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Improved Flexural Strength, Modulus, K_{IC}, and Compressive Strength of Epoxy Foam Modified with Natural Rubber

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ABSTRACT

The study developed new epoxy composites enhanced with natural rubber (NR) and sodium bicarbonate (SB) as a foaming agent. Adding NR improved the epoxy's toughness, flexural strength, modulus, and fracture toughness (K_{IC}), with optimal results at 10 vol% NR for flexural properties and 15 vol% NR for compressive strength. Increasing NR content led to greater expansion but reduced crosslinking and mechanical strength due to increased porosity. The improvement was attributed to small, uniformly dispersed NR phases that acted as energy dissipation centers, enhancing elasticity and load-bearing capacity. SEM analysis confirmed uniform dispersion of NR particles within the epoxy matrix, which significantly influenced mechanical performance.

Keywords: epoxy foam, natural rubber, depolymerized natural rubber, foaming agent, mechanical properties.

تحسين مقاومة الانحناء ومعامل المرونة ومعامل متانة الكسر (K_{IC}) ومقاومة الضغط لرغوة الإيبوكسي المعدلة بالمطاط الطبيعي

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ملخص البحث

تبحث الدراسة في تحسين مادة الإيبوكسي بإضافة المطاط الطبيعي وبيكربونات الصوديوم لتكوين رغوة خفيفة الوزن. أظهرت النتائج أن إضافة 10–15% من المطاط الطبيعي حسّنت قوة الانحناء والضغط، بينما أدت الزيادة المفرطة إلى ضعف الخصائص الميكانيكية بسبب المسامية. وأكدت فحوصات المجهر الإلكتروني أن توزيع جزيئات المطاط كان منتظمًا، مما زاد من مرونة المادة وقدرتها على تحمل الأحمال.

الكلمات الدالة: رغوة الإيبوكسي، المطاط الطبيعي، المطاط الطبيعي المتحلل والمفكك بوليمريًا، عامل الرغوة، الخواص الميكانيكية.

1. INTRODUCTION

Due to the increased benefit of using renewable raw materials for the investment of new high-volume plastic systems, natural rubber is a material of choice among identified rubbers [1]. NR latex of numerous rubber-producing trees, including Hevea brasiliensis and Parthenia argentatum, is converted into cis-1,4-polyisoprene, NR by coagulation with chemicals, drying, and electrical coagulation, among other procedures. NR is a depolymerized natural rubber (DNR) that is obtained by depolymerizing a natural rubber latex. One technique for enhancing epoxy toughness involves adding a rubber phase to the epoxy as an extra component [2]. According to Riew & Smith (1989), PS toughening steps were the focus of the first publications on rubber as a plastic toughening agent that were published in 1957 [2]. The process of polymer depolymerization depends on a reaction that uses a reagent containing reactive polar groups to open the active linkage in the polymer backbone. It has the capacity to reduce the length of polymer chains.

Epoxy resins are a significant class of industrial thermoset resins. they are typically the most often utilized resins in polymer composites that have been employed as a matrix. Epoxy resin also offers superior characteristics than some other thermoset resins, including good chemical and insulating properties, good adhesive, and environmental endurance. Low creep, excellent strength, resistance to corrosion, thermal and acoustic insulator capabilities, low cost, and effective electrical insulating are further characteristics compounds [3] , [4], [5], [6]. They are primarily employed in electronic (microencapsulation) and structural (structural adhesives) applications. The most important epoxy resin is diglycidyl ether of bisphenol (DGEBA) which it is commonly used by most researchers [7]. Epoxy resins form a highly crosslinked three-dimensional network structure after curing with the suitable hardener. However, because of its brittleness, low toughness, low impact strength, and poor resistance to crack propagation, its range of applications is limited. Due to brittleness is known to be a drawback in epoxy matrix and its use is limited by their brittleness; it can cause premature failure during application. As a result, industry and research development have shown a great deal of interest in modifying epoxy resin. By trying to deal with this matter, one of the successful approaching to toughen the epoxy resin is by introducing rubber phases into the brittle epoxy resins.

Polymer foams, which are frequently low toughness and very brittle and are used primarily as basic materials in sandwich structures, create a weak link in those systems. The micro-mechanisms within the spreading crack control the K_{IC}. It is possible to depict plastic foam as a structure rather than a uniform substance. Depending on the plastic matrix, plastic foam can also be divided into thermoset and thermoplastic foams. Foam morphology allows for additional classification of plastic foam into closed cell foams and opened cell foams arrangements. Open cell foams are made from the cell edges (struts), whereas closed cell foams have membranes that connect the cell edges. As surface tension pulls a significant portion of the solid material into the cell while the foams are being created, the membranes appear to be thin [8]. For applications involving impact, the polymeric cell walls' ability to contain the gas phase offers good features. This is because gas has a fantastic energy-absorbing function as a blowing agent.

Depolymerized natural rubber or liquid natural rubber is natural rubber that has been depolymerized [9]. Vk et al., (2016) was produced (DNR) and used as toughening agents for epoxy resins. As a result, the toughness of epoxy resin, as well as other tensile properties, increased significantly. With mixed DNR, hardness rises as well. [10]. On the other hand, brittleness or a lack of crack development resistance, low toughness, low impact resistance, and notch sensitivity prevent epoxies from being utilized more frequently. To develop toughening materials that don't affect the thermo-mechanical properties of the epoxy polymeric matrix, numerous research initiatives have been made. Epoxies must now be toughened in order to ensure their appropriateness for real-world applications [11].

A variety of toughening chemicals have been used to modify epoxy polymers in order to increase fracture toughness without sacrificing other important characteristics. Natural rubbers are used to modify epoxy resins in order to increase their toughness. A more flexible epoxy backbone can be created chemically. Based on structure-property relationships, crosslink density can also be decreased by increasing the molecular weight of the epoxy monomers and/or reducing the functionality of the curing agents. The approach that is most prevalent is the addition of distributed toughener phase(s) as a second phase into the curing epoxy matrix [12].

Phase separation results from the epoxy losing configurational entropy during cure, which is caused by an increase in molecular weight. The outcome is a shift in the free energy of mixing, which lowers the solubility of the rubber. This is the force that separates the phases of matter. As a result, the functionality of the matrix monomers, which control the development of networks and crosslink density of the epoxy matrix, has an effect on the phase-separation process [13]. A DNR can generally be produced using mechanical, chemical, photo-depolymerization, metathesis reaction, biodegradation, ultrasonic irradiation, and ozonolysis processes. Through the photo-chemical the decomposition of natural rubber utilizing hydrogen peroxide (H_2O_2) and irradiation to both medium pressure the vapors of lights and direct sunshine, Ravindran et al. (1988) examined the creation of hydroxyl telechelic natural rubber [14]. Both resources, according to their examination, were effective in the degradation and produced oligomers with hydroxyl endgroups. Isa et al. (2007) found that sodium nitrate, formic acid, and H2O2 were used to degrade NR. After a long process and exposure to higher temperatures, they revealed that degraded NR with low molecular weight was formed. The epoxide (C-O-C) and hydroxyl (-OH) groups replace the C=C bonds in the rubber molecules' chains. FTIR and H-NMR techniques can be used to investigate the products' structure [15].

2. MATERIALS AND METHODS

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) utilized in this investigation was provided by Euro Chemo-Pharma Sdn Bhd, and Zarm Scientific & Supplies Sdn supplied the NR. The ratio of epoxy to hardener was set at 100:60. Sodium bicarbonate (SB), a white powder with a micron-sized particle size, was utilized as a foaming agent. It was provided by Malaysia's Kasihku Marketing Sdn Bhd in Penang. HmbG Chemical provided the hydrogen peroxide that was used as a sensitizer in the photo-depolymerization of NR with a ketone-end group. Sigma Aldrich Sdn Bhd provided the acetic acid, which has the chemical formula CH3COOH, that was used as the acidic medium in the photo-depolymerization.

Preparation of DNR by using Photo- chemical Depolymerization method; Natural rubber depolymerization, also known as photo-degradation, is a process which promotes the chain scission of long polymer chains (high molecular weight) into short polymer chains (low molecular weight). According to Nair et al. (2007) [16], the NR photo-chemical depolymerization method used in this study was carried out in the sunlight. Due to possible changes in viscoelastic characteristics, solid natural rubber was first weighed and then masticated in a two roll mill to lower viscosity.

Finally, natural rubber was cut into dimensions of 5 x 5 mm before the depolymerization reaction. Toluene and 25 g of NR were added, and the mixture was agitated with a mechanical stirrer at a speed of 400 rpm for two hours at 50 oC. The NR solution followed by 0.35 mol of acetic acid and 30 weight percent of H_2O_2. For an hour, the milky natural rubber solution was stirred. The depolymerization process occurs in the sunlight at room temperature. After 100 hours, the milky NR solution has changed to a yellow pale color. The required quantity of methanol was added to the solution in order to coagulate, precipitate, and eliminate residues. The coagulants were then dried in an oven for 24 hours at 90 °C.

Preparation of Epoxy and NR Samples; to make the epoxy/NR foam, DNR was first dissolved in epoxy resins using magnetic stirring at 60 °C and 200 rpm. Considering the findings of previous research, the optimal SB content, 15 phr, was determined to be the best content for providing enough porosity. Different amounts of NR (5, 10, 15, 20, and 25 vol%) were used as a toughening agent. Initially, a total of 250 ml of epoxy (95 phr) and DNR (5 vol%) were mixed together until the rubber was completely dissolved in the epoxy solution. $NaHCO_3$ at 15 phr was added to the mixture once the NR had completely dissolved, and the mixture was then stirred for 10 minutes to achieve homogeneity. After adding the hardener, the mixture was rapidly agitated for a further 10 minutes. Acetic acid was added to the remaining the amount and thoroughly mixed at 400 rpm. The mixture was then put into a mold and heated at 100 °C for an hour to cure it. The finished item was then removed from the mold after curing. The experiment was repeated using 10, 15, 20, and 25 vol% NR, respectively.

3. CHARACTERIZATION

The flexural properties were measured using a three-point bending method and applied load was 50 kN, as described in ASTM D790 using an Instron machine, Model 5569, with a crosshead speed of 2 mm/ min. The rectangular samples have dimensions of 60 x 12.7 x 3 mm. The standard used in fracture tests under tensile mode followed the procedure as stated in ASTM D638 and was performed using Instron machine, Model 5569 at crosshead speed of 1 mm/min. Scanning electron microscopy (SEM) was used to study the morphology of the flexural fracture samples. It can produce an image of the surface of polymer; the samples were characterized using JEOL JSM-6460 LA. Palladium was coated to the flexural fractured surfaces of cross-sectional samples to avoid the formation of electrostatic charge. The epoxy foam composite was cut into compression testing size, compression testing was run based on the ASTM D3575 by using the Instron machine.

4. RESULTS AND DISCUSSION

4.1 Flexural Strength

Figure 1 displays the flexural strength of epoxy/NR foam as a function of NR content; the flexural strength of the foam decreased with the addition of NR. The decline was ascribed to the porous character of the epoxy foam because of the toughening induced by the NR. Epoxy foam's flexural strength decreases as its porosity increases because it loses density. It displayed a somewhat higher flexural strength value with a 10% NR content. Consequently, epoxy foams with 15 and 20 vol% NR showed medium porosity but decreased flexural strength when compared to epoxy foams with 5 and 10 vol% NR. Flexural strength increased when less than 10 vol% NR was applied.

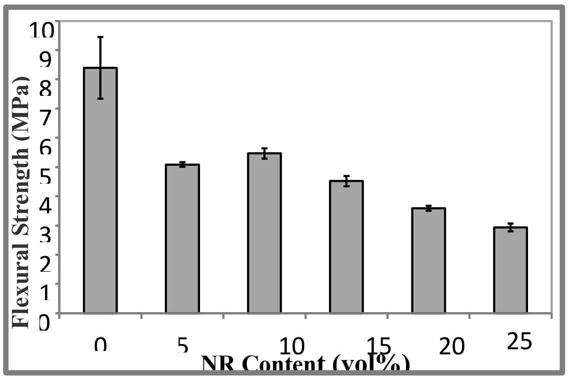


Fig 1. Effect of NR contents on the flexural strength of epoxy/NR foam.

The crucial NR threshold caused the flexural strength of the epoxy foam to decrease when NR level exceeded 15 vol%. The maximum flexural strength of the foam was 55 MPa at 10% NR addition, compared to 50 MPa at 5 vol% NR. The increase, which was about 10%, was mostly due to the toughening that was continued by the NR addition of the polymer matrix, which successfully enhanced capacity for energy absorption. Following the NR addition, rubber domains were formed to increase the composite's flexibility and toughness. The mechanical properties of the foam are influenced by the amount and distribution of the rubber phase. Because more rubber particles are close to one another and can interact over the critical content, the flexural strength is reduced. The NR phases' capability to act as energy dissipation centres in the epoxy significantly enhanced the load transfer from the epoxy matrix to the NR phases. Similar results were found by Thomas and colleagues et al. (2008), who found that the phases of rubber particle sizes have significant effects on the epoxy/NR properties [17].

Results for the epoxy/NR foam's flexural modulus are shown in Figure 2. As the NR used in this study had a rubber reactive group, it was mixed with pure epoxy to see how the flexural properties were impacted. Rubber is less dense than epoxy, hence adding NR causes the flexural modulus of epoxy foam to decline. The flexural modulus of the epoxy foam at 10 vol% NR was 1351 MPa, while it was 866 MPa at 5 vol% NR and 3000 MPa for plain epoxy. All of the load generated when stress is applied to the epoxy foam is carried by the small NR phases inside the epoxy system. About 56% of the improvement at 10 vol% NR was due to epoxy foam. In this case, the addition of NR produced a strong interface that raised the flexural modulus of the foam at low concentrations. If NR mixes with epoxy while being mixed, the reaction's path might change.

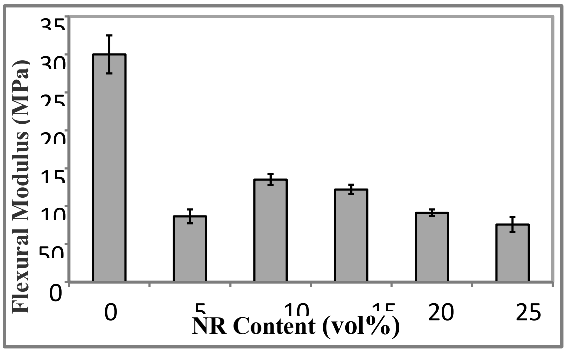


Fig 2. Effect of NR contents on the flexural modulus of epoxy/NR foam.

In an epoxy/foam system, stresses are concentrated on both the pore surface and the rubber particles, while in an epoxy matrix, stress is localised only on the brittle epoxy network. The contact between rubber particles and pores might lessen the toughening effect of rubber particles because pores cannot transfer energy. Therefore, it is necessary to keep rubber particles away from pores and other rubber particles. According to Back et al. (2018), this indicates the critical amount of rubber particles in epoxy/foam was lower than in epoxy matrix[18]. As the NR percentage rose, the flexural modulus decreased and peaked at 10% NR content as opposed to 0% NR content. A decrease in the flexural modulus at high NR loading (25 vol%) indicates that the epoxy foam has grown more ductile with bigger rubber segments, disrupting the cross link density of the epoxy network. Furthermore, the rubbery particles reduce the flexural modulus of the epoxy foam by acting as stress concentrators.

The SEM of a flexural fractured epoxy/NR surface is shown in Figure 3. The flexural fractured foam surfaces in Figure 3 (a), (b), and (c) displayed two distinct phases; the epoxy structure included a continuous epoxy blend and rubber phases; and the NR phase sizes inside the epoxy system were thinner and more evenly distributed. This matrix morphology demonstrates that the epoxy foam has NR phase separation at 5 vol% and 20 vol% of NR. Figure 3 (b) shows that a little of NR phases in the epoxy system at 10 vol% of NR content. At low NR content, the NR phases functioned as an energy dissipation centre, absorbing and releasing the applied load into the surrounding matrix. Effective toughening is frequently promoted by lower NR content as opposed to greater NR levels [19]. As seen in Figure 3 (b), the outcome at 10% NR's surface was rougher and had more fracture lines, indicating that it took more effort to break the sample and that its flexural strength was higher. Epoxy toughened by NR at 10 vol% provides advantages compared to other epoxy types, as shown in Figure 3 (a) and (c) with 5 vol% and 20 vol% of NR content. The lowest flexural strength was caused by more brittle failure with fewer fracture lines at 20 vol% NR. The increased surface area of the epoxy matrix interacts with the surrounding epoxy matrix as a result of the decreased NR element content [2], [20] .

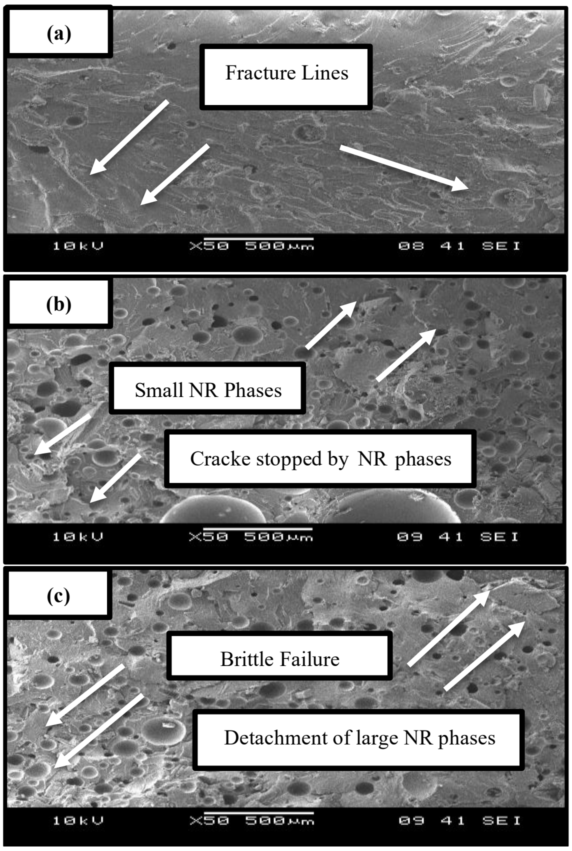


Fig 3. SEM micrographs of the flexural fractured surface for (a) 5vol %, (b) 10 vol %, (c) 20 vol % NR.

Significantly, the blends of epoxy foam with NR 10 vol% had the highest flexural modulus (1351 MPa), whereas epoxy foam with NR 15 vol% had a higher flexural modulus than epoxy foam with NR 5vol%. Therefore, adding NR 5 to 15 vol% could enhance porosity by 29.7% while maintaining flexural properties. Interestingly, the epoxy matrix, which supports the entire stress applied to the materials, has a sizable contact surface area inside tiny NR [21]. While stress would be concentrated on both the cell surface and the rubber particles in an epoxy/rubber foam system, it is possible to concentrate load just on the rubber particles in an epoxy/rubber system. Because pores cannot transmit energy, interaction between them and rubber particles may reduce the toughening effect of the rubber particles. Therefore, it is necessary to keep rubber particles away from pores and other rubber particles. This indicates that compared to epoxy/NR foam, the crucial rubber volume in epoxy/NR foam was smaller.

4.2 Fracture Toughness (K_{IC})

The fracture toughness of epoxy/NR foam is shown in Figure 4 as a function of NR content; All of the load generated when stress is applied to the epoxy foam is carried by the small NR phases inside the epoxy system. About 56% of the improvement at 10 vol% NR was due to epoxy foam. In this case, the addition of NR produced a strong interface that raised the flexural modulus of the foam at low concentrations. If NR mixes with epoxy while being mixed, the reaction's path might change. The few NR phases avoided the fractures from expanding and allowed a large deformation of the epoxy matrix. The maximum K_{IC} value actually appeared at 15 vol% NR content due to an enhancement in the interaction between the epoxy and NR phases, according to the facts. It's interesting to note that the rubber particles seem to be spread more evenly throughout the epoxy in the case of 15 vol% modified epoxy. The K_{IC} did not increase further as the rubber content was increased, but the excess NR content caused some cavitated rubber particles to agglomerate throughout the epoxy and the cavitated rubber particles to increase.

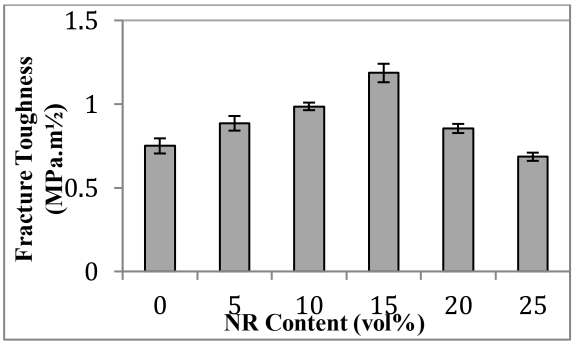


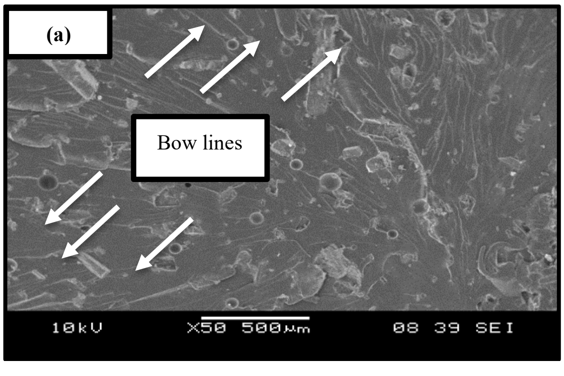
Fig 4. Effect of NR contents on the fracture toughness of epoxy/NR foam.

Depolymerized NR's active epoxide group could interact with the epoxy ring and improve interphase adhesion, leading to a noticeable increase. According to Seng's report, rubber acted as a plasticizer or flexibilizer when it was added to the epoxy matrix [22]. The flexibility of the secondary NR phase was impacted by dissolved NR in the epoxy matrix, whereas the toughening process was influenced by NR particles' ability to experience cavitation in the epoxy matrix [23]. The polymer matrix's ability to deform under shear will be enhanced by both of these processes [17].

The energy saved in the NR phases served increase toughness during stretching. Small NR phases generated considerable plastic in the epoxy matrix to deform while preventing the formation of cracks. Conversely, NR increased the K_{IC} value of the epoxy foam system. This occurs as a result of the epoxy matrix and the small NR particles developing a strong bond. However, matrix softening as a result of matrix flexibilization is frequently coupled with the use of rubber modifiers to improve fracture toughness. Given that the modulus of the modifier is significantly less than that of the matrix, this is not surprising. To achieve toughness properties in epoxy systems, 10 to 15 vol% of rubber are often needed [24].

SEM micrographs of epoxy containing 10, 15, and 20 vol% NR were taken in order to link the K_{IC} values with the morphological research. SEM micrographs depicting the fracture toughness surface are shown in Figure 5. The NR phase was significantly smaller and more uniform, as seen in Figure 5 (b). Cell wall bending might cause low density foam to collapse, and low K_{IC} could damage the pores. High density foams have smaller pores, which suggests that their cell walls are thicker and more robust. This reduces the matrix transfer load when the epoxy foam deforms. Consequently, the matrix properties will have a significant impact on the characteristics. Effective load transfer from the epoxy matrix to the NR phases was made possible by the small NR phases' strong interfacial interaction with the surrounding epoxy matrix. The blend morphology illustrates the immiscibility of NR and epoxy, and these morphologies support the fracture results displayed in Figure 5. (b) Toughening is frequently promoted more at 15 vol% NR amount than at other levels.

A rich continuous phase along the epoxy matrix phase and a higher K_{IC} were both evident in the result obtained with 15 vol % NR. It is clear that epoxy toughened with NR at 10 vol% causes a crack, signifying the formation of a fracture surface and the expansion of the crack length as a result of the shape change. While the sample contains some bow lines, which use more energy, as compared to those with 15 vol% and 20 vol% NR in Figure 5 (a) and (b), energy is needed to generate new fractures and nonlinear crack fronts, which improved fracture toughness, as shown in Figure 5 (a) and (b).



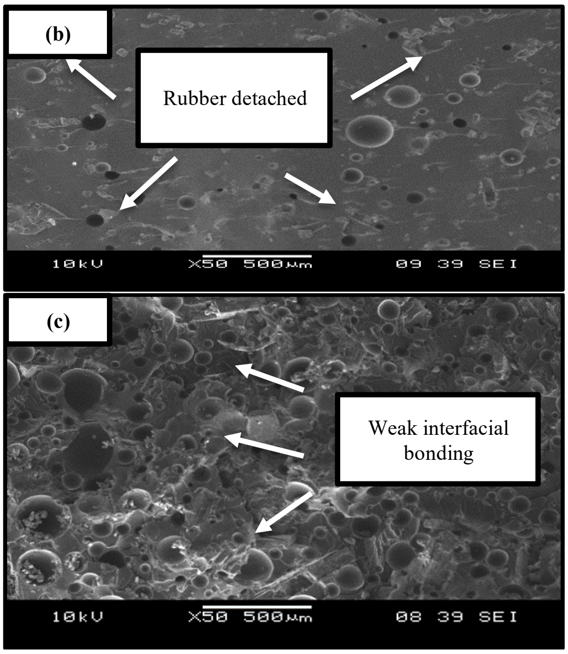


Fig 5. SEM micrographs of the fracture surface for (a) 10 vol %, (b) 15 vol % and (c) 20 vol %, of NR toughened epoxy.

4.3 Compressive Strength

Figure 6 shows how NR contents affect the compressive strength property of epoxy foam. The general reduction in compression strength was caused by the addition of 5 to 25vol% NR. In other words, the compression strength decreased by 21.8, 24.2, and 39.5%, respectively, with the addition of 5, 10, and 15% vol.% of NR. Figures 1, 2, and 4 show how mechanical properties were accepted by the higher foam density. NaHCO3 was also demonstrated to reduce the compression strength of epoxy foams because it causes cells to grow larger as a result of larger void volumes. On the other hand, increasing the amount of NR in the epoxy matrix reduced compressive strengths while enhancing foam flexibility by showing high toughness as a result of the growth in rubbery segments. However, as can be seen in Figure 6, the compressive strength of the less rigid epoxy phase within the foam matrix was low.

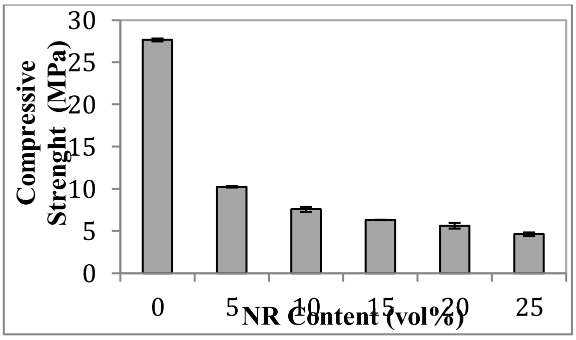


Fig 6. Effect of NR contents on the compressive strength of epoxy/NR foam.

As shown by the little increase in size of the foam with a medium ratio of NR to epoxy resin, the growth of bubbles in softer matrix was easily generated around the rubber particles due to a drop in epoxy fraction. NR content, which rose from 5 to 25 vol% as the ratio increased, was larger than that of the low ratio. The pores in the epoxy foam with a 15% NR content expanded more easily than those in the epoxy foam with a high epoxy content, which was the cause of this.

Lower foam density was the outcome of the increase in pore size. Foam with a 15% NR percentage was found to have a somewhat lower density than foam with a 5% NR content. The cell walls and struts of epoxy foam have the greatest impact on its mechanical characteristics. In 25 vol% NR, either the cell struts or the cell walls were weak due to an increase in pore size, which led to a decrease in compressive strengths [25].

5. CONCLUSIONS

Elastomeric materials must be added to thermoset materials to enhance their mechanical properties. The incorporation of NR has successfully induced cellular materials. In this research, NR was used to toughen epoxy matrix in varying amounts (from 5 to 25vol.%), and the impacts of NR on the properties of the epoxy/NR foam were compared. The findings demonstrate that NR is an effective, promising, and efficient elastomer filler and toughening agent for modifying epoxy foam systems.

Epoxy/NR foam has good mechanical properties when compared to its density. The epoxy/NR foam with 10 vol% NR provided the greatest results, with flexural strength, modulus, K_{IC}. In terms of compressive properties, the epoxy/NR foam had the good compressive strength value than others at 15 vol% of epoxy/NR. The findings show that the NR phase, enhanced the flexural strength, modulus, and K_{IC} of the epoxy foam. The size of the rubber phase (NR) has a significant impact on the mechanical properties of epoxy foam. The NR phase size was more constant and smaller which is attributed to the plastication and flexibilization effect of natural rubber in the epoxy matrix. The tough and elastic NR phases worked as an efficient energy dissipation centre, preventing the growth of cracks. In the meantime, the growth of significant interfacial bonds between the epoxy matrix and tiny NR phases is crucial for the filled system's high strength and modulus. SEM investigations showed that, in contrast to high content, the NR phases' particle sizes were uniformly smaller.

6. ACKNOWLEDGMENT

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