

Research Article

Application of r-GO-MMT Hybrid Nanofillers for Improving Strength and Flame Retardancy of Epoxy/Glass Fibre Composites

Vijayananth Kavimani ¹, Balasubramaniam Stalin ²,
Pudhupalayam Muthukutti Gopal ¹, Manickam Ravichandran ³, Alagar Karthick ⁴,
and Murugesan Bharani ⁵

¹Department of Mechanical Engineering, Karpagam Academy of Higher Education, Coimbatore, 641 021 Tamilnadu, India

²Department of Mechanical Engineering, Anna University, Regional Campus Madurai, Madurai, 625 019 Tamilnadu, India

³Department of Mechanical Engineering, K.Ramakrishnan College of Engineering, Tiruchirappalli, 621 112 Tamilnadu, India

⁴Department of Electrical and Electronics Engineering, KPR Institute of Engineering and Technology, Coimbatore, Tamilnadu, India

⁵School of Textile Leather and Fashion Technology Kombolcha 208, Kombolcha Institute of Technology, Wollo University, South Wollo, Ethiopia

Correspondence should be addressed to Murugesan Bharani; bharani.murugesan@kiot.edu.et

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The application of nanomaterials as a strengthening agent in the fabrication of polymer nanocomposites has gained significant attention due to distinctive properties which can be utilised in structural applications. In this study, reduced graphene oxide (r-GO) and montmorillonite (MMT) nanoclay were used as filler materials to fabricate hybrid epoxy-based nanocomposites. The synergistic effect of nanomaterials on flammability and mechanical behaviour of nanocomposites were studied. Results revealed that the addition of nanofiller showcases 97% and 44.5% improvement in tensile and flexural strength. However, an increment in the percentage of filler material over 0.3% exhibits a decremental mechanical property trend. Likewise, the addition of nanofiller increases the nonignition timing of the glass-fibre-reinforced epoxy composites. Fracture surface morphology displays the occurrence of the ductile fracture mechanism owing to the presence of hybrid fillers.

1. Introduction

In modern industries, epoxy-based resins are considered one of the most promising thermosetting polymers with outstanding merits, including low shrinkage, better adhesion, admirable chemical stability, and corrosion resistance [1–4]. These facts made them a candidate material to employ widely as coating materials, laminates, microelectronic materials, and aerospace materials [5–9]. Nevertheless, similar to other polymer resins, its extreme flammable nature and lower mechanical strength act as the potential barriers to limiting its prevalent applications. Consequently, it is authoritative to form a high strength and flame retarded epoxy resin to expand its future applica-

tions among the areas that demand outstanding mechanical properties and flame retardancy. Composite fabrication is considered one way to improve the basic and functional properties of epoxy by adding suitable reinforcements and filler material [10–14]. Herein, glass fibres are commonly used as reinforcements to improvise polymer materials' basic strength and performance. Glass-fibre-reinforced polymers (GFRP) have been practised in several industrial applications that include transport and electrical appliances with better mechanical properties [15–18].

Nevertheless, the inherent flammability of epoxy-based matrix induces potential fire hazard for GFRP products. It brings large concern in enlightening the flame retardancy of GFRP. Flame retardancy of GFRP can be improved by various

techniques such as retardant coating and charring method [19, 20]. Several researchers used inorganic and organic retardants to decrease the flammability of epoxy. In recent halogen and non-halogen-based flame retardant materials, it is combined with epoxy resin to increase its flame retardant properties. However, the usage of halogen-based retardants results in the formation of harmful toxic gases that results in environmental pollution. To overcome these effects, halogen-free elements such as silicon and boron and some nanocarbon materials, hydroxides, and clays are used. In some research, hybrid fillers with different sizes and shapes are used to enhance the thermal properties of epoxy-based polymers by simplifying the development of compacted and effective heat transfer network [21–24].

Montmorillonite (MMT), a form of clay with various sodium and calcium ions, has more attraction in use as flame retardant filler material in polymer matrix composite due to its low cost. MMT can act dual role viz. shielding and insulating in the improvement of desirable properties of polymer matrix material [25–28]. It further assists in decreasing the smoke and formation of toxic gases during combustion. However, MMT has only minimal ability to increase the expected limiting oxygen index. Hence, there is a need to use additional filler material to make the epoxy-based resin pass in the flammability test. Nanomaterials such as graphene and carbon nanotubes are considered better filler materials to improve the flame retardancy of epoxy-based resins. Usage of nanofiller needs suitable dispersion in loading level to achieve the expected properties [29–34]. Graphene and its derivatives viz. graphene oxide, graphene nanoplates, and graphene nanosheet consisting of a carbon atom with lattice structure have been broadly applied in various multidisciplinary domains owing to its better electrical, thermal, and mechanical properties. Graphene derivatives work as an inhibiting barrier over char residues in the condensed phase and restraining its volatiles. Graphene oxide (GO) is broadly employed as a modified substrate due to its carbon-layered structures [35–38]. Incorporating favourable functional groups in a graphene oxide sheet is also considered an effective way to improve the desired properties of polymer while using it as filler material. To improve polymer composites' thermal behaviour, synergistic effect strategies are considered a feasible approach to resolve interface and dispersion-based issues in developing polymer composite. Herein, easily dispersed nanocarbon materials like graphene and CNT are used in property enhancements [39–41]. There are few kinds of research in which MMT and graphene derivatives are used as filler material to increase the mechanical and flame retardancy characteristics. He and coworkers used MMT-based hybrid filler material to improve the flame retardancy behaviour of epoxy matrix. Results reveal that hybrid nanofiller usage increases the limiting oxygen index of epoxy and attained V0 rating in the flammability test [42]. Kim et al. used MMT to develop basalt fibre-reinforced composite and observed that the addition of MMT improves the interfacial interaction between the matrix and reinforcement.

Further, it was observed that the saline treatment in composite exhibits 33% decrement in fatigue strength [43]. Souza and coworkers utilised MMT as filler material to improve the

thermomechanical behaviour of epoxy resin. The results indicate that MMT's addition influences the delamination and uniformity of matrix, and addition of MMT up to 3 wt.% showcases betterment in dynamic mechanical properties [44]. Kamar and coworkers investigated the effect of graphene nanoplatelets over epoxy matrix. Their observation depicts that incorporating 0.25 wt.% of graphene nanoplatelets improves the flexural strength of matrix up to 29%, and 25% improvement in fracture toughness is also observed [45]. Naeem and coworkers developed a graphene-based composite and investigated its dynamic thermomechanical properties. Results revealed that the addition of graphene increases the fracture toughness and stiffness of matrix material up to 78.41% and 16.29%. Usage of graphene up to 0.25 vol.% improves its glass transition temperature [46]. Based on a clear-cut literature survey, the addition of graphene and MMT improvises the basic and functional properties of the polymer. However, there is limited research in the investigation on the effect of hybrid MMT-graphene fillers in the epoxy composite. Hence, this research focuses on developing MMT-graphene hybrid filler-based composites and examining their mechanical and flame retardancy behaviours.

2. Materials and Methods

The polymer matrix used for developing the novel composite is epoxy resin diglycidyl ether of bisphenol A grade LY 556 with the hardener of HY951 grade ($C_6H_{18}N_4$), which has the gel time of 2 to 3 h. Smaller viscosity array, fibre impregnations, and outstanding dimensional steadiness are the key factors that lead to the selection of the material above as matrix. In improving the mechanical properties of the matrix material, 200 GSM glass fibre is utilised as a reinforcement. The percentage of glass fibre reinforcement is fixed as 30% based on the literature where the composites with 30% glass fibre addition yielded better properties. The nanofiller materials used in this research are reduced graphene oxide (r-GO) and MMT nanoclay, where the amount of MMT is fixed as 1.5% and the graphene percentage is varied from 0.1 to 0.4 with an equal interval of 0.1%. Herein, r-GO is synthesised through the modified Hummers method, and detailed r-GO synthesis procedure was already reported in previous works [45]. MMT is received from Ad-Nano Technologies Private Limited with the average size of 50–100 nm with alumina silicate layers of 1 nm thickness. XRD of as purchased MMT is depicted in Figure 1, and the purchased MMT is used directly without any modification.

2.1. Composite Fabrication. The fabrication methodology followed to fabricate the nanofiller-added glass-fibre-reinforced polymer matrix composite is the simple and low-cost fabrication method process, namely, compression moulding. The measured amount of nanofillers, namely, r-GO and MNT, as per the research hypothesis, is mixed with ethanol and ultrasonicated for an hour and mixed with the matrix resin. The matrix material mixed with nanofillers is stirred mechanically at 1200 rpm for two hours because of attaining uniform distribution of fillers in the matrix.

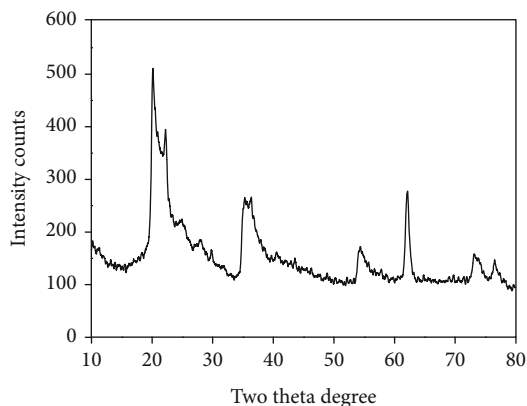


FIGURE 1: XRD of as received montmorillonite nanoclay.

Subsequently, a hardener (HY951) is added in a 1:10 ratio, and mixing is done for 5 minutes. The prepared mixture is then coated over the glass fibre (both sides) initially through the hand layup method. Process variables and their operating range for composite fabrication are fixed based on the trial and error method. Stacking is done in the flat mould of $270 \times 270 \times 30$ mm size, where the number of coated glass fibre layer is ten followed by degassing. Then, the stackings were hot-pressed under the pressure of 15 MPa and allowed to cure. Other nanofiller-added epoxy-glass fibre composites were prepared with various r-GO filler percentage viz by following the same procedure, 0.1, 0.2, 0.3, and 0.4.

2.2. Material Characterisation. X-ray diffractometer (BRUKER) was utilised to authorise the presence of graphene oxide and MMT. Fourier transform infrared spectroscopy (FTIR) was used to confirm functional groups' presence in the developed composite. ASTM D 638 standard was adopted to understand the tensile behaviour of the developed composite. ASTM D 790 standard is used to find the flexural strength of the fabricated composite. In contrast, ASTM D 256 standard is adapted to understand the impact strength behaviour by using a Charpy impact testing machine with a sample dimension of $65 \times 13 \times 3$ mm. Experiments were repeated four times, and the average value is noted for the exactness of the attained results. Scanning electron microscopy (JEOL JSM6610LV) is used for fracture surface analysis. The UL-94 vertical burning test is accompanied by adopting ASTM D3801 standard to analyse the flammability nature, and tests were conducted over five samples, and the average values are noted. A limiting oxygen test was conducted on developed samples based on ASTM D2863.

3. Results and Discussion

3.1. XRD and FTIR of the Developed Composite. XRD and FTIR analysis results of the developed composites are depicted in Figures 2(a) and 2(b), which confirm the presence of r-GO and MMT in the epoxy matrix. The peak near 25.4° is respective to 111 planes, and small peaks near 43.6° are related to 002 planes of graphene, and other minor peaks

are respective peaks of MMT and base matrix (Figure 2(a)). The diffraction peaks at 19.8° and 20.9° are the related peaks of MMT with 100 planes, and the attained peaks are matched with JCPDS no. 13-0135. Figure 2(b) depicts the presence of functional groups and their state of vibration in the developed composite. The absorption peaks near 3395 cm^{-1} reveal O-H stretching vibration, peaks near 1652 cm^{-1} associated with C=N bonds, and peaks near 1387 cm^{-1} related to C=C stretching vibration. Minor peak near 1738 cm^{-1} belongs to the COOH group. Absorption peaks near 3618 cm^{-1} related to Al-O-H stretching vibrations of MMT and smaller peaks at the range of $1089\text{--}1035\text{ cm}^{-1}$ are correlated peaks of Si-O-Si stretching vibrations. Likewise, a peak near 1625 cm^{-1} represents the bending vibration of the H-O-H group and the absorption peak around 1030 cm^{-1} was associated with stretching vibration of Si-O-Si [47–50].

3.2. Tensile Behaviour of Developed Composite. Stress-strain curves and tensile behaviour of the developed nanofiller-added composites are given in Figures 3(a) and 3(b). It is evident from the figure that the added r-GO and MMT have a greater influence on the epoxy-glass fibre composites' tensile behaviour. Tensile modulus and strength are increased significantly with the addition of nanofillers.

The addition of r-GO resulted in increased tensile modulus from 1.2 GPa to 2.2 GPa, where sudden improvement is found while adding 0.1% r-GO. It increases gradually for further addition of r-GO. On the other hand, the tensile strength increased up to 150 MPa for 0.3% r-GO addition, and it decreased by 0.4% r-GO addition. This improvement in tensile modulus and strength can be attributed to the nanofillers' restriction for polymer chain mobility. Higher-strength, modulus, and aspect ratio and healthier interfacial bond among the nanofiller and matrix are also the key factors that contributed to the enhancement of tensile properties. The decrease in tensile strength for the 0.4% rGO-added composite might be attributed to the agglomeration and void formation tendency of nanoparticles when reinforced in higher quantity [28].

3.3. Flexural Strength of Developed Composite. Stress-strain curves and flexural strength of the developed composites are depicted in Figures 4(a) and 4(b). It indicates that the flexural properties of the epoxy-glass fibre composites increase up to 0.3% r-GO addition, and further addition of r-GO resulted in decreased properties. Flexural strength and modulus of the composite increase gradually up to 0.3% of r-GO addition, and small decrement is observed for further r-GO addition. This enhancement in flexural properties is due to two possible mechanisms, enhancement in the strength of the matrix due to modification due to added r-GO and better interfacial bonding.

To withstand higher loads, the load transmission from the matrix to reinforcement should be as high as possible. This increment in strength of the matrix mainly depends on the interfacial area between the matrix and r-Go. As the reinforced r-GO and MNT are in nanosize, they are having a high surface area which resulted in a higher interfacial area between the matrix and nanofillers. This higher interfacial

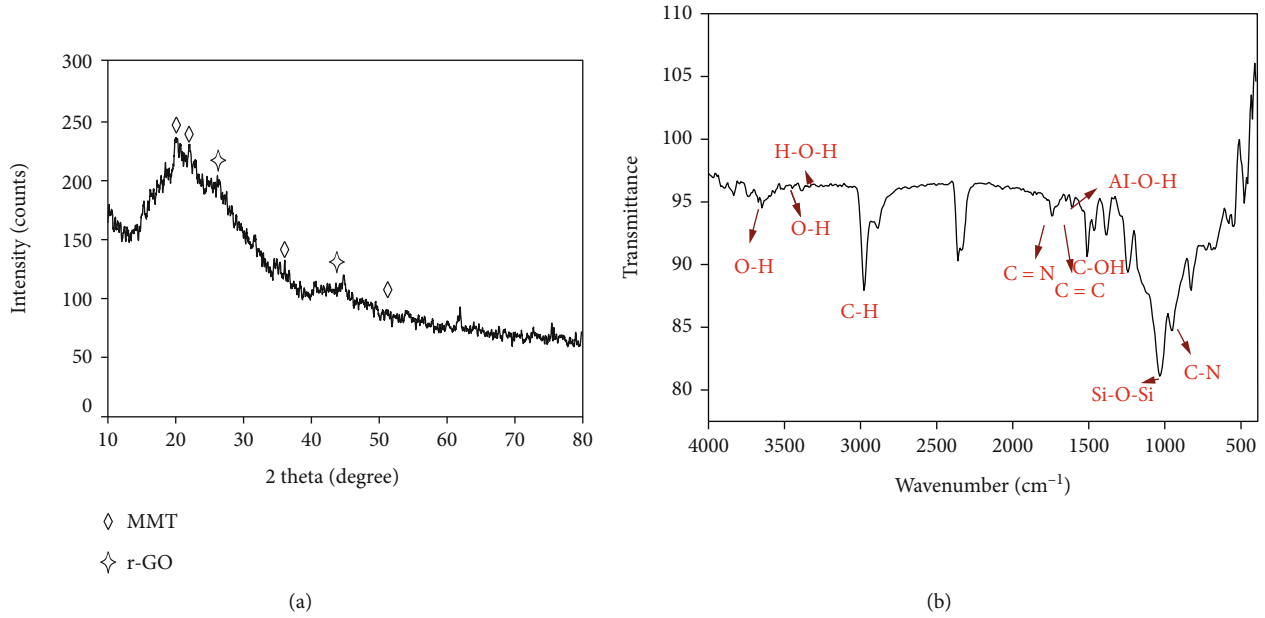


FIGURE 2: Epoxy-r-GO-MMT composite: (a) XRD; (b) FTIR.

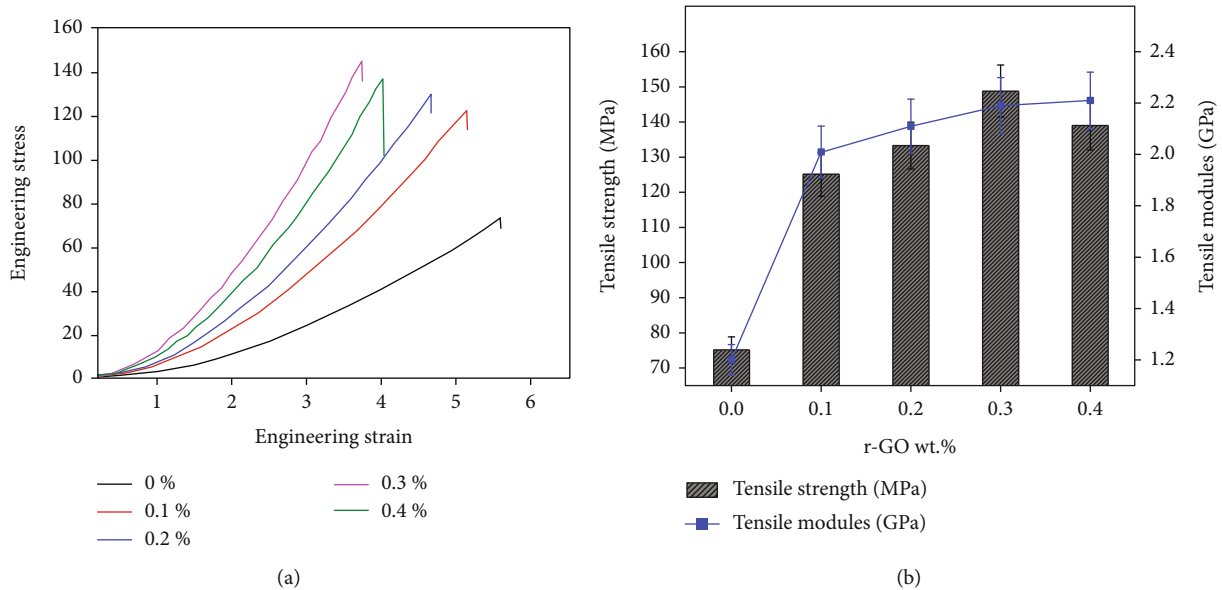


FIGURE 3: (a, b) Variation of tensile strength with respect to r-GO addition.

area enables more stress transfer among them which facilitates the polymer composites to bear more stress, i.e., strength increases. In addition to this, generally, the nanofiller-added polymers possess better strength than the raw polymers as a result of physical absorption of polymer onto the surface of added nanofiller. When higher amount of r-GO is added with the matrix, there are possibilities of particle agglomeration owing to the higher surface area. This particle agglomeration decreases the effective surface area of the nanofillers which results in decreased load-bearing capability of the composites [51].

3.4. Impact Strength of Developed Composite. The impact strength of the developed composites, i.e., the capability of absorbing energy, is illustrated in Figure 5. It is evident from the depiction that the addition of r-GO improves the composites' energy absorption capability up to 0.3% addition. The process where the energy is absorbed during sudden loading of polymer composites is matrix deformation, interface debonding, and reinforcement fracture. Among these phenomena, which absorbs lower energy is said to be the key factor for fracture. However, the crack length during fracture is high in the case of particulate-reinforced

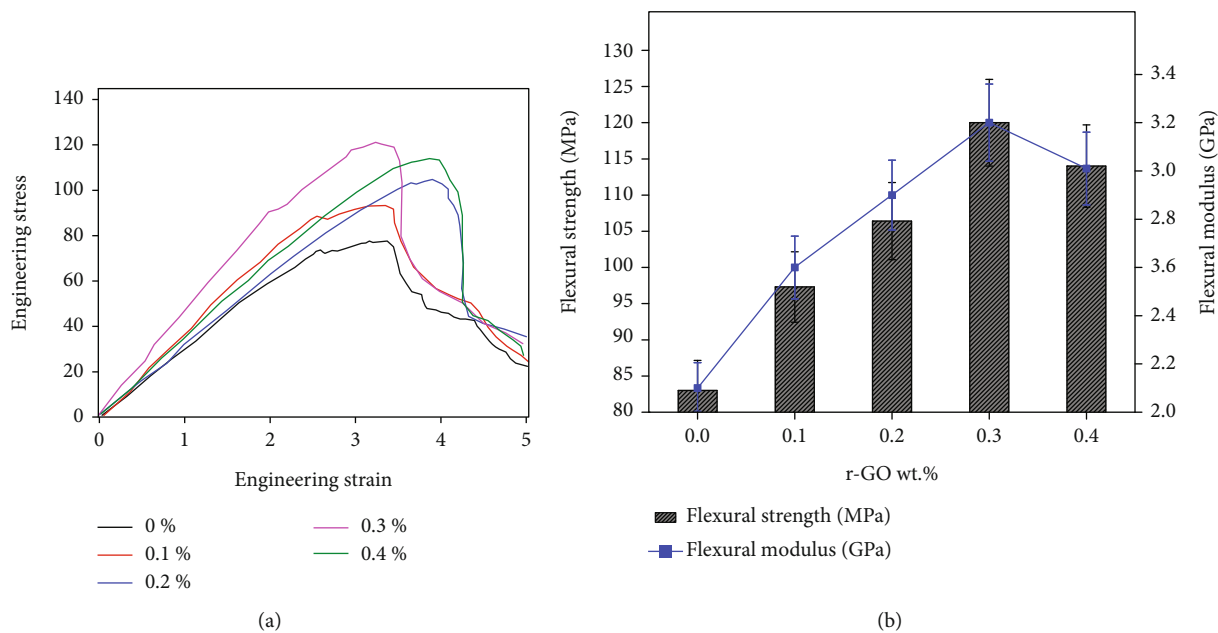


FIGURE 4: (a, b) Variation of flexural strength with respect to r-GO addition.

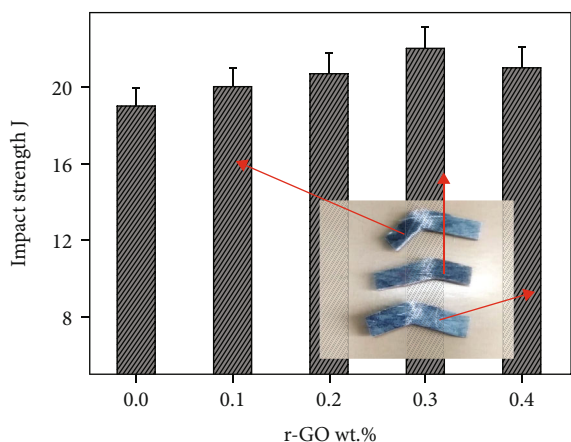


FIGURE 5: Variation of impact strength with respect to r-GO addition.

polymers. Since the reinforcing fillers are nano in size, their surface area is also high, leading to an increase in crack length, i.e., more energy absorbed. These reinforced fillers restrict the advancing cracks and branch the cracks that lead to more energy absorption.

3.5. Fracture Surface Morphology of the Developed Composite.

The fractography of the developed composites subjected to the tensile test is shown in Figures 6(a)–6(d) that aids to analyse the mechanism of failure. The fracture surface micrographs illustrated in Figures 6(a) and 6(b) belong to the unfilled composite material, i.e., the composite without hybrid nanofillers clearly shows the matrix and fibre debonding. This debonding of fibre from the epoxy matrix due to the lack of interfacial bonding leads to failure of

composite material when subjected to smaller loads, i.e., the composite exhibits lesser strength. Huge fibre pull out, as depicted in Figures 6(a) and 6(b), occurs due to inadequate fibre matrix bonding and subsequently effortless fracture. The fractured surface of 0.2 wt.% of r-GO-added epoxy-glass fibre-MMT composite is given in Figure 6(c), which also has the composite EDS spectrum.

The EDS spectrum of the composite endorses the r-GO and MMT nanofillers’ existence, and Figure 6(d) depicts the fractured surface of the 0.3 wt.% of r-GO added epoxy-glass fibre-MMT composite. It is clearly evident from Figures 6(c) and 6(d) that the addition of nanofillers highly influences the adhesion of fibre with epoxy matrix. Better adhesion among the epoxy matrix and glass fibre can be easily visualised from Figures 6(c) and 6(d). It is visible from Figures 6(c) and 6(d) that the matrix material and fillers are sticking with fibres which are attributed to the improvement in strength for the filler-added composite when compared to composite without filler.

Conversely, only minimal matrix material is stick with fibre for unfilled composite shown in Figure 6(b) and complete debonding of fibre from the matrix is evident from Figure 6(a). When the load is applied over the composite material, a crack is formed initially and it has to propagate across a huge number of nanofillers and fibre matrix interface which act as a hindrance to the crack propagation. As a result of these hindrances offered by the fillers and interface, the crack propagation changes the direction several times, increasing crack length, which results in improved strength. The fillers that hinder the crack propagation have to be fractured, which also required additional load, i.e., strength increases. So the improvement in strength is achieved with the addition of r-GO and MNT nanofillers with the epoxy-glass fibre composites [30–33].

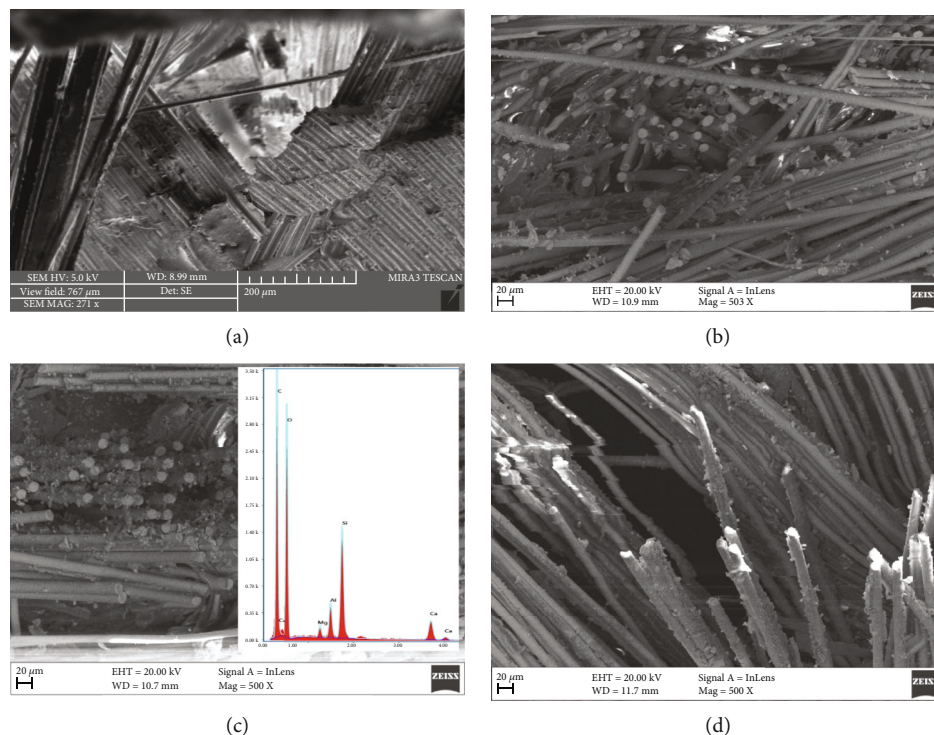


FIGURE 6: Fracture surface of composite with (a, b) 0 wt.% filler, (c) 0.2 wt.% of r-GO, and (d) 0.3 wt.% of r-GO.

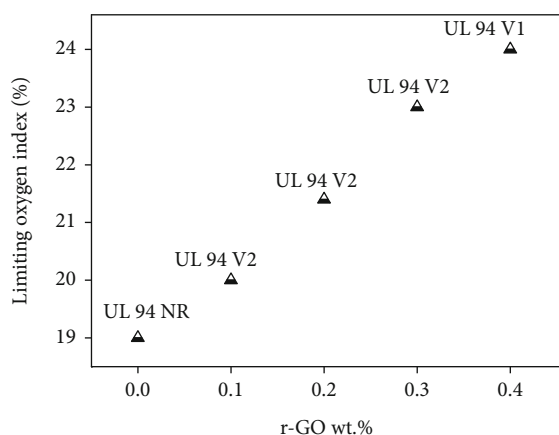


FIGURE 7: Limiting oxygen index and vertical flammability ranking of composites.

3.6. Fire Retardancy Behaviour of Developed Composite. The limiting oxygen index and UL 94 rating are depicted in Figure 7. Herein, UL 94 ratings are given based on the self-sustained burning duration. Based on the available literature, burning time less than 10 seconds is termed as V0 rating and the material is considered the best for flame retardant applications. V1 ratings are given to the sample with a burning time of fewer than 30 seconds, and burning is not sustained. V2 rating is given to the samples with burning seconds of 30, and burning is not sustained, but flaming drips ignite the cotton below the specimen. NR rating is given to the samples which have more than 30 seconds of burning. The limiting oxygen index value is an important pointer on polymer and

plastic materials' flammability as in general, materials with higher limiting oxygen index are considered the flame-retardant material. The limiting oxygen index is defined as the volume percentage of minimum oxygen consideration that assists in material combustion. It can be notified from Figure 7 that the addition of nanofillers showcases better influence in the limiting oxygen index of epoxy polymer matrix composite. In this, the addition of 0.4 wt.% of r-GO improves the limiting oxygen index values up to ~26.32% and ~21.01% of improved limiting oxygen index values was attained for 0.3 wt.% of r-GO filler. It can also be noted that matrix material has a low limiting oxygen index, indicating the poor flammability resistance of the epoxy polymer. It can also be observed that the addition of r-GO showcases excellent performance on the developed composite's flame retardant behaviour. This might be due to the effect of intermolecular and intramolecular interaction among the matrix and filler phase.

During testing conditions, the presence of r-GO fillers helps in limiting the rapid burning of composite in thermal condensation of functional groups present in composite structures. The presence of hybrid filler forms as a passive barrier over the burning surfaces that hinders the proclamation of noncombustible gases and further limits the destruction of dripping properties of the composite. It can be notified that the increase also of hybrid filler reduces the burning timing of advanced composite. The samples with higher r-GO wt.% have V1 rating in vertical flammability testing, and it depicts that increasing in r-GO improves the flame-retardant performance of the developed composite. V2 rating is attained for the composites

made with 0.3, 0.2, and 0.1 wt.% of r-GO; this might be due to the insufficient supply of filler material that reduces the burning process. The matrix material has an NR rating that means the samples have burned more than 30 seconds or burned completely [52–55].

4. Conclusions

Reduced graphene oxide and MMT are used as filler material in epoxy-based glass fibre composites to improve mechanical and flame retardancy properties. The results observed from the various characteristic studies conducted are as follows:

- (i) Addition of 0.3 wt.% of r-GO improves the tensile strength up to ~97%
- (ii) Addition of r-GO results in 44.5% improvement of the flexural strength and 15.6% impact behaviour of composite
- (iii) V1 rating was attained for 0.4 wt.% r-GO incorporated samples, and a decrease in r-GO reduces UL 95 rating
- (iv) These composites may be suggested in potential applications like roofing sheet and car dashboards

Data Availability

The data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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