Laporan Akhir Projek Penyelidikan Jangka Pendek

MODIFICATIONS OF NANOSILICA-EPOXY FILLER AND ITS PROPERTIES IN THE COMPOSITE MATRIX

No. Akaun Geran : 304/PKIMIA/637 004

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Kandungan

Penemuan projek/Abstrak

Silika nanofumed dengan purata saiz partikel dalam lingkungan 15-30 nm telah diubahsuaikan permukaannya dengan menggunakan molekul epiklorohidrin dan molekul resin epoksi. Nanokomposit-nanokomposit silika fumed-epoksi terubahsuai ini diperoleh dengan merefluks molekul epoksi bersama silika fumed dengan menggunakan imidazola sebagai pemangkin dan juga diubahsuai dengan molekul epiklorohidrin menggunakan eter mahkota dan kalium iodida sebagai pemangkin. Sifat permukaan silika yang telah diubahsuai dan kesan keberkesanannya bertindak sebagai bahan pengisi dalam komposit epoksi telah dianalisis dengan menggunakan Analisis Infra Merah (FTIR), Analisis Termogravimetri (TGA), Analisis Pekali Pengembangan Terma (CTE), analisis kekuatan regangan dan Mikroskop Elektron Pengimbasan (SEM). Berdasarkan daripada analisis FTIR dan TGA, didapati bahawa gelangan epoksi berikatan secara kimia dengan permukaan silika. Analisis CTE dan analisis kekuatan regangan pula, menunjukkan bahawa dengan penambahan 5 % bahan pengisi silika terubahsuai dengan molekul pengubahsuai yang berbeza ke dalam matriks komposit, amat mempengaruhi sifat keupayaan terma bagi komposit-komposit tersebut. Komposit epoksi yang telah diisi dengan bahan pengisi silika-epiklorohidrin (MSEpi) terubahsuai mempunyai nilai kestabilan CTE yang lebih baik berbanding komposit epoksi yang diisi dengan silika asli (PS) tetapi ia mempunyai kestabilan yang rendah berbanding komposit epoksi dengan pengisian silika- epoksi (MSE) terubahsuai. Matrik komposit dengan sistem pengubahsuaian dan tanpa pengubahsuaian tidak menunjukkan sebarang perubahan dalam sifat kekuatan regangan. Sifat-sifat ini disokong selanjutnya melalui analisis SEM, yang menunjukkan bahawa taburan partikel-partikel silika dalam komposit epoksi yang diisi dengan bahan pengisi MSEpi mempunyai taburan yang lebih baik berbanding komposit yang diisi dengan bahan pengisi MSE.

Kata kunci: Silika nanofumed, molekul epoksi, epiklorohidrin dan silica terubahsuai.

Abstract

Nanofumed silica with mean particle sizes in the range of 15-30 nm was surface modified using epichlorohydrin and epoxy resin molecule. Modified fumed silica-epoxy nanocomposites were obtained by refluxing epoxy molecule with fumed silica using imidazole as catalyst and the other one was surface modified by epichlorohydrin using crown ether and potassium iodide as catalyst. The properties of surface modified silica and their effect as fillers in epoxy composite were characterized by Fourier Transform Infrared spectroscopy (FTIR), Thermogravimetri Analysis (TGA), Coefficient of Thermal Expansion (CTE), tensile testing and Scanning Electron Microscopy (SEM). From FTIR and TGA analysis, it was found that the epoxide ring was chemically bonded onto the silica surface. The CTE analysis, shows the addition of 5 % modified silica fillers with different modifier into the composite matrix, highly influences their thermal properties. Epoxy composite filled with modified silica-epichlorohydrin fillers (MSEpi) has higher stability of CTE value than epoxy composite filled with pure silica (PS) but lower than that of epoxy composite filled with modified silica-epoxy filler (MSE). The composite matrix with and without modification show no significant differences in their tensile properties. This behavior was further supported by SEM analysis which indicates that the distribution of silica particles in epoxy composite filled with MSEpi has better dispersion than that of epoxy composite filled with MSE.

Keywords: Nanofumed silica, epoxy molecule, epichlorohydrin and modified silica.

Penghargaan

Dengan lafaz Alhamdullillah, syukur ke hadrat Ilahi kerana dengan berkat dan izinNya, saya telah berjaya menamatkan projek penyelidikan ini pada masa yang lebih awal daripada masa yang ditetapkan. Penghargaan setinggi gunung khas buat ibu bapa saya yang berada di Ranau (Sabah), isteri dan anak-anak saya atas kesabaran, ketabahan dan sokongan yang diberikan selama ini walaupun kadang-kala berjauhan dengan mereka. Rakaman setinggi-tinggi terima kasih saya ditujukan kepada Pengerusi and Ahli-Ahli jawatanKuasa Penyelidik Universiti, Universiti Sains Malaysia kerana peluang yang diberikan kepada saya untuk melaksanakan projek penyelidikan ini. Bantuan kewangan dan sokongan daripada bahagian penyelidikan dan pembangunan Universiti Sains Malaysia amat saya hargai. Akhir sekali terima kasih saya ucapkan kepada pembantu pelajar yang bekerja dalam projek ini iaitu Cik Norhayati Ahmad serta pelajar projek tahun akhir di bawah penyeliaan saya pada sidang akademik 2006/2007 iaitu Husna Abd. Halim, Nur 'Aini Raman Yusuf, Nur Farhana Ibrahim, Siti Rosdiana Rahuni, Nur Syafawati Bt. Abdul Rahman, Firda Zahila Binti Zakaria, Norfaezah Bt. Sharifuddin, Norzie Hani Binti Ramli dan Farah Hani Binti Shahari.

Output dan Faedah projek

(a) Penerbitan

Conference : *"The effect of modified silica-fillers with different organic compound in epoxy composites properties"*, Abstract 1st Penang International Conference for Young Chemist 24th – 26th May 2006, Universiti Sains Malaysia, Penang.

International journal publication: *"Properties and Morphology of Modified Fumed Silica-Epoxy Nanocomposites"*, Journal of Applied Sciences 7(1):27-34, 2007. Journal of Applied Sciences 7(1):27-34, 2007.

International journal publication: *"Preparation and Characterization of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites"*. Submitted to Journal of Applied Polymer Sciences.

(b) faedah-faedah lain

Penemuan teknik baru untuk meningkatkan daya tahanan komposit epoksi terhadap haba dengan melalui pembentukan interaksi ikatan antara bahan organik (epoksi) dan bukan organik (silika).

(c) Latihan Gunatenaga Manusia

(i) Pelajar Prasiswazah

- Husna Abd. Halim
- Nur 'Aini Raman Yusuf
- Nur Farhana Ibrahim
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Penerbitan I

Jamil bin Ismail

N. Ahmad, C. S. Sipaut*, R. Adnan, I. Ab. Rahman, M. A. Bakar, J. Ismail and C. K. Chee:" The effect of modified silica-fillers with different organic compound in epoxy composites properties", Abstract 1st Penang International Conference for Young Chemist 24th – 26th May 2006, Universiti Sains Malaysia, Penang.

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THE EFFECT OF MODIFIED SILICA-FILLERS WITH DIFFERENT ORGANIC COMPOUND IN EPOXY COMPOSITES PROPERTIES

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Nanofumed silica with mean particle sizes in the range of 15-30 nm was surface modified by epichlorohydrin and epoxy resin molecule. The properties of surface modified silica and their effect as fillers in epoxy composite were characterized by Fourier Transform Infrared spectroscopy (FTIR), Thermogravimetri Analysis (TGA), Coefficient of Thermal Expansion (CTE), tensile testing and Scanning Electron Microscopy (SEM). From FTIR and TGA analysis, it was found that the epoxide ring was chemically bonded onto silica surface. The CTE and tensile testing analysis, shows the addition of 5 % modified silica fillers with different modifier into the composite matrix, highly influences their thermal properties. Epoxy composite filled with modified silica-epichlorohydrin fillers (MSEpi) has higher stability of CTE value than epoxy composite filled with pure silica (PS) but lower than that of epoxy composite filled with modified silica-epoxy filler (MSE). The composite matrix with and without modification show no significant differences in their tensile properties. This behavior was further supported by SEM analysis which indicates that the distribution of silica particles in epoxy composite filled with MSEpi has better dispersion than that of epoxy composite filled with MSE.

Keywords: Nanosilica, Modified Fumed Silica, Nanocomposite and Epoxy Composite.

Penerbitan II

N. Ahmad, C. S. Sipaut*, R. Adnan, I. Ab. Rahman, M. A. Bakar, J. Ismail and C. K. Chee:" Properties and Morphology of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites, Journal of Applied Sciences 7(1):27-34, 2007.

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Properties and Morphology of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites

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Abstract: Modified fumed silica-epoxy nanocomposites were obtained by refluxing epoxy molecule with fumed silica using imidazole as catalyst. The modified fumed silica was then used as filler in epoxy resin with amine as curing agent. The properties of the surface modified silica and their effect as fillers in bulk epoxy composite were characterized by Fourier Transform Infrared spectroscopy (FTIR), Proton Nuclear Magnetic Resonance Spectroscopy (^{1}H-NMR), Thermogravimetri Analysis (TGA), Differential Scanning Calorimeter (DSC), Coefficient of Thermal Expansion (CTE), Tensile testing, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectrometry (EDX). From FTIR, ^{1}H-NMR and TGA analysis, it was found that the epoxy resin was chemically bonded onto silica surface. From the DSC and CTE analysis, the addition of modified silica filler in the composite matrix highly influences thermal properties. This new synthesis filler shows higher glass transition temperature and more stable CTE data compared to unmodified filler when introduce into composite matrix. The tensile properties of composite matrix with and without the addition of filler show no significant difference in their tensile properties. SEM-EDX analysis show modified fillers have better adhesion with composite matrix compared to unmodified filler.

Key words: Nanosilica, modified fumed silica, nanocomposite, epoxy composite

INTRODUCTION

Epoxy composites with silica are normally used in encapsulation materials for semiconductor devices (Zheng and Ning, 2003; Chen *et al.*, 2004; Ritzenthaler *et al.*, 2000; Ho and Wang, 2001; Wang *et al.*, 2002). The used of silica in semiconductor provide significant properties changes in the epoxy composite. Normally, silica provides high thermal stability, strength, hardness and low cost. These combinations strongly can achieve the high performanee in electrical applications (Bauer *et al.*, 2004; Kickelbick, 2003; Becker *et al.*, 2003; Salahuddin *et al.*, 2002).

Fumed nanosilica based materials are normally low cost and it can be manufactured by high-temperature hydrolysis of silicon tetrachloride in a flame producing smaller sizes 15-30 nm (Wu *et al.*, 2005). The silanol and siloxane group are covered on the silica surface, leading to many applications of silica such as rheological controlling agent in epoxy resin systems, as fillers in toothpaste and car tires and as starting material for optical fibers (Barthel, 1995; Shirono *et al.*, 2001; Stesmans *et al.*, 2005).

However, the intrinsic properties, namely interaction between organic and inorganic phase and disperaibility of the filler in organic media largely affect the properties of composite matrix. Therefore, for excellent properties, strong interfaces between components are required that are normally achieved by the introduction of silica with modification using some organic molecules (Kang *et al.*, 2001).

Kang *et al.* (2001) reported that in the preparation of epoxy composites filled with functionalized nanosilica particles, surface modified particles highly affect the particle dispersion and interface in bulk epoxy composite. They investigated the behavior the Coefficients of Thermal Expansion (CTE) and glass transition temperature (T_g) of bulk epoxy composite with different silica contents and found that the CTE of bulk epoxy composites are reduced and T_g increased with increasing filler contents.

The compatibility effect of poly(propylene-g-maleic anhydride) copolymer (PP-g-MA) on isotactic poly(propylene)-silica nanocomposites on the morphology behavior and mechanical properties of the nanocomposites has been studied by Bikiaris *et al.* (2005). The authors indicate that the addition of modified silica

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Penerbitan III

N. Ahmad, C. S. Sipaut*, R. Adnan, I. Ab. Rahman, M. A. Bakar, J. Ismail and C. K. Chee: "*Preparation and Characterization of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites*". Submitted to journal of applied polymer science.

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Preparation and Characterization of Bulk Epoxy Composites Filled with Modified Fumed Silica-Epoxy Nanocomposites

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ABSTRACT

To study the interfacial effect on properties of bulk epoxy composites, fumed nanosilica were modified by substituting surface silanol groups into epoxide ring. The modified silica (MS) and bulk epoxy composites filled with modified silica were characterized by Fourier Transform Infrared spectroscopy (FTIR), Elemental Analysis (CHN), Thermogravimetric Analysis/Fourier Transform Infrared spectroscopy (TGA/IR), Differential Scanning Calorimeter (DSC), Coefficient of Thermal Expansion (CTE), Mechanical properties, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectrometry (EDX). From FTIR, CHN and TGA/FTIR analysis, it was found that the epoxide ring was chemically bonded onto silica surface. DSC and CTE data show that the modified silica filler highly influence the thermal properties of bulk epoxy composites, such as the modified silica has lower T_g *and lower CTE value compared to bulk epoxy composites with pure silica (PS) filler. The mechanical properties however, are more complicated and no regular results can be found because of the inhomogeneous distribution of the modified silica filler. This finding was also further supported by SEM and EDX analysis.*

INTRODUCTION

Epoxy resins are widely used industrially due to their unique properties like good heat, solvent, lightweight, moisture and chemical resistance, good adhesion to various surfaces and good mechanical properties [1-5]. The epoxy resins are normally used in aerospace, automobile, electrical and electronic industries and chemical process industries. However, epoxy resin cannot be used alone, because of their low thermal properties. Therefore, numerous attempts have been made to improve their thermal properties. The introduction of epoxy/silica nanocomposites systems is a general approach to increase the thermal properties and to have the suitable mechanical requirements [6-10].

Inorganic materials like silica particles have excellent thermal stability, economical and easy to produce. Fumed silica is one type of pyrogenic silica and is commonly used as fillers in epoxy molding compounds (EMC), rheological controlling, thickening and thixotropic agents and starting material for optical fiber [11-12].

Kang et al. [13] investigated the thermal and mechanical properties of bulk epoxy composite filled with funtionalized inorganic-organic nanocomposite silica and found that the intrinsic properties namely the nature of the interface between organic and inorganic phase, highly affect the thermal and mechanical properties of bulk epoxy composite. The silica particles used were precipitated silica obtained from sol-gel method. It has been also reported that surface modification of silica with organic material could improve the dispersity of the filler in bulk epoxy composite [14-18]. The dispersity of filler is known to highly influenced the mechanical properties of bulk epoxy composite [19].

Wu et al. [20] reported that the introduction of modified silica nanoparticles with polypropylene highly improved the mechanical performance of the composite. Besides, the size and surface area of the nanoparticles are also important influencing factors with the smaller nanoparticles lead to higher Young's modulus and impact strength of the composite.

In this research, the effect of surface modification of fillers on bulk epoxy composite properties, especially on thermal and mechanical properties using fumed silica was investigated. In the first stage, the fumed silica was surface modified by epichlorohydrin using crown ether and potassium iodide as catalyst. The modified silica was then characterized by FTIR, CHN and TGA/IR techniques. In the second stage, the effectiveness of modified silica as filler was tested by dispersing it into bulk epoxy composite with different silica content. The thermal and mechanical properties were examined by DSC, CTE, tensile and SEM analysis. The filler dispersity and interfacial properties in bulk epoxy composite were also investigated.

EXPERIMENTAL

MATERIALS

Surface modification of silica

Nanoscale silica particles used was fumed nanosilica (99.8 % purity) manufactured by Sigma. The mean particle sizes are in range of 15-30 nm. Epichlorohydrin used as surface modifier was manufactured by Acros Organic. Reaction catalysts used were 18-crown-6-ether (99 %) and potassium iodide (99 %), manufactured by Acros Organic and R & M Chemicals, respectively.

Bulk epoxy composites preparation

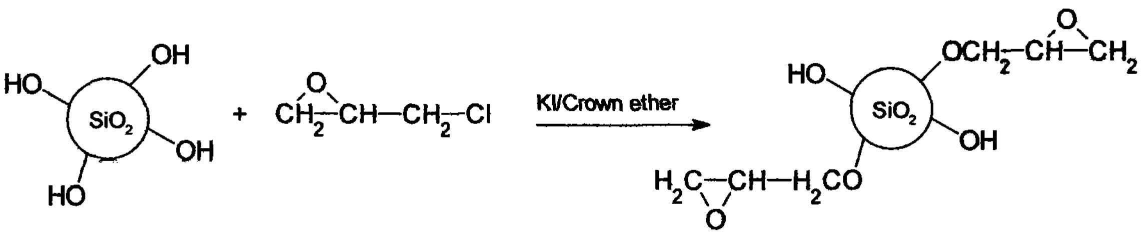
Bulk epoxy composite used to prepare using epoxy resin clear 331 (bisphenol type) manufactured by Echemo Trading with an average molecular weight of 700 g mol^{-1} and the amine hardener (Epoxy hardener clear 2963) of Echemo Trading, is a mixture of trimethylhexamethylene diamine (5-11%), isophorone diamine (30-42 %) and benzyl alcohol (30-42 %).

SAMPLE PREPARATION

Surface modification of fumed silica

1 wt. % of potassium iodide, 18-crown-6-ether (0.87 wt. %) and 50 ml of epichlorohydrin were mixed together. After the solutes were completely dissolved, three grams of fumed silica was added into the solution. The mixture was refluxed at 40 °C for 24 hours. The solvent was removed by acetone under

centrifugation and the collected product was dried in an oven at 110 °C. The collected product (MS) was pretreated at 110 °C under vacuum for 24 hours to eliminate water on the particles surface [13]. The surface modification is shown in Scheme 1.



Scheme 1: The surface modification of nanosilica particles

Bulk epoxy composite

A fixed amount of silica nanoparticles and amine hardener (Table 1) were dispersed by ultrasonic instrument at 70 ± 2 °C. After the silica nanoparticles were completely dispersed, the desired amount of epoxy resin was added into the silica/amine hardener solution. They were mixed gently to minimize any air bubbles and follow the earlier steps. The mixture was stirred at room temperature for 15 minutes to obtain a homogeneous solution. Later, the mixture was poured into a preheated polytetrafluoroethylene (PTFE) mould and cured at room temperature for 5 hours.

Table 1: Preparation of bulk epoxy composite

Sample	Epoxy resin (ml)	Amine hardener (ml)	PS (%)	MS (%)
Blank	5	5	-	-
PS-5	5	5	5	-
PS-10	5	5	10	-
PS-20	5	5	20	-
PS-30	5	5	30	-
MS-5	5	5	-	5
MS-10	5	5	-	10
MS-20	5	5	-	20
MS-30	5	5	-	30

CHARACTERIZATIONS

Surface modification of fumed silica

In the modification stage, all samples either pure silica or modified silica particles were characterized to verify that the chemical reactions occurred as planned. The techniques of characterization are includes Fourier Transform Infrared (FTIR) measurements were recorded on a Pelkin Elmer 2000 from 4000 to 400 cm^{-1} region on KBr discs. Elemental analysis for Carbon, Nitrogen and Hydrogen was carried out using

Perkin Elmer 2400 Series II model of CHNS/O analyzer. The samples were heated in combustion column at 925 °C under helium atmosphere. Thermal analysis was performed using a Mettler Toledo (TGA/SDTA 851^{e}) model under nitrogen flow at heating rate of 10 °C min^{-1} from 50 to 950 °C. The output of the inert gas from the TGA was connected to a Nicolet 5700 FTIR system through a heated line. This technique was conducted to study of the qualitative evolution of the composition of the gases evolved during heating process.

Bulk epoxy composite

The effect of pure and modified silica as filler in bulk epoxy composite on thermal and morphology properties were investigated. Differential Scanning Calorimetry (DSC) temperature scans were taken after cure from -50 to 300 °C at a scanning rate of 20 °C min^{-1} under nitrogen atmosphere using Pelkin Elmer Pyris 1 DSC. The CTE was measured using a dilatometer (NETZSCH DIL 402 C) in the temperature range of 30 to 60 °C. A sample of approximately 2 mm in length was used at a heating rate of 10 °C min^{-1} under nitrogen atmosphere. The mechanical properties (tensile properties) were determined using a Hounsfield Test Equipment Ltd using a crosshead speed of 100 mm min^{-1}. The rectangular bar samples is 80 x 10 x 2 mm^3 and gauge length was set at 40 mm. The morphology of nanocomposites was examined by Leo Supra 50 VP Field Emission model SEM. Samples were gripped by pliers, chilled in liquid N_2 and then fractured. The specimen was deposited on double-sided scotch and examined at the fractured surface. The fractured surfaces were coated with thin layer (20 nm) of gold to improve SEM imaging using a Polaron SC 515. EDX analysis used to determine the elemental compositions of the particles was conducted using Oxford INCA 400 energy dispersive x-ray microanalysis system attached to SEM.

RESULTS AND DISCUSSIONS

Results and discussions was divided into two main sections namely, the characterization of the surface modified silica and their effect as fillers in bulk composite.

MODIFIED FUMED SILICA

1. Analysis of modified silica by FTIR

This analysis was conducted to verify that the chemical reactions occurred as illustrated in Scheme 1. Figure 1 shows the FTIR spectra for the pure material (PS), together with that of the surface-modified fumed silica (MS).

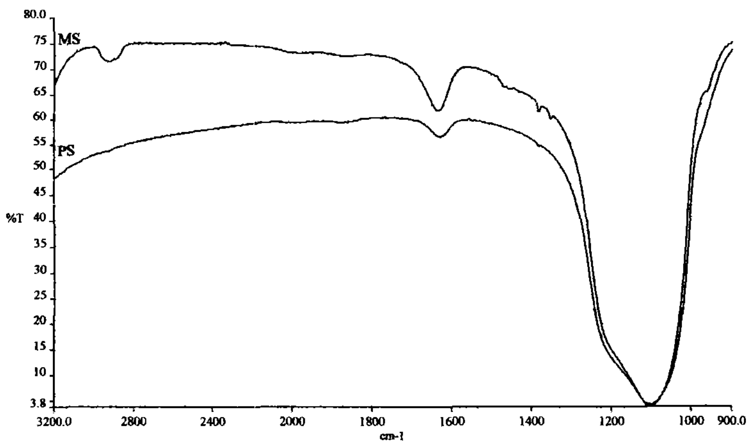


Figure 1: FTIR spectra of PS and MS.

A significant appearance of new peak was observed in the spectra of MS compared to PS. The new peaks, such as weak peaks near 2924 and 1350 cm^{-1} correspond to the absorption of aliphatic C-H stretching from the inclusion of epoxide ring onto silica surfaces. Moreover, a shoulder peak near 960 cm^{-1} was expected to be oxirane ring [13].

Based on the appearance of these new peaks, it was strongly suggested that fumed silica could have been chemically bonded with organic molecule. This phenomenon was further investigated by CHN analysis.

2. CHN analysis on modified nanosilica

CHN analysis was conducted on the sample to determine the compositions of carbon, hydrogen and nitrogen in the samples. Table 2 shows the percentage of C, H and N in PS and MS. As expected PS, has 0.00 wt. % of C but the modification of silica surface (MS) has increased the percentage of C compositions in the silica particles to 2.08 wt. %. This finding highly supports our previous assumption that reaction between silica and epichlorohydrin had taken place.

Table 2: CHN analysis of PS and MS.

Sample	**Weight , %**		
	Carbon (C)	**Hydrogen (H)**	**Nitrogen(N)**
PS	0.00	-	-
MS	2.08	-	-

3. TGA/FTIR analysis on modified nanosilica

TGA/FTIR analysis was conducted on the product to further support that the modification of silica surface has taken place. Figure 2 shows the TGA thermogram of PS and MS. TGA thermogram of PS shows no weight loss during the heating temperature from 50 to 950 °C and also no release of any other substances which is in accordance with Liu et al. [15]. However, the TG curve for MS shows three stages of weight loss steps with total weight loss of 8.3 %. In the first step, weight loss of approximately 1.9 % at temperature below 100 °C was due to H_2O molecules. In the second and third step, total weight loss of 6.3 % at temperature above 285 °C was due to the organic molecules. This is because thermal decomposition of epoxy resin normally occurs between 200 to 400 °C [19].

This phenomenon was confirmed by TGA/FTIR results. Figure 3 shows the TGA/FTIR spectra of evolution of specific gasses from MS at different heating time. All TGA/FTIR spectra give a similar peak pattern. The generation of CO_2 and H_2O were observed over the whole range. The other absorption peaks are attributed to gasses released by pure epoxy material. This is supported by TGA/FTIR spectra in Figure 4 (TGA/FTIR spectra of evolution of specific gasses from PS, pure epoxy and MS). PS sample shows no gasses are released during heating process. Absorption peak of gasses released by MS shows a similar pattern as that of pure epoxy. This finding highly suggests that the surface silica was modified by epoxide ring.

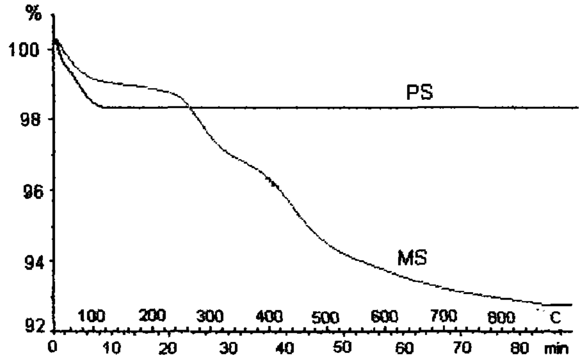


Figure 2: TGA thermogram of PS and MS.

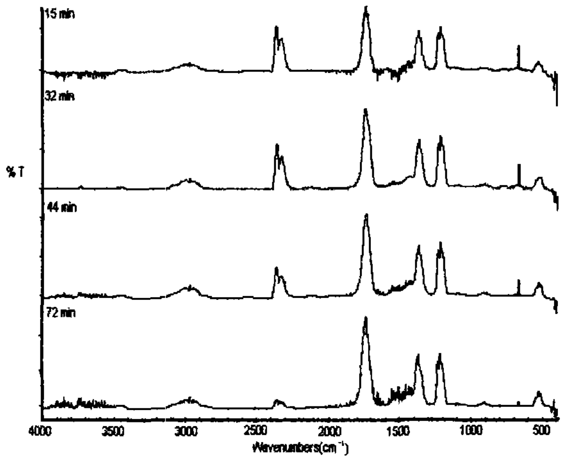


Figure 3:FTIR-windows:evolution of the evolving gasses released by MS during heating process..

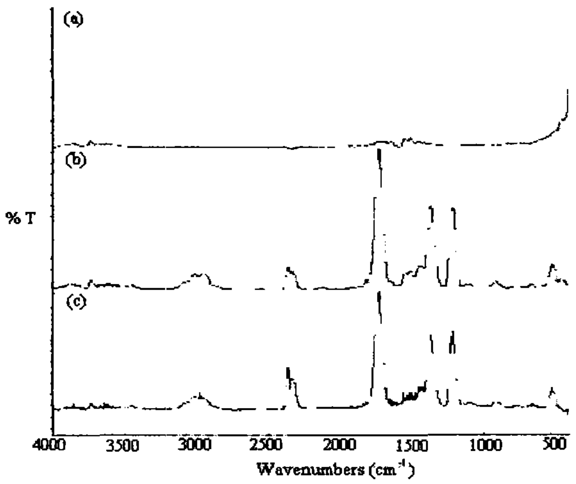


Figure 4 . FTIR-windows: evolution of the evolving gasses released by: (a) PS, (b) Pure Epoxy, and (c) MS.

THE EFFECT OF MODIFIED SILICA AS FILLER IN BULK EPOXY COMPOSITE

1. The effect of glass-transition temperature

T_gs of the samples were determined from the tangents of DSC spectra as a function of temperature. Figure 5 shows the plot of the glass temperature (T_g) as a function of filler concentration for bulk epoxy composite with addition of PS and MS fillers at different concentration. At each specific filler concentration, composite prepared with PS have high T_g value compared to composite prepared with MS filler. The lower T_g values for MS filler composite were possibly due to decreased in formation of the covalent bond between silica surface and the matrix to form chemical crosslink compared to the other system (PS) which is in accordance with Kang et al [13]. This behavior can be described using the free volume concept which is the decreasing of crosslink in polymer matrix will increase the specific volume, more mobility of the molecular motion required less energy for rotation therefore decrease T_g value. The composite prepared with MS filler have relatively, more mobility tends to have low values of T_g compared to composite filled with PS filler. This finding gave the further evidence of the successfulness of the modification of the silica surface with epoxide ring [21].

Both systems show a similar trend as the filler's concentration was increased. When the filler concentration was increased to 5 wt. %, the T_g value increases and then reduces at 10 wt. %. After the addition 20 wt. % of filler the T_g value become constant. This indicates that the optimum concentration of bulk epoxy is 5 wt. %.

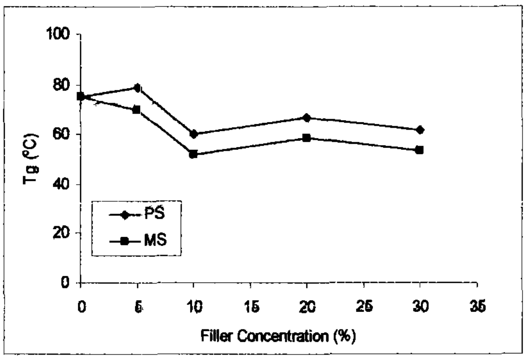


Figure 5 : T_g of composite filled with PS and MS as a function of filler concentration.

2. The effect of CTE

The CTE value was correlated as the expansion or shrinkage of sample on heating phenomenon depends on internal interaction namely intermolecular forces. Figure 5 shows the plot of CTE values as a function of filler concentration. The curve shows that for CTE of blank composite sample give higher negative value. This behavior indicating that sample tends to shrink i. e unstable sample. Both curves show a similar trend. With the addition of 5 wt. % of filler, the CTE value of both composite matrixes decreased. This behavior shows that the addition of silica filler in the composite matrix can reduce the degree of shrinkage. Increasing filler concentration, the curve of composite filled with PS and MS filler were approximately zero. It is known that increasing filler concentration in epoxy matrix, tends to reduce the degree of shrinkage in the epoxy thus stabilizes the matrixs [22]

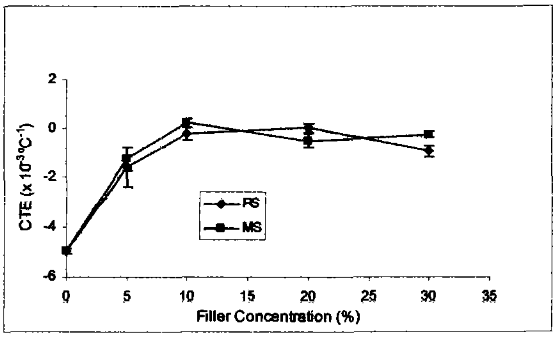


Figure 6: CTE value of pure and modified silica.

3. Mechanical properties

The blank composite and composite filled with PS and MS filler were subjected to a tensile properties test. The modulus at 5 %, Ultimate Tensile Strength, UTS and elongation at break value with blank, PS and MS composite are shown in Figure 7, 8 and 9 respectively. Based on Figure 7, both curves show no changes in UTSs at 0 to 5 wt. % filler concentration. Increasing the percentage of filler, both of curve drops to change of UTSs becomes slowly. However, after 20 wt. % of silica, the PS curves increase dramatically while the MS curves show no significant changes in UTSs value. The PS has higher UTSs value than MS.

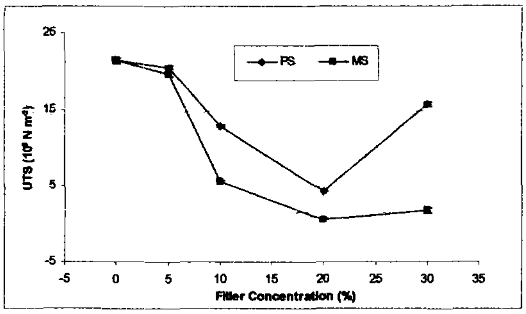


Figure 7 : Effect of silica modification on tensile strength of bulk epoxy composites at different filler contents.

Similar results can be found about the modulus at 5 % (Figure 8). According to the results given in Figure 7, the modulus at 5 % of bulk epoxy composite with PS filler generally have higher modulus value compared to MS filler. This demonstrates that the mechanical property of bulk epoxy composite is also not significant.

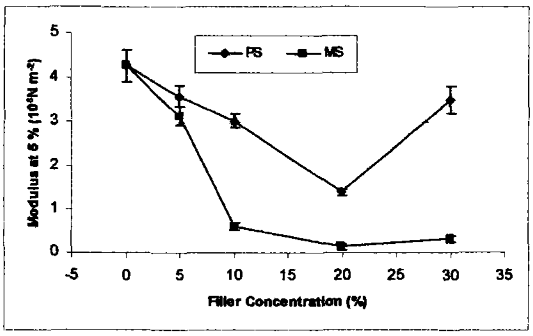


Figure 8: Effect of silica modification on Modulus at 5 % with different filler contents

The break elongation for bulk epoxy composites with different filler and concentration are shown in Figure 8. Both curves show that no changes in elongation at break from 0 to 5 wt. % of filler concentration. After 5 wt. % addition of filler, the bulk epoxy composite with MS filler has higher elongation at break values compared to bulk epoxy composite with PS filler. However, at higher filler concentration, the situation is more complicated and no regular results can be found.

From the results, it shows that within the limit of experimental error there is no significant different on the tensile values for all formulations. This phenomenon might be caused by inhomogeneous mixing during sample preparation. However, in the presence of either PS or MS filler, the silica particles tend to form agglomerate in the composite matrix. As evident from the work of Xing et al. [17] good dispersion of particles in the epoxy matrix can be achieved at lower percentage of filler (below 2.5 %). However, at higher filler concentration (up to 4 %) loading, it tends to form agglomerate. According to the findings of Wu et al., the changes in the tensile properties could be found if the loading capacity is less than 1 % [20]. Hence, the preparation technique namely good mixing need to be improved so that it can leads to breakdown of the agglomerate and improves the interaction between particles and polymer matrix.

This finding shows that the dispersity of filler in the matrix greatly influences the mechanical behavior of the matrix. To confirm this, SEM analysis was preformed to investigate the dispersity and interface interaction between filler and epoxy matrix.

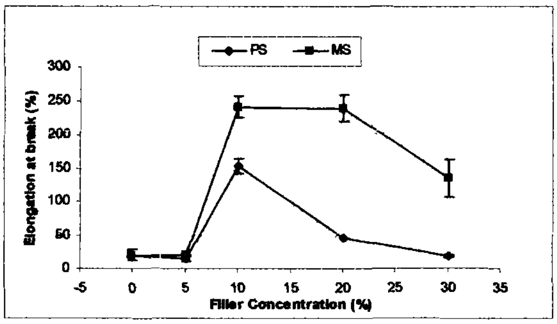


Figure 9: Effect of silica modification on failure elongation with different filler concentration.

3. SEM-EDX of fractured surface.

The good dispersity and strong interface between filler and epoxy matrix are of great factors to improve the mechanical behavior of composite. SEM micrograph of pure epoxy resin in Figure 10(a) shows the clear river lines with smooth surface on the fracture surface part. However in Figure 10(b), the SEM of composite filled with 5 wt. % of PS illustrates the river lines crowded were together. The additions of PS into epoxy matrix highly affect the fracture surface and brittle behavior of bulk epoxy composite [1]. At higher silica concentration the composite matrix becomes brittle which is discovered by Wang et al. that the nanocomposite film becomes opaque and very fragile when the silica content increases to 12 wt. % [19]. This behavior is attributed to the formation of hydrogen bonding between the silanol groups with the matrix which generate more ordered molecule i.e more river lines.

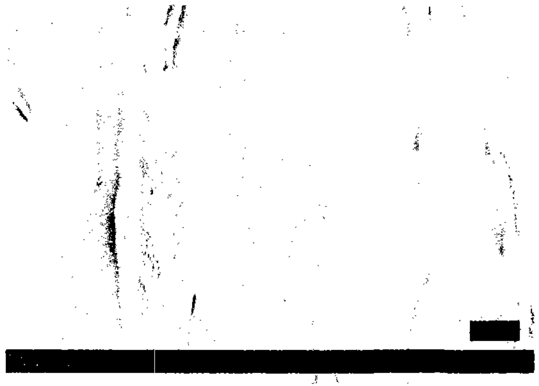


Figure 10(a) : Unfilled composite matrix.

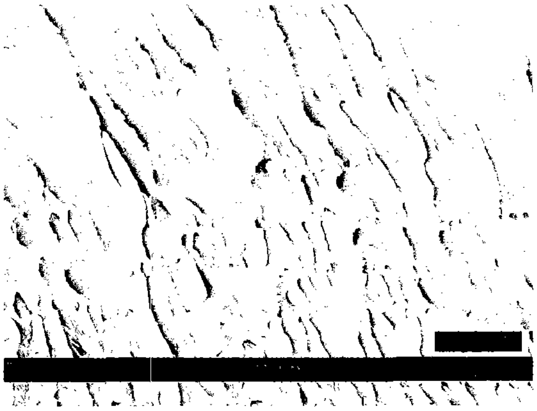


Figure 10(b) : Unmodified filler composite matrix.

The morphology of modified silica particles dispersed into epoxy matrix with different silica content was observed by SEM are shown in Figures 10(c-e).

Figure 10(c): 5 wt % Modified filler composite matrix.

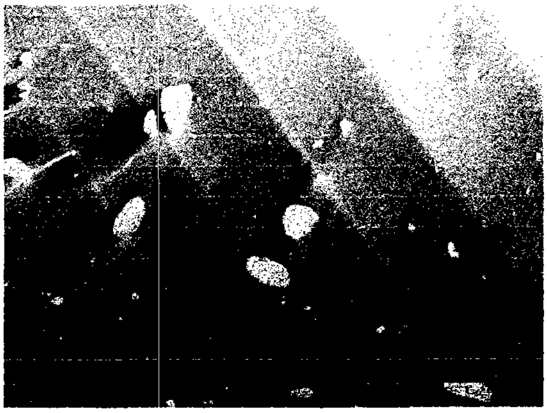


Figure 10(d): 20 wt % Modified filler composite matrix.

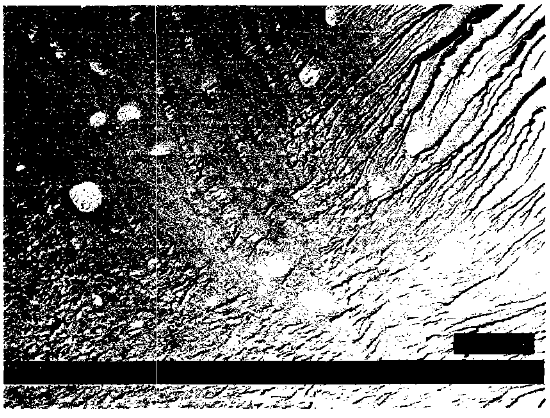


Figure 10(e): 30 wt. % Modified filler composite matrix

SEM micrograph (Figure 10(c)), shows that silica nanoparticles were well dispersed, however Figure 10(d-e) show that silica particles are spherical in shape and agglomerates. It is suggested that the silica particles are well bonded in the epoxy matrix but the particles have a strong tendency to agglomerate due to weak interaction between the two phases. This resulted in the particles dispersion into the epoxy matrix not being homogeneous. The agglomerate is caused by the formation of a silica-silica aggregated structure due to hydrogen bonding.

Is it also noted that there is no silica agglomeration formed in unmodified filler (Figure 10(b)). This hinted that the weak interaction between silica and matrix during sample preparation for SEM analysis (fracturing). This observation was supported by EDX analysis, which showed there is no silica detected in the sample (Figure 11). However, the strong interaction of modified silica particle with matrix shows the 1.41 % of silica remains intact in composite matrix (Figure 12) which is in accordance with Kang et al. [13]. This phenomenon gave further support to the above-mentioned matter. Figure 12 shows the composition of silica compound on fractured MS composite with 5, 20 and 30 %. Percentage of silica weight increased with the increased of silica content in the composite but not linearly. This shows that the mixing of the sample were not homogeneous during the preparation stage.

Element	Weight (%)	Atomic (%)
C K	82.89	86.58
O K	17.11	13.42
SiK	0.00	0.00
Total	100.00	

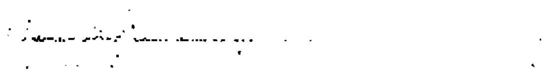


Figure 11: EDX spectra of fumed silica (5 % PS)

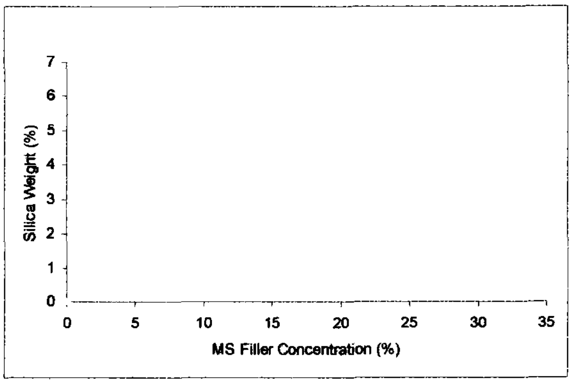


Figure 12: Percentage of silica weight of composite filled with at various MS filler concentration.

CONCLUSIONS

In this paper, we report our successful attempt to surface modified fume silica with epichlorohydrin molecules using crown ether and potassium iodide as the reaction catalysts. This modification was characterized and confirmed by FTIR, CHN and TGA/IR analysis. The effectiveness of modified silica (MS) composite as filler in epoxy matrix was also presented. The MS composite highly affects the thermal properties, namely T_g and CTE values by reducing the T_g value and also slightly stabilizes the CTE of bulk epoxy composite. However, the mechanical property of the composite with MS filler is complicated and no regular result can be found. This phenomenon was supported by SEM.

ACKNOWLEDGEMENTS

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Lampiran Abstrak-abstrak Penyelidikan Projek Tahun Akhir

Abstrak I: Kesan Penambahan Resin Epoksi dan Imidazola terhadap Pembentukan Busa Poliuretana.

Abstrak II: Pengaruh Agen Pemanjangan Rantai terhadap Pembentukan Busa Poliuretana.

Abstrak III: Kajian Kesan pelarut dalam Pembentukan Busa Poliuretana.

Abstrak IV: Kesan Pengisi terhadap Pembentukan Busa Poliuretana.

Abstrak V: Kesan Penambahan Di-(3-Aminopropil) Eter dan Polietilenaglikol dalam Tindakbalas antara Resin Epoksi dengan Agen Pematangan Amina Industri.

Abstrak VI: Kesan Perbezaan Bilangan Kumpulan Berfungsi bagi Amina sebagai Agen Pematangan di dalam Pembentukan Komposit Epoksi.

Abstrak VII: Comparison between Carbon Black and Silica as a Filler in Epoxy Composite

Abstrak VIII: Perbandingan di antara Lignin dan Silika sebagai Pengisi di dalam Komposit Epoksi

Abstrak ~~VIII~~: Perbandingan antara Karbon Teraktif dan Poliuretana sebagai Pengisi dalam Komposit Epoksi. x
IX

Abstrak I:

Abstrak

Kajian ini dijalankan untuk mengkaji kesan resin epoksi dan imidazola terhadap pembentukkan busa poliuretana. Sampel-sampel telah dicirikan menggunakan beberapa kaedah seperti analisis Inframerah (FTIR), ketumpatan busa, analisis Mikroskop Pengimbas Elektron (SEM), analisis set mampatan dan analisis kekuatan mampatan. Daripada analisis FTIR telah menunjukkan bahawa ikatan uretana terbentuk dengan kehilangan puncak 2275 cm^{-1} yang mewakili kumpulan NCO dan kemunculan puncak baru iaitu pada 1600-1594 cm^{-1} dan 1729-1708 cm^{-1} yang masing-masing mewakili ikatan C=O dan N-H sekunder. Kehilangan puncak 915 cm^{-1} yang mewakili sifat utama epoksi juga menunjukkan bahawa telah berlaku tindak balas pembukaan gelang epoksi. Daripada analisis-analisis sifat busa poliuretana yang dihasilkan, didapati apabila peratus jisim resin epoksi dan imidazola ditingkatkan, ketumpatan busa menurun menyebabkan saiz sel dan keanjalan meningkat disamping penurunan pada kekuatan dan modulus mampatan. Berdasarkan keputusan yang diperoleh, penambahan resin epoksi dan imidazola telah mengurangkan darjah rangkai silang yang terhasil dalam pembentukkan busa poliuretana.

Kata kunci: Busa poliuretana, Resin epoksi, Imidazola, Rangkai silang.

Abstrak II

ABSTRAK

Tujuan kajian ini dijalankan adalah untuk mengkaji pengaruh amina sebagai agen pemanjangan rantai terhadap pembentukan busa poliuretana (PU). Ancamida 503 (AC3) dan di-(3-aminopropil) eter dietilena glikol (DCA 221) telah dipilih sebagai agen pemanjangan rantai. Sampel-sampel telah dicirikan melalui analisis infra merah (IR), ketumpatan busa, struktur dan saiz sel, set mampatan serta kekuatan mekanikal. Analisis infra merah telah menunjukkan bahawa pembentukan ikatan uretana telah berlaku antara kumpulan isosianat, poliol dan amina melalui kehilangan puncak 2276 cm^{-1} bagi kumpulan N=C=O serta kewujudan puncak baru pada 3414 cm^{-1} dan 1717 cm^{-1} masing – masing merupakan ciri utama bagi NH dan C=O. Hasil kajian menunjukkan bahawa apabila peratus AC3 dan DCA 221 ditingkatkan, penghasilan rangkai silang dikurangkan melalui pengurangan pengikatan hidrogen walaupun terbentuknya ikatan kovalen. Kajian telah menunjukkan bahawa apabila kepekatan AC3 dan DCA 221 ditingkatkan, ketumpatan busa, keanjalan dan kekuatan mekanikal berkurang di samping peningkatan pada saiz sel. Dapat diperhatikan juga pada setiap peratus kepekatan agen pemanjangan rantai yang sama, ketumpatan busa PU yang mengandungi AC3 lebih tinggi berbanding dengan DCA 221.

Kata kunci : Poliuretana, Ancamida 503, Di-(3-aminopropil) eter dietilena glikol.

Abstrak III

ABSTRAK

Tujuan kajian penyelidikan ini dijalankan adalah untuk mengkaji kesan pelarut dalam pembentukan busa poliuretana serta sifat-sifat busa yang dihasilkan. Terdapat tiga jenis pelarut digunakan iaitu air (H_2O), aseton (C_3H_6O) dan kloroform ($CHCl_3$). Sampel dicirikan dengan analisis Inframerah (IR), ketumpatan busa, saiz sel, set mampatan dan kekuatan mampatan. Daripada analisis Inframerah, menunjukkkan bahawa tindak balas antara kumpulan isosianat (NCO) dengan kumpulan hidroksil (OH) dan pelarut H_2O telah berlaku membentuk ikatan uretana. Hal ini dapat diperhatikan dengan pengurangan puncak pada frekuensi 2279 cm^{-1} yang menunjukkkan kehadiran kumpulan berfungsi NCO. Selain itu, terdapat puncak yang tajam pada frekuensi 3325 cm^{-1} menunjukkan kehadiran kumpulan berfungsi N-H dan puncak yang lebar pada frekuensi 3391 cm^{-1} menunjukkan kehadiran kumpulan berfungsi hidroksil (OH). Keputusan juga menunjukkan bahawa dengan peningkatan peratus jisim pelarut dalam sistem busa poliuretana, ketumpatan busa dan kekuatan mampatan berkurangan. Hal ini diikuti dengan pembesaran saiz sel dan keanjalan busa poliuretana bertambah baik. Pada peratus jisim pelarut yang sama, pelarut C_3H_6O memberikan kesan ketumpatan busa yang tinggi, saiz sel yang besar dan keanjalan yang baik berbanding pelarut $CHCl_3$. Daripada keputusan yang diperoleh, ia juga menunjukkan pada peratus jisim pelarut H_2O yang tinggi (>1.5 %) akan menyebabkan pemecahan dinding sel busa.

Kata kunci : Poliuretana, Busa, pelarut H_2O, pelarut $CHCl_3$, pelarut C_3H_6O

Abstrak IV

ABSTRAK

Kajian ini dijalankan untuk mengkaji kesan penambahan agen pengisi terhadap sifat-sifat busa poliuretana (PU). Dua agen pengisi yang digunakan adalah Tandan Sawit Kosong (TSK) dan lignin. Pencirian sampel busa PU dilakukan dengan analisis Inframerah (IR), ketumpatan busa, saiz dan struktur sel, keanjalan dan kekuatan mampatan. Analisis FTIR menunjukkan ikatan uretana terbentuk dengan kehilangan puncak pada 2275 cm^{-1} yang mewakili kumpulan fungsi NCO dan kewujudan puncak baru pada 3325 cm^{-1} dan 1733 cm^{-1} yang masing-masing mewakili ikatan N-H dan ikatan C=O. Daripada analisis yang dijalankan ke atas penambahan peratus pengisi ke dalam busa PU, didapati apabila peratus pengisi ditingkatkan, ketumpatan busa menurun menyebabkan penurunan kekuatan mampatan disamping peningkatan saiz sel dan keanjalan. Selain itu, bagi setiap peratus pengisi yang sama, didapati ketumpatan busa PU yang ditambah lignin adalah tinggi berbanding dengan busa PU yang ditambah pengisi TSK.

Kata kunci : Busa poliuretana (BPU),pengisi, Tandan Sawit Kosong (TSK), Lignin.

Abstrak V

ABSTRAK

Tujuan kajian ini dijalankan adalah untuk mengkaji kesan penambahan polietilenaglikol (PEG) dan Di-(3-aminopropil) eter (DG) bagi tindak balas antara resin epoksi dengan agen pematangan amina industri. Sampel-sampel telah dicirikan menggunakan beberapa kaedah seperti Analisis Inframerah (FTIR), Analisis Kalorimeter Pembezaan Pengimbasan (DSC), Analisis Terma Gravimetrik (TGA) dan Ujian Kekuatan Regangan. Daripada analisis inframerah menunjukkan bahawa tindak balas antara resin epoksi, agen pematangan amina industri dan Di-(3-aminopropil) eter telah berlaku. Hal ini boleh diperhatikan dengan kewujudan puncak kumpulan O-H pada 3587 cm^{-1} manakala puncak-puncak pada 3345 cm^{-1} dan 3269 cm^{-1} menunjukkan kumpulan amina dan puncak 915 cm^{-1} yang menunjukkan gelang epoksi telah hilang. Manakala dengan kehadiran polietilenaglikol dalam sistem menunjukkan bahawa tindak balas antara kumpulan O-H daripada polietilenaglikol atau kumpulan amina daripada agen pematangan amina industri dengan molekul epoksi adalah tidak dapat dipastikan melalui analisis inframerah. Keputusan juga menunjukkan penambahan polietilenaglikol dalam komposit epoksi memberikan tahap pembentukan rangkai silang yang lebih tinggi berbanding dengan penambahan Di-(3-aminopropil) eter. Keputusan ini disokong dengan nilai modulus dan kekuatan regangan muktamad yang lebih tinggi bagi sampel campuran polietilenaglikol berbanding Di-(3-aminopropil) eter melalui ujian kekuatan regangan. Walau bagaimanapun, melalui analisis TGA dan analisis DSC tiada sebarang keputusan seragam diperhatikan.

Kata kunci : resin epoksi, agen pematangan amina industri, Di-(3-aminopropil) eter, polietilenaglikol dan rangkai silang

Abstrak VII:

ABSTRACT

The purpose of this research was to study the comparison and the effect of carbon black and silica filler in the epoxy composite. Epoxy composite sample at a ratio 1:1 (epoxy resin to amine hardener) were prepared by introducing the carbon black and silica filler at different loading capacity. All samples were characterized by Fourier Transform Infared (FTIR), Differential Scanning Calorimetry (DSC), Thermal Gravimetry Analysis (TGA), Scanning Electron Microscope (SEM) and tensile strength. FTIR analysis shows no significant different spectra with or without the present of carbon black filler in the epoxy composite. However, a broad peak was observed at 1106 cm^{-1} attributed to OH group of silica filler in epoxy composite. In DSC analysis, it shows that the glass transition, T_g value for reference sample is higher compare to the sample in the present of filler. Furthermore, increasing filler loading either carbon black or silica filler in epoxy composite shows no significant trend in the glass transition temperature. From the TGA analysis, the result shows that increase filler content, the decomposition temperature of the sample increased. An uneven surface of sample were observed in SEM analysis as well as bubbles are formed in some part of the sample. For mechanical properties, increasing the percentages of filler (either carbon black or silica) in epoxy composite show higher Young's Modulus and lower elongation at break. However, no significant trend was observed for Ultimate Tensile Strength values.

Key words : Epoxy resin, Silica, Carbon black, Amine hardener, Filler

Abstrak VIII

ABSTRAK

Kajian ini dijalankan untuk membuat perbandingan penambahan pengisi di dalam komposit epoksi. Dua jenis pengisi yang digunakan adalah lignin dan 'fumed' silika. Sampel dicirikan dengan menggunakan beberapa kaedah iaitu analisis infra merah (FTIR), analisis terma gravimetri (TGA), kalorimeter pengimbasan pembezaan (DSC), mikroskop pengimbasan elektron (SEM) dan kekuatan regangan. Nisbah terbaik yang dicadangkan dalam kajian ini adalah 1 : 1 bagi epoksi kepada agen pematangan amina. Analisis FTIR menunjukkan bahawa pembentukan rangkai silang telah berlaku antara gelang epoksi dengan agen pematangan amina melalui kehilangan puncak untuk gelang epoksi 915 cm^{-1} dan 3300 cm^{-1} – 3200 cm^{-1} pada puncak amina serta kemunculan puncak baru pada 3360 cm^{-1} yang menunjukkan kehadiran puncak O-H. Daripada analisis TGA yang dijalankan, sampel rujukan melibatkan tiga tahap pembakaran dan sampel dengan pertambahan pengisi hanya melibatkan dua tahap pembakaran. Penambahan pengisi ke dalam sampel komposit epoksi akan memberikan nilai T_g yang lebih rendah berbanding dengan sampel komposit epoksi tanpa pengisi. Didapati bahawa apabila peratusan pengisi ditingkatkan, akan berlaku penyebaran pengisi yang tidak seragam di dalam matriks. Analisis kekuatan tensil pula menunjukkan penambahan lignin akan meningkatkan Modulus Young dan mengurangkan pemanjangan muktamad. Keadaan ini tidak dilihat pada pengisi silika.

Kata kunci : komposit epoksi, agen pamatangan amina, lignin, 'fumed' silika.

Abstrak VIIII:

ABSTRAK

Tujuan kajian ini dijalankan adalah untuk mengkaji kesan penggunaan pengisi karbon teraktif dan poliuretena ke atas komposit epoksi. Pencirian komposit epoksi dilakukan dengan menggunakan lima jenis analisis iaitu analisis infra merah (FTIR), analisis terma gravimetri (TGA), kalorimeter pengimbasan pembezaan (DSC), mikroskop pengimbasan elektron (SEM), dan kekuatan regangan. Analisis FTIR menunjukkan bahawa pembentukan rangkai silang telah berlaku antara gelang epoksi dengan agen pematangan amina melalui kehilangan puncak untuk gelang epoksi 915 cm^{-1} dan 3300 cm^{-1} - 3200 cm^{-1} pada puncak amina serta kemunculan puncak baru pada 3360 cm^{-1} yang menunjukkan kehadiran puncak O-H. Kehadiran pengisi karbon teraktif dalam komposit epoksi dibuktikan kehadirannya apabila panjang gelonbang pada 1557 cm^{-1} untuk ikatan C=C telah hilang dan begitu juga seperti kehadiran poliuretena didalam komposit epoksi dibuktikan apabila panjang gelonbang yang dipunyai oleh poliuretena pada 1050 cm^{-1} untuk regangan C-O telah hilang. Keputusan analisis TGA menunjukkan tahap pembakaran yang berbeza diantara karbon teraktif dan poliuretena. Daripada analisis DSC yang dijalankan terdapat dua peringkat bacaaan T_g yang diperoleh bagi kedua-dua pengisi iaitu karbon teraktif dan poliuretena. Secara umumnya poliuretena memberikan bacaan T_g yang tinggi berbanding dengan karbon teraktif dalam komposit epoksi. Bagi analisis SEM, kehadiran pengisi poliuretena dalam komposit epoksi memberikan permukaan patahan yang lebih kasar berbanding dengan kehadiran pengisi karbon teraktif. Manakala, bagi ujian regangan ia menunjukkan kehadiran pengisi karbon teraktif memberikan nilai Modulus Young yang lebih tinggi dan kekuatan muktamad lebih tinggi daripada pengisi poliuretena dan pengisi poliuretena pula menunjukkan pemanjangan muktamad yang lebih rendah dari karbon teraktif.

Kata kunci : komposit, epoksi, poliuretena, karbon teraktif, ujian polimer