

POLYMER COMPOSITES RESEARCH & TECHNOLOGY

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INSIDE THIS ISSUE:

STYRENE BUTADIENE RUBBER (SBR): EMERGING PROCESS TO MET GLOBAL DEMAND (PART 1) 2

THE COCOON 3

SYNTHESIS AND CHARACTERIZATION OF HYBRID MWCNT-ALUMINA FILLED EPOXY NANO-COMPOSITES 3

THE EFFECT OF PULVERIZATION PROCESS TO THE PHYSICAL PROPERTIES OF KENAF 4

THE EFFECT ON TENSILE PROPERTIES TOWARDS THE AMOUNT OF EPOXIDISED PALM OIL (EPO) IN STYRENE BUTADIENE RUBBER (SBR) 5

LICQUID RUBBER MODIFIED EPOXY 6

EFFECT OF MASTER-BATCH METHOD ON GEL TIME AND THERMAL PROPERTIES OF THE UNSATURATED POLYESTER/ P84 (POLYIMIDE) COMPOSITES 7

BOOK REVIEW 9

ACHIEVEMENTS — TOP RESEARCH SCIENTIST MALAYSIA (TRSM); PROF DR HAZIZAN MD AKIL (USM)

We are glad to announce that one of the associate researchers at the Center of Polymer Composites Research & Technology (PoCResT) have received a Top Research Scientists Malaysia (TRSM) 2014 awarded by the Prime Minister Datuk Seri Najib Abdul Razak as an initiative driven by the Akademi Sains Malaysia (ASM). Prof Hazizan is one of the 34 scientists receiving the awards. They were given the recognition for pushing the boundaries of research and development to generate new knowledge, make discoveries, create value-added opportunities and translate their research into meaningful and impactful outcomes that significantly contribute to the socio-economic transformation of the nation.



Prof. Dr. Hazizan Md Akil—TRSM award receivers. Area of expertise; polymer composites, impact mechanics and sandwich structures.



The TRSM award.

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NEW APPOINTMENT — DR MOHAMMAD AZIZOL ABDUL WAHAB, SABIC PRODUCT & APPLICATION DEVELOPMENT CENTER



One of PoCResT associates, **Dr Mohammad Azizol Abdul Wahab** recently has been appointed as Lead Scientist (Elastomer) for SABIC Functional Polymer Research & Product, Technology & Innovation (T&I) which is based in Riyadh, Saudi Arabia. SABIC overtook Shell Chemicals to become 4th largest petrochemical company in the world after BASF, SINOPEC and DOW.

Dr Azizol was previously a Rubber Technologist at TARRC (formerly MRPRA) in Hertford, UK be-

fore joining PETRONAS Research, Technology & Engineering (T&E) as Senior Material Scientist specializing in polymer materials. His last position at PETRONAS was Senior Technology Specialist (Elastomer) and Manager for PETRONAS elastomer project.

Dr Azizol is a UK registered Chartered Engineer (CEng) with 17 years' experience in polymer and elastomer product development for automotive components and oil & gas applications. He obtained his PhD from London Metropolitan University and MSc in Polymer Science & Engineering from University of North London.

We wish Dr Azizol a successful career and we are looking forward to his continuous contribution to PoCResT. He can be reached by email at azizol@scientist.com.

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ELASTOMER BUSINESS AND TECHNOLOGY

STYRENE BUTADIENE RUBBER (BR): EMERGING PROCESS TO MEET GLOBAL DEMAND (PART 1) — DR M. AZIZOL A. WAHAB

Introduction

SBR consumption is dominated by the automobile industry for tyres and tyre products, where it is mainly used in the manufacture of the tread. The trends of size, type, and quality of tyres all impact the SBR market. Within the SBR market, different grades show different growth profiles. SBR in the tyre market is mainly segmented between solution and emulsion grades:

- Emulsion SBR (eSBR) is a commodity material sold in standard grades. It is consumed in the majority of tyre production, particularly low-end, low-cost, or general-purpose products
- Solution SBR (sSBR) is required for high-performance tyre treads where better abrasion, wet-dry grip, and rolling resistance performance are important

In contrast, due to its more costly manufacturing process, sSBR is used predominantly in tyre production.

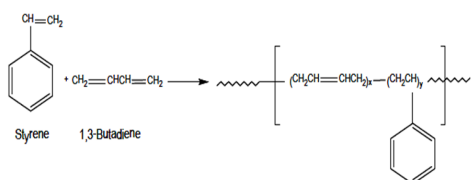
SBR was the first major synthetic rubber (SR) to be produced commercially. Early grades produced in Germany by Buna S and in the United States by Standard Oil Company/The Rubber Reserve Company during the Second World War. It was based on the emulsion polymerization technology developed by I.G. Farbenindustrie AG of Germany.

In 1960s, the first solution polymerized random SBR grades were produced commercially by Firestone and Phillips. The original aim was to produce SBR with lower styrene content to counteract the increase in styrene prices.

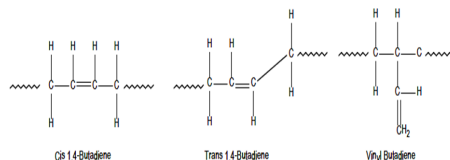
SBR is the workhorse of the rubber industry, even though some of its properties do not match those of natural rubber (NR). What it lacks in elongation, hot tear strength, hysteresis, resilience and tensile strength, it makes up for in better processability, slightly better heat aging and better abrasion resistance than NR. Probably the most important factors in the commercial viability of SBR have been its wide availability, low cost compared with those of all other SRs, ability to accept high filler levels, relatively stable price compared to NR and overall properties on a cost/performance basis.

Chemistry

SBR is produced by the reaction of styrene with butadiene monomers as follows:



Once polymerized, each butadiene component in the SBR takes one of three forms. It is important to note that the ratio of these governs the properties of the rubber.



The peculiar nature of the insertion of butadiene on the growing chain, i.e. the 1,4 and 1,2 additions, together with the two possible 1,4 addition isomers, cis and trans, some technologists suggest it would be more appropriate to refer to SBR as a four monomer copolymer.

Production Technology

There are three conventional routes used in producing solid SBR:

- Emulsion polymerization (Hot and Cold process) – usually known as eSBR
- Solution polymerization – usually known as sSBR.

Each process produces SBR grades with different properties:

- Hot emulsion polymerization is the original SBR process. The major advantage of this process is that these grades have exceptional processing characteristics in terms of low mill shrinkage, good dimensional stability and good extrusion characteristics. However, high levels of microgels are also produced so this process has almost entirely been replaced by cold emulsion. However, hot emulsion grades are still used in applications such as adhesives and flow modifiers for other elastomers where good flow properties are required.
- Cold emulsion polymerization produces SBR grades with superior mechanical properties, especially tensile strength and abrasion resistance compared to grades produced by the hot emulsion polymerization route. This process has largely replaced hot emulsion polymerization for the production of emulsion (eSBR) grades.
- Typical hot eSBRs contain 18.5 % cis, 65 % trans and 16.5 % vinyl butadiene forms, while typical cold eSBRs contain 12 % cis, 72 % trans and 16 % vinyl butadiene forms.
- Since emulsion SBRs are produced by a free radical polymerization process, their molecular structure can only be statistically controlled. In contrast, solution SBR processes are more versatile, and provide greater latitude for inde-

pendently controlling polymer microstructure and sequence distribution.

- Solution SBR (sSBR) grades have superior mechanical properties, particularly tensile strength, low rolling resistance and handling (encompasses traction under a variety of conditions and performance at different speeds and when cornering), when used in tyre applications. However, sSBR typically displays a narrower molecular weight distribution, which negatively impacts processability, when compared to eSBR.
- The ratio of butadiene configurations varies. Generally speaking, sSBR grades have a lower trans and vinyl butadiene content, and a higher cis than eSBR grades. Conventional sSBR has the following stereochemistry: cis: 35 to 50 %; trans: 35 to 55 %; vinyl: about 10 %; copolymer gel content is negligible. Vinyl content affects glass transition temperature and macrostructure, which in turn affects processability.

At the very beginning of their development, sSBR suffered from processing problems (mixing and compounding), due to their narrow molecular weight distribution (MWD). As a matter of fact, the main problem for sSBR to overcome has always been the non-interchangeability with the eSBR commonly used by tyre producers; the limited number of grades coming from the emulsion process makes it easier to switch from a supplier to the other, without any re-configuration of the processing machines and procedures. So one of the main efforts of sSBR producers has been the search for a better processability, which is obtained by modifying polymer macrostructure (i.e. MWD and long chain branching). In this sense, the use of coupling agents like SiCl_4 and SnCl_4 in the batch anionic synthesis provided a way to broaden the MWD, adding a star-shaped structure at the same time.

[In the next publication - SBR Part 2, Production Technology will be discussed in slightly more details which includes some comparison between technologies].

This article is not meant to cover every aspect in great details but rather providing readers with a snapshot and relationship between application, chemistry, production technology and market. If you have any question or would like to find out more information related to this article, please feel free to contact Dr Azizol at azizol@scientist.com.

RESEARCH IDEAS & PRELIMINARY WORK

THE COCOON — DR AHMAD ZAFIR ROMLI

The 'Cocoon' will be a capsule like isolation product for calm and quality sleep in which will re-energized people for the next day.

For human, exposure to light will stimulate a nerve pathway from the eye to parts of the brain that control hormones, body temperature and other functions that play a role in making us feel sleepy or wide-awake. Too much light, right before bedtime may prevent human from getting a good night's sleep. In fact, one study recently found that exposure to unnatural light cycles may

have real consequences for human health including increased in the risk for depression. Regulating exposure to light is an effective way to keep circadian rhythms in check. Due to the above factors, the cocoon to be designed in such a way that it would be practical, high customization ability, light weight and safe. This product will be using glass fiber reinforced composites system as its shell and inner side coupled with proper and comfortable mattress lining for sleep-

ing comfort. On top of that, there is also the possibility of having semi-cocoon design where it can be attached to the existing mattress but having the same effect as the full cocoon.

This project will requires experts from different fields such as the electronic & electrical engineers, computer programming, finite element experts and polymer composites experts.

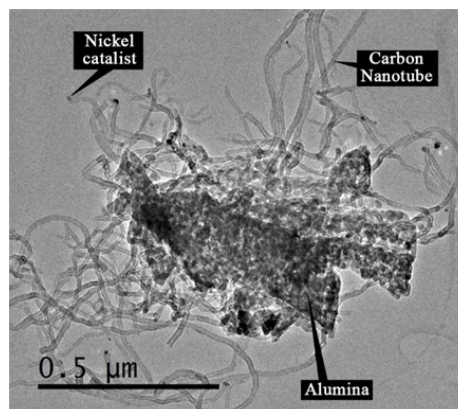
According to Thomas J. Balkin, Ph.D., Chairman of the National Sleep Foundation, "The hour before bed is an important time to relax and wind-down before going to sleep."

ESTABLISHED RESEARCH WORKS

SYNTHESIS AND CHARACTERIZATION OF HYBRID MWCNT-ALUMINA FILLED EPOXY NANOCOMPOSITES — MUHAMAD RAZLAN ZAKARIA, HAZIZAN MD AKIL & HELMI ABDUL KUDUS

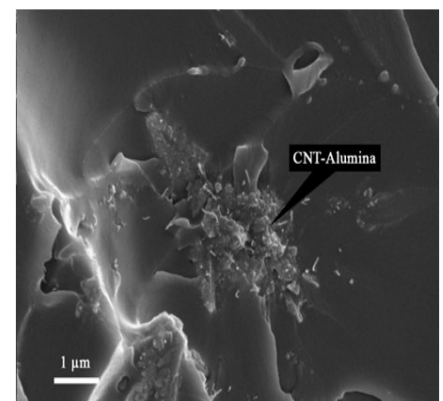
The multi-scale hybridization of carbon nanotube (CNT) with microparticles in polymers offers new opportunity to develop high performance multifunctional composites. The hybrid carbon nanotube-alumina (CNT-Al₂O₃) compound was synthesized via chemical vapor deposition (CVD) by direct growth of CNT on alumina particles. This hybrid CNT-Al₂O₃ compound was incorporated into the epoxy matrix at various filler loadings (i.e., 1–5%). The CNT-Al₂O₃ hybrid epoxy composites showed higher hardness compared to the neat epoxy. This enhancement was associated with the homogenous dispersion of CNT-Al₂O₃ hybrid compound in the epoxy matrix

CNT deposits.

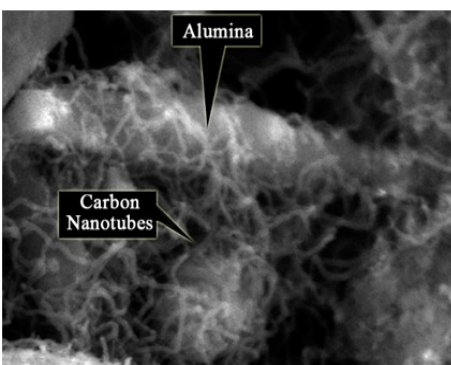


HRTEM images of CNT-Al₂O₃ hybrid

reached the maximum value at 5 wt% filler loading with an increase of 17% compared to the neat epoxy. The improvement of the micro-hardness may be attributed to the rigid CNT-Al₂O₃ fillers in the epoxy matrix. The presence of CNT-Al₂O₃ was restrict the plastic deformation during the indentation. In addition, the increase in CNT-Al₂O₃ filler loading would also reduce the interparticle distance of the fillers.

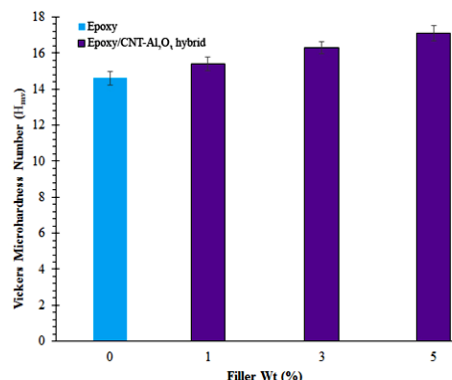


SEM images of the state of dispersion in Epoxy/CNT-Al₂O₃ hybrid



SEM images of CNT-Al₂O₃ hybrid

The CNT successfully grown on alumina particles. The CNT was attached on the alumina particles with the diameter of approximately 10 - 30 nm. The CNT distributed homogenously on the alumina particle and wrapped around the alumina particle. The alumina surface was fully covered by the CNT due to the large amount of the



Vickers micro-hardness of Epoxy/ CNT-Al₂O₃ hybrid

The vickers micro-hardness number increased gradually after the addition of the CNT-Al₂O₃ hybrid filler into the epoxy matrix. The Vickers micro-hardness number

The CNTs seem to be dispersed evenly around the alumina particle with minimal amount of unattached CNTs. The good dispersion of the CNT-Al₂O₃ hybrid in the epoxy matrix which alumina works as transport for the CNTs to disperse homogenously and leading to enhancement of epoxy composites hardness.

ESTABLISHED RESEARCH WORKS

THE EFFECT OF PULVERIZATION PROCESS TO THE PHYSICAL PROPERTIES OF KENAF — MUHAMMAD MUSTAKIM MOHD GHAZTAR & DR AHMAD ZAFIR ROMLI

Many breakthrough research for better understanding on the impact of short fiber were done to impart flexibility and stiffness of the bio-based polymer composite to suit various applications [1-5]. However, there are many challenges that need to be emphasized qualitatively. The major aspects that hinder the usage of natural materials in the composite systems are the variations of the plant growth condition (humidity, sunlight exposure, water absorbed and minerals permeated by the plants) and variability of fiber used (parts of fiber plant taken, maturity of the plants, place where the plants cultivated, types of fiber plants taken and retting process) [5-7].

Apparently, these aspects only focus on the commencement stage of the process. There is, however lacking of attention on the materials preparation process [8, 9], to be specific, on the natural fiber size reduction stages. This is a crucial stage whereby it will later determine the effectiveness of the natural fiber reinforced composite to transfer stress from the matrix itself. Uniformity of the fiber length and diameter are the important features in ensuring uniformity of the physical and mechanical properties of the composite system.

This study is to determine the effect of pulverization process (using different sieve size) to the aspect ratio of untreated and chemically treated kenaf fiber. The aspect ratio of the kenaf fiber will refer to the lengths and diameters measured after the pulverization process. The variability of aspect ratio is expected to affect the physical and mechanical properties of the composites system. This is because the aspect ratio will be the guide in determining the effective surface to interact with the polymer matrix.

Another thing that should be considered is the damage due to the mechanical cutting of the pulverize knife. The mechanical damage to the fiber will surely affect the bio-based polymer composites as there are possibilities for the fiber in becoming the crack initiator (weak point due to the mechanical damage).

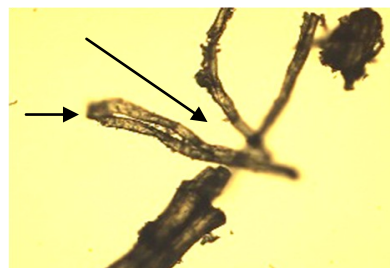
The treatments process.

The fiber was immersed in 6% Sodium Hydroxide (NaOH) for 3 hours then washed thoroughly with ionized water to remove any excess of NaOH on the fiber surface. The treated fiber was dried in an oven at 70°C and further treated using aminosilane dissolved in Ethanol and water with ratio of 1:95:5 respectively for 30 minutes. The pH of immersion was con-

trolled in the range of 3.5 to 5.5 using acetic acid. The completely dried fiber was pulverized into 2 different lengths using 5mm and 0.25mm sieve sizes of Fritsch Power Cutting Mill Pulverisette 15. Only the 0.25mm sieve size fiber was cut staggered from 5.0mm, 1.0mm, 0.5mm until 0.25mm pulverize sieve size. The process was repeated 3 times to analyze the effect of repeated pulverize process to the kenaf fiber. Samples for each process were taken for microscopic analysis. The fiber was analyzed using to determine the fiber condition, length and diameter.

The fibre condition

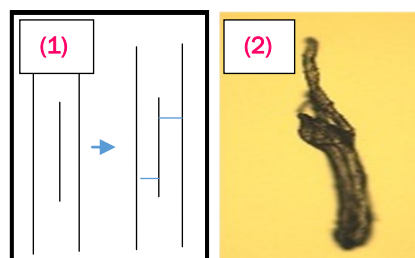
Some of the fiber damages can be identified as fiber breakage, axial splits, fibrillar failure, fiber peel off and others. Examples of the types of damage are as shown in the following diagram.



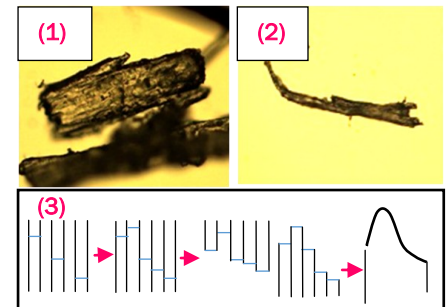
A) Fiber long axial splits at the middle part of the fiber captured at 10x magnification.



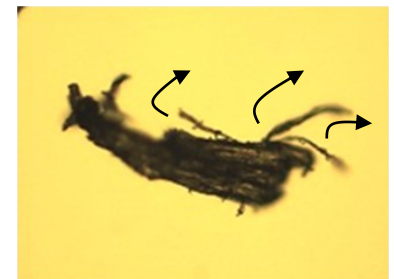
B) Fiber axial splits propagated from the edge of the fiber at 10x magnification



C) Failure mechanism (1) for fiber granular fracture at fiber end (2) at 10x magnification.



D) Complete structure of fiber fibrils (1) and fibrillar failure (2) at 10x magnification and the schematic illustration of fibrillar failure (3)



E) Fibrillar peel off

There are other types of fibre damages that were identified and will be published in the near future.

But, by looking at the type of the fibre damages due to the mechanical cutting (pulverisation process), the consideration of the causes of failure upon mechanical deformation of the polymer composites must be taken into account the effect of the fibre damages as it may create the weak point to the composites.

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ESTABLISHED RESEARCH WORKS

THE EFFECT OF EPOXIDISED PALM OIL (EPO) TOWARDS TENSILE PROPERTIES OF STYRENE BUTADIENE RUBBER (SBR) VULCANIZATES –

ANIS NAZURAH MOHD NASIR, AHMAD ZAFIR ROMLI & M. AZIZOL A. WAHAB

There are various types of plasticiser such as aromatic, paraffinic, naphthenic and vegetable oils. [1] stated that oils and fats are the renewable resources that can be treated to create new materials. The new materials have been used to replace the petroleum based product. EPO is derived from palm oil and undergo the process of epoxidation [2]. In addition to that, epoxidation is the most important process to treat the fats and oils so that their performance, such as thermal properties can be improved [1]. The functional group that presence in EPO makes the EPO easily converted into useful derivatives such as plasticiser [3] and it has been used at about 20% [4]. As an alternative plasticizer, it provides outstanding mechanical properties and harmless to human comparing to petroleum based aromatic oil [5]. Ganga Chandrasekara [6] reported that EPO showed enhanced processing properties, interaction between polymer filler and dispersion properties and also best heat resistance ability and also reported that petroleum based aromatic oils often used as extender oils in rubber compounding. The main function in rubber compounding is to provide better processing and the most important properties is good compatibility with natural rubber or synthetic rubber. In addition to that, palm oil has been selected due to its high yield and also availability [7]. Jayewardhana, Perera [8] reported that natural oil such as palm oil has shown better heat resistance and oils like soybean and sunflower oil can be used instead of petroleum as processing aids in rubber compounding.

The formulation and the cure time are as in Table 1. One must take note that this experiment is meant to see the effect of the EPO alone without the presence of fillers such as carbon black, talc, calcium carbonate and others. The cure time was observed to be increasing with the increasing of the EPO content in the rubber compounding. This is due to reduction in shear forces absorbs by the rubber chains during the curing process. The EPO act as buffer in between the chains thus reducing the friction resulting to longer cure time.

On the other hand, the tensile results in Figure 1 a) & b) shows interesting

relationship between the tensile strength and the tensile strain. For the tensile strength, slightly increasing trend was observed at 25 & 30 phr EPO loading in SBR before sudden increase of the tensile strength at 35 & 40 phr EPO loading before dropping to the lowest tensile strength for 45 phr EPO loading. In contrast to the tensile strain, which improves gradually as the loading of the EPO increases.

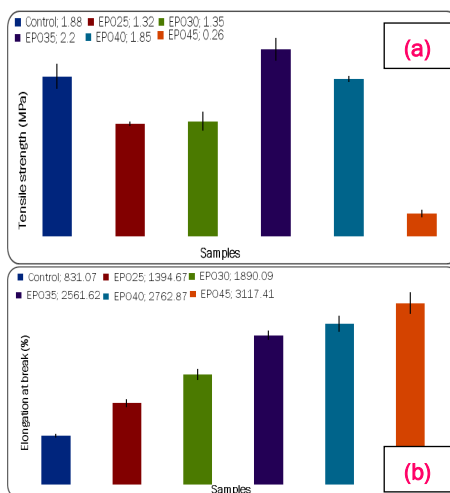


Figure 1 a) The tensile strength results b) The tensile strain results at various phr EPO loading.

The reason for this can be explained using Figure 2 a), b) and c). At low loading (25 & 30 phr), the EPO expected to spread evenly in between the rubber chain assisting the rubber chain to slide between one and another yielding lower tensile strength compared to control sample. Having said that, the same cause does not help much on the plasticizing effect which results to lower tensile strain. As the loading of the EPO increases to 35 & 40 phr, the effect of dispersion reduces due to the suppressing effect of the rubber chain and convert to the accumulation of the EPO in between certain rubber chains. The reduction in the dispersion of the EPO increases the rubber-rubber chain interaction and the accumulation of the EPO increases the lubrication effect resulting to higher tensile strength and also higher tensile strain. The final

effect was observed as the EPO loading was increased to its maximum loading (beyond this point, the mixing process has become impossible) at 45 phr. At this point, due to a very high loading of the EPO, the dispersion and the accumulation of the EPO increases drastically causing the rubber compound to be over lubricated and the presence of the EPO is suspected to interrupt with the cross linking process resulting to very low tensile strength and the highest tensile strain.

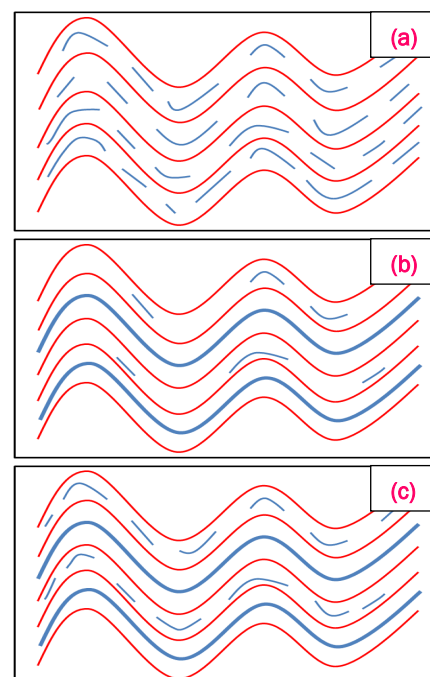


Figure 2 a), b) & c) showing the possible EPO/ SBR interaction. The possible interaction at a) 25 & 30 phr EPO loading, b) 35 & 40 phr EPO loading and c) 45 phr EPO loading. The red line would be the rubber chain and the blue line represent EPO in SBR.

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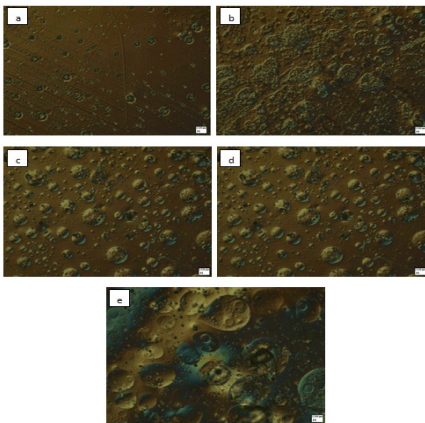
Ingredients	Amount [pphr]					
	Control	EPO25	EPO30	EPO35	EPO40	EPO45
Styrene Butadiene Rubber (SBR)	100	100	100	100	100	100
EPO	0	25	30	35	40	45
Zinc oxide	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Antioxidant	3	3	3	3	3	3
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5
CBS	1.5	1.5	1.5	1.5	1.5	1.5
Cure time [min]	20	14	12	16	18	20

ESTABLISHED RESEARCH WORKS

LIQUID RUBBER MODIFIED EPOXY —DR SITI NUR LIYANA MAMAUD & WAN NORSYAHIRA WAN ZULKIPALI

Liquid rubbers (LRs) are nowadays widely used as a toughening agent in many thermo-setting materials. It can be synthetic or natural based. The type of liquid rubbers was selected based on the polarity and reactivity of functional group. The same polarity of functional groups of both resin and liquid rubber were enhancing the chemical interaction and hence improve the epoxy's behaviors. Amine terminated butadiene acrylonitrile (ATBN) was used as a liquid rubber and introduced as a dispersed phase into the brittle epoxy resin networks. ATBN was chemically interacted with oxirane ring of epoxy resin and well dispersed in the epoxy matrix. Fig.1 (a) -(e) shown the degree of rubber dispersion in different percentage of ATBN in the epoxy matrix.

Figure 1: Optical micrographs of unmodified



(a) and modified epoxy resins at different percentage of ATBN liquid rubber (b) 3%, c) 5%, c) 7% and d) 10%.

Figure 1.a-e shows the unmodified and modified epoxy resins using liquid rubber ATBN ranging 3-10%. The pattern of mor-

phology observed for the unmodified epoxy resin formulation (Fig. 1a) was the behaviours of epoxy's brittle system and it can be seen that the presence of small void inside the epoxy matrix. For the modified epoxy resins (fig.1 b-e), it can be observed the presence of precipitated elastomeric particles of ATBN which were homogeneously dispersed in epoxy matrix cause the formation of two phases. The soft elastomeric phase was phase separated from the hard epoxy matrix. The size of elastomeric particles increased with the increasing of ATBN content. Fig.1e shows the increment of size of elastomeric particles about 50% (Ep/ATBN₁₀) as compared Ep/ATBN₃. The size of the elastomeric particles for Ep/ATBN₃, Ep/ATBN₅, Ep/ATBN₇ and Ep/ATBN₁₀ was shown in Fig. 2.

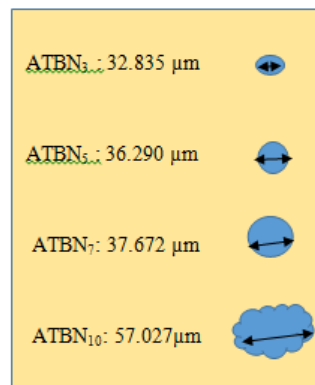


Figure 2: Size of elastomeric particles in epoxy matrix.

The presence of elastomeric particles was enhance the impact properties of

epoxy resin. It is proved that the increasing of ATBN content determines an improvement of the impact strength because of the presence of rubber particles which are homogeneously dispersed in the epoxy matrix. The impact strength of these materials increased as the percentage of ATBN increase where the best performance was achieved with 10% of liquid rubber in epoxy matrix. The Ep/ATBN₁₀ showed higher impact strength about 64.58% as compared to unmodified Ep resin. The unmodified epoxy resin (Ep) shows poor impact strength due to brittle behavior (high crosslinking density). The higher the impact strength of the samples means more energy absorbed by the sample before it breaks. In other words, it can be said that the increase in toughness was due to the amount of elastic energy stored in the rubber particles during the stretching process. Therefore, it can be said that, the addition of ATBN in epoxy matrix gave a positive effect upon the impact strength and the liquid rubber can be used as an effective impact modifier to increase the toughness behavior of epoxy resin. In other words, it can be said that the elastomeric particles in epoxy matrix were considered to bridge the crack as it propagates through the material and therefore, the elastomeric particles were able to prevent the crack growing to a catastrophic size. The crack building of elastomeric particles along with shear yielding is classified as the main toughening mechanism that enhances the impact behavior.

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 – PoCR**), 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.

ESTABLISHED RESEARCH WORKS

EFFECT OF MASTERBATCH METHOD ON GEL TIME AND THERMAL PROPERTIES OF THE UNSATURATED POLYESTER/ P84 (POLYIMIDE) COMPOSITES—NIK NOOR IDAYU NIK IBRAHIM, DR SITI NURLIYANA MAMAUOD & DR AHMAD ZAFIR ROMLI

Unsaturated polyester (UPE) resins are one of the most versatile synthetic copolymers which are the most commonly used resins and have long been known as the premium polymers for composites materials. UPE resins are the condensation products of unsaturated acids or anhydrides and diols with/without diacids which produced the final thermoset products that having a wide range of chemical and mechanical properties depending upon the choice of reactants, cross-linking agents, cross-linking parameters and others additives [1, 2]. The unsaturation points present in UPE provide sites for the subsequent cross-linking. The cross-linking process is initiated by the curing agents that control the reaction rate and the performance characteristics of the finished part. The most common curing agent used is Methyl Ethyl Ketone Peroxide (MEKP). MEKP is an organic peroxide which initiates the crosslinking or curing of the resins through the exothermic chemical reaction that causes heat to build up [3].

UPE resins can be reinforced with short fibers, continuous fibers, or particles to form composites materials. The addition of particles to the resin materials increases the stiffness, reduces the shrinkage and modified the rheological properties [4]. Due to the low viscosity of UPE resins, they are easy to handle and allow high filler contains. In this work, P84 (Polyimide) was incorporated into the UPE resin in order to increase the thermal stability and strength. The effect of P84 on the gel time (t_{gel}) and glass transition temperature (T_g) was studied beforehand since its can affect the end-properties of the composites. t_{gel} is the time taken for the resin to reach gel point once the curing agent is added. The gel point is determined by measuring the resistance to the motion of a stirring rod [5]. T_g is the temperature range where a thermosetting polymer change from a hard and rigid state to a more pliable, compliant or "rubbery" state [6]. The T_g can be determine using calorimetry techniques where the enthalpy changes accompanying chemical events in the sample are monitored as a function of temperature or time [7].

The analysis

The addition of P84 into the UPE resin seems to give no significant affect on the t_{gel} as shows in Table 1. The average t_{gel} is about 8.33 minutes. The unaffected t_{gel} of the UPE resin with the incor-

poration of P84 might be due to the reason that both of them are physically mixed. Thus, there are no chemical interactions involved. P84's particles are not covalently bound to the UPE resin [11]. However, the t_{prod} is clearly much shorter when the composites were prepared through the masterbatch method as compared to the standard method. Table 1 shows the t_{prod} of M_2 are much shorter than M_1 .

Samples	Gel time (min)	Production time (min)
M_0	8.38	8.38
M_1	8.40	11.40
M_2	8.21	8.21

Table 1 Gel time and production time of unsaturated polyester resin

Table 2 show the T_g values for the M_0 , M_1 and M_2 samples detected through the DSC testing. The values of T_g increase from M_0 to M_1 , to M_2 . The pristine unsaturated polyester, M_0 shows T_g about 126.9°C. The T_g increase about 10.6% when the P84 Polyimide was added into the unsaturated polyester resin were M_1 shows T_g around 140.2°C. T_g of the thermosetting materials give insight on the degree of cure. The degree of cure is important parameter in determining the end properties of the materials. Higher degree of cure means that cross-link points in the polyester network is high. High cross-links density produce high strength materials [12].

Samples	T_g (°C)	ΔC_p at T_g (J/g°C)
M_0	126.9	0.406
M_1	140.3	0.173
M_2	163.2	0.055

Table 2 Glass transition temperature (T_g) values for the cured unsaturated polyester

The present of P84's particles inside the UPE cross-links network are the possibly of the increasing of the T_g by absorbing the heat applied since the thermal stability of P84 Polyimide is high. Shen and Lua stated in their study that the T_g of P84 Polyimide is around 337°C to 364°C and decompose around 536°C [13]. Other possibility that could explained the increase in T_g of the unsaturated polyester is that the P84 Polyimide's particles which is located in between the cross-links network

have hindered the rotation of the molecules thus lead to the increment in T_g as illustrated in Figure 1 [7, 14-16].

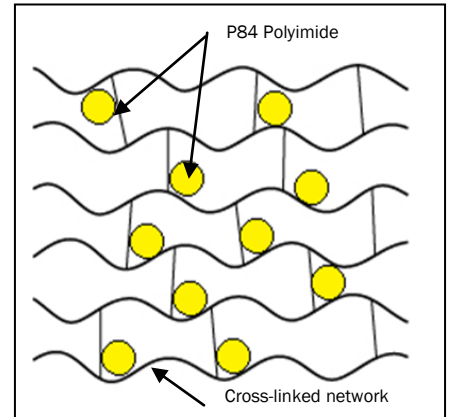


Figure 1 Illustration on how the P84 Polyimide's particles penetrate the cross-links network of unsaturated polyester

The preparation of P84 Polyimide in masterbatch method by mixing with MEKP beforehand gave positive result since the T_g of M_2 increase about 16.3% which is 163.2°C as compared to M_1 and 28.6% higher than M_0 . This phenomenon are possibly due to the better physical interaction of the P84 Polyimide's particles with unsaturated polyester since the particles is 'coated' with MEKP. Another advantage of using masterbatch technique is that it can serve as a simple auto-detector in the resin system for the dispersion of MEKP and P84 Polyimide since the P84 Polyimide is the carrier of the MEKP.

The increases in T_g for both of the samples containing P84 Polyimide, M_1 and M_2 , compared to the pristine sample M_0 are supported by B. Szolnoki et al. [17]. With the homogeneity and rigidity of the P84 Polyimide's particles, they are able to block the segmental movements of the cross-linked unsaturated polyester matrix. Since the colour of the P84 Polyimide is yellow, the dispersion of P84 Polyimide in the pale-purple unsaturated polyester resin can be easily observed with naked eyes.

From Table 2, the increasing pattern of T_g and diminishing of $\Delta C_p(T_g)$ indicating the increasing in cross-links density [14, 18]. Based on the T_g and $\Delta C_p(T_g)$, can be said that the cross-links density of the samples increase in order of $M_0 < M_1 < M_2$. As studied by Montserrat, high T_g is produced when the molecular mobility reducing which is caused by the high cross-links density.

ESTABLISHED RESEARCH WORKS

Reducing molecular mobility inside the composites leads to the reduction in $\Delta C_p(T_g)$. The increment in T_g and reduction in $\Delta C_p(T_g)$ of the samples, M_0 , M_1 and M_2 accordingly suggests that the incorporation of P84 Polyimide into the unsaturated polyester resin promote to the higher cross-links density and induced 'strengthening' effect which decrease the configurational entropy of the unsaturated polyester resin network [14].

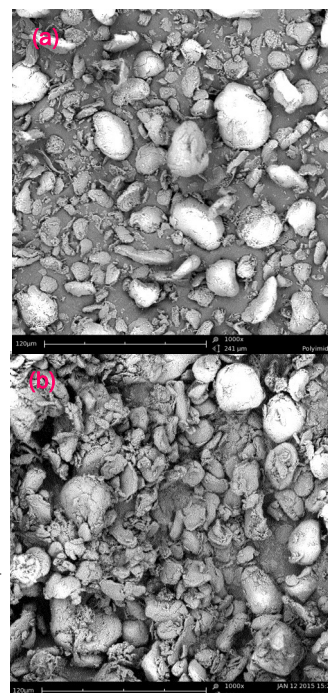
The results obtained shows that the t_{gel} for the 3 samples were about the same since the P84 Polyimide and unsaturated polyester are just physically mixed without involving the chemical bonds. However, in the aspect of the t_{prod} , masterbatch method, M_2 , is much shorter compared to the M_1 . Beside of much shorter production time, other advantages of masterbatch method over standard method are the simplicity of the manufacturing process by reducing one step in the preparation of the composites. It also can serve as a simple auto-detector in the UPE curing for the dispersion of MEKP and P84 since the P84's particles is the carrier of the MEKP. Given that the P84 is

in the form of fine powder, the masterbatch technique will help reducing the difficulty of working with fine powder fillers which tends to get airborne easily and can cause messy workplace.

Throughout the DSC test, the T_g values were increasing from M_0 to M_1 to M_2 . The presence of P84's particles inside the UPE cross-links network are possibly increasing the T_g by absorbing the heat applied since the thermal stability of P84 is high.

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Images on the appearance of P84 polyimide particles (a) before and (b) after being treated with MEKP showing no physical differences.

EQUIPMENTS AND FACILITIES AT PoCRest

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

Testing and analysis

- Tensile, 3-point bending and compression testing machine – SHIMADZU AG-X Series 50kN
- Hydraulic fatigue/ dynamic testing machine with hydraulic wedge grip and com-

pression ability – SHIMADZU SERVO-PULSER 50kN Dynamic and 75kN Static

- 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)

PoCRest Areas of Interest

- Polymer composites fabrication process
- Polymer composites mechanical testing
- Polymer composites characterization

- Thermoplastic elastomers
- Natural fiber/ filler polymer composites
- Elastomers/ rubber composites

- Polymer matrix modification
- Thermoplastic blends
- Polymer nano-composites
- Polymer composites failure analysis

BOOK REVIEW

Compositional and Failure Analysis of Polymers; A Practical Approach**Writer: John Scheirs****Publisher (Years): John Wiley & Sons, Ltd. (2000)****Number of pgs: 766**

This is a very good book for polymer technologist, materials engineers and R&D executives as it comprises of everything related to polymer testing, identifications, characterizations and problem solving. This book has 22 main chapters in a well arranged manner according to the targeted subject or types of failure leading an easy access to the information required. The book starts with the chapter detailing about approaches in solving polymer problems. Few good questions were asked and discuss, such as the pattern of failure location, recurring problems and the trend and timing of the failure. This chapter was concluded with the discussion on the product life cycle approach and the checklist and worksheet for the organizing the problem-solving process. The sampling method and sample preparations are well explained for the common sampling methods and miscellaneous sampling methods such as for intractable samples, brittle thermosets, viscous samples, thin films and rubbers.

The microscopy techniques on the polymer samples involve two most common methods that are the optical microscopy and the electron microscopy. Clear explanation of the techniques on illumination and sample prepara-

tions related to optical microscopy were included. The electron microscopy is briefly being discussed and they are divided into two that is the scanning electron microscopy and the transmission electron microscopy. Nevertheless, at the end of this chapter, the readers were supplied with handful of examples on the uses of the microscopy in polymer analysis.

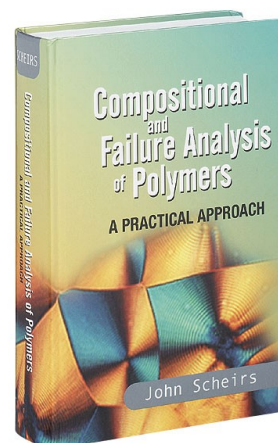
The main characterization of the polymeric materials involves are the Fourier-Transform Infrared Spectroscopy (FTIR) and thermal analysis which were discussed quite thoroughly. The FTIR discussions start with the understanding of the working principle, analysis of common samples, special techniques for difficult sample till the interpretation and validation of the FTIR data. Thermal analysis, on the other hand were separated into few subtopic based on the type and method of the thermal analysis. The thermal analysis involves the differential scanning calorimetry (DSC), dynamic mechanical thermal analyser (DMTA) and thermo gravimetric analyser (TGA).

The best part about this book is about the identification and analysis of polymers. These parts alone acquire about

1/3 of the overall contents of the book. Many tests were described in this section including the density measurement, flammability test, thermal analysis, FTIR, solubility test, structural information and molecular weight measurement. Detail analysis and examples were given for common polymeric material.

Reading further into the book, open up to other interesting analysis, such as analysis of the contaminants in polymer, mechanical failure mechanism, chemical attack on polymer, oxidative degradation of polymers, failure of fiber reinforced composites, environmental stress cracking and including most of the defect in the polymer production such as residual stress and weld lines, voids, blisters and discoloration.

**Dr. Ahmad Zafir Romli,
PoCResT, IOS, UITM**



Front cover of the book.

"This book is highly recommended as it would help on the analysis and ideas in tackling problems or damage related to polymeric materials and products."

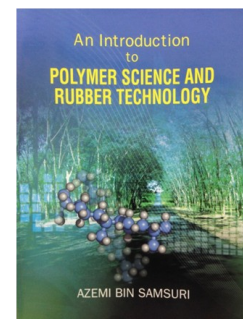
An Introduction to Polymer Science and Rubber Technology**Writer: Azemi Samsuri****Publisher (Years): Pusat Penerbitan Universiti (UPENA) (2009)****Number of pgs: 270**

The book is divided into three main sections. The first section addresses the polymer science by offering a general introduction of a polymer. In this section, which is divided into six chapters, the authors inform readers on the basic and fundamental of polymer science such as viscoelasticity and rheology. It is very important that readers are able to comprehend the basic and fundamental concepts of polymer science first.

The second part of the text is comprised of four chapters that are centered on the rubber technology. Having understood the basic and fundamental of polymer science, the technological part will be much easier to comprehend. The rubber technology section covers the general properties of rubber and their applications, mastication and mixing of rubber compounds. The selections of correct compounding materials and the quantity used are very important to design compounding formulation. The final section deals with physical testing of rubber. It covers most of the standard tests normally required for product specifications as well as for quality control tests in the production.

Overall, the book is effective, in part because it is easy to understand. As it is intended for an introductory study, this book will be of particular interest and utility to those who do not have adequate background in polymer science and rubber technology and not for advanced research.

Dr. Norazura Ibrahim, UITM



Front cover of the book.

"The book is effective, in part because it is easy to understand."

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Thank you.

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Imagine
With all
your mind.
Believe
With all
your heart.
Achieve
With all
your might.



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