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Physical Properties of Epoxy Foam Prepared Using Natural Rubber As Toughening Agent

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***Abstract*— Sodium bicarbonate was used as a chemical foaming agent to fabricate a variety of pores epoxy foams toughened by natural rubber (NR), and the appropriate formulation was found to ensure good physical properties. In this study, we added NR as a toughening agent at contents ranging from (5 to 25 vol.%) to examine the properties of epoxy foam. 15 phr of SB was added as the optimum content foaming agent. The characteristics of epoxy foam's viscosity, density, porosity, shape, and pore size were determined. Through pores within the foam, cellular polymers minimize polymer weight and mechanical strength loss. By increasing the expansion, adding more NR to the material reduced the crosslinking reaction and lowered its mechanical strength. For epoxy-foam containing 10 to 15 vol% NR, a satisfactory expansion and pore size were noted. By adjusting the NR concentration from 5 to 25 vol%, foam densities can be adjusted in a range up to 0.0.787 g/cm3. There was a reduction in porosity between 6.48 to 31.98 %.**

Keywords—epoxy, foam, natural rubber, sodium bicarbonate, toughening agent

I. INTRODUCTION

Since their introduction in the 1930s, epoxy resins have been the thermosetting polymers with the broadest application. Epoxy resins contain a variety of intrinsic properties because of the highly reactive epoxy groups in the terminal chains [1]. The most widely used epoxy resin is a thermoset polymer with enhanced characteristics, including less cure shrinkage, good electrical and chemical insulating characteristics, good adhesion and environmental resistance [2]. Due to their brittleness, lack of impact resistance, and lower processing fracture energy than industrial thermoplastics and metals, epoxy resins have a limited range of industrial applications [3]. Because of its inherent fragility, epoxy composites have poor damage tolerance, poor shear strength, and adhesive peeling [4]. It was originally shown that the fracture toughness of epoxy can be increased by adding a dispersed rubber phase to glassy, highly cross-linked epoxy resins, which are widely recognized as brittle thermosets and need to be toughened for a number of applications. Due to the increased benefit of using renewable raw materials for the investment of current high plastic systems, NR is a material of choice of identified rubbers [5]. NR is made up of long polymeric chains with a high degree of flexibility and mobility that are linked together to form a network structure [6]. Natural rubber has a number of distinct mechanical, chemical, and physical properties [7]. One technique for enhancing epoxy toughness involves adding a rubber phase to the epoxy as an additional content [8]. The modulus and strength properties have been enhanced by taking into account and studying the concentration, disruption of rubber in the epoxy system, rubber particle size, curing, and interaction between epoxy and rubber. The earliest publications on rubber as a plastic toughening agent were written about PS toughening stages, according to [9]. In order to increase the toughness of the brittle matrix, rubber particles have been widely studied as a second phase to the epoxy resin [10]. According to the general description, there is a chemical interaction between the NR and the epoxy matrix. It has been established that these chemical interactions are quite minimal. In other words, the rubber modified epoxy has a low modulus and strength because there is relatively little interfacial interaction between the rubbery phase and the matrix. It is well known that interfacial adhesion plays a key role in enhancing desirable mechanical properties in a multi-phase system [11]. Using hydroxyl functionality, Mathew [10], investigated how to modify epoxy using liquid natural rubber (HLNR). The results show that as rubber volume increased, the number, volume, area, and weight average zone diameters of HLNR also increased. The critical size of rubber particle that 6 μ was shown to have to highest toughness with a rubber composition of 15 wt % [10]. Modification can be done by dispersing a little amount of rubber as a detachable phase [4], [12]. Epoxy has fracture energies that are orders of magnitude lower than those of thermoplastics and other strong polymers in their cured condition, making it a brittle polymer [13]. Epoxies should be toughened to improve their properties such as fracture toughness in order to preserve their status as the polymer of choice for various applications such as adhesives and composite matrixes [14]. It has been proposed that shear banding, crazing, and elastic partial deformation can all increase epoxy toughness. These techniques can be applied singly or in combination to produce a toughening influence [14]. Natural rubber was used by Kumar and Kothandaraman to toughen the DGEBA kind of epoxy [13]. By grafting maleic anhydride onto depolymerized NR, it was created together. Various concentrations of epoxy were added to it, with the epoxy resins portion taking precedence over the NR component. When NR was added to epoxy, the (Tg) didn't change much, but the blends' mechanical properties, such flexural and elongation, improved [13]. The fracture and mechanical features of mixes with submicron powdered and liquid rubber were investigated and assessed by Tang et al. in 2014. Although liquid rubber is more thermally resistant than powdered rubber, there are some processing and toughening performance limitations [15]. Epoxy foams are low-density materials that are favored for usage in shock, thermal, and vibration applications because of their high specific characteristics (property divided by density). For the transportation sector, isolation of electronic components and production of lightweight materials [16]. Stefani created a closed-cell epoxy foam using a chemical foaming agent to test the thermal conductivity of composites made of EP and carbon black with varied carbon contents. They found that as foaming agents' rises, the size of the cells increases while density, compression strength, and modulus all fall [17]. In unsaturated samples, Lye showed that supercritical CO_2 predominates as a foaming agent, and that with increasing CO_2 concentration, cell coalescence causes the average cell diameter to increase and the cell density to decrease [18]. The role of acetic acid as a promoter in increasing the decomposition of $NaHCO_3$ in epoxy foam was effectively examined. To make rigid epoxy foam, a mechanical mixing approach was used. According to the findings, adding acetic acid made the solution viscous as the amount of $NaHCO_3$ rose. Although adding acid to epoxy decreased density, increasing porosities decreased the dielectric constant, cross-link density, and mechanical density [19]. Chang and colleagues used the new foaming agent to test the morphology, porosity, mechanical property, thermal conductivity, and adhesive property of foams with different polysilazane contents. The development of high-strength, low-density epoxy foams was successful [20]. Epoxy foams with a higher percentage of $NaHCO_3$ have larger cells and more porosity, according to Fauzi [21]. Processing and final physical properties are affected by the degree of chemical degradation and cure (crosslinking). The density might alter depending on how much foaming agent is employed. To reach its maximum strength, the foam needs to be postcured for a long time [21].

II. EXPERIMENTAL

A. Materials

Euro Chemo-Pharma Sdn Bhd provided clear epoxy (DER 331), a liquid reaction product of epichlorohydrin and bisphenol A. The crystal clear epoxy hardener (A062) used in this study was provided by Euro Chemo-Pharma Sdn Bhd, and the ratio of epoxy to hardener was set at 100:60. Sodium bicarbonate was utilized as a foaming agent. Kasihku Marketing Sdn Bhd in Penang, Malaysia, provided it. HmbG Chemical provided the hydrogen peroxide that was used as a sensitizer in the photo-depolymerization of LNR with a ketone-end group. Sigma Aldrich Sdn Bhd provided the acetic acid, which has the chemical formula CH_3COOH, that was employed as the acidic medium in the photo-depolymerization. Nnatural rubber (NR) was supplied by the Malaysian company Zarm Scientific & Supplies Sdn. Bhd.

B. Sample Preperation

Photo-Chemical Depolymerization of NR Long polymer chains (high molecular weight) can break apart into shorter polymer chains during depolymerization of NR, a process that is well-known as photo-degradation (low molecular weight). According to Nair [22], the photo-chemical depolymerization of NR was performed in this study outside in the sunlight. In a 1000 ml glass beaker with a mechanical stirrer at a speed of 400 rpm at 50 ^oC for two hours, a specific volume of NR was dissolved by adding the proper amount of toluene as a solvent (25 g of NR equivalent 166 ml of toluene). The NR solution was then treated with hydrogen peroxide (30 weight percent H_2O_2) and 2 phr acetic acid. For an hour, the milky liquid natural rubber solution was swirled. For 100 hours, the depolymerization reaction occurs at room temperature in the sunlight. After 100 hours, the milky NR solution has turned a pale yellow color.

To prepare of epoxy and NR samples, Prior to that, NR was first completely dissolved in epoxy resins using the magnetic stirring method at 60 ^oC and 200 rpm, this could take some time. Based on the findings of a previous study, the optimum sodium bicarbonate loading, 15 phr, was chosen as the loading that would result in the highest possible porosity content. As a toughening agent, natural rubber in various volumes (5, 10, 15, 20, and 25 vol.%) was utilized. A magnetic stirrer was used to combine the epoxy (95 vol.%) and NR (5 vol.%) in a total of 250 ml of solution until the rubber was completely dissolved. The mixture was given a fixed amount of 15 phr sodium bicarbonate and stirred for 10 minutes to achieve perfect homogeneity. After another 10 minutes of vigorous stirring, hardener (with an epoxy/hardener ratio of 100:60) was added. A portion of the mixture was then used to measure the viscosity using a viscometer with various rotor speeds of 1, 2, 5, 10, 20, 50, and 100 rpm. While adding the remaining half, acetic acid was added and well mixed at 400 rpm. The mixture was then put into a mold and cured at 100°C for an hour to cure it. The finished product was then removed from the mold after curing. The experiment was repeated with respective amounts of 10, 15, 20, and 25 vol.% of depolymerized natural rubber.

III. CHARACTREIZATIONS

Using a Viscometer, the viscosity of each uncured sample was determined (Model: Brookfield DV-II, spindle size 6). Using a Gas Pycnometer (Model: Micrometrics AccuPyc 1330) and helium medium, the density of the polymer samples was determined in accordance with ASTM D1895, and the porosity percentage was then computed using the resulting densities. Using optical microscopy, pore characterizations, including pore sizes, were measured. Image software was used to analyze the optical microscopy images in order to calculate the average pore size.

IV. RESULTS AND DISSCUSIONS

A. Viscosity

Although it is generally known that the sequence of epoxy hardening involves complex viscosity fluctuations, linear macromolecule growth takes precedence at the beginning of the process [23]. Epoxy resin curing and $NaHCO_3$ decomposition must be precisely balanced in order for epoxy foams to be effectively prepared. Bubbling won't be achievable if the epoxy matrix is dried first since the polymer matrix would lose its flexibility. If bubbling occurs first without a subsequent cure, bubbles congeal and even collapse [24]. Fig. 1 illustrates the epoxy foam viscosity as a function of NR content and rotor speed. The viscosity often increases slightly as NR content increases. The fraction of toughened rubber rose, leading to an increase in mixture viscosity, when NR in amounts ranging from 5 to 25 vol.% was completely mixed with NaHCO3. The viscosity of the resin is essential for effective foaming, and curing can speed up the gelification process, creating a significant processing problem. Literature indicates that the gel point is when the best viscosity ranges for producing a suitable pore size are obtained [25], [26]. The ideal viscosity ranges for obtaining an acceptable pore size are obtained at the gel point. By increasing the NR content, viscosity generally tends to increase slightly. When NR was extensively included into the mixture with $NaHCO_3$ at various contents ranging from 5 to 25 vol.%, the proportion of toughened rubber increased, increasing the mixture's viscosity. A controlled pore size ensures a consistent foam quality and great mechanical properties [27]. At a fixed 100 rpm rotor speed and 10 vol.% NR content as opposed to 5 vol.% NR, Fig. 1 demonstrates a 22% increase in viscosity. However, the findings showed that viscosity rose in the presence of NR, indicating that epoxy foam crosslinking was to cause the increase in viscosity. The rise is assumed to be caused by the NR molecule being activated by the presence of $NaHCO_3$. According to earlier study, introducing NR to the plastic system increased the melt blending's viscosity [10]. The sample containing 15 vol.% NR had the greatest pore achieved, with a maximum of 15% NR, as pore size values increased with the rise of NR due to reaction delays. It's interesting to note that the size of the pore values significantly decreased despite the addition of 20 vol.% NR to the reaction.

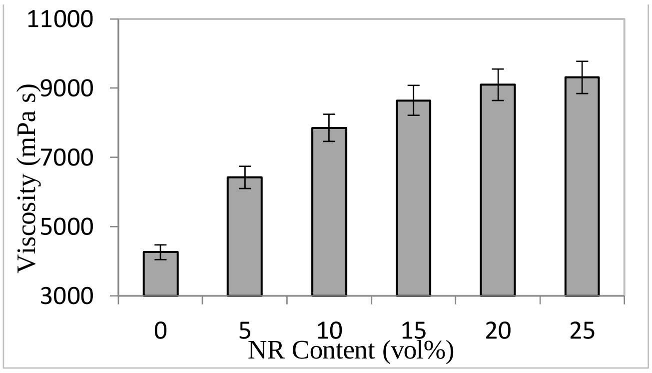

Fig. 1: The viscosity of epoxy foam with 100 rotor speed and NR content

B. Density

Fig. 2 illustrates how the NR content affects the density of an epoxy/NR foam system. Reduced density, which is related to the level of voids created in the polymer matrix, is a top priority in foaming operations. In this study, PDA was used to determine the density of the epoxy foam; the densities of neat epoxy and NR were 1.157 and 0.945 g/cm^3, respectively. To examine the effect of NR content on the density of the epoxy foam, various NR and $NaHCO_3$ amounts were added to epoxy samples. These samples had NR contents that ranged from 5 to 25 vol.% in 5 vol.% increments, whereas the NaHCO3 contents remained constant at 15 phr. The density value of 5 vol.% NR stands out among the others as having the highest value. More NR was added, and the density decreased. This is because the density of the epoxy/NR foam blend was decreased by the addition of NR because raw NR has a lower density than pure epoxy (1.157 g/cm3). If the initial NaHCO3 level was lower, foam density would be expected to be lower since the NaHCO3 eventually decomposes out of the foam. Despite the fact that this is the case, a higher NR content may cause the composition to continue to exist in a rubberier phase, which would cause more expansion and, thus, a lower foam density (SEM micrographs). Hemmasi et al. (2011) [28] suggest that the amount of void created in the polymer matrix controls density reduction. The figures 4.20, 4.21, 4.23, and 4.25 show that it has been found that the density of foams significantly affects their mechanical properties, with higher density foams having superior mechanical properties. The higher viscosity of the mixture as a result of the higher NR content appears to have a conflicting effect (as demonstrated by the difficulty of hand mixing). The tiny NR phases have hindered and impeded the reaction between the reactive epoxide groups of the epoxy matrix and the reactive groups of the hardener. Jianing Zhang and his colleagues claim that, especially in cured epoxies with low cross-linking density, rubber phase separation is not necessary to get the best rubber toughening effect [29](Jianing Zhang et al., 2016). When a result, as the density of the foam system decreases, so does the density of the foam cross-links. When the rubber component reached 10%, Thomas et al. (2007) [30]claimed that this became more relevant [30], [31](R. Thomas et al., 2007; Wei et al., 2019). Depending on the density of the foam, polymer foams have different mechanical properties. In general, higher density is associated with better mechanical properties, while lower density is associated with better insulation [32](Chand & Sharma, 2012).

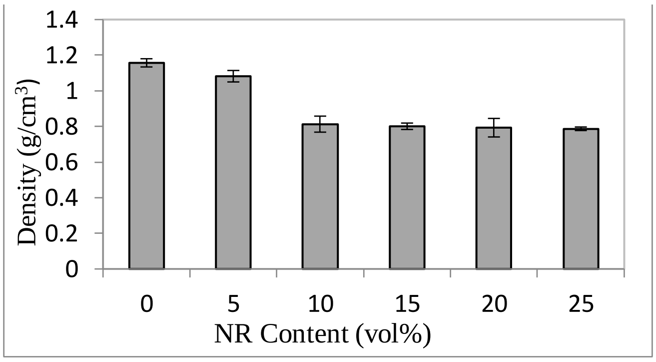

Fig. 2: Density of epoxy foam at different content of NR

The decrease was attributed to the epoxy foam's porous nature as a result of the NR's toughening effect. Epoxy foam loses mechanical characteristics as its porosity decreases with increasing density.

C. Porosity

The effect of NR loading on the porosity of an epoxy/NR foam is depicted in Fig. 3. Epoxy foam density and porosity can be varied from 1.157 g/cm^3 and 7% to 0.787 g/cm^3 and 32%, respectively, by changing the concentration of NR from 5 to 25vol.% The porosity properties of epoxy/NR foam have generally been unaffected by the addition of NR. Porosity is the exact opposite of density since it is the amount of empty space per unit volume as opposed to density, which is the quantity of material per unit volume. Pores in the material are necessary for particular applications, and this affects the material's mechanical properties [33]. When $NaHCO_3$ is added, the possibility of rubber developing porosity and phase separation rises up to a point where a sufficiently high NR content allows the foam to separate from the epoxy matrix, which increases porosity. Findings show that the sample with 10 vol.% loading of NR had the lowest porosity with good dispersion due to the crosslinking that occurred between epoxy and NR [12]. In other words, the porosity rose along with the increase in NR content, eventually plateauing at roughly 30% above a 10 vol.% loading. This is because the rubber particles in epoxy foam are smaller. As a result, the porosity of the porous epoxy reduced while the density grew, increasing the porous epoxy's overall ability to absorb stress during fracture and decreasing the carrying capacity of the materials.

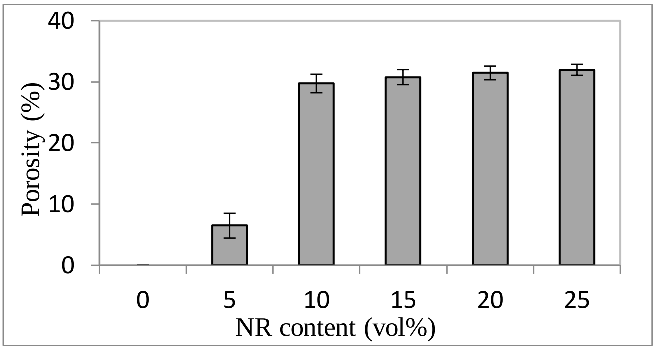

Fig. 3 : Porosity of epoxy foam at different content of NR

D. Average Pore Size

Fig. 4 shows that NR content had a considerable impact on cell structure. At a constant $NaHCO_3$ content, the impact of NR content on pore size was investigated (15 phr). Additionally, the average pore size was established. The results showed that it rose as the NR content increased. Two things account for this. First, when the NR content of the matrix rises, so does the matrix's melt viscosity, which leads to a greater barrier to cell expansion during foaming. Second, dispersing NR throughout the epoxy matrix to improve the cell morphology of epoxy foams.

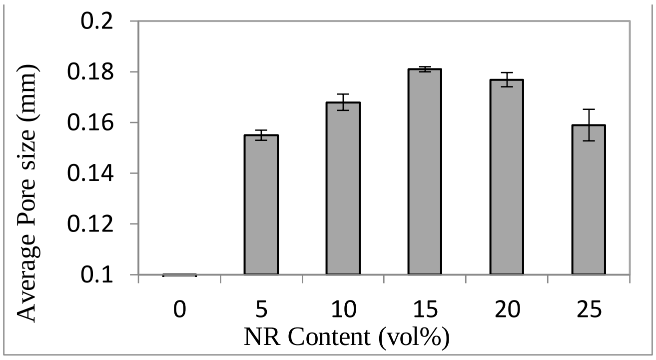

Fig..4: The average pore size of epoxy foam at different content of NR

The pore size values were improved by using NR as a toughening agent, with a maximum NR content of 15% obtained. The reaction was 20 vol.% NR slower, but the average pore size decreased significantly. The amount of gases needed to produce foam remains constant with constant $NaHCO_3$ loadings. A heterogeneous method leads to smaller sizes by increasing the number of nucleation sites [34]. In other words, when the NR content rose, cell growth was constrained by larger pores that ate up available gas more quickly. As a result, the pore size distribution reduced and pore size decreased. The epoxy foams with 10 vol.% and 15 vol.% of NR exhibited the biggest and smallest pore sizes, respectively, as was to be expected. The density of epoxy foam decreases to 0.813 g/cm^3 at 10 vol.%, resulting in a 30% increase in porosity (foams' physical and morphological characteristics were all affected). Fig. 4 shows the open-cell structure of epoxy foams with NR toughened agent. This is owing to the presence of an NR, which slowed the draining of bubbles during the crosslinking reaction in the epoxy system.

V. CONCLUSIONS

In this study, the impact of NR loading on the physical properties of epoxy foam was the main concern. Elastomeric materials must be added to thermoset materials to increase their properties. In this research, NR was used to toughen epoxy matrix in varying amounts (from 5 to 25vol.%). The study demonstrates that NR is a potentially significant elastomeric and toughening agent in the modification of epoxy foam. When compared to viscosity, porosity, density and pore size of epoxy/NR foam has good physical properties. The findings showed that viscosity rose in the presence of NR, indicating that epoxy foam crosslinking was the cause. The rise is assumed to be caused by the NR molecule being activated by the presence of $NaHCO_3$. In general, higher density is associated with better mechanical properties, while lower density is associated with better insulation. The decrease was attributed to the epoxy foam's porous nature as a result of the NR's toughening effect. Epoxy foam loses characteristics as its porosity decreases with increasing density. Because of this, epoxy foams with 15 and 25 vol.% NR had a sizable amount of porosity (32%). The sample containing 15% NR had the greatest pore achieved, with a maximum of 15% NR, as pore size values increased with the rise of NR due to reaction delays. It's interesting to note that the size of the pore values significantly decreased despite the addition of 20 vol % NR to the reaction.

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