FOAMING AND CURING AN AQUEOUS EPOXY FOAM USING MICROWAVE

ERNY RAUDHOH BINTI MOHD SHAFIE

SCHOOL OF MATERIALS ENGINEERING
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Foaming and Curing an Aqueous Epoxy Foam Using Microwave

by

Erny Raudhoh Bt Mohd Shafie
(1431621183)

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DECLARATION OF THESIS

Author's full name : ERNY RAUDHOH BT MOHD SHAFIE

Date of birth : 09 MAC 1986

Title : FOAMING ANG CURING AN AQUEOUS EPOXY FOAM USING MICROWAVE.

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LIST OF ABBREVIATIONS

W/O	Water-in-oil
W/O/W	Water-in-oil-water
DGEBA	Diglycidyl ethers of bisphenol A
Phr	Part per hundred resin
ASTM	American Standard for Testing and Materials
Min	Minutes
H	Hours
OM	Optical Microscope
SEM	Scanning Electron Microscope
DSC	Differential Scanning Calorimetry
DTG	Derivative thermogravimetry
TGA	Thermogravimetry Analysis
P	Microwave energy
W	Water content
AC	Ammonium Carbonate
CBA	Chemical Blowing Agent

LIST OF SYMBOLS

%	Percentage
°C	Degree Celcius
mW	Heat Flow
wt	Weight
cm	Centimetre
mm	Milimetre
g/mol	Molecular weight
g/cm^3	Gram per centimetre cube
$(NH_4)_2CO_3$	Ammonium Carbonate
Pa.s	Pascal-second
Ti	Initial thickness of epoxy emulsion
Tf	Final thickness epoxy foam

Pembusaan dan Pematangan Busa Epoksi Akuas Menggunakan Gelombang Mikro

ABSTRAK

Penyelidikan ini melaporkan kesan-kesan tenaga gelombang mikro, kandungan air, dan kepekatan agen pembusaan terhadap morfologi, sifat-sifat mekanikal dan termal bagi busa epoksi akuas berasaskan poliamida-epoksi aduk. Sampel-sampel variasi tenaga gelombang mikro disediakan pada had tenaga 160 hingga 320 watts dan sampel-sampel variasi kandungan air diformulasikan dengan menggunakan kandungan air 25 hingga 200 phr. Seterusnya, sampel-sampel untuk variasi kepekatan agen pembusaan dihasilkan menggunaan 0 hingga 4 phr ammonia karbonat sebagai agen pembusaan. Air ditambah sebagai pelarut mesra alam dan berperanan sebagai pembentuk lompang. Proses pencampuran semua bahan dilakukan berdasarkan resepi yang ditentukan menggunakan pengaduk berkepala atas IKA pada kelajuan 300 hingga 1200 ppm. Proses pembusaan and pematangan dilaksanakan menggunakan ketuhar gelombang mikro. Sampel-sampel busa telah melalui pasca-matang selama 4 jam pada suhu ketuhar 70°C. Morfologi menggunakan mikroskop optik (OM), mikroskop pengimbas elektron (SEM) menunjukkan penggunaan tenaga yang tinggi dan kepekatan agen pembusaan yang banyak akan menghasilkan pembusaan yang baik dan wujud lebih interaksi antara sel. Penambahan kandungan air melebihi 100 phr, campuran berakuas berubah daripada sistem W/O (air di dalam cecair epoksi) kepada sistem (W/O/W) (air di dalam cecair epoksi di dalam air). Ini memberi kesan kepada pengembangan yang rendah dan meningkatkan masa penjelan bagi sampel W100 dan W200. Ciri-ciri dan sifat-sifat busa dijumpai daripada refleksi morfologi busa tersebut. Secara teori, keliangan yang banyak akan mengurangkan tegasan mampatan tetapi sedikit peningkatan terhadap rintangan termal pada suhu penguraian di bawah 450°C kerana berlaku pemindahan haba yang kurang di antara spesimen busa. Ujian set mampatan pada suhu bilik menunjukkan pemulihan bentuk yang baik iaitu 100%, yang mana berkurang kepada 95-68% bergantung pada formulasi busa-busa jika dimampatkan pada suhu ketuhar 70°C. Penyelidikan ini membuktikan proses akuas adalah mesra alam, selamat dan tiada sebatian organic meruap (VOCs). Selain daripada itu, menggunakan kepekatan ejen peniupan yang rendah untuk menghasilkan busa epoksi dengan prestasi yang baik.

Foaming and Curing an Aqueous Epoxy Foam Using Microwave

ABSTRACT

This study reports the effects of microwave energy, water content, and blowing agent concentration to the morphology, mechanical and thermal properties of aqueous epoxy foam based on polyamide-epoxy adduct. Samples of microwave energy were prepared at 160 to 320 watts of different microwave energy level and the samples of variation water content were formulated by using 25 to 200 phr of water content. Furthermore, the samples for variation of blowing agent concentration were produced using 0 to 4 phr of ammonium carbonate as blowing agent. Water was added as a green solvent and played as the void template. Mixing process of all ingredients was carried out based on their determined recipes by using an IKA overhead stirrer at speed 300 to 1200 rpm. Foaming and curing process were implemented using microwave oven. The foam samples were undergone 4 h post-cure at 70°C oven temperature. Morphology using Optical microscope (OM), Scanning electron microscope (SEM) showed that higher microwave energy and higher blowing agent concentration were better foaming and exist more cell interconnection. Increase of water content more than 100 phr, the aqueous mixture was changed from W/O system (water in epoxy resin) to W/O/W system (water in epoxy resin in water). This affects lower expansion and increase the gel time of sample W100 and W200. The foam characteristic and properties was found to be well reflecting the foams morphology. Theoretically, higher porosity caused lower compression stress but slight improved the thermal resistance at degradation temperature below 450^{o}C due to lower heat transfer within the foam specimens. Compression set at room temperature exhibited an excellent recovery of 100%, which reduced to 95 – 68 % depending on foams formula if compressed at 70^{o}C oven temperature. The study proves that the aqueous process is green, safe and no volatile organic compounds (VOCs). Besides of that, uses low concentration of blowing agent to produce epoxy foams with good performances.

CHAPTER 1

INTRODUCTION

1.1 Historical Background

Thermosetting foam is defined as the polymer material possessing porous structure in a thermosetting matrix. The crosslink structure in thermosetting foam enhances the thermal resistance of the foam. The foam does not melt and turns to char by heating. Polyurethane is one of the most common thermosetting foam, which produced from polyol and isocyanate. The reaction between the two chemicals produces polyurethane and carbon dioxide as a by-product, which foams the polyurethane. In the other case, thermosetting foams could be prepared by the simultaneous occurrence of two mechanisms such of polymer formation and gas generation. Figure 1.1 shows the principle of preparation of thermosetting plastic foams (Ashida & Iwasaki, 1980).

The both mechanisms need to be optimum in order to produce good thermosetting foams. It believes that polymerization of the thermosetting monomers could be occurred first (but not fully), then gas generation could be happened so that the foaming process can be stable. Fully crosslink will turn the thermosetting polymer into solid so the expansion is hard to occur, leading high internal pressure and causing the foam crack. However, too early gas generation will induce the foams having big voids and collapsed.

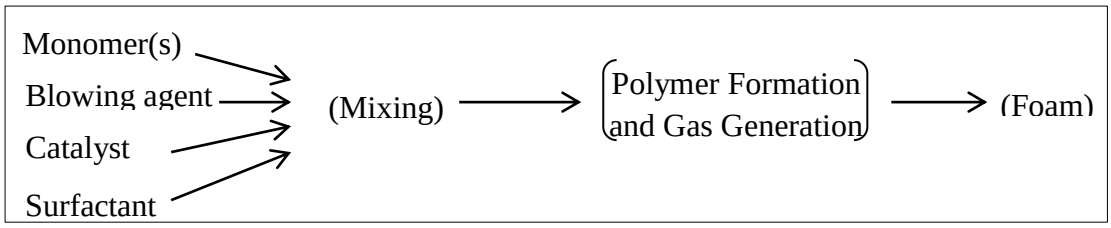

Figure 1.1 : Mechanism of thermosetting foam preparation (Ashida & Iwasaki, 1980).

The idea to change the crosslink density as well as the length of the crosslink to produce a flexible thermoset matrix is exploited. This matrix could be expandable under the gas generation, hence the foam is obtained. Polyamide-epoxy adduct is a suitable representative polymer for this case. Using invert ratio of epoxy and polyamide hardener, the polymerization occurs faster gel time and very low crosslink density.

Beside the use of blowing agent as the chemical foaming process, physical foaming process is usually used in foam engineering. Natural latex foam is the most suitable example for this process. For thermosetting, the use of water in epoxy could produce an emulsion system. If water content is less than epoxy resin, then single emulsion of water in oil (W/O) is produced. If water is used more than epoxy resin volume, double emulsion of water in oil in water (W/O/W) is produced. In this system, water can play as the void template to produce epoxy foams.

This research is to produce an aqueous epoxy foam using microwave with optimal composition of polyamide-epoxy adduct, water content and blowing agent loading as well as the microwave energy. The ratio of epoxy polyamide are 1:3, water content from 25 phr to 200 phr, blowing agent concentration from 0 phr to 4 phr and the range of microwave energy to be apply for curing and foaming around 20% to 40% of 800 watts.

1.2 Problem Statement

Time-consuming is one of the problems for conventional thermal method. Consequently, it contributes to the expensive cost of production to complete the project. Arisen of the alternatives for curing method has been studied such as microwave, UV rays, gamma rays, and electron beam (Bogdal et al., 2003). For UV light curing method, it has limited application because of limited dose rate and poor penetration as the thick material cannot cure completely (Wiesbrock & Hoogenboom, 2004). However, applying gamma rays as curing method is not safe to execute since it can generate radiation hazard as well as the environmental issues (Sinwell & Ritter, 2005). The electron beam curing requires high capital cost for initial setup although it has tough efficient and fast curing (Sinwell & Ritter, 2006). So, the most recommended alternative for curing is microwave heating since it more economically and consumes the energy efficiently (Soane & Martynenko, 1989).

Some other foaming processes have to use solvent to produce foam. So, the idea using aqueous method as medium to form epoxy foams is a green and pioneer. Foaming mechanism depends significantly on the blowing agent, matrix ratio, and blowing conditions. Furthermore, the use of ammonium carbonate via this advanced aqueous method as blowing agents is safe. The foaming and curing of thermoset foam using microwave energy could promise some advantages as faster gelling time and complete foaming. The process could generate better morphology and foam properties.

1.3 Objectives

The main objectives for the epoxy foam preparation by using aqueous methods are written as below:

i. To investigate the effects of curing and foaming of epoxy foam using different microwave energy on optimum gel time & expansion ratio.

ii. To study the effect of different water content to epoxy foam on the expansion ratio.

iii. To study the effect of different blowing agent loading on the morphology, mechanical properties and thermal stability.

1.4 Scope of Study

The scope of this study including the purpose of the experimental and testing to be carried out is to determine the properties of the epoxy foam. The investigation of the curing and foaming process of epoxy foam using different microwave energy is carried out. The range of microwave energy to be used is 20, 30 and 40% of 800 watts. After that, the study of the effect of water content in the formation of epoxy foam which has 25, 50, 100 and 200 phr. Furthermore, the effect of different blowing agent loading is investigated. The loading of blowing agent to be used are 0, 1, 2, 3 and 4 phr. The Ammonium Carbonate, $(NH_4)_2CO_3$ was chosen as blowing agent to implement this research. Finally, Pcynometer Density, Optical Microscope, Scanning Electron Microscope, Universal Testing Machine, Compression set, Thermogravitmetric Anaysis and Differential Scanning Calorimeter were used to determine the gel time, expansion ratio, morphology, physical, mechanical and thermal properties of epoxy foam.

CHAPTER 2

LITERATURE REVIEW

2.1 Foam and Foam Formation

Generally, the plastic foams refers to expanded sponge consists at least a minimum of two phases, a gaseous phase and a solid-polymer matrix develop from blowing agent. Inorganic, organic or organometallic can be the phase content of solid-polymer. The chemical composition, degree of crystallinity and degree of crosslinking contribute to the foam characteristic either shows flexibility, rigid or semi-flexibility. Moreover, the open or closed cell, size and shape give effect to the foam properties. In addition, the best application for open cells is acoustical insulation while for thermal insulation application suited to closed cell foams (Okoroafor & Frisch, 1995).

There are two optional fundamentals method in preparing the foam. The first method involves gas such as nitrogen or air is dispersed in a continuous liquid phase to produce a colloidal system with the gas as the dispersed phase. For the second fundamental methods, the emerged of separate bubbles dispersed in the liquids phase is generated by gas. Thermal decomposition of blowing agent is another technique to generate a gas in the liquid phase. The released gas can be nitrogen or carbon dioxide or both. The factor that affects the bubbles stability is temperature, as increasing the temperature the surface tension and viscosity will reduce. Consequently, promote cell rupture and the cell membrane turn into thinning (Frisch & Saunders, 1972).

2.2 Foaming and Curing Process

Curing and foaming is the process to change the polymer into valuable product provided with good properties. The properties can be tailored according to the required applications. In general, the curing process can be explained as chemical reaction with the presents of heat, radiation exposure (light, UV, electron beam and gamma radiation), also due to exposure to the external chemical such as moisture. However, the foaming process is used to produce lightweight foams by applying several methods with the existent of blowing agent or gas injection. In addition, the structural, syntactic as well as soft and rigid polyurethane foams are made by foaming process with different method and blowing agent (Biron, 2013). According to the report by Wang et al. (2014) at high pre-curing extent and used low foaming temperature, may resulting to produce small cell size of foam (Wang et al., 2014). In fact of that, when used high pre-curing extent with high foaming temperature, this condition promote to a homogeneous distribution of cells.

The existing of varies method for curing and foaming being an advantages to the industrial developer. The demand for accelerated curing process has arisen due to increasing application in microelectronic and aerospace industries. Besides, electron beams, photo curing and curing with γ-rays are an alternative (Fink, 2013). Instead of using oven as conventional method, the microwave can be the optional if requiring short time curing period. Supported by Rangari et al. (2010), the research discovered that the mixture of epoxy resin system where as EPON-862 (Diglycidyle Ether of Bisphenol-F) and Epikure W (Aromatic Amine based curing agent) with the presence of carbon nanofibers (CNFs) was undergo curing process using microwave only 10 min compared to 8 h by conventional oven heating (Rangari et al., 2010). Curing using microwave has

several advantages if compared to conventional thermal processing. Decreasing in time is one of the advantages, because the time is necessary for manufacturing industry. Moreover, the power can be directed to the sample is another advantage. The material can absorb the microwave energy rather than relying on the convection and conduction thermal. Thus, this process required less energy compared to thermal curing. The products cured with microwave energy exhibit lower glass transition temperature if compared to thermal methods (Boey et al., 2001). In other case, when curing using microwave techniques the interfacial shear strength in those composites are comparable with being thermally post cured (Day et al., 1998). However, the curing agent used strongly affects the curing performance (Boey et al., 1999).

2.3 Double Emulsion Technique

The foam made by single emulsion or double emulsion technique will presents different characteristic as well as the properties. According to the McClements et al. (2007) a mixture based on at least two immiscible liquids with the small droplets found inside other droplets (McClement et al., 2007). The droplets can be oil and water or other substances. The single emulsion can be described into two conditions, whereas (O/W) system is oil-in-water emulsion and (W/O) system is water-in-oil. Furthermore, the double emulsion can be categorized into two conditions as well. (W/O/W) emulsions indicate that water-in-oil-in-water and (O/W/O) emulsions refer to system oil-in-water-in-oil (Van Der Graaf et al., 2005). Refer Figure 2.1 for better understanding the system.

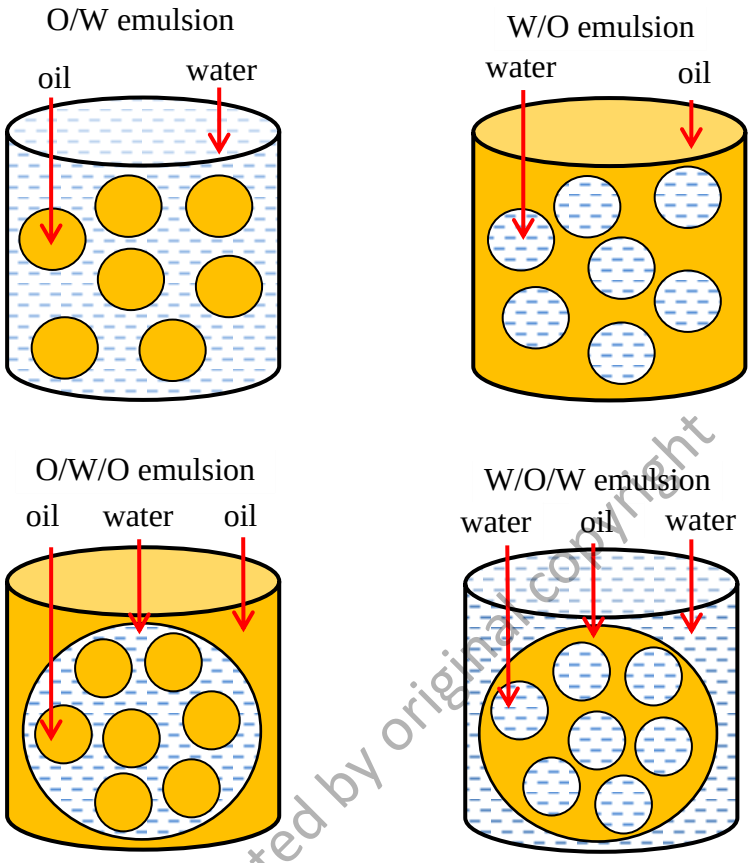

Figure 2.1: Two types of emulsions (Vladisavljevic & Williams, 2005)

2.4 Additives for Epoxy Foam

2.4.1 Ammonium Carbonate as Blowing Agent

A particular agent can cause plastic to foam called blowing agents. The following are two type common reaction of blowing agent:

i The gases were introduced into liquid plastic material or molten.

ii The decomposition of gases from chemical substance occurred in the matrix resin at given temperature (Schwartz, 1995).

Besides, the gas dispersed well and expands to form the cells in the plastics. Generally, the formation of cellular structure depends on the loading or type of blowing

agent, particular process being used as well as the type of plastic resin. In addition, another factor should be considered depend on application of end product such as K-factor and R-factor. K-factor refers to thermal conductivity and R-factor indicates the resistance of the material to the transmission of heat. Consequently, the foams having lower K-factors show superior thermal-insulating properties. Then, higher R-factor shows better insulating properties of plastics foams (Schwartz, 1995). There are seven different blowing methods that generally used to fabricate the plastic foams (Schwartz, 1995). :

i Decomposition of gas at elevated temperature caused by incorporating chemical blowing agent (CBA) into polymer.

ii In injection molding machine, when the nitrogen gas injected into resin and pressure is decreased cause the gas to expand and produce cellular structure.

iii Formation of cells caused by releasing of gas from bifunctional material such as isocyanate combined with polyester.

iv Applied heat externally or by exothermic reaction can cause volatilization of a low-boiling liquid. Chlorofluorocarbons (CFCs) was common liquids used to produce rigid polyurethane foams.

v The foamed latex rubber made by whipping air into a colloidal-resin suspension and then gelling the porous mass.

vi Produce plastic cups, packaging and mannequin heads by expanding the small polystyrene beads of thermoplastic resin. Heat up an internally controlled blowing agent such as pentane to implement the expanding.

vii The incorporating of nonchemical such as gas liberating agent into the resin mix cause the mixed to release a gas after heating.