-3%SiOx	71.8 - 95.8	0.59	79.8
0.40 -	E 0.40 -	Ep-4%SiOx	73.0 - 94.2	0.54	81.9
0.20 -	0.20 -	Ep-5%SiOx	71.2 - 96.3	0.57	82.9
			45 minutes sonicate		
Temperature /º C	Temperature /°C	Ep-1%SiOx	69.7 - 93.9	0.63	79.7
Figure 1: a), b), c) and d) shows Tan delta, δ of 5 different loading of nanosilicon which were sonicated at 15, 30 and 45 minutes and unfilled epoxy respectively.		Ep-2%SiOx	70.4 - 93.8	0.63	81.2
		Ep-3%SiOx	73.8 - 96.1	0.51	83.0
		Ep-4%SiOx	72.4 - 94.0	0.49	81.2

Ep-5%SiOx

Table 1: Summary of DMA data for epoxy-nanosilicon composites system

71.0 - 98.3

0.51

82.0

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POLYMER COMPOSITES RESEARCH & TECHNOLOGY

ESTABLISHED RESEARCH WORKS

THE ACCURACY OF TGA AS TOOLS IN MEASURING THE PERCENTAGE OF FILLER LOADING IN POLYMERS – MOHD HANIF KASA@ABDUL GHANI & AHMAD ZAFIR ROMLI

TGA (Thermogravimetric Analyzer) is one of the most common tools in measuring the percentage of filler in polymers. The simple setup couple with a very small amount of sample required make it the method of preference. But before we go further, let us recap the fundamental of the TGA. Basically the TGA consist of a micro balance coupled with the sample holder that was placed in a furnace (various temperature ranges depending on the TGA system). During the test, the furnace will be heated at a pre-determined heating rate to the desired end temperature (beyond the degradation temperature of the material to be analyzed). The sample required usually within the range of 5 - 10 mg and some system able to accept up to 20 mg either solid or liquid form. Now the question - Can we analyze the filled polymer sample? The answer is YES, but how accurate is the result? That will be discussed further in this article.

Most of the polymer products available are filled with fillers as to feed various purposes such as cost reduction, mechanical properties enhancement, physical properties enhancement and others. Therefore, it is critical to know the amount of filler being added into the polymer especially for quality control of product or for the purpose of reverse engineering. In order to study this, a simple experiment was setup by using unsaturated polyester as the matrix and CaCO₃ as filler at 10%, 30% and 50% w/w accordingly. Both materials are mixed using a mechanical stirrer for 4 minutes before methyl eter ketone peroxide (MEKP) was added and stir for another 4 minutes and poured into a mould of 200mm length X 200mm width X 10mm thickness. The sample was left at room temperature for 24 hours before demoulding and post cured for another 24 hours at 70°C in an air circulated oven. The schematic diagram and the labelling of the samples are as shown in Figure 1 where the pink coloured dot is the location of sampling for the TGA analysis.

Referring to Figure 1, the sampling was taken at various places as to indicate the relevancy of the location of the sampling for the TGA analysis.

As to ensure homogeneity of the samples to be taken, drilling method was used where the sample taken is at the center and not on the surface as shown in Figure 2. The debris will be taken and weight accordingly prior to the TGA tests. For the TGA test, the test will be carried out at 10 °C/min and stop at 600 °C. The result obtained are as shown in Figure 3, 4 and 5.

Looking at Figure 3, the scattered trend of the histogram showing the variability of the result obtains ranging from

about 9% up to 15% of CaCO₃ being detected. The variability in the results obtain are most probably due to the uneven mixing of the CaCO₃. It seems that the filler is much harder to evenly mix at a lower percentage compared to at a higher percentage as shown in Figure 4 and 5. The graph shown in Figure 4 and 5 indicates a stable mass of filler being detected and the amount shown are the same as the amount of filler being incorporated in the polymer during mixing.

As the conclusion by looking at the results - Is it possible for the TGA to accurately measure the percentage of filler loading in polymers? The answer is YES and NO. The YES answer is at high filler loading and NO is when the low filler loading in polymer in this case at 10%. It is, however debatable where one can argue about the effectiveness of the mixing method. In whatever case, we can say that the lesser the percentage of filler in the polymer the higher the possibility of having uneven mixing. In additional to that, one has to take note that even mixing does not mean that good dispersion of the fillers will be obtained. We might have even mixing of agglomerate filler in the polymer system.





Drill bits

Figure 2: The drilling method used for the TGA sampling



Figure 3: TGA results for 10% loading of $CaCO_3$ in polyester



Figure 4: TGA results for 30% loading of CaCO₃ in polyester

Figure 1: The schematic diagram of the CaCO_3 filled unsaturated polyester composite sampling for TGA analysis



Figure 5: TGA results for 50% loading of $CaCO_3$ in polyester

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ESTABLISHED RESEARCH WORKS

LIQUID RUBBER MODIFIED DGEBA REINFORCED WITH CARBON NANOTUBES (CNTs) - SITI NORQHALIDA MOHD FAUDZI & SITI NUR LIYANA MAMAUOD

Epoxy resins (EPs) are one of the most utilized thermosetting used nowadays as matrices for many industrial applications. However, they are inherently brittle and hence have reduced damage tolerance. In order to remain competitive as materials of choice for many applications such as adhesives and composite matrices, epoxies should be modified to improve their fracture toughness.

Over the years, many attempts have been made to modify epoxy by adding either rubber particles or fillers in order to improve the matrix-dominated composite properties. One of the successful methods to toughen EPs is the incorporation of rubber phase into the brittle epoxy matrix, which may be achieved by the use of reactive liquid rubber. Carboxyl, epoxy and amine terminated butadiene-acrylonitrile copolymers (CTBN, ETBN and ATBN) are widely used as toughening agents. Amine-Terminated Butadiene Acrylonitrile (ATBN) is initially miscible with the epoxy but during the polymerization the rubber phase separates due to slight immiscibility with the matrix.

The addition of 5% ATBN improved the fracture toughness of epoxy, but somehow decreased its modulus and strength. Thus, the research effort has been made towards the modification of matrix resin system. Of the various nanofillers used to modify polymer matrices, CNTs are one of the stiffest known fibers which possess a high tensile strength and modulus. By using mechanical testing such as tensile test and impact test, the results showed that 0.05% CNT is the optimum amount of loading to increase the tensile strength of modified epoxy by 73% and its modulus by 95%. However, 1.0% CNT is the optimum amount of loading to increase the toughness of modified epoxy by 41% and the impact strength by 24%. By doing the density test, it shows that the incorporation of CNTs can produced a lightweight material which is suitable for structural applications. The morphology analysis was conducted by using Field Emission Scanning Electron Microscopy (FESEM) to study the structures of epoxy-ATBN/CNT blend.

A two-phase network was observed with the epoxy as the continuous matrix and the ATBN as a dispersion phase. FE-SEM analysis indicated that the dispersed liquid rubber particles act as stress concentrators during fracture, which induces delocalized plastic deformation of the matrix around the particles. At 0.05% CNT, the cavitation sizes are bigger. As the percentage of CNT added increased, the number of cavitation of rubber particles became smaller in size but many in numbers. The incorporation of CNT led to significant improvement of fracture toughness of the unreinforced modified epoxy resin. The toughening effect becomes more apparent at high CNT% loading due to its high aspect ratio and ATBN flexible elastic behavior. It can be observed that the higher the CNT% added, the rougher the surface of fracture samples. This is in contrast with smooth surface of the unmodified epoxy provides a clear indication for the brittle fracture behavior. The strongly bonded CNTs significantly absorb fracture energy when pulled out from the matrix. CNTs serve as effective crack bridges inducing mechanical interlocking with the matrix material.



Figure 2: SEM image shows dispersion of ATBN in epoxy





Figure 1: The shiny and very smooth surface of the unmodified epoxy provides a clear indication for the brittle fracture behavior

Figure 3: Micrograph of tensile fractured surfaces of reinforced modified epoxy: at 0.05% CNT: (a) X5,000, (b) X20,000, at 0.3% CNT: (c) X5,000, (d) X20,000, at 0.5% CNT: (e) X5,000, (f) X20,000, at 0.7% CNT: (g) X5,000, (h) X20,000, at 1.0% CNT: (i) X5,000, (j) X20,000

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ESTABLISHED RESEARCH WORKS

COMPRESSIVE PROPERTIES OF THREE LAYERS GLASS FIBRE REINFORCED UNSATURATED POLYESTER FILLED WITH P84 POLYIMIDE COMPOSITE - NIK NOOR IDAYU NIK IBRAHIM, AHMAD ZAFIR ROMLI & SITI NUR LI-YANA MAMAUOD

Compressive test is the opposite of tensile test where the material was subjected to compression force intending to reduce size. In this study, the three layers glass fibre laminates were fabricated using hand lay-up technique where the matrix unsaturated polyester was filled with P84 Polyimide at three different loading amounts; 1, 3, and 5 wt%. The laminates were then cured at 60°C for one hour and post-cured at 90°C for 24 hours. The cured sample then cut into 78 mm in length and 14 mm in width. The compression test was performed on AG-X Shimadzu Universal Testing according to ASTM D695 at 1.3 mm/min test speed.

Figure 1 and 2 shows the graph of compressive strength and compressive modulus. The addition of 1 wt% P84 polyimide as the reinforcement inside the matrix resulted in the improvement in the compressive strength and compressive modulus at about 7% and 35%, respectively. The incorporation of P84 Polyimide particles inside the matrix system had assisted in bearing the compressive load applied on the sample since the P84 Polyimide itself is the high mechanical strength material. As the loading amount of P84 Polyimide was increased to 3 and 5 wt%, the compressive strength and modulus started to decline. The reduction in strength and modulus with the increased of filler loading was believe due to the filler agglomerations that cause the stress concentration sites. In addition, P84 Polyimide is usually used as low level filler where a small amount of P84 Polyimide can extend the performance of standard material. Since the composite system consist of glass fibre as the main reinforcement, the addition of excess P84 Polyimide resulted in the crowded composite system which can be the weakness point. 1 wt% of P84 Polvimide seems to be enough to increase the compressive strength and compressive modulus since the powder P84 Polyimide can be categorized as a light material, thus a high amount of P84 Polyimide was already obtained at low weight percent.

The fractured compressive samples then observed under Olympus Optical Microscope BX51. Figure 3 shows the image of sample which failed under compressive mode. The dominant failure observed on all composite samples was the kinking and fibres delamination. Kinking is defined as the highly localized fibre buckling. When the composite was subjected under compressive load, the stress bear by the matrix was divided or shared with the reinforcement material (fibre and filler). At certain point, when the maximum stress achieved, the composite developed microbuckling. As the load was kept applied, the composite can no longer contain the stress and started to kink. Figure 4 shows the illustration on the kinking failure mode. As can be seen from Figure 3, the composite failed due to the kinking and the delamination of fibre from the matrix. Filler agglomeration can be the stress concentration sites that will initiate the fibre-matrix delamination and crack propagation.







Figure 2: Compressive modulus of three layer composite laminates



Figure 3: Kinking failure and fibre-matrix delamination of the composite sample observed under optical microscope (5x magnification)



Figure 4: Illustration on the kinking failure

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POLYMER COMPOSITES RESEARCH & TECHNOLOGY

- PRENEUR VERSUS - PRENEUR - MARIATINI OTHMAN

The term 'entrepreneur' dated back to the 17th century to describe qualities of leadership and innovation in business. It has long since been established that certain skill sets are required for one to be an entrepreneur.

If 'businessman' is described as someone who invests in an established business or product, driven by profit and able to bring it to greater heights; an entrepreneur is described as a somewhat different animal. This unique species invests in original ideas, driven by the desire to induce change (that may not necessarily be profitable), and may be just as capable to bring the new idea to a great height. The effort and resources required in pushing an original idea in an untested market will naturally be more than venturing into a currently-hot business in a receptive market - but this is palatable to an entrepreneur.

A businessman seizes opportunities as they come along. Entrepreneurs create them.

A businessman conforms to market demands. Entrepreneur does more than just conforming – he introduces more than what the market desired, he attempts to change the market.

There is something rebellious about entrepreneurship – they walk on rugged terrain, their path unchartered and they are mavericks in their own rights. That said, there is also something romantic and heroic about them.

And then there is a special form of entrepreneurs called 'technopreneurs'.

Schumpeter in 1934 defined technopreneur as a person who destroys the existing economic order (creative disruption) by introducing new products or services, by creating new forms of organizations and by exploiting new raw materials.

In short, technopreneur is simply an individual who is technically competent as well as entrepreneurally inclined.

If entrepreneur is a subset of businessman, technopreneur is its subset.

In 2012, Prof. Dr Amin Soebandrio from the Indonesian Ministry of Research and Technology in his opening speech at the International Conference on Biomedical Science held in Bandung sent a strong message to the participants, 'Indonesia needs Researchpreneur', he insisted. By 'researchpreneur', he means the ability to integrate technology with market demands; a scientist who understands the market needs and does his research towards meeting them.

If technopreneur is defined as a businessman with technical competency, Prof. Soebandrio defined 'researchpreneur' as a researcher with entrepreneurial inclination.

The idea of an entrepreneur venturing into technology-based ventures is one that is easy to digest. A technopreneur would have the same skill-set as that of an entrepreneur and the technical field in which he would be playing in would be one that he is most knowledgeable about or interested in.

Would a researcher have the skill sets required to be an entrepreneur? Would he be entrepreneurial enough to be a researchpreneur?

A good researcher must be openminded and thinks critically as well as analytically. The person should also be hard working, focused and maintains current knowledge in the chosen field. The person has to be resourceful and inventive, honest and a team-player too. Will these traits fit the researcher to be a researchpreneur? But then, aren't those the common traits of an entrepreneur too?

In the Venn Diagram of –preneurs, where would researchpreneurs be?

There are a lot of studies conducted by various research groups from all over the world in trying to understand what makes a successful entrepreneur. There are conflicting school of thoughts on whether entrepreneurship is even trainable.

What makes entrepreneurs so entrepreneurial? Is there such a thing as 'entrepreneurial behaviour' and 'entrepreneurial thinking' that can be applied across all ventures, transcending all boundaries? Does one have to be an entrepreneur to be entrepreneurial?

Until researchers can come up with a precise and fool-proof formula to classify them, entrepreneurs, technopreneurs, reserchpreneurs, and all other -preneurs will continue to be dissected, their entrails read and scrutinised under the electron microscope.

The true entrepreneur is a doer, not a dreamer.

- Nolan Bushnell

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ACHIEVEMENTS



Invention, Innovation & Design Exposition (IIDEX) is an annual International Exhibition and Competition organized by Universiti Teknologi MARA (UiTM). The aim for IIDEX is parallel to the third thrust of the National Higher Education Strategic Plan which is Enhancing Research and Innovation. For the year 2016, it was held on 27 to 30 April 2015 at Dewan Agung Tuanku Canselor (DATC). The themed for IIDEX 2015 was "Bridging Innovation Commercialisation to Maximise". This exhibition was organised by the



Silver Medal

Research Innovation Business Unit (RIBU).

POLYMER COMPOSITES RESEARCH & TECHNOLOGY

For the IIDEX 2015, a team from PoCResT which including Mrs Roslinda Fauzi, Dr Siti Nur Liyana Mamauod and Dr Ahmad Zafir Romli was participated as one of the team that registered under Faculty of Applied Sciences (FSG) and Institute of Sciences (IOS) by presenting a project entitle "The Modification of Diglycidyl Ether Bisphenol A (DGEBA)/Amineterminated Butadiene Acrylonitrile (ATBN) Blend" which won a silver medal.



Left : Dr Siti Nur Liyana Mamauod, Right : Pn. Roslinda Fauzi

Livana Mamauod and mem-Two teams from PoCResT had participated in IIDEX 2016

which registered under Institute of Science (IOS). The first team which was led by Dr Ahmad Zafir Romli and members: Dr Siti Nur Liyana Mamauod, Muhamad Faizal Abd Halim and Noriman Selamat was presenting their project entitled "The Light Weight IBS (L-IBS)" and won bronze medal. The second team whom led by Dr Siti Nur

bers; Dr Ahmad Zafir Romli and Mohd Ismail Rifdi Rizuan was presenting their project entitled "ePO Plasticised SBR/r-NBR" and won silver medal. This annually event is one of the right track to nurture creativity and innovative thinking amongst researcher in sustaining a culture of innovation, creativity and commercialisation in UiTM.







4th PRIM Student Chapter Carnival, 8th-10th Oct 2016, USM Engineering Campus

On 8th to 10th Oct 2016, 4th PRIM Student Chapter Carnival was successfully held at USM Engineering Campus, Nibong Tebal. Dr Siti Nur Liyana Mamauod, one of PoCResT researcher had brought an undergraduate team of five; Noor Amirah Nabillah Ab Rahim, Nurhasanah Norhisam, Noriman Selamat, Rabiatul Adawiah Shahalan, and Siti Nurhidayah Che Aziz participating in this

event to encourage innovation, entrepreneurship, and creativity among undergraduate student. They participated in poster presentation presenting a project entitled "Influence of n-CaCO3/n-SiO2 on the Physicomechanical Properties of m-Ep/CTBN. They won fifth placed out of 20 teams including from Universiti Sains Malaysia (USM) and Universiti Malaysia Perlis (UniMAP).



Sample fabrications

- 25-tonne hot press
- 50-tonne cold press
- 20-tonne high temperature hot and cold • press
- Vacuum bagging system
- Pulveriser
- Steel grinder
- Precision sample cutting machine
- Digital Ultrasonic Cleaner

Testing and analysis

- · Tensile, 3-point bending and compression testing machine - SHIMADZU AG-X Series 50kN
- · Hydraulic fatigue/ dynamic testing ma-

chine with hydraulic wedge grip and compression ability - SHIMADZU SER-VOPULSER 50kN Dynamic and 75kN Static

EQUIPMENTS AND FACILITIES AT PoCResT

- Moisture balance analyzer
- Thermal conductivity machine (TeDCaS) RT up to 150°C with heater power variable
- Electronic density measurement of solid samples
- Dart drop weight impact testing machine INSTRON Dynatup® 9200HV capable of doing up to 70km/h of impact speed with variable tub
- Thermal gravimetric analyzer (TGA)-Netzsch (RT to 1000°C)

- Differential scanning calorimetry (DSC)— Netzsch (-150°C to 600°C)
- Metallurgical microscope Olympus with DIC capability
- Mini-Scanning Electron Microscope -FEI Phenom (up to 20k magnifications and with 'charge' reduction holder)
- · Creep testing machine

Others

- Balance/ analytical balance
- Air circulated oven
- Thickness gauge
- Thermal static chamber (fabricated)
- 20 channel temperature acquisition systems (fabricated)

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JNTEGRATJON OF KNOWLEDGE FOR ADVANCED RESEARCH OUTPUT



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Management of Institute of Science (IOS)

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Thank you.

INVITATION TO PUBLISH IN THE POLYMER COMPOSITES RESEARCH & TECHNOLOGY TECHNICAL BULLETIN

The "*Polymer Composites Research & Technology Technical Bulletin*" is an initiative from the Center of Polymer Composites Research and Technology (PoCResT) at the Institute of Science (IOS) with the aim of dissipating research information and findings related to polymer & polymer composite systems to other researchers/ students. This bulletin will be published biannually (June & December).

You can send articles about your research work to this email (tb.pocrest@gmail.com) and our editors will go through and notify if your articles were accepted to be published in the bulletin. There will be no fees as we believe in the sharing of knowledge.

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Please send your article with good English in MS Office format and we will do the final typesetting. The articles to be submitted must be not more than a full 1 page A4 size including image(s). The font must be either Franklin Gothic Book or Times New Roman at size 9 with single spacing and justified text alignment.

For any inquiries, please e-mail to: tb.pocrest@gmail.com

ABOUT THE CENTER OF POLYMER COMPOSITES RESEARCH AND TECHNOLOGY (PoCResT)

Center of Polymer Composites Research and Technology (PoCResT) in Institute of Science is established to be a base for researches that will have an impact to the academic and industrial practices and productivity through the understanding and application of fundamental and advanced polymer composites technology. Supported by a comprehensive research laboratory **(Polymer Composites Research Laboratory – PoCRe)**, researches under this center are focusing on the development of innovative polymer composites materials (thermosets, thermoplastics and rubber) which include the usage of functional fillers (bio-based and synthetic), advanced mechanical and physical testing and analysis, thermal and dynamic studies, and the studies on the innovative polymer composites fabrications and testing.

The members and associate members of this center are from various research backgrounds such as applied sciences and engineering. As to maintain the standard of researches to be accepted by the industries, few associate members were appointed from various private sectors. The center has dedicated external professors as academic advisors for the academic courses run by the center.

Until today, the center has produced many postgraduate students by research at MSc. and PhD. levels. On top of that, the center has many collaborative works and provides continuous testing services to researchers from within the university and also to the outsiders. With proper planning and support, the center aims to be one of the leading reference centers in the field of polymer composites in this country.