

POLYMER COMPOSITES RESEARCH & TECHNOLOGY

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WELCOMING ADDRESS

This technical bulletin is an idea from a group of polymer and polymer composites researchers under the Center of Polymer Composites Research and Technology – PoCResT, Institute of Science, UiTM in the sharing of latest news, knowledge and research findings in this area.

This bulletin will be published biannually covering the market trends of the polymers, preliminary finding and latest finding in research involving the elastomers, polymers and polymer composites system. It is hope that this may also become a platform for other researchers to contribute and share their research findings in relation to the topics.

The members of the Center of Polymer Composites Research and Technology – PoCResT

are as follows:

Head:
Dr Ahmad Zafir Romli (UiTM)

Associate members:
Prof. Dr. Hazizan Md Akil (USM)
Dr Mohd Azizol Wahab (Petronas)
A.P. Dr Mohd Hanafiah Abidin (UiTM)
A.P. Dr Siti Zaleha Saad (UiTM)
Dr Mimi Azlina Abu Bakar (UiTM)
Dr Norazura Ibrahim (UiTM)
Dr Siti Nur Liyana Mamaoud (UiTM)

Assistant research officer:
Muhamad Faizal Abd Halim

Let us inculcate the research culture and sharing of knowledge to everyone.

EDITORIAL TEAM

Editors:
Prof Dr. Hazizan Md Akil
(hazizan@usm.my)
A.P. Dr. Siti Zaleha Saad
(sitizaleha@salam.uitm.edu.my)
A.P. Dr. Mohd Hanafiah Abidin
(mohdh615@salam.uitm.edu.my)

Editorial coordinator:
Dr Ahmad Zafir Romli
(ahmad349@salam.uitm.edu.my)

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QUICK FACT—BUTADIENE RUBBER (BR)

Global production and consumption in 2013 were both approximated at 4.5 million metric tons, increased from 2.8 million metric tons in 2008. Leading global BR technology are Styron, Lanxess, Goodyear, Versalis, JSR, Zeon, Kumho, Sinopec, Petrochina, Michelin, NKNK and Sibur.

ELASTOMER BUSINESS AND TECHNOLOGY

BUTADIENE RUBBER (BR): NEW CATALYST TO MEET GLOBAL DEMAND —DR AZIZOL WAHAB

Introduction

Butadiene rubber (BR) or also known as Polybutadiene rubber (PBR) is a Homopolymer of 1,3-butadiene ($\text{CH}_2=\text{CH}-\text{CH}=\text{CH}_2$). It is the double bonds in the butadiene molecule that are they key to polymer formation. They are attacked by the catalysts to maintain a repetitive chain growth process which continues until something is added to terminate the reaction at the desired molecular weight. For a typical BR, the number of average molecular weight is usually >100,000 grams per mole. This represents a chain that contains over 2,000 butadiene monomers.

In the production of tyres, BR is generally used for the sidewalls, due to its low susceptibility to flex cracking and its high weather resistance. BR is also used for tread compounds because of its high abrasion resistance and low rolling resistance. The polymerization of butadiene results in the formation of a number of stereo isomers. The most important commercial isomer is cis 1,4, whose configuration is similar to that of natural rubber. Natural rubber has properties of high tack and high green (unvulcanized) strength which are hard to replicate in synthetic rubbers. Five main catalyst types are used to produce BR, and the catalyst type affects the proportion produced of cis 1,4 (Table 1).

Table 1: Typical Composition of Butadiene Rubber: Different Catalyst Systems

Catalyst	Cis 1,4 (%)	Trans 1,4 (%)	Vinyl (%)
Neodymium (Nd)	98	1	1
Cobalt (Co)	96	2	2
Nickel (Ni)	96	3	1
Titanium (Ti)	93	3	4
Lithium (Li)	36	52	12

Commercially, Ziegler Natta catalyst systems based on transition metal compositions and on the rare earth neodymium are used to manufacture high cis types due to their selectivity in promoting polymerization of this type of stereo regularity. High cis 1,4 polymer has a very low glass transition temperature (T_g), leading to high resilience, good low-temperature properties, and low heat build-up on repeated deformation. High cis rubber (i.e., 4% or less of 1,2 vinyl) does not crosslink readily and withstands high temperatures without increase in melt viscosity, making it preferred for tyres where significant heat build can take place. High cis BR is used widely in tyres, where its properties are of benefit in sidewalls, carcass stocks, and tyre treads. However, the high cis BR has poor wet grip performance to counter the advantages of the low rolling resistance and high abrasion resistance. Blending with other polymers such as SBR is typically used

to achieve the desired mix of properties in the tyre tread.

The properties of high cis BR vary between catalyst systems and even for one catalyst system. Products with unbranched chains and narrow molar mass distributions tend to provide high strength, but are difficult to process. Long linear chains tend also to be subject to greater 'cold flow', which is when green rubber flows and distorts in storage. Polymers made with neodymium catalyst systems are highly linear with broad molar mass distribution, giving good properties except for extrudability and cold flow. Nickel systems tend to produce rather more highly branched products, with better processability, but lower tensile strength and fatigue resistance. Titanium and cobalt based products are between these two extremes, and the degree of branching can be varied in the cobalt catalyzed system. Branching can be intentionally introduced into high cis BR by post-polymerization reactions such as with sulfur dichloride. High molar mass BR has improved tensile strength, abrasion resistance and fatigue resistance. However, mixing and processing is more difficult with high molar mass product. Oil extension with naphthenic or aromatic oil is used to make this material more easily processed.

High cis butadiene rubber will usually have cis content greater than 95% which gives rise to better 'green strength' and increased cut growth resistance in the cured product (high trans BR is a crystalline plastic material similar to high trans polyisoprene or balata, which is used in golf ball covers). 'Green Strength', which is the strength of the uncured rubber compound, is important for the tyre building process and cut growth resistance is necessary for tyre performance. Cut growth resistance is the resistance to the propagation of a tear or crack during a dynamic operation like the flexing of a tyre in use. Neodymium catalyst butadiene rubber (NdBR) and 'solution' styrene butadiene rubbers (sSBR) are essential in the production of 'green' tyres that are more fuel efficient, safer and durable than standard tyres.

The characteristic of NdBR compared with cobalt catalyst butadiene rubber (CoBR) and nickel catalyst butadiene rubber (NiBR) are: lower rolling resistance (less fuel consumption), higher resilience (low heat build-up/safety), and higher on its excellent properties of low hysteresis (low heat build-up), and abrasion resistance (higher mileage/longer lifespan). The neodymium catalyst system produces the highest cis content of about 99% and also makes the most linear chain structure (no

branching) producing a polymer with the best tensile and hysteresis (low heat build-up) properties of all the high cis types. The cobalt system produces a highly branched BR with a low solution viscosity that makes a good polystyrene and ABS modifier. The nickel catalyst produces BR with an intermediate level of branching.

For tyre application, BR must be blended with Natural Rubber (NR) and/or 'emulsion' styrene butadiene rubber (eSBR) or sSBR. The recent trends are to use sSBR and NR in the blend. The most common grades of NR used are Technically Specified Rubber 20 (e.g., Standard Malaysian Rubber 20 or SMR20) or Ribbed Smoked Sheet 3 (RSS 3). Using 100% BR can causing tyre treads or parts to disintegrate (chunking). New generation NdBR, which have the highest cis content and the lowest trans and vinyl contents, is increasingly used in higher proportions in tyres (hence capitalize lower fuel consumption) and high abrasion resistance (higher mileage). There are other ingredients in a rubber compound which have a significant effect on the final properties which include the reinforcing filler, (carbon black, silica) cure (vulcanization) system, processing aids and anti-degradant system. Another consideration is the processing of the rubber and the building of a tyre which comprises rubbers, textiles and metal components.

Chemistry

Most BR are made by a solution process, using either a transition metal (Nd, Ni, Co) complex or an alkyl metal like n-butyllithium, as catalyst. Since the reaction is very exothermic and can be explosive, particularly with alkyllithium catalysts, the reaction is normally carried out in solvents like hexant, cyclohexane, benzene or toluene. The solvents are used to reduce the rate of reaction and control the heat generated by the polymerization. It is also to lower the viscosity of polymer solution in the reactor. A typical BR polymerization would be run at about 20% monomer and 80% solvent.

Batch vs Continuous Process

The polymerization can either be a batch process or a continuous process. In batch mode, monomer, solvent and catalyst are charged to the reactor, heated to initiate the process, and then allowed to continue to completion. The polymer solution is then transferred to another vessel or process unit to remove the solvent. In continuous mode, monomer, solvent and catalyst are continuously fed into the bottom of the first of a series of reactors

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at a temperature suitable for polymerization. The polymerization progresses as the solution flows through reactors and polymer solution is taken off at the top of the last reactor without stopping the process. The continuous process is the most economical. In both processes, the finished product is usually in the form of bales, which weigh from 20 – 35 kg each.

Emulsion vs Solution Process

In 1930s and 1940s, BR was introduced using the emulsion polymerization system. Although this is not considered to be satisfactory today, it is used in some plants mainly due to historical reasons. The solution polymerization process developed in 1950s is the most popular today.

Process Description

An outline process flow for batch production of high cis BR is illustrated in Figure below. All the catalyst systems are sensitive to impurities, and monomer and recycled solvent must be purified. Carbonyl and acetylenic impurities must be reduced to parts per million levels, and polymerization inhibitor must be removed from the monomer. The feed streams of monomer and solvent are usually dried by azeotropic distillation. 1,3-butadiene and solvent are mixed before passing to the first of a series of polymerization reactors where the butadiene polymerizes exothermically. The catalyst systems and mixing requirements differ between process type; catalyst may be pre-mixed into the monomer and solvent stream as shown, or added separately into the first reactor. Reaction heat is removed with a refrigerated cooling system. After polymerization, a short stop such as water or an alcohol is added, as are components such as anti-oxidants.

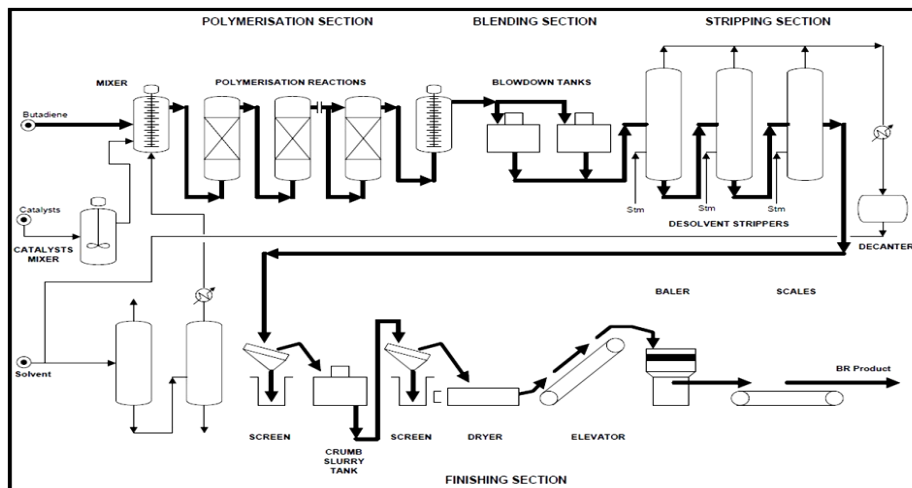
When oil extended types of rubber are being produced, the oil is continuously metered into the stream of polymer cement, homogenized through the line blender, and fed to the first of three steam strippers. In the first stripper, the polymer cement is coagulated and converted into a slurry by the addition of recycle water and by vigorous agitation, then flows into the second and third strippers under similar conditions and pumped into the crumb slurry tank. Vapours of solvent and unreacted butadiene removed by steam stripping from polymer are condensed and separated from water by decantation, then recycled to the respective purification sections. From the crumb slurry tanks, which are provided as a buffer, crumb slurry is pumped to the shaker screen where water is removed from crumb slurry and partly recycled to the stripping section (further dewatering may be effected by an extruder – not shown).

Catalyst Systems

There are variations between processes based on different Ziegler Natta catalyst

systems, including in the solvents used. The main catalyst metal is present in compounds which are soluble in the process liquids. A wide range of possibilities

are achieved in 30 minute polymerization tests. As a result, the butadiene rubber has cis contents in excess of 98%, and the rubber molecules exhibit a



for catalyst systems is presented in the literature.

Neodymium systems typically consist of neodymium carboxylates with alkyl aluminium and a source of chlorine, such as an alkyl aluminium chloride. The neodymium catalyst reacts with a chlorine donor, such as a Di-alkyl Aluminium Chloride, or Ethyl Aluminium Sesquichloride, as well as an aluminium activator, such as a Di-alkyl Aluminium Hydride. The liquid neodymium catalyst is stable at high solids contents over long time periods, and can maintain its catalytic efficiency in liquid storage for a couple of years (Didymium, a combination of neodymium and praseodymium, can be substituted, aliphatic or cycloaliphatic solvents, not aromatics, must be used)

Cobalt systems consist of cobalt compounds such as carboxylates, larger quantities of alkyl aluminium chloride compounds, and carefully controlled small amounts of water. Solvents may be aromatics or, with implications for product characteristics and process efficiency, cyclohexane or substituted aromatics

Nickel systems can include nickel carboxylate, alkyl aluminium, and fluorine compounds. Solvents may be aromatic, such as benzene, or aromatic-aliphatic mixtures

Titanium systems are based on a titanium iodine compound and an alkyl aluminium. The ratio of titanium to aluminium influences the product characteristics and process efficiency. Benzene or toluene are typical solvents, but others can be used.

The neodymium catalyst exhibits a very high activity in polymerizing butadiene, typically over 95 percent conversion effi-

ciency, contributing to high impact resistant butadiene rubber suitable for tyre manufacture and other applications.

International Market Scenario

BR is the second largest end-use for butadiene consumption worldwide, accounting for 26-28% of total butadiene consumption. It is also the second largest volume synthetic rubber produced, next only to SBR. The major use of BR is in tyres with over 70% of BR produced going to treads and sidewalls. Cured BR imparts excellent abrasion resistance (good tread wear), and low rolling resistance (good fuel economy) due to its glass transition temperature. However, this also leads to poor wet traction properties, therefore BR is usually blended with other elastomers like NR and SBR for tread compounds. BR also has a major application as an impact modifier for polystyrene (PS) and acrylonitrile butadiene styrene (ABS), with about 17-21% of total volume going into these applications. Typically about 7% of BR is added to the polymerization process to make rubber-toughened resins. PS modification accounts for the majority of uses. In this application, BR provides toughness and impact resistance for high impact polystyrene (HIPS). In fact, HIPS is the largest non-tyre end-use for BR. The next largest market is ABS, where BR is again add impact resistance to the copolymer. High cis BR is generally used worldwide for manufacturing of golf balls. This application is growing since the golf ball industry is seems to have moved away from traditional wound ball technology to the two-piece, solid core construction. Specialty application such as carboxyl- and hydroxyl-terminated BR grades are used in binders for rocket propellants and in sealants and water-proof membranes.

PRELIMINARY WORK

PLASTIC MADE OF CHICKEN FEATHER—DR AHMAD ZAFIR ROMLI

Chicken feathers are protein in the form of keratin with α and β structures that give the mechanical and physical properties to the feathers. Since protein is a natural occurrence type of polymer thus, there is possibility that it might be process as the conventional plastics.

From literature, there are few researchers tried in the making of plastic from chicken feather through chemical denaturation process and some using synthetic oil to form the plastic. In this preliminary studies,

the chicken feather was washed and pulverized and hot pressed at a specific temperature with bio-based plasticizer to form the required sample before being analyzed.

The preliminary analysis is to see the fusion of the pulverized chicken feather particle upon heating. It was found that the chicken feather was able to fuse between one and another and was able to form sample for further testing.

From the preliminary data, it

is believed that this research will open to new opportunities in refining the techniques and determining the physical and mechanical properties of the 'new' plastic material.

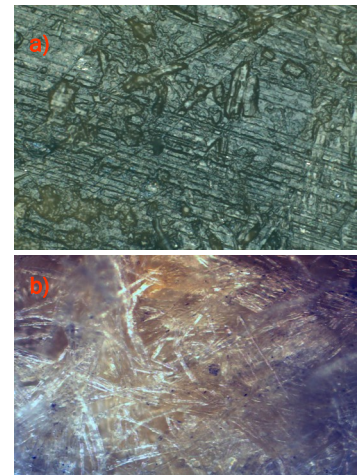


Figure (a) the fusion of the chicken feather particle and (b) the un-fused chicken feather

HBU & DMA; HOW CAN THEY MEET? —DR AHMAD ZAFIR ROMLI & MOHD ISMAIL RIFDI

Heat built up (HBU) machine being used to test rubber article especially tire truck compound by exposing it to sinusoidal wave deformations at specific frequency, time and temperature. The results obtained and evaluated based on the final features of the sample; sample break means fail and vice versa.

Dynamic mechanical analyzer (DMA) on the other hand have the capability of

doing the same test procedure as the HBU in terms of sinusoidal deformation, frequency and temperature. The only different is the sample shape and size.

Due to that, Mohd Ismail Rifdi an MSc. candidate will spend for the next 12 months or so trying to find the correlation between both test. From the preliminary work, the data obtain shows that there are high possibilities in correlating

both test.

The success of this project will open to new dimension where small sample from DMA will be adequate in forecasting the HBU analysis behavior. This will save time and cost of preparing the sample. Not only that, investment in machinery can be save where by having the DMA machine, two analysis can be carried out.

THE COCOON—DR AHMAD ZAFIR ROMLI

The 'Cocoon' is 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 stimulates 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 design 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 sleeping comfort.

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

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

STRUCTURE-PROPERTY RELATIONSHIP OF POLYLACTIDE/ORGANOCLAY NANOCOMPOSITES — DR NORAZURA IBRAHIM

Numerous studies of polylactide (PLA) nanocomposites have been reported. However, the role and importance of processing conditions in preparing the nanocomposites is the subject of very few papers. The effect of filler loadings on nanocomposites also has been widely reported but previous studies either use a narrow range of filler or report a limited range of properties.

In this study, PLA/organoclay nanocomposites with concentrations of 2-10 wt % of montmorillonite (MMT) were prepared by melt intercalation technique and tested for a wide range of properties. One novel contribution of this study is the optimisation of processing conditions by using statistical analysis. A Box-Behnken Design (BBD) of experiments was applied to investigate three variables; processing temperature, rotor speed and mixing time. Results showed that the optimum processing conditions were found to be at 175 °C, 100 rpm and 7 min. These nanocomposites exhibit significant improvement of practical materials properties, such as Young's modulus and thermal stability, as compared to the neat PLA.

Mixing of the PLA with Cloisite® 30B (the MMT modified with a quaternary ammonium salt) resulted in formation of a nanocomposite material (PLA-30B), with characteristic intercalated peaks seen in small-angle x-ray scattering (SAXS) results and high aspect ratio tactoids or exfoliated layers in transmission electron microscopy (TEM). Rheological behaviour of the PLA nanocomposites showed effective interaction between polymer and organoclay. The rheological properties correlated with the nanostructure evolution and material property enhancement observed in other tests. The percolation threshold was reached around 4.2 wt % filler. Measurement of thermal properties showed that increasing clay content did not influence the glass transition temperature (T_g) significantly compared to the pure matrix. However, the melt temperature (T_m) and degree of crystallinity increased significantly in the presence of organoclays, indicating the filler acts as a nucleator. An increase in thermal stability with the increase in clay loading level was also observed for PLA nanocomposites. The Young's modulus of the PLA nanocomposites improved very significantly. At 10 wt % of filler, the increase in Young's modulus compared with the unfilled PLA was around 54 %. Tensile strength and elongation at break decreased, attributed to presence of agglomerates. Overall the properties of the nanocomposite PLA-30B were consistent with an exfoliated/intercalated morphology, with good dispersion and interfacial adhesion.

A further novel contribution of this work is the comparison of these nanocomposites to microcomposites made with natural MMT. Microcomposites were made with two types of natural MMT, Cloisite® Na⁺ and Cloisite® Ca⁺⁺ DEV, to produce samples PLA-Na and PLA-Ca. The main difference between these two types of MMT is that the Na⁺ grade is water dispersible whereas the Ca⁺⁺ grade is not. Results observed by SAXS shows the composite has a morphology that is neither exfoliated nor intercalated. Scanning electron microscopy (SEM) suggested the morphology has a predominance of agglomerates and aggregates while agglomerates were also observed in TEM. This confirmed that the morphology of these samples was that of a micro- rather than a nanocomposite.

In contrast to PLA-30B, both PLA-Na and PLA-Ca show a decrease in G' , G'' , and η^* in the terminal region (low frequency) compared to neat PLA. This suggested poor dispersion and/or interfacial adhesion between polymer and filler. For the microcomposite samples, the percolation threshold was much higher, around 9 wt %. This is consistent with the lower aspect ratio of filler in these samples compared to a nanocomposite. TGA curves recorded for PLA filled with Cloisite® Na⁺ and Cloisite® Ca⁺⁺ show that increasing the filler content triggered a substantial decrease in thermal stability, also suggesting poor dispersion. The addition of unmodified clay provided only a modest improvement to the Young's modulus of PLA. At the same 10 wt % filler loading, PLA-Na and PLA-Ca improved modulus by 18 % and 17 %, respectively. The inferior performance of the micro filler was attributed to the low interaction between the low aspect ratio microparticles and surrounding polymer matrix, as well as the creation of aggregates and/or agglomerates as shown by SEM and TEM. Overall the properties of the microcomposite were consistent with an agglomerated morphology, poor dispersion and interfacial adhesion.

Finally, the composite theory of Halpin-Tsai was applied to predict the Young's modulus of the nano and microcomposite as a function of the organoclay concentration and aspect ratio. The higher the filler concentration and aspect ratio, the higher the modulus. The objective was to evaluate whether the Young's modulus of PLA composites (both nano

and micro) can be modelled using the Halpin-Tsai micromechanical model. A comparison of the experimental data of Young's modulus with values predicted from a model suggested that the aspect ratio of the clay layer is about 4 for Cloisite® 30B, and about 2 for Cloisite® Na⁺ and Cloisite® Ca⁺⁺. The model predicted the behaviour of PLA composites satisfactorily with the appropriate material parameters obtained from the literature. However, a more accurate estimate of aspect ratio by quantitative examination of multiple TEM micrographs is recommended.

Other areas suggested for further work include evaluation of barrier properties and bio degradability, whether mixing could be optimised better with a statistical analysis based on a different design of experiments, as well as comparison of properties with other micromechanical models.

ESTABLISHED RESEARCH WORKS

SINTERING OF AQUEOUS BASED VPP (VISCIOUS POLYMER PROCESSED) ALUMINA —ASSOC PROF DR SITI ZALEHA SAAD, T. W. BUTTON & C. B. PONTON

In all sintering processes of ceramics, the bodies are normally fired at an appropriate sintering temperature in order to achieve high density which is near to 98% relative densities. However heterogeneities originated during the ceramic processing would remain during the sintering, inhibit the development of high densified bodies and deteriorated the mechanical properties of the final bodies. In the current work the approach of slow heating rate and long preheating hours at temperature below 500°C prior to sintering were carried out to develop high sintered density of aqueous based VPP alumina with PVA binders. For the sintering, two firing controls denoted as regime A and regime B were used and the effect of the sintering controls on the microstructures and final properties were discussed. Under regime A the sintering control was carried at 5°C/min heating rate above 500°C and short dwell of two hours with the samples placed on alumina foam. Under regime B, the sintering was carried out at a slower heating rate of 2.5°C/min above 500°C/min and long 9 hour dwell at 225°C for partial debinding, with the samples mostly fired within a zirconia powderbed. Few samples were fired on alumina foam under regime B at maximum temperature 1550°C. All the optimised VPP alumina AST samples (pressed) are sintered by using both regimes and the effect of sintering on the surface texture and microstructural evolution is discussed. The effect of processing variables on the properties and microstructures of bulk ceramics before and after sintering for various consolidations were highlighted. The heterogeneities that originate during the powder processing stage sometimes remain after the sintering and degrade the final properties of the parts. These heterogeneities must be reduced by optimising the processing especially during the early stages of processing. Generally ceramic processing consists of three main stages:- raw materials preparation, consolidation of powder to form green compacts, and densification of the green compacts to produce dense ceramics. Each of these processing steps greatly influences the properties of the material by altering the microstructure of the material, which in turn affects properties. Many work have highlighted on the issues on raw materials and their preparation which involve various powder processing and synthesis [1-2]. Although different types of powder such as silicon carbide, barium titanate and alumina have been used in their work but the results led to the same conclusion that improve sintered bodies could be developed with improve powder processing and synthesis. Various powder synthesis and powder processing techniques were developed to improve bar-

ium titanate powders properties with high specific surface area and high purity [3-4]. Tripathy [5] has reported on the hydrothermal processing to produce high purity barium titanate powder and Gao also reported on the processing technique by using high gravity reactive precipitation to produce high specific surface area powder. Although specific powder routes have been developed for the improvement of the final parts but the powder still needs to be characterised by the following method. First of all chemically; the stoichiometry and the amount of impurities present. Secondly, crystallographically; to detect non reacted phases and second phases. Thirdly, morphologically; to detect the presence of agglomerates, the size (distribution) and shape, and the specific surface area. Having the powder characterised could aid in the optimisation during the processing and the green and sintered properties were expected to be improved. One work has reported on the effect of powder property such as agglomerates which led to a strength-degrading flaw population [5-7] and the morphology of a powder directly affects the particle packing density. It is highlighted that the frictional forces that oppose spherical particles from rolling over one another will be much lower than those opposing the sliding motion of highly angular particles. Hence, controlling the morphology of the raw powder is very important in order to enhance the properties of the final material, and suitable processing methods must be chosen in order to obtain the desired particle morphology. Highly densified bodies can be obtained by using powder pressing and the powder particle packing density in the green bodies should be as high as possible whereby particle size distribution is known to affect particle packing. Very high packing densities can be achieved by mixing the appropriate volume fractions of different particles since the finer particles can fill the interstices between the coarser particles [8]. For Viscous polymer Processing pastes must be prepared prior extrusion. A paste is a mixture of solid and liquid, the relative amounts being such that the resulting materials can be readily moulded [9].

Experimental procedures**Viscous polymer Processing (VPP)**

The powder and dry organic compounds were weighed and dry mixed thoroughly for two minutes. The liquid plasticizer

(which was glycerol) the lubricant, stearic acid and distilled water were added and the mixture was wet milled for another two minutes by using a spatula. Half of the mixture was poured into the nip between the water-cooled (10°-11° C) twin roll mills with the gap set to 0.25mm and milled for five minutes. The twin roll mills were then stopped and the gap size increased to 0.65 mm and another half of the mixture were added and milled for another five minutes. The process was repeated until a smooth, flexible surface was obtained. The milling time optimised to 10 minutes milling time to develop a homogeneous paste. The paste sheet was reintroduced at right angles of the roll mill for three times to improve homogeneity. It was then removed off the mill and uneven edges were cut. The sintering of alumina VPP paste sheet was carried out in an electric furnace, sintering control denoted by regime A and regime B at high and low heating rate (>500°C) respectively. All the sintering used regimes are described in Figure 1.0 :

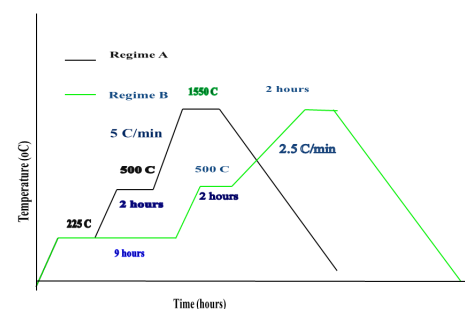


Figure 1.0: The schematic diagram of sintering control regime A and regime B with heating rates (Both Regimes, 1°C/min below 500°C Regime A, 5°C/min above 500°C (c) Regime B, 2.5°C/min above 500°C)

It is claimed that at too high sintering temperatures and too long sintering time can hinder materials properties due to microstructure coarsening [8]. However the burnout process has to be taken into consideration in order to reduce the defects after sintering. The grain boundary and abnormal grain growth at 1500°C and 1550°C sintering temperatures are shown in the transverse section of thermal etched sample in Figure 2.0 (a) and (b) respectively. The grain boundaries are clearly revealed in the thermal etched microstructures.

ESTABLISHED RESEARCH WORKS

Conclusion

Homogeneous structure obtained after optimised sintering led to the enhanced properties of viscous polymer processed (water-based binders) Alumina.

Future Work

The potential utilisation of Alumina for medical application especially for body implant should be further investigated with fully densified Alumina.

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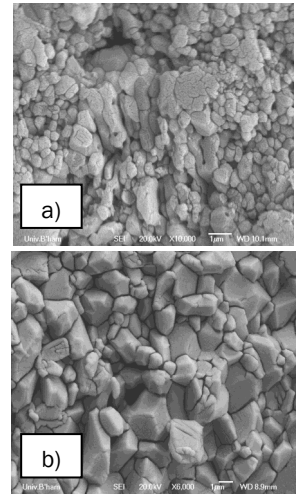


Figure 2.0: (a) and (b) Transverse section of thermal etched (fracture surface) of VPP (Viscous Polymer Processed)

TENSILE PROPERTIES OF EPOXY/ CHICKEN FEATHER COMPOSITES SYSTEM—DR AHMAD ZAFIR ROMLI

Production of bio-composites using biodegradable filler/ fibre or matrix has been steadily increased for the past decade. In the case of fibre reinforced polymer composites, natural fibres such as jute, hemp and kenaf have widely reported. Apart from cellulosic based natural fibre, there are other potential filler from animal based by-product such as chicken feather. In this study, epoxy feather composite was produced via pressure assisted hand lay-up technique to form flat and homogenise board. The percentage of the feather content by weight percent was varied accordingly (i.e. 10%, 20%, 30%, 40% and 50%). The board then was cut and prepared for the tensile test according to the standard testing method. From the tensile test results it shows that the ultimate tensile strength reduces as the percentage of the feather increase and this pattern also applies to the strain value. The Young's modulus on the other hand shows little differences as the percentage of the feather is increased. The fractured piece of the sample due to the tensile test was then observed and evaluate under

the scanning electron microscope (SEM) for fracture behaviour. From the results of the tensile test, the maximum percentage (50% feather incorporation) shows promising results, consumes the most chicken feather and has the possibility to be used in non-structural applications.

Ultimate tensile strength (UTS)

By incorporating the chicken feather into the epoxy decreases the UTS value to more than half of its virgin epoxy. This happens due to the fibre pullout which can be observed in Figure 1. Even though the amount of fibre pull out is extensive at 10% chicken feather incorporation, there is evidence of fibre fracture observed in the fractography image. The fibre fracture shows that there is some interphase bonding happens between the chicken feather and the epoxy. As the amount of chicken feather inside the epoxy resins reached 50%, the epoxy resins is being forced to fill and penetrate to parts of the chicken feather as shown in Figure 2 due to fixed mould

In 2003 a statistical report produces by the government of Malaysia stated that by average, each Malaysian from the aged 18 to 59 years old consume about 31.66 gram of chicken per day. Meaning that by average, each Malaysian from the aged 18 to 59 years old consume about 949.8 gram of chicken per month.

If the chicken feather consist of 5% - 7% of the chicken weight then each Malaysian from the aged 18 to 59 years old will waste between 1.6 gram - 2.2 gram per day of chicken feather and between 48 gram - 66 gram per month (30 days each month)

volume. This phenomenon has somehow improved the bonding between the chicken feather and the epoxy itself.

Samples	% Epoxy	% Chicken Feather
Ep0F	100	0
Ep10F	90	10
Ep20F	80	20
Ep30F	70	30
Ep40F	60	40
Ep50F	50	50

	Ep0F	Ep10F	Ep20F	Ep30F	Ep40F	Ep50F
Ultimate tensile strength (MPa)	48.25 ±0.32	20.46 ±0.69	16.36 ±0.51	14.68 ±0.68	12.93 ±0.21	14.08 ±0.21
Young's modulus (GPa)	1.49 ±0.02	1.14 ±0.02	1.13 ±0.03	1.19 ±0.06	1.18 ±0.06	1.21 ±0.02
Strain @ ultimate stress value (%)	5.16 ±0.06	4.03 ±0.69	2.53 ±0.12	2.71 ±0.37	2.80 ±0.17	3.08 ±0.24

ESTABLISHED RESEARCH WORKS

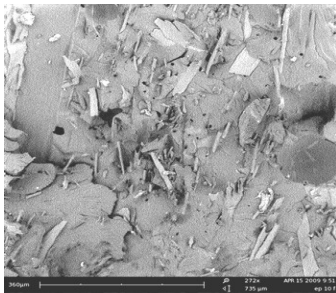


Figure 1: Fractography images of the Ep10F samples viewed using SEM at 272X. This fractography images showed the fibre pullout and fibre fracture during the tensile test.

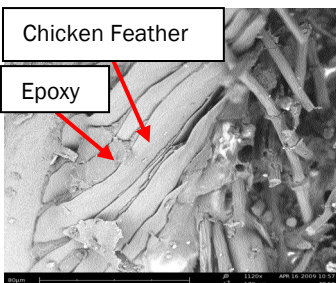


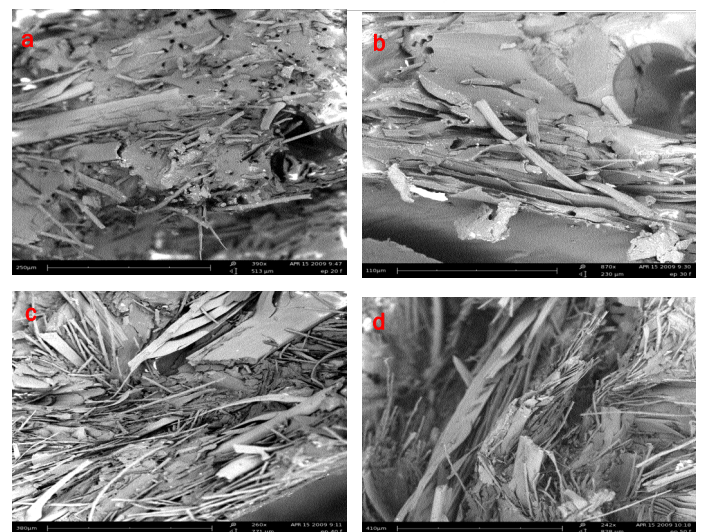
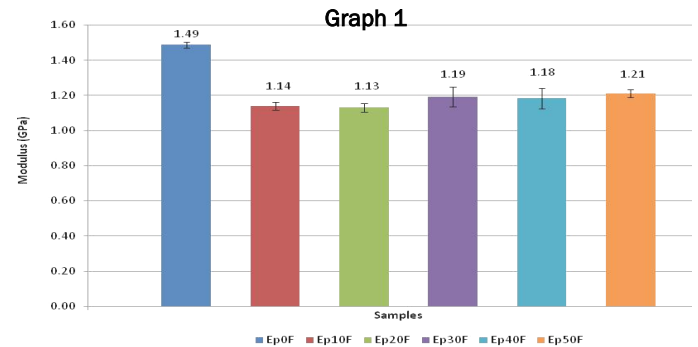
Figure 2: The scanning electron micrograph image showing the maximum filling and penetration of the epoxy matrix in between the chicken feather as a result of high loading of the chicken feather (sample Ep50F at 1120 X magnification).

Young's Modulus

The Young's modulus drop from blank to the first incorporation of the chicken feather is obvious as referred to Graph 1. The Young's modulus was taken at the range of 10N to 110N. It is however after 10% incorporation of the chicken feather, the value of the Young's modulus seems to show no significant difference. This phenomenon is interesting since there are not many known fillers maintain or have the same modulus as the percentage incorporation of the filler increase. With no significant change of the modulus thus, indicates that the toughness of the epoxy/ feather bio-composites is somewhat the same for the tested percentage (10% till 50%).

Conclusion

The tensile properties drop with the increasing amount of chicken feather being loaded into the epoxy. The factors contributing to the decreasing trend of the tensile properties are the interfacial properties between the chicken feather and the epoxy and the possibility of polymer network distortion.



The fractography images of epoxy/ feather bio-composites; a) The observation of the fibre pull out in Ep20F sample, b), c) and d) Fibre fracture in the tested sample of Ep30F, Ep40F and Ep50F consecutively.

PREPARATION AND THERMAL PROPERTIES OF HYBRID LAMINATED COMPOSITE

—DR SITI NUR LIYANA MAMAOU

Epoxy resins are one of the brittle thermosetting polymers which need to be toughened by the introduction of some types of dispersed phases such as liquid rubbers, thermoplastic resins and epoxidized vegetable oils. In this study, the toughening properties of epoxy resin was improved by modifying epoxy resin with epoxidized palm oil in the present of suitable catalysts. There are three types of catalyst (tetrabutyl hydro peroxide, TBHP; tetrabutyl ammonium bromide, TBAB and imidazole, I) were used to identify the best catalyst that gave good impart to the curing system of modified epoxy resin.

After the curing process was carried out, it was found that the composition of polymer blends at ratio 50:40:10

No. of blends	Composition (%)			Types of catalyst		
	EPO	EP	A	TBHP	TBAB	I
1	70	20	10	2	-	-
				-	2	-
				-	-	2
2	60	30	10	2	-	-
				-	2	-
				-	-	2
3	50	40	10	2	-	-
				-	2	-
				-	-	2

showed the cured condition using imidazole catalyst. Therefore, imidazole was classified as an effective catalyst for this system. The compositions of imidazole catalyst were also studied in order to obtain the correct amount requires in this system and it varies at 5% to 35%. The results of TGA showed that the polymer blends that consists of 5% of catalyst had high T_{onset} and residual mass. The transition of T_{onset} from high to low value was presumably due to the reduction of the crosslinking density. The crosslinking density of cured polymer was related to the crosslinking reaction between epoxide ring of Ep and

EPO. The three dimensional networks of cured polymer blends yielding the structure which are more tightly packed and thus, increase the T_{onset} .

% of catalyst	C-5	C-10	C-25	C-35
T_{onset} [°C]	367.5	363.9	359.7	354.8
Residual mass [%]	9.55	1.94	1.92	1.89

Polymer blend of Ep-EPO was used as a matrix in a composite system. Glass fiber reinforced composite was fabricated using dipping method. The techniques to dip the woven glass fiber were studied and it can be divided into three sections which are immersion, hanging and curing. The period was varies to get the sufficient time for each section and the base line of dipping method was obtained.

ESTABLISHED RESEARCH WORKS

Properties of laminated composite samples were studied and according to the DMTA results, it shows that the T_g of hybrid laminae composite was negative (-31.25°C) value as compared to unmodified Ep laminae composite (47.5°C). It happens due to flexibility effect of long fatty acid chains of EPO and low oxirane content in hybrid resin cause a reduction on degree of crosslinking. The T_g value of EPO was in the range of -44.8°C to -50.6°C and it indicates that the modified epoxy resin had the flexibility and rubber phases.

Material designation	Time (min)		
	Immersion (t_1)	Hanging (t_2)	Curing (t_3)
EPO/Ep/A/I	5	5	10

BOOK REVIEW

Introduction to Industrial Polypropylene; Properties, Catalyst, Process

(ISBN 9781118062760)

Publisher (Years): Wiley (2012)

Number of pgs: 316

Polypropylene is common to most engineers with material-related background. The subject of polypropylene is always assumed as a small sub-section of polymer materials, although it is a huge subject in its own right dictated by the infinite variables in an ever-developing science. Different types and properties of polypropylene can be produced with the right understanding on its chemistry, catalysts and process technology. The book has 13 chapters focusing on polypropylene polymerization and catalysts used in the production of industrial polypropylene.

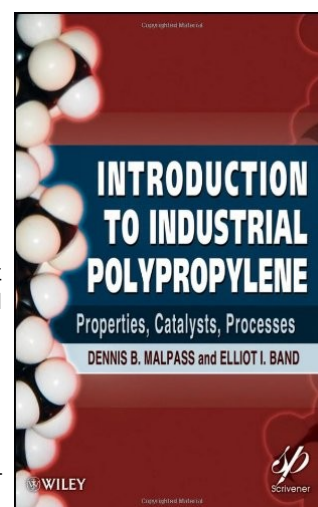
Chapter 1 - 2 focus on history of crystalline polypropylene and describe basic properties and nomenclature for this versatile polymer. The most important industrial catalysts used for its manufacture are introduced. Also covered, overview on its stereochemistry which is a crucial aspect that underpins properties and how polypropylene is used in fabrications. Key characterization methods to support research and commercial production of polypropylene are also discussed. Chapter 3 - 5 concentrate on features of catalysts and co-catalysts i.e. key characteristics of Ziegler-Natta (ZN) catalysts and mechanistic features of ZN polymerization. Chapter 4 describes the various generations of industrial polypropylene catalysts as well as

reviews on intermediate catalysts developments and its evolution. It also covers safe handling of hazardous aluminium alkyls which is critical to all polypropylene manufacturers. Chapter 6 and 7 covers single site catalysts i.e. metallocene and large-scale manufacture of polypropylene catalysts respectively. This includes a description in term of equipment required and importance of its recycle streams. This followed by Chapter 8 which provides and introduction on wide range of process technologies, reviews on industry trends and movement towards 'hybrid' processes. Chapter 9 - 10 describes some laboratory synthesis as well as laboratory polymerization testing protocols. The last three chapters provide some overview on the downstream aspect of polypropylene such as additives, fabrication methods and environmental issues. It also discussed about global market, major producers of polypropylene and how it has shifted in recent years.

This book is suitable for chemists, engineers and students especially in providing an extended overview to the essentials of industrial polypropylene beyond what they usually learn in classroom - what is polypropylene, how it is

made and fabricated, how it is characterised, market segment it serves, and its environmental related matters. It will provide some appreciation on relationship between catalysts, process technology and how it affect the structure, properties and the applications of polypropylene. The technical aspect is described beautifully with minimal discussion of esoteric theory such that a person with a modicum of training in chemistry should be able to understand. Extensive theoretical discussions have been largely omitted, but details are available in excellent handbooks and encyclopaedia articles included as references at the end of each chapter. I would strongly recommend this book as a reading in this subject area.

*Dr Azizol Wahab, PhD CEng
MIMMM*



Front cover of the book.

“This book is suitable for chemists, engineers and students especially in providing an extended overview to the essentials of industrial polypropylene beyond what they usually learn in classroom.”

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LIST OF ARTICLE CONTRIBUTORS FOR THIS ISSUE

Dr Ahmad Zafir Romli, Head of PoCResT, IOS, UiTM; ahmad349@salam.uitm.edu.my OR ahmadzafirr@gmail.com

Dr Mohd Azizol Wahab, PETRONAS; azizol_wahab@petronas.com.my OR azizol@consultant.com

Dr Norazura Ibrahim, Assoc Member PoCResT, IOS, UiTM (Polymer Technology Programme, Faculty of Applied Sciences, UiTM); noraz371@salam.uitm.edu.my

Assoc Prof Dr Siti Zaleha Saad, Assoc Member PoCResT, IOS, UiTM (Polymer Technology Programme, Faculty of Applied Sciences, UiTM); sitizaleha@salam.uitm.edu.my

Dr Siti Nurliyana Mamaoud, Assoc Member PoCResT, IOS, UiTM (Polymer Technology Programme, Faculty of Applied Sciences, UiTM); nurliyana2219@salam.uitm.edu.my

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