Production and Characterization of Shape Memory Epoxy Foam by an Advanced Aqueous Method

by

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LIST OF ABBREVIATIONS

AHEW	Amine Hydrogen Equivalent Weight
ASTM	American Society Testing and Material
CIPS	Chemically induced phase separation
Cl	Chlorine
CO_2	Carbon Dioxide
Cr_2O_3	Chromium (III) oxide
DDM	4, 4'-methylenedianiline
DEG	Diethylene glycol
DGEBA	Diglycidyl Ether of Bisphenol A
EEW	Epoxy Equivalent Weight
H_2O	Water
IPNs	Interpenetrating polymer networks
MnO	Manganese oxide
$NaHCO_3$	Sodium bicarbonate
NH_4Cl	Ammonium chloride
O/W/O	Oil in water in oil
P	Phosforus
PACM	Methylenebiscyclohexanamine
PAPI	Polymer assisted phase inversion
PEG	Polyethylene glycol
phr	Per hundred resin
PIPS	Polymerization induced phase separation

PP	Polypropylene
PS	Polystyrene
PU	Polyurethane
rpm	Rotating per minute
S	Sulfur
SEM	Scanning Electron Microscopy
SiO_2	Silicon dioxide
SMP	Shape memory polymer
TGA	Thermogravimetry Analysis
THF	Tetrahydrofuran
TIPS	Thermally induced phase separation
W/O/W	Water in oil in water

LIST OF SYMBOLS

%	Percentage
^{o}C	Degree Celcius
µm	micrometer
bsr	Bahagi seratus resin
cm^3	Centimeter cubic
g/eq	Gram per equivalent
g/ml	Gram per mililiter
g/mol	Gram per mol
mm	milimeter
mmol/kg	Milimol per kilogram
phr	Per hundred resin
rpm	Rotating per minute

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Penghasilan dan Pencirian Busa Epoksi Memori Bentuk oleh Kaedah Akues Termaju

ABSTRAK

Busa memori bentuk dianggap sebagai suatu rekacipta berfaedah yang sedang diberi tumpuan oleh penyelidik baru-baru ini. Keupayaan busa memori bentuk adalah ia akan berubah bentuk di bawah daya-daya mekanikal tertentu dan perlahan-lahan pulih kembali ke bentuk asalnya seolah-olah ia dimemorikan. Kajian ini telah mencadangkan dengan kaedah akues termaju untuk menghasilkan epoksi busa memori bentuk. Kaedah ini sangat mesra alam kerana ia bebas daripada pelarut. Pertama sekali, campuran epoksi, poliamida, natrium bikarbonat dan pengisi telah disediakan dengan menggunakan pengacau. Kemudian, campuran diemulsikan dengan air suling menggunakan pengacau yang berkelajuan tinggi (1200 rpm – 2 minit), diikuti oleh penambahan larutan ammonium klorida ke dalam campuran tersebut dan dikacau pada kelajuan 600 rpm selama 2 minit. Akhirnya, emulsi epoksi dituang kedalam acuan PP dan dimatangkan. Kegunaan poliamida-epoksi adduct sebagai nisbah terbalik daripada poliamida dan epoksi telah diterokai bagi menghasilkan epoksi yang fleksibel. Oleh kerana poliamida diperolehi daripada dimer asid lemak minyak tinggi (TOFAS), poliamida-epoksi adduct mampu untuk beremulsi pada pengacauan yang berkelajuan tinggi tanpa menggunakan pengemulsi. Ia akan menghasilkan emulsi epoksi sama dengan sistem air / minyak / air (A /M / A). Di mana, air bekerja sebagai fasa berterusan di dalam sistem emulsi dan juga dibentangkan sebagai fasa tersebar di dalam resin epoksi. Tambahan, kulit telur telah digunakan sebagai pengisi eko-hijau, yang terdiri daripada 95 % berat Kalsium Karbonat ($CaCO_3$). Proses pembuihan berlaku melalui dua mekanisma: penguraian ejen peniup dan pengewapan air yang tersebar pada matrik epoksi. Mekanisma pertama menghasilkan buih-liang yang besar dan saling berhubung manakala yang kedua menghasilkan liang yang kecil pada dinding sel. Oleh itu, busa epoksi akan menghasilkan liang yang banyak. Ciri-ciri ini ditunjukkan pada span melalui ujian set mampatan yang cemerlang (0 %) dan juga ciri-ciri memori bentuknya. Didapati bahawa semakin bertambah nisbah epoksi:poliamida daripada 1:3 ke 1:2 akan menghasilkan liang yang banyak dan kekuatan mampatan terlentang yang lebih baik (disebabkan oleh mampatan sambung silang yang tinggi). Walaubagaimanapun busa epoksi dengan nisbah epoksi:poliamida 1:3 menghasilkan memori bentuk yang lebih baik berbanding 1:2. Kesan daripada kandungan agen peniup yang menunjukkan bahawa penambahan agen peniup daripada 5 ke 15 bsr menghasilkan liang yang banyak dan saiz liang span yang tinggi serta ketumpatan yang rendah. Busa epoksi dengan 15 bsr natrium bikarbonat menunjukkan memori bentuk yang terbaik tetapi rendah ketahanan haba, sedangkan 10 bsr adalah kandungan yang optimum bagi menghasilkan set mampatan yang 0 %. Fokus kepada kesan pengisi kulit telur, penambahan kulit telur meningkatkan emulsi epoksi, mengurangkan flokulasi dan menyimpan lebih gas agen peniup untuk menghasilkan liang busa yang banyak. Kulit telur menurunkan ciri-ciri set mampatan tetapi meningkatkan ciri-ciri memori bentuk secara ketara untuk semua komposit busa epoksi.

Production and Characterization of Shape Memory Epoxy Foam by an Advanced Aqueous Method

ABSTRACT

Shape memory foam is consider as an advantageous invention that has being gained researcher's focus recently. The ability of shape memory foam is it will deform under certain mechanical forces and slowly recover back into its original shape as if it memories. This research suggested an advanced aqueous method to produce epoxy shape memory foam. This method is environmentally friendly because it is free from solvent. First of all, mixture of epoxy, polyamide, sodium bicarbonate and filler was prepared by using over-head stirrer. Next, the mixture was emulsified with distilled water using high stirring speed (1200 rpm – 2 minutes), following by addition of ammonium chloride solution into the mixture and stirred at 600 rpm for 2 minutes. Finally, the epoxy emulsion was casted into PP mould and cured. The uses of polyamide-epoxy adduct as the reversed ratio of polyamide and epoxy was explored to produce flexible epoxy. Due to polyamide derive from dimerize tall oil fatty acids (TOFAS), the polyamide-epoxy adduct was able to be emulsified at high speed stirring and without using emulsifier. It resulted in an epoxy emulsion similar to water/oil/water (W/O/W) system. Which, water worked as the continuous phase in emulsion system and also presented as the dispersed phase in epoxy resin. In addition, eggshell was used as an eco-green filler, which consisted of 95% by weight of calcium carbonate ($CaCO_3$). Aqueous emulsion foaming process occurred via two mechanisms: decomposition of blow agents and vaporization of dispersed water in epoxy matrix. The first mechanism produced large foam-pores and inter-connection while the second produced small pores on cell wall. Therefore the epoxy foam obtained possessed high porosity. These advantageous features in foam structure exhibited the excellent compression set (of 0 %) and well-shaped memory property. It was found that increase epoxy: polyamide ratio from 1:3 to 1:2 produced more porosity and also better flatwise compression strength (due to the higher crosslink density). However epoxy foam with epoxy: polyamide ratio of 1:3 had better shape memory to that of 1:2. The effect of blowing agent content showed that increase in blowing agents from 5 to 15 phr produced higher porosity and the foam pore size as well as lower foam density. Epoxy foam with 15phr sodium bicarbonate expressed the best shape memory but low thermal resistance, while 10 phr was also the optimum content in order to produce foam with 0% compression set. Focusing on the effect of eggshell filler, addition of eggshell enhanced the epoxy emulsion, reduced flocculation and kept more blowing agent gases to produce higher porosity foam. Eggshell reduced the compression set property but enhanced significantly shape memory property for all epoxy composite foams.

CHAPTER 1

INTRODUCTION

1.1 Introduction

In the last 50 years, porous material received a lot of attention by competing with long established solid materials (Hao, Kai, Huan & Ming, 2008). Until now, there were a lot of porous materials created and produced based on different in matrix, different in porous structure, and application. For example of porous materials which being produced were metallic foam and polymeric foam. In application of light weight, low density, and energy absorption, polymeric foam could be more potential than metallic foam. Polymeric foam can be divided into elastomer foam, thermoset foam and thermoplastic foam. The most common thermoplastic foams are polystyrene foam, polyvinyl chloride foam and other thermoplastic based foam, while thermoset foams are polyurethane foam and epoxy foam. Polymeric foams have many applications such as absorbing the energy during impact events, lightweight structures, aerospace, thermal insulation products and shape memory foam. Recently, shape memory polymer and shape memory polymeric foam (SMP) are considered as new materials and have drawn intensive scientific attentions as functional materials with ample potentials applications (Kim, 2008).

Versatile synthesis methods have been developed for the preparation of SMP (Chung, Chang, Pei & Ju, 2010). SMP, with the advantages of low density, good thermal and electric insulation, and high specific surface, has been found wide applications in membranes, filters, chromatography media, and solid supports. Those with pore size in the order of micrometers are also of interest for applications in catalysis, sensors, size- and shape-selective separation media, adsorbents, and scaffolds for bone and tissue in medical applications (Safinia, Mantalaris & Bismarck, 2006), (Nakanishi, Amatani, Yano & Kodaira, 2007).

Recently, the concept of shape memory is same with the self-healing of structural damage and has been a tremendous interest in the scientific community. The ability to heal wounds is one of the truly remarkable properties of biological systems. A significant challenges faced by the materials science community is to design smart synthetic systems which can mimic this behaviour by not only sensing the presence of a wound or defect, but also by actively re-establishing the continuity and integrity of the damaged area. Such self-healing materials would significantly extend the lifetime and utility of a vast array of manufactured structures. Because of the widespread use of thermoset polymers in structural applications, self-healing of damage in thermosetting polymers has been a research focus for years, especially epoxy resin (Li & Nettles, 2010).

Epoxy is well-known as a brittle polymer due to its three-dimensional crosslink, which is obtained with the curing reaction between epoxy and hardeners. Optimum crosslink density will be obtained when epoxy and hardener are used in stoichiometric ratio based on epoxy equivalent weight and functional group equivalent weight.

Furthermore, the three dimensional crosslinking could be varied by changing the ratio of epoxy and hardener. Excess of epoxy or hardener content will result in the adduct system, which possesses low 3D crosslinking and exhibits different properties. One of the common adduct system is polyamide-epoxy adduct, which is a product of the reversed ratio of epoxy-polyamide ratio using excess stoichiometric polyamide content. Due to low polymer chain between crosslink, polyamide-epoxy adduct exhibits as a flexible three-dimension crosslink polymer.

Ahead of screening a considerable amount of porous polymer-related literatures, six approaches can be concluded or summarized to prepare porous materials. They are of chemically and/or thermally induced phase separation (Schugens, Maquet, Grandfils, Jerome & Teyssie, 1996), fiber bonding (Mooney et al., 1996), solvent evaporation, gas foaming (Xuan, Chen, Zhongjie & Hangquan, 2008) freeze drying, electro-spinning and so on. Design of porous polymeric materials is continuously developing for such applications and entails controlling the permeability by tailoring the pore size, structure, and interface chemistry.

Double emulsion can be considered as one the most suitable method to prepare porous materials because it has the emulsion systems that can double the porosity. It can be divided into two types which are water in oil in water (W/O/W) and oil in water in oil (O/W/O) (Brodin, Kavaliunas & Frank, 1978). Thermoset porous materials can be produced from preparing its double emulsion, which is water dispersing in thermoset resin dispersing in water environment (Water/Thermoset Resin/Water: W/O/W). Further heating will induce curing and solidifying of thermoset resin and water's evaporation so that the thermoset porous are formed. Normally, foaming mechanism could be enhanced by using blowing agent. Selection of blowing agents is based on their decomposition mechanism, solubility, safety and cost. One of the common blowing agents is sodium bicarbonate, which is cheap and edible.

Najib, Ariff, Manan, Bakar & Sipaut (2009) have used sodium bicarbonate as a blowing agent. They found that the controlling of the gas phases within the polymeric cell walls provides excellent properties for applications that involve impact. This is due to the fact that foam has excellent energy-absorbing characteristics as compared to solid polymeric materials. The decrease in relative density also played a role by increasing the number of cells per unit volume. As the blowing agent concentration increased, the number of cells per unit volume also increased. An increase in the blowing agent concentration resulted in smaller, finer, and more uniform cells. The decomposition of high concentrations of carbon dioxide gas occurs simultaneously for a given time. Thus, more cells formed at that same time. Consequently, the number of cells per unit volume increased, resulting in a smaller average cell size in the foam (Najib et al., 2009).

In production of polymer's product, filling or reinforcement of the polymer is one of the most important methods in order to enhance the properties of the materials. It possesses the necessary mechanical and physical properties for any given practical applications. The additions of filler give fast and cheap methods to modify the properties of the base materials. The addition of inorganic filler to polymer has received considerable attention lately (Fu, Feng, Lauke & Mai, 2008). Inorganic fillers are added to thermoset and thermoplastic resins because of economic reasons and to modify certain properties such as mechanical, electrical and thermal properties (Gonzalez, Albano, Ichazo & Berenice, 2002).

1.2 Problem Statement

Most of thermoset foams are stiff and rigid due to the three dimension crosslink networks so there are some limit of thermoset foam in high impact and flexible application. Thermoplastic foams are the most suitable candidate for that. However, thermoplastic foams have poor chemical resistance and poor compression set, which means the foam will collapse after certain usage time. To surmount these problems, flexible and tough thermoset foam is investigated, which is expected to exhibit better properties via the light three dimension crosslink networks.

The foaming process of thermoset foam involves both mechanisms such of curing and foaming mechanism. Both mechanisms are common exothermic reaction. Therefore, it is difficult to control the process. Optimum condition is necessary to produce good foam morphology and achieve desirable properties of thermoset foam. Foaming processes for epoxy resins are very complex and expensive, and chemical and processing details of the materials are generally proprietary (Quadrini, Santo & Squeo, 2012). In order to control foaming process, aqueous method is introduced to prepare the polyamide-epoxy adduct emulsion so that the foaming and curing are carried out mildly. Further practice to reduce cost is to use cheap and green materials such as sodium bicarbonate and eggshell powder as fillers.

1.3 Objectives

The research objectives are as below:

i. To investigate the effect of epoxy-polyamide ratio on the foam morphology, mechanical strength, memory shape properties and thermal properties of shape memory epoxy foam.

ii. To study the effect of blowing agent (sodium bicarbonate) content on foaming behavior as well as on foam morphology, mechanical strength, memory shape properties and thermal properties.

iii. To study the effect of eggshell filler's content on the foam morphology, mechanical strength, memory shape properties and thermal properties of shape memory epoxy foam.

1.4 Scope of study

In this study, production and characterization of shape memory epoxy foam by an aqueous emulsion foaming method was done. Three ratios of the epoxy : polyamide content such of 1:3, 1:2.5 and 1:2 were studied to modify the crosslink density of the epoxy matrix hence changed the foam characteristic and properties. Blowing agent content was varied from 5, 10, 15 and 20 phr in order to observe the effect on the different morphology of the samples. Eggshell filler of 10, 20 and 30 phr was used to produce shape memory epoxy composite foams. Foam morphology was observed using optical light microscope and scanning electron microscope. The foam mechanical properties were investigated using compression and compression set. Thermal decomposition of the foam was done by using Thermogravimetry Analysis (TGA). Shape memory properties were carried out by in-house developed testing procedure, in which, the foam specimen was folded into four times and the time of folded foam recovering from its original shape was recorded and reported.

CHAPTER 2

LITERATURE REVIEW

2.1 Polymer Foam

Polymer foam or plastic foam also called as cellular foam. They are referred to an expanded or sponged plastic (Arthur, 1995). The macroscopic constitutive behaviour of polymer foams is determined by a subtle interaction of the essential constitutive behaviour of the polymeric material and the complex microstructure. There are two types of polymer foams which are open cell flexible foam and close cell structure. The macroscopic constitutive behaviour will partially be determined by the intrinsic constitutive behaviour of the polymeric material of which the foam is made. There are a lot of models in literature that relate material properties of the polymer foam to the polymeric material of which the cell walls of the foam are made (Nigel, 2007), (Lorna & Michael, 2001).

The other contribution that partially determines the material behaviour of foams is the complex microstructure. Besides that, there are a lot of external conditions that can influence the material behaviour of the foam, like temperature and pressure. As a result, the strain rate and flow of air through cells will affect the macroscopic constitutive behaviour of the foams. In expectation, for open-cell foams the influence of air flow will be higher than for closed cell foams, because the air in foams with an open structure can be forced to flow out of the foam. For larger length scales the air will pass