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CHEMICAL AND MORPHOLOGICAL EFFECTS OF POLYURETHANE AND LIQUID SILICONE RUBBER ON EPOXY COMPOSITES

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ABSTRACT

This study aimed to improve the morphological properties of Sikadur 52 epoxy resin by incorporating polyurethane (PU) and liquid silicone rubber (LSR) as toughening agents. The modifiers were added at various weight percentages (3, 6, 9, and 12%) and cured with a hardener. SEM analysis revealed microphase separation within the epoxy matrix, with distinct PU and LSR domains. FTIR analysis verified that addition of LSR strengthened some chemical bonds in the epoxy matrix, and SEM analysis provided direct information about phase separation behaviors of the modified epoxies. This micro-phase separation is believed to contribute to the enhanced mechanical properties of the modified epoxy resins.

KEYWORDS

Epoxy, FTIR, SEM, liquid silicon rubber, polyether polyol.

1. INTRODUCTION

Epoxies are a thermoset polymer that has found widespread application as a structural material, coating, and adhesive in diverse fields, including the aerospace, automotive, and electronics industries, on account of their high mechanical properties, high heat resistance, and solvent resistance. Their unique qualities result from the chemical bonds that make up their cross-linked structure. However, the cross-linked structure's restriction of molecular mobility also contributes to the material's low fracture toughness in comparison to other engineering thermoset polymers. Therefore, they need to be reinforced so that new uses can be realized with them (Kishi et al., 2011). Extensive studies during the last few decades have focused on the toughening of epoxy resin using functionalized rubbers, thermoplastics, and block copolymers. Adding the rubbery inclusions, severely decrease the modulus, while adding thermoplastic inclusions, slightly improves the mechanical properties (Akın, E., et al., 2021; Mathew and George, Soney C., Parameswaranpillai, Jyotishkumar , Thomas, 2014).

Toughened epoxy is commonly used in scientific studies because of its capacity to raise the finished material's strength. Epoxy resins' key advantages are their rigid shape and non-slip coating. Epoxy's toughness can be increased by mixing it with liquid rubber (Aiza Jaafar et al., 2021). Thus, epoxies typically require a second component to toughen it. Researchers used a variety of methods, such as chemical changes involving chain extenders and plasticizers, to toughen brittle epoxy-based systems. The addition of a second phase, typically liquid rubber, is the next most frequent approach. Epoxy resins (EPs) mechanical properties could be improved by a variety of means, one of the ways is the addition of reactive liquid rubbers (Parameswaranpillai et al., 2017a). It was also possible to modify such EPs by the introduction of rubbers with terminal functional groups, like HTPB (Yi et al., 2018), EHTPB (Zhou, W., et al., 2020), MPB (Muthalif and Choe, 2022) with aliphatic or aromatic linkers between the amine group and the polybutadiene chain. Besides, it was also possible to use CTBN as modifier (Januszewski et al., 2021). Siloxanes are critical toughening agents owning their flexible Si-O-Si backbone, low (Tg), very good oxidative and thermal stability, excellent weather ability, and low surface tension. However, the use of pure PDMS is of limited utility as a standalone toughening agent due to its limited compatibility with the polar hard segments of epoxy resins, mainly ascribed to a lack of hydrogen bonding (Singh, S.K., et al., 2024).

On the other hand, numerous investigations have been linked to modified epoxy resin silicone rubber in an attempt to enhance the material's flexibility, thermal stability, water resistance, and flame retardancy. silicone rubber is one of few elastomers that have been shown to increase the impact resistance of epoxy. Elastomers such as silicone rubber are notable for their low glass

transition temperature (Tg), excellent elastic characteristics, hydrophobic nature, and resistance to heat and oxidation (Bai, Y., et al., 2024). Combining epoxy resin with silicone rubber shows great promise for uses that call for high impact strength. Therefore, it is of significant interest to investigate and assess the mechanical properties of the composite material resulting from the combination of epoxy resin and silicone rubber. (Farhan and Jaffer, 2020; Kumar et al., 2021). When it comes to polymer blends and their practical uses in industry, there has been a lot of research done before. explored the effects of adding different amounts of RTV silicone rubber (0%, 5%, 10%, 15%, and 20%) to epoxy resin on its thermal stability and mechanical properties. The addition of 15% silicone rubber significantly improved impact strength and energy absorption while slightly decreasing tensile strength, elongation at break, and hardness. Additionally, the thermal stability of the epoxy was reduced with increasing silicone rubber content (Pakaya et al., 2017a). Another study investigated the impact of adding silicone rubber to epoxy and unsaturated polyester resins. The results showed that increasing silicone rubber content generally improved impact strength but negatively affected other mechanical properties like hardness, Young's modulus, and flexural strength (Jasim, Z.M. and Abdulsamad, H.J., 2025; Farhan and Jaffer, 2020). Alternatively, PU (polyol) was used to create overlapping polymer networks (IPNs) with epoxy (EP), enhancing the toughness and elasticity of the EP (Kostrzewa et al., 2019). Polyols (PU) are one of the most adaptable thermoplastics, that used in a wide variety of applications (Mondal, S., 2021). Polyol's soft segments, which are made up of linear long-chain molecules, make it a useful material in many applications (Kim et al., 2018) with an abundance of hydroxyl groups ready for chemical reaction (Kausar, 2019). The incorporation of reactive polyol (PU) and amino-functionalized multi-walled carbon nanotubes (CNTs) significantly enhances the toughness of epoxy composites, especially in the hybrid system that includes both modifiers. However, adding PU can decrease the glass transition temperature (Tg) and tensile strength, while increasing elongation at break. SEM analysis indicates potential phase separation in PU-modified epoxies, which may affect overall strength. While these modifications improve toughness, it's crucial to carefully consider the trade-offs in other mechanical properties for optimal material design (Jin et al., 2014).

This study synthesized a modified epoxy blend and examined the effect of different toughening agents (polyol and liquid silicone rubber, LSR) on its properties. Fourier Transform Infrared Spectroscopy (FTIR) was employed to characterize the structural properties of the PU/epoxy and LSR/epoxy blends. Scanning Electron Microscopy (SEM) used to analyze morphological characteristics of these blends.

2. EXPERIMENTAL WORK

2.1. Materials and Apparatus

Epoxy Sikadur 52 (TR Producted) based on bisphenol F-(epichlorohydrin) and hardener (izoforon diamine), (1-methylethyl)-1,1'-biphenyl) was purchased from Sikadur. Polyol toughening agent is the Quickmast 120 type (PU base resin), a yellowish green color, with a relative density at 25°C of 1.1-1.2 (g/cm), manufactured by DCP Saudi Co. Room temperature vulcanization silicone rubber, LSR, vulcanizes at room temperature with good self-leveling ability, was purchased from HONG YE JIE, China (Shenzhen Hong Ye Jie Technology Co., n.d.).

Fourier Transform Infrared Spectroscopy (FTIR) Test. The epoxy system samples were tested using Fourier transform infrared spectroscopy (FTIR), that provide detailed information about the chemical bonding and molecular structure of the tested materials. An FTIR instrument of the IR Affinity-1 type was utilized for this analysis (made in Japan). The spectrum of the infrared light was utilized, namely between 400 and 4000 cm-1.

Scanning electron microscope (SEM). Scanning electron microscopy (SEM) was utilized to evaluate the fracture surface of the unmodified epoxy and toughening agents/epoxy blends analyzed. An accelerating voltage of 20 kV was used in the tests. To prevent charging, the samples were covered in a gold coating before the examination. It was an SEM device used to analyze the fracture surface of epoxy/blends. It was from Seron Technology Company, South Korea Manufacturing, AIS2100 Model.

2.2. Experimental Procedure

Table 1 illustrates the preparation procedure of an unmodified epoxy blend (epoxy resin/hardener). A mechanical stirrer was used to mix the hardener and epoxy (at a ratio of 2:1) for (10 minutes). The mixture then placed in the vacuum degassing system at room temperature for 10-15 minutes to remove bubbles. Afterward, the mixture was cast into pre-prepared silicon molds and left at room temperature to cure for 24 hours, resulting unmodified epoxy samples, as shown in Fig. 1.

In order to prepare modified epoxy systems, for the two types of toughening agents (PU base and LSR) used in this study (see Table 1), a certain amount of each type of toughening agent was added in epoxy resin using different ratios of 3%,6%, 9%, and 12% wt. Toughening agents were added to epoxy with continues mixing for 10–30 minutes. After that, the hardener (with a ratio of 2:1) was added and mixed for 10 minutes. The prepared mixture is put in the degassing system under vacuum at room temperature for 10–15 minutes to remove bubbles. Afterward, the mixture was discharged into silicon molds and left at room temperature to cure for 24 hours.

This process yielded various samples of modified epoxy polymer blends. These steps are applied to each case of the two types of toughening agents. The procedure used to prepare the modifier epoxy polymer blend samples described in Fig. 2.

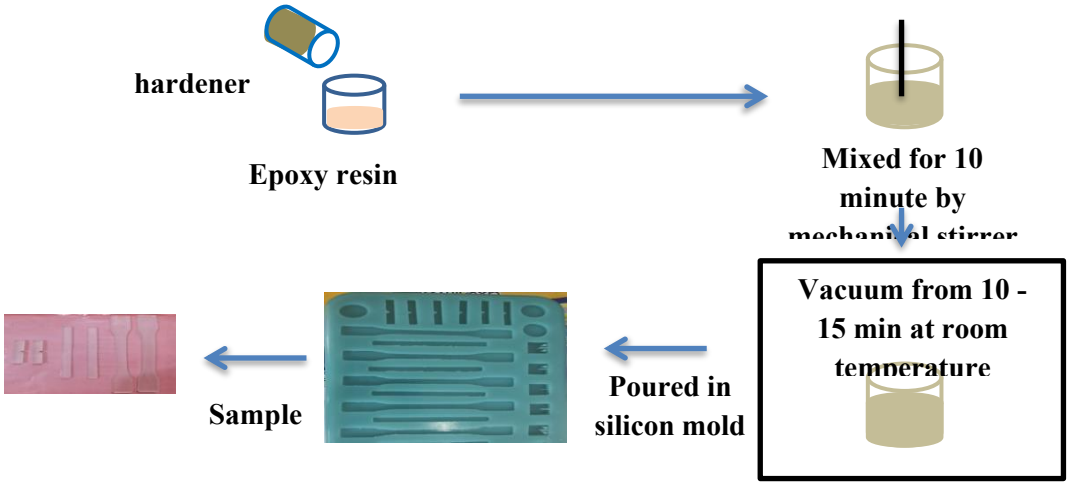

Fig. 1. The preparation procedure of unmodified epoxy samples

Table 1 Sample composition of toughening agent /epoxy blends prepared in this study

Sample	Matrix	Toughening agent	Samples
1	Unmodified epoxy	No toughening agent	EP
2	Modified epoxy	(Quick mast 120)	3% PU/EP
3	=	=	6% PU/EP
4	=	=	9% PU/EP
5	=	=	12% PU/EP
6	=	Liquid silicon	3% LSR/EP
7	=	=	6% LSR/EP
8	=	=	9% LSR/EP
9	=	=	12% LSR/EP

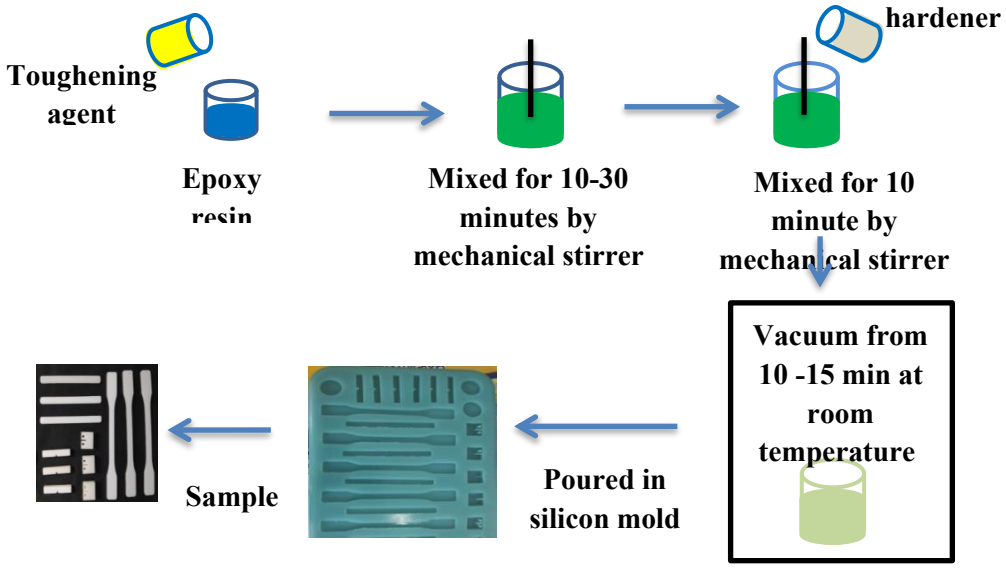

Fig. 2. The procedure used for preparing modified epoxy blends

3. RESULTS AND DESCUSSION

3.1. Results of Fourier Transform Infrared Spectroscopy (FTIR).

The structural function groups for quickmast (120) polyol were analyzed by Fourier Transform Infrared Spectroscopy (FTIR). The FTIR spectra in Fig. 3 (a) were recorded for the quickmast (120) polyol, which shows the main peaks. Peaking at 3487 cm-1 and broadening due to an increase in hydroxyl concentration, this spectrum region is associated with the vibration stretching of-OH (free hydroxyl groups) (Patel et al., 2016). Asymmetrical and symmetrical stretching vibrations of (-CH) match to (CH2) with a frequency of 2962 and 2839 cm-1 (Hassanajili and Sajedi, 2016), respectively. Additionally, the bending vibration of the symmetrical (-CH) group in (CH3) appears at the absorption peak of 1373 cm^-1. The absorption at 1273 cm^-1 is characteristic of the stretching vibrations in C-O-C (ether bonds). The absorption at 1643 cm^-1 corresponds to the stretching vibration of C=O (carbonyl group) (Wu et al., 2017). Polyol has a specific molecular structure that was defined by the presence of hydroxyl groups (-OH) and ether bonds (C-O-C) (Jia and Gu, 2020; Ajibade, O., et al., 2019), and 120 quickmast (PU base) are thought to have a polyether structure, which agrees with (Datta et al., 2017; Jin et al., 2020).

The FTIR spectra of the room temperature vulcanization silicone rubber (LSR) are shown in Fig.3 (b). The peak around 3425.58 cm-1 is related to the O-H group in (silicon rubber) and the peaks at about 2962.66 cm-1, 802.39 cm-1, and 694.37 cm-1, which indicated the (C-H) bonds, Si(CH3)2, and Si(CH3)3, respectively (Cheng, L., et al., 2022). The presence of methyl silicon is indicated by an absorption peak at 1265.30 cm-1 and three peaks around 1100-1000 cm-1 are related to silicon-oxygen bonds (Wu and Chen, 2017). Bands in the infrared spectrum with wavelengths of about 890–860 cm-1 and 1100-1000 cm-1, respectively, are indicative of the existence of Si-H bending and Si-O-Si stretching modes (Kassu et al., 2020).

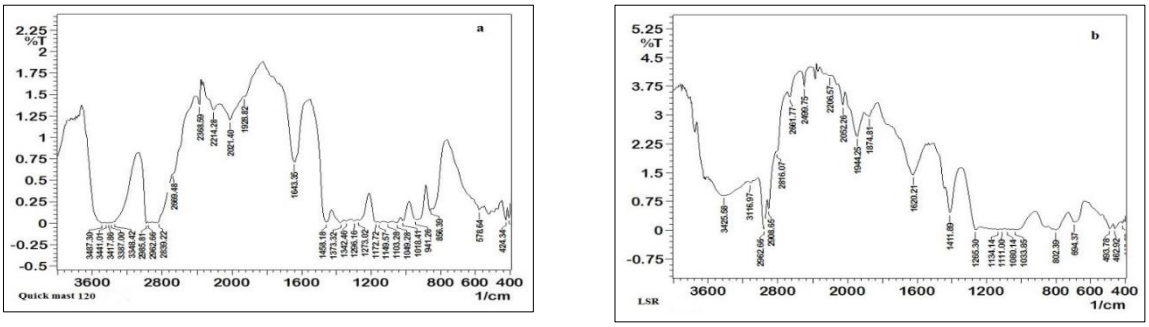

Fig.3. The FTIRs Spectrum for (a) Quick mast120 (PU base) and (b) liquid silicon rubber, toughening agents

The FTIR spectra of the unmodified epoxy (epoxy/hardener) system are shown in Fig. 5(a). Typical absorption bands, including the OH-bond absorption band at 3417.86 cm-1, are seen in

the spectra of the neat epoxy system (between 3600 and 3200 cm-1). There are many OH groups in the molecule of this resin, hence the observed peak is broad. The spectral peak at 1110 cm-1 suggests the existence of an aromatic ether group, while the band at 933.55 cm-1 (in the 950 to 860 cm-1 range) confirms the presence of the epoxy group (Balaji, N., et al., 2022).

The FTIR of unmodified epoxy and its blends are illustrating in Fig. 4. FTIR spectra shown in Fig. 4 (b) are obtained to indicate potential intramolecular hydrogen bonding interactions between the epoxy matrix and the quick mast (120) modifier. The typical peaks of epoxy (EP) functional groups may be seen to emerge around 3417.66 cm-1 to the amine and hydroxyl functional groups and roughly (933.55 cm-1) for epoxy functional groups (Kostrzewa et al., 2019). Fig. 4 (b) shows that the peak of unmodified epoxy group appears at (933.55 cm-1), which has nearly the same epoxy blends intensity. The ether bonds (C–O–C) stretching vibrations are presented at 1230 cm-1 to 1099 cm-1 (Datta et al., 2017). The FTIR spectra confirm the complete curing of the epoxy-amine system, indicated by the absence of a peak at 915 cm-1 and the presence of a peak at 3620 cm-1. This indicates that the secondary hydroxyl groups formed during curing primarily engage in intramolecular hydrogen bonding. As a result, intermolecular hydrogen bonding between the ether groups of the PU polyol and the free hydroxyl groups of the cured epoxy is impeded, causing phase separation and immiscibility in the PU/epoxy complexes (Parameswaranpillai et al., 2017b). The formation of the 3D network structure, facilitated by ether bond network during curing, can enhance the mechanical properties of the modified epoxy (Zhang W. et al., 2021).

Fig. 4 (c) depicts the FTIR spectrum of thermosetting epoxy with 3, 6, 9, and 12 wt% silicone rubber additions. The distinctive absorption peaks of LSR/EP are located at 1438, 1247, 1132, 913, and 933.55 cm-1, which may be traced back to the Si-C6H5, Si-CH3, Si-O-C, and the end-group epoxy functionalities [18–20]. When compared to blends including unmodified epoxy peaks, these peaks are essentially identical. This means some of epoxy groups interacted with functional Si-O-H, slightly, the stretching intensity of Si-O-Si and Si-O-R at their peak of 1018 cm-1 increases with a higher ratio of silicone rubber to thermoset mixture as measured in terms of weight percent. The FTIR spectrum shows an increase in the intensity of the peak at 780 cm-1, indicating a higher (C-H) group ratio from (Si-CH3) groups in the thermoset blend of epoxy/silicone rubber. Additionally, increasing intensity of the peaks at 1018 and 780 cm-1 suggests a strengthening of the Si-O-Si, Si-CH3, and Si-O-R bonds after addition of silicone rubber (Pakaya et al., 2017b).

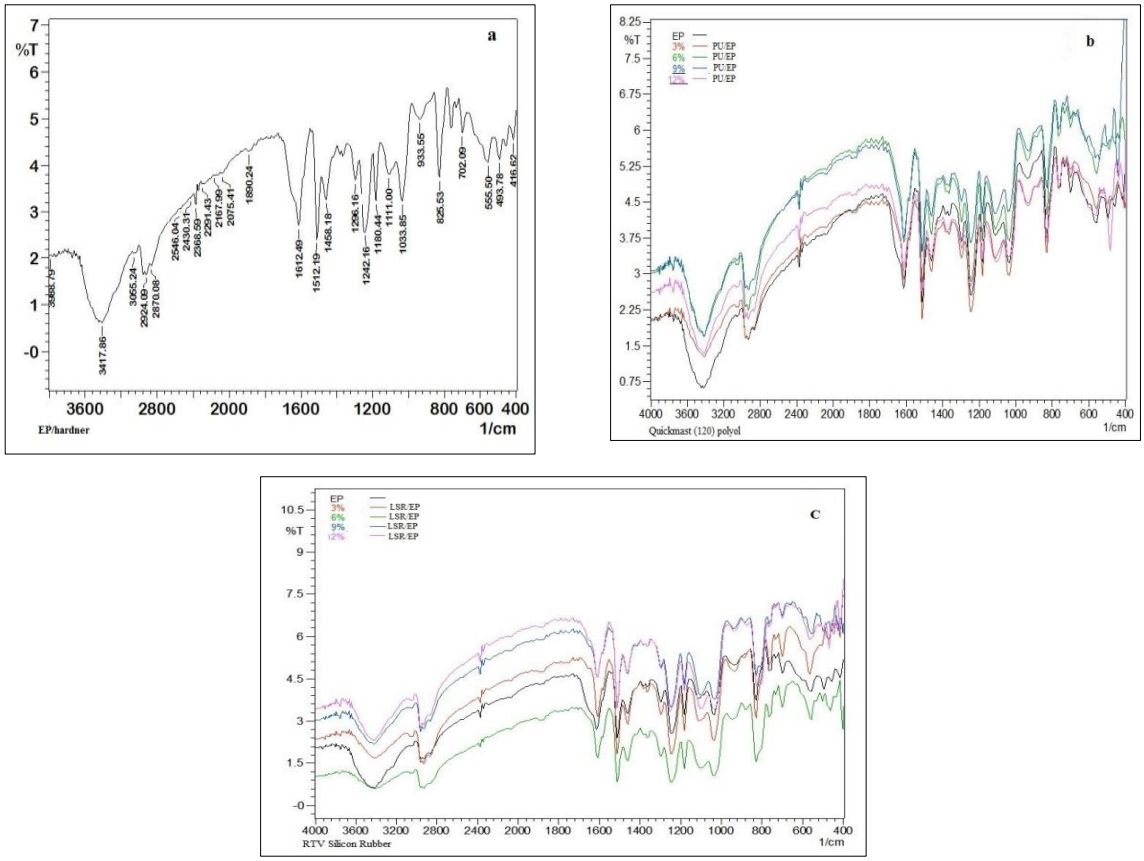

Fig. 4. The FTIR Spectrum for (a) unmodified epoxy, (b)PU/epoxy blends, and (c) LSR/ epoxy blends

3.2. Results of the SEM

SEM examined the surfaces fracture of all epoxy. Representative SEM micrographs illustrated in Fig. 5. SEM micrographs, Fig. 5. (a and b), reveal a typical featureless morphology the unmodified epoxy (neat epoxy), consistent with the amorphous, single-phase structure of epoxy networks. These observations align with previous studies (Alhabill et al., 2018; Nguyen et al., 2015). Fig. 5. (c and d) displays SEM morphology of the fracture surface of epoxy blended with quick mast120 (PU base) at (3%wt) load content and (12%wt) load content of PU polyol, respectively. It is indicated to be an elastically deformed matrix with branching cracks on a number of different fracture planes. As the curing process advances, the polyol phase becomes less miscible with the epoxy matrix, leading to phase separation. The inset image reveals finely dispersed submicron-sized polyol domains within the epoxy matrix. Additionally, microcracks presence, shear bands, and riverbed markings suggests mechanisms for absorption of strain energy (Burhan, S.K., Abed, M.M. and Salih, M.A., 2019; Islam et al., 2015; Jajam et al., 2014). As is characteristic of ductile systems, the matrix of epoxy exhibits homogeneous plastic deformation and significant shear yielding due to the dispersion of spherical polyol boundaries within the matrix (Rahman et al., 2012).

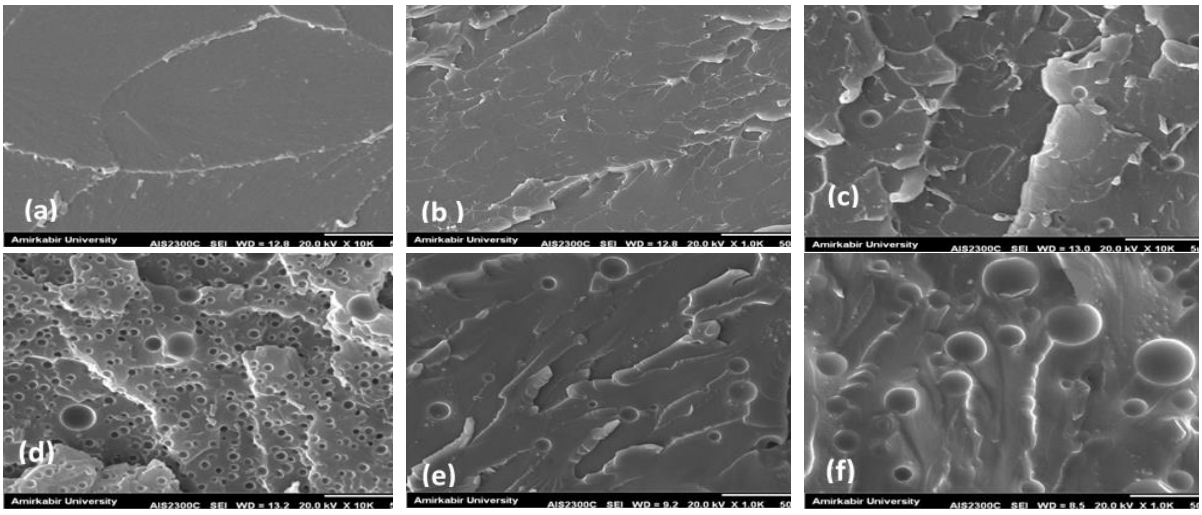

Fig.5. SEM micrographs of a) and b) unmodified epoxy, c) (3%wt) load content and d) (12%wt) load content of PU /epoxy blends, respectively, e) (3%wt) load content and f) (12%wt) load content of LSR/epoxy blends, respectively

The fracture surface of liquid silicon rubber (LSR)/epoxy blend at (3%wt) load content and (12%wt) load content of LSR was analyzed using SEM, as shown in Fig. 6 (e and f). The SEM images reveal a sea-island structure within the epoxy resin (Heng et al., 2015). The image clearly shows two-phase separation, with the thermosetting epoxy forming the majority of the material and the RTV silicone rubber providing flexibility and durability in smaller quantities. As the LSR content reaches 12 wt%, the dispersed phase elements begin to grow significantly in size (Pakaya et al., 2017b). When liquid silicone rubber (LSR) is mixed with epoxy resin, tiny rubber spheres form within the cured samples. While these rubber particles enhance toughness by preventing crack propagation, the poor compatibility between LSR and epoxy resin leads to uneven distribution of the LSR particles (Wang et al., 2020).

Fig. 5 (e and f) demonstrate rubber cavitation and subsequent void growth in all epoxy-LSR formulations. The diameter of these spherical zones varies with the LSR content, increasing as the LSR concentration rises from 3% to 12% wt. This indicates the presence of plastic void growth mechanisms. These findings align with previous research on rubber-toughened epoxies, which have also exhibited rubber cavitation and void growth. The correlation between increased LSR concentration and higher KIC values supports the role of these mechanisms in enhancing fracture toughness (Leelachai et al., 2017).

4. CONCLUSIONS

In this study, modified epoxy resins were developed using liquid silicone rubber (LSR) and polyether polyol (PU) as structural agents. FTIR analysis revealed that the absence of hydrogen bonding in PU-modified epoxy caused phase separation, while LSR addition strengthened specific bonds. SEM analysis showed distinct phase separation in modified epoxies, with PU-

modified epoxy displaying a sea-island morphology and LSR-modified epoxy showing rubber cavitation and void growth, enhancing strength of functional groups. However, LSR and epoxy compatibility issues led to uneven rubber particle distribution.

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