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Analysis of the Physical and Mechanical Characteristics of Hybrid Filler Composites Containing Rice Husk and Aluminium Nitride

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Abstract: The purpose of this study was to investigate how the presence of hybrid filler affected the physical and mechanical characterization of epoxy matrix composites. The construction of this hybrid composite was accomplished through the use of the hand layup method, and the filler content featured variable weight loading in addition to weight ratios. Rice husk and aluminium nitride were both elements that went into the construction of the hybrid system. The results of the tensile, flexural, and micro-hardness tests were consistent with the findings of the surface morphology, void content, chemical composition, and crystalline structure analyses performed on the manufactured samples. Because of the formation of agglomeration, which was previously visible in SEM micrographs, the hybrid composite made of rice husk, aluminium nitride, and epoxy did not show any discernible improvement in tensile or flexural strengths. In addition, the existence of voids is an indication of insufficient adhesion and incompatibility between the hybrid filler and the epoxy matrix. Because of this, the strength of the hybrid filler composites is negatively impacted.

Keywords: *Hybrid filler; Bio composites; Aluminium nitride; Rice husk; Physical and Mechanical Properties*

1. Introduction

Interest in novel composite materials with enhanced capabilities to satisfy specific application requirements has been gradually increasing recently. Typically, polymer is strengthened with organic filler during the development of new materials. Polymers have continued to be used in a variety of organizations because of their great corrosion resistance, low cost, and lower weight (Sadashiva *et al.,* 2022, Dinesh, *et al.,* 2020; Vitaliy Datsyuk, et al., 2013). Development of novel polymer composites, including hybrid filler, has grown to be a popular area of study in the field of materials science because of a variety of advantages (Balaji *et al.,* 2022, Sadashiva *et al.,* 2022). Polymer composites have a substantially higher internal interfacial area than traditional composites because the filler is dispersed on a Nanoscale, which maximizes the interactions between the polymer and the particles (Vassiliou *et al.,* 2008). A good thermal stability was obtained by mixing hydroxyapaptite (HAp) nanoparticles with hydroxyethyl cellulose acetate (Azzaoui *et al.,* 2014). This kind of composite represent a new, efficient, and inexpensive natural-based adsorbent with high efficacy for the cationic dye methylene blue (Akartasse *et al.,* 2022). Furthermore, the synthesis of hydroxyapatite, polylactic acid (PLA) and poly-caprolactone membranes "HAp/PLA/PCL" was successfully achieved through dispersion of HApt particles homogeneously in the composite to provides the "bioactive" character desired for bone regrowth (Azzaoui *et al.,* 2016). Polymer composites are a crucial material property for many applications, despite not having been extensively explored. For specific applications, such electronic applications, where the majority of polymers exhibit a relatively low thermal conductivity, it is crucial to make improvements (Zhou *et al.,* 2009). When filler loadings are extremely large (up to 70% vol %), the critical concentration for micron-sized filler is frequently achieved. Large filler loadings impair the mechanical toughness and processing capabilities of polymers (Bao et al., 2009; Cui et al., 2011). The mechanical properties of epoxy polymer, a range of thermoplastic and thermoset resins, are improved along with their dimensional and thermal stability. Epoxy resins also go through a phase of solidification, which makes them brittle (Luo et al., 2015; Zhang et al., 2013; Kavya H et al., 2021). The use of different recyclable and renewable reinforcement materials is advocated, including plant leaves, jute, kenaf, bamboo dust, wood dust, flax, and crop. Despite numerous attempts to employ biodegradable and renewable polymer for industrial application, there is a significant disadvantage established in the thermo-mechanical properties. In recent years, synthetic and natural fibres have been used to make the majority of polymer laminates (Jaszkiewicz et al., 2013; Andrzejewski et al., 2016; Jenish, et al., 2020). Due to their adaptability, accessibility, biodegradability, and ability to provide sustainable solutions to support technical innovation, not just in the textile, automotive, electrical, and construction industries, characterizing new natural fibres from various plant parts has gained popularity (B. Gurukarthik et al., 2019). Making epoxy composites was the main focus of the current research, which aimed to describe how they behaved in terms of their physical and mechanical characteristics when they were filled with various ratios of rice husk and aluminium nitride. Additionally, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were utilised to analyse the molecular structure before using EDX to analyse the chemical composition of hybrid particle composites. The tensile, flexural, and hardness strengths of the produced composites were thoroughly examined to ascertain the impact of hybrid particles on the pure epoxy. The amount of voids in the hybrid composites was assessed

2. Experimental

2.1 Materials

The composite was mostly made of epoxy, aluminium nitride, and rice husks. Rice husks and 25 micron-sized aluminium nitride were purchased from Vruksha composites in Telangana. The matrix material is epoxy (LY 556), commonly known as Bisphenol-A-Diglycidyl-ether (BADGE or DGEBA). A solvent-free room temperature curing system is created when epoxy is combined with the tri-ethylene-tetramine (TETA) hardener, which is related to essential amine and is sold under the trade name HY 951. Both the epoxy resin LY 556 and the hardener HY-951 were provided by Nano Technologies Bangalore.

2.2 Treatment of fillers

Rice husks were chemically treated to remove dirt and other particles. By being immersed (soaked) in NaOH solution for 24 hours while maintaining a temperature of 25 °C, the rice husks underwent chemical treatment. The nursing rice husks were properly washed in regular water before a few drops of ethanoic acid were added to balance the NaOH. RH was put into the processor's operation with a strainer that was 1000 microns in size to generate fine grains. The rice husk was treated (using a strainer with a size of 250 m) before sieving. The RH particles were ground and strained into various grain sizes less than and equal to 100 m. Aluminium nitride surface was treated with silane solution. In order to function as a coupling agent or adhesive promoter, silane is added to an ethyl solution at a predetermined amount and stirred for approximately 10 minutes with a magnetic stirrer in a glass cup.

AlN molecules were added to the mixture and stirred for 20 minutes at a temperature of up to 80 o C. The product is filtered, rinsed with alcohol, and dried with the use of an oven at 110 °C for 12 hours after being brought to room temperature. These methods help to enhance mechanical qualities by using an epoxy matrix to increase the adhesive property.

2.3 Sample preparation

Hybrid composites consisting of epoxy are created by following the traditional hand lay-up method. Two different kinds of fillers, one synthetic and the other organic, were used for this experiment and were both distributed at random. production of specimens with different weight proportions (10–30 wt%) while keeping the weight ratios of RH and AlN at 1:1, 1:3, and 3:1 Accordingly, **Table 1** below displays the positioning and names of numerous composite specimens made using epoxy resin as the basic matrices. In accordance with recommendations, LY556 (Epoxy) and HY951 (Hardener) are mixed in this composite matrix at a weight ratio of 10:1. To guarantee uniform particle dispersion and avoid big molecule aggregation, the Epoxy matrix was appropriately enhanced with RH and AlN as needed. All mixtures were mixed into one batch and mechanically stirred at room temperature for 30 minutes. The mixtures were heated to a temperature of 50 °C before being degassed. The composites were created in a wooden mould using the conventional casting procedure. To make it simple to remove the composite material, the mould has been coated with a releasing agent. The 30x30x0.3 cm wood mould was filled gradually with the hybrid filler mixture. The curing process was carried out in an oven for 1 hour at 80° C, followed by 2 hours at 145° C, and then gently cooled to ambient temperature. The castings were then left to cure for 24 hours at normal temperature. After that, the composite was taken out of the mould and mechanically characterized without causing any damage by being cut with a water jet in accordance with ASTM standards.

Table 1: Fabrication of Samples for various ratios and wt. %

Specimens	Composites	Ratios	Compositions
1	CE7	1:1	Epy (70 wt.%) + RH (15 wt.%) + AlN (15 wt.%)
2	CE8	1:1	Epy (60 wt.%) + RH (20 wt.%) + AlN (20 wt.%)
3	CE9	1:3	Epy (70 wt.%) + RH (7.5 wt.%) + AlN (22.5 wt.%)
4	CE10	1:3	Epy (60 wt.%) + RH (10 wt.%) + AlN (30 wt.%)
5	CE11	3:1	Epy (70 wt.%) + RH (22.5.5 wt.%) + AlN (7.5 wt.%)
6	CE12	3:1	Epy (60 wt.%) + RH (30 wt.%) + AlN (10 wt.%)

2.4 Investigation Techniques

According to ASTM standard D3039, which demands that the specimen's length be 250 X 25 X 3 mm and that the crosshead operate at a speed of 10 mm/min (UTM), the hybrid filler composites sample underwent tensile testing. Each grip's distance from the other was kept at 25 mm. Flexural strength of hybrid particle composites was evaluated using specimens with dimensions of 100 x 12.7 x 3 mm and a loading rate of 2 mm/min in line with ASTM standard D790. Hardness is tested for resistance to deformation or damage in accordance with ASTM standard D785. A diamond-shaped indenter is pressed into the specimen under pressure. Three composite specimens were utilised for each test, and the three outcomes' averages were computed. X-ray diffractometer employ a specific pattern of hybrid filler composite in which the y-axis denotes intensity and the x-axis represents a range from 0 to 80o when determining the crystalline peak of a specimen with Ni-filtered CuK radiation. The volume proportion of voids in hybrid composites was determined using the link between the theoretical and experimental densities of the composites.

3. Results and Discussion

3.1 Density & Void fraction (%)

In comparison to epoxy and aluminium nitride, rice husk (RH) is less dense. The experimental and theoretical densities of the hybrid composites are shown in **Table 2**, together with the corresponding volume fraction of voids. The discrepancy between predicted and experimentally measured values suggests that the composite has pores or voids. As the RH and AlN content in the matrix increased from 30 to 40 wt% in varied ratios, the density and void fraction percent of the specimens (1 to 4) grew, but those of the specimens (5 to 6) declined. Density, which depends on the proportions of resin and filler components, should be taken into account while evaluating the properties of composites. The voids have a significant impact on certain of the mechanical properties and usefulness of composites in the services. Knowing the void content is ideal for making a more accurate assessment of the composites' quality because superior composites have fewer voids.

Table 2: Experimental and Theoretical Density of the hybrid filler composite

Specimen	Composites	Experimental density (gm/cc)	Theoretical density (gm/cc)	Void fraction (%)
1	CE7	1.334	1.290	3.298
2	CE8	1.412	1.361	3.612
3	CE9	1.541	1.461	5.191
4	CE10	1.688	1.562	7.464
5	CE11	1.127	1.103	2.130
6	CE12	1.136	1.109	2.377

3.2 Scanning electron microscopy

The composite material's scanning film produced by SEM reveals the presence of dispersed hybrid tiny particles in the resin. **Figure 1** shows that the RH & AlN particles are distributed uniformly across the resin's surface area at weight percentage concentrations of 1:1 (Specimen 2). Due to the smooth surface and tiny holes created by the combustion reaction between the fillers and resin, there is no sign of agglomeration. The detection of a chunk or collection of particles in **Figure 2** as well as the uneven distributions of hybrid fillers on the resin surface (Specimen 4) are both shown. The Vander Waals interaction between the hybrid fillers and the resin's viscosity led to agglomeration developing on the resin surfaces. When the AlN particle concentration rose due to high density relative to RH & resin, the interparticle distance reduced, causing uneven dispersion, and the bond between fillers and resin composite weakened. The composite exhibits a constant distribution of microparticles in the resin with a ratio of 3:1, as shown in **Figure 3** (Specimen 6). Rice husk particles develop a strong dispersion and interlink among themselves in the resin matrix by lengthening the interparticle distance and overcoming the AlN without agglomerating.

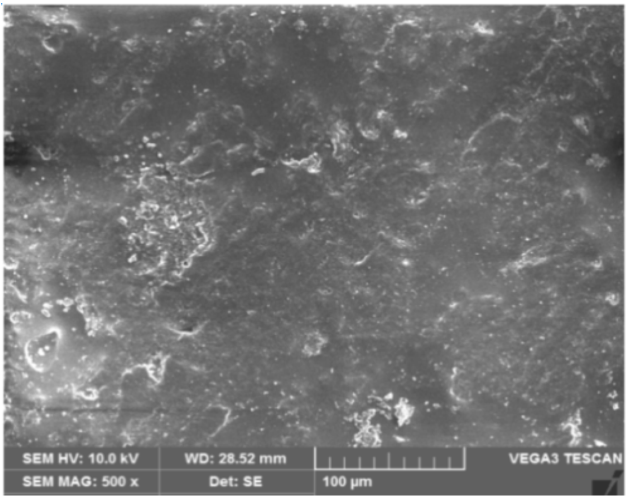

Figure 1 (CE08)

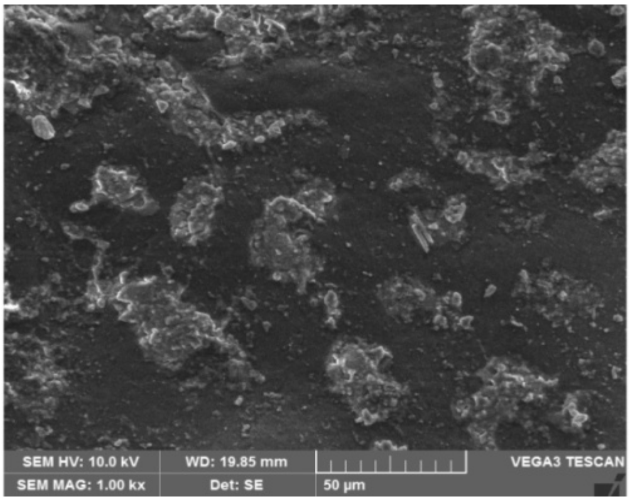

Figure 2 (CE10)

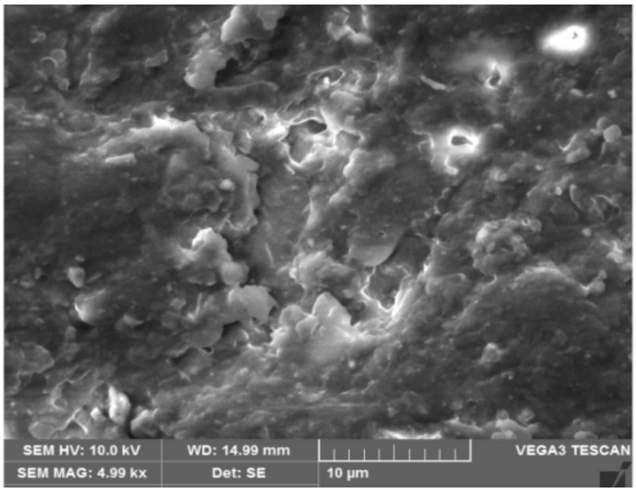

Figure 3 (CE12)

3.3 Energy Dispersive X-ray (EDX)

An elemental analysis of the hybrid filler composites was carried out using an EDX analyzer that is connected to the SEM. EDX spectrum of burning-produced Si and AI particles. The existence of the Al, C, and O peaks revealed that the composites contained Al particles with a weight percentage of 5%. Peak indexing for the Al particle, the C particle, and the O particle are shown in **Figure 4** at 1.6 kev, 2 kev and 6 kev, respectively. Similarly, **Figure 5** shows that Si particles made up 5 weight percent of the composites and Al particles made up 15 weight percent. **Figure 6** shows that the composite contained Si particles in an amount of 15 weight percent and Al particles in an amount of 5 weight percent. It has now been established that hybrid composites comprised of rice husk and aluminium nitride exist.

3.4 X- Ray Diffraction (XRD)

It is a sophisticated, non-destructive method for figuring out the material's composition and atom arrangement, as well as its crystalline phase. To differentiate between chemicals that are present in solid and fine powder form, the diffractometer compares the X-ray pattern. The average size of crystallite particles was calculated using the Scherer **Eqn 1**.

$$D = \frac{k.\lambda}{\beta \cos\theta} \qquad \textbf{Eqn. 1}$$

Where D = crystallite size, k = 0.89 (Scherrer constant), β = FWHM (full- width at half maxima) of diffraction peak, λ = 1.540598 Å (the wavelength of X-ray) and θ = Bragg angle.

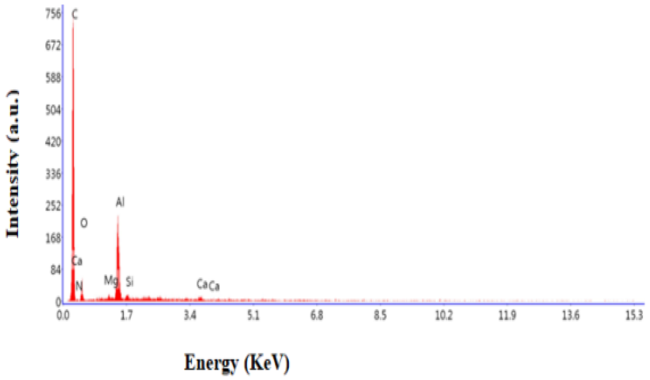

Figure 4 (CE08)

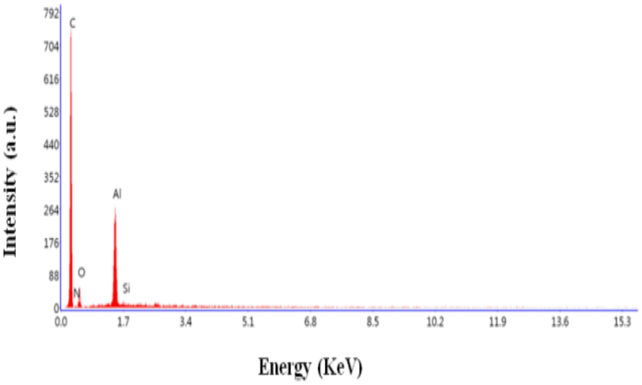

Figure 5 (CE10)

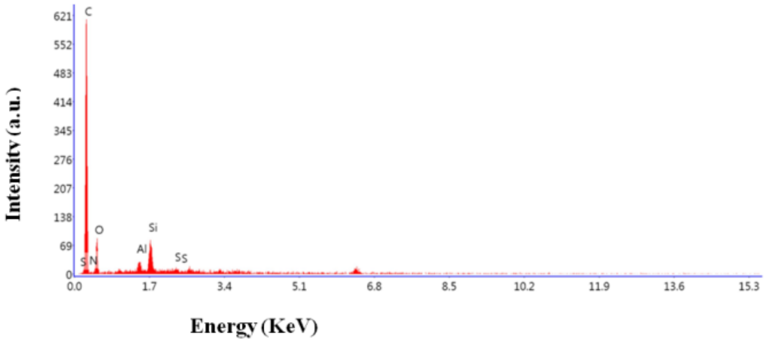

Figure 6 (CE12)

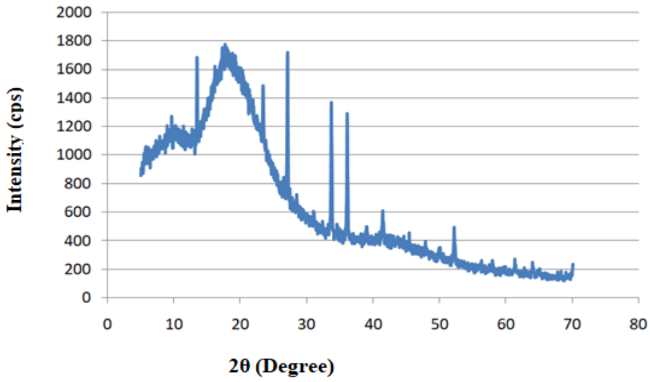

Figure 7 (CE8)

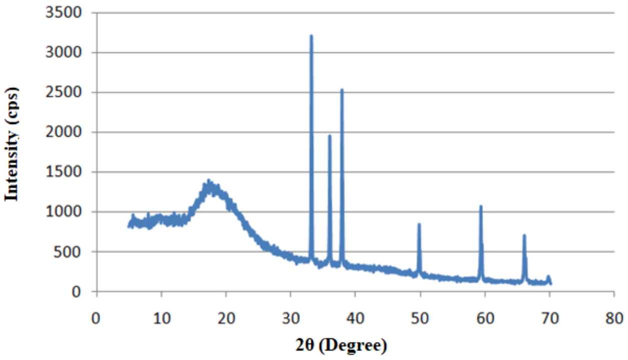

Figure 8 (CE10)

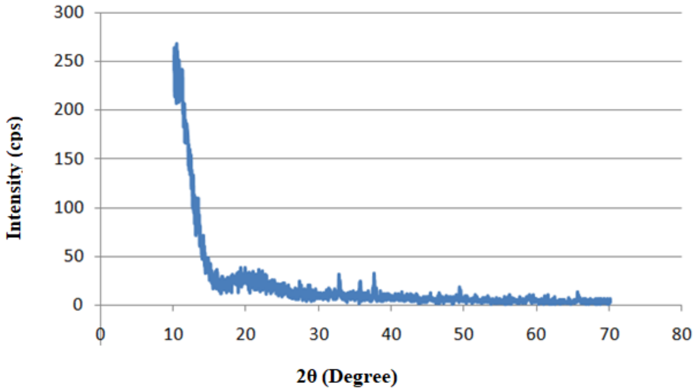

Figure 9 (C12)

The hybrid composite's X-ray pattern, where the X-axis represents range in 2 and the Y-axis the intensity of the radiation, was used to find the crystalline peak with Ni-filtered CuK radiation. The hybrid composite's crystalline peak changed after the inclusion of rice husk and aluminium nitride particles. The graph above shows that the crystalline peaks were fairly high for the 1:1 hybrid composites that included AlN (20% weight), RH (20% weight), and epoxy resin (60% weight) in an amorphous form. This composite is depicted in **Figure 7**. As shown in **Figure 8**, when RH (10% weight), AlN (30% weight), and resin (60% weight) are present, a ratio of 1:3 results in a decrease in the amorphous peak band and an increase in the crystalline peaks at 2=33, 36, and 38. The crystalline band shrinks and the amorphous peak increases in the ratio 3:1 of RH inclusion (30% weight), AlN inclusion (10% weight), and resin inclusion (60% weight), as seen in **Figure 9**. This is because RH overlaps AlN. It was known that the 1:1 and 1:3 ratios have strong crystalline peaks, and the inclusion of AlN verifies that the hybrid composites' amorphous band is reduced and their crystalline peak is raised.

3.5 Tensile Strength

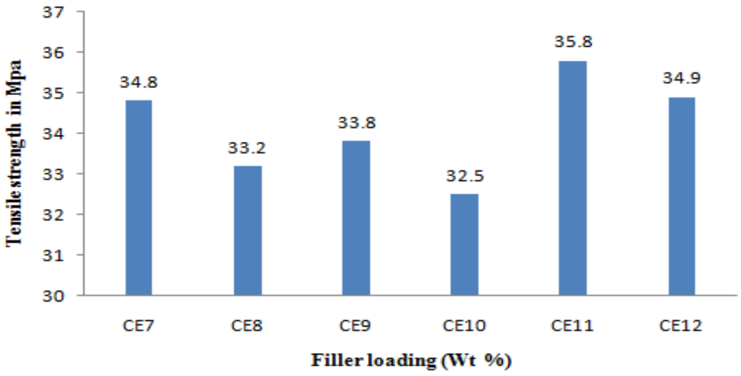

Figure.10 Tensile (MPa) v/s Filler loading (wt %)

Figure 10 displays the results of tensile strength tests conducted on six hybrid composite specimens, with the ratios 1:1 (CE7 & CE8), 1:3 (CE9 & CE10), and 3:1 (CE11 & CE12) respectively. This illustration shows how the tensile strength gradually decreases as the amount of filler material in the material increases. When compared to the other composition, the specimen CE11 composite that had both fillers added in a weight ratio of 3:1 had the highest tensile strength possible. The tensile strength of CE10 composites decreased when RH and AIN were added to the mixture at a weight ratio of 1:3, as expected. Figure 10 demonstrates that although CE7 and CE8 composites perform less well than CE11 and CE12, they have a better tensile strength than CE9 and CE10.In compared to AlN with epoxy resin, RH has superior adhesive characteristics, with the fillers functioning as stress raisers in tensile loading scenarios.

3.6 Flexural Strength

In the current study, the flexural resistance of hybrid epoxy-based materials reinforced with rice husk and aluminium nitride particles is investigated. Under flexural loading, the hybrid filler composite specimen is subjected to compression, tension, and shear stress. **Figure 11** illustrates, with examples, how filler loading and weight ratios affect the flexural values of resin-based composites. It has been proven that adding RH & AIN particles boosts composites' flexural strength by up to 20% of the filler's weight. The loss in flexural resistance at the weight ratio of 1:3(CE9 & CE10) could be caused by a number of factors, including greater particle-to-particle contact, an increased likelihood of void formation, and lower resin and particle adhesion. Flexural resistance has improved by a factor of 3:1 (CE11) as a result of an increase in RH content in the resin, which helps to diminish particle-to-particle contact and strengthens the adhesive force between AlN and resin. The best composition is provided by the 1:1 ratio when compared to other ratios.

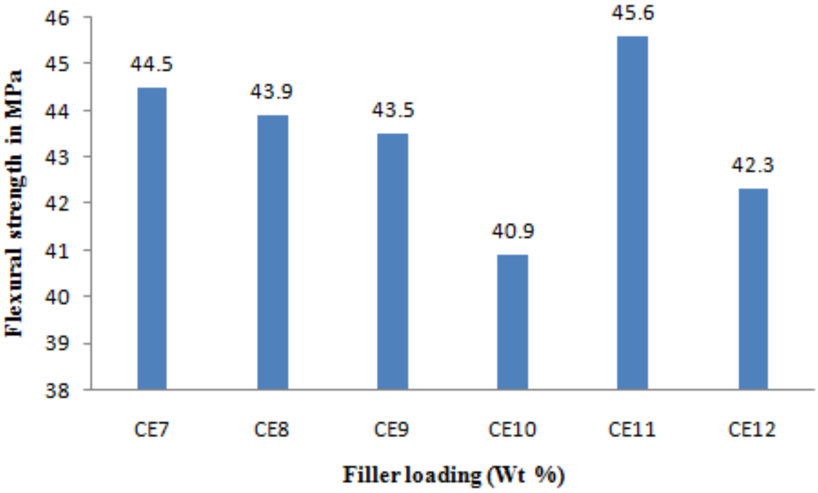

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Figure.11 Flexural (MPa) v/s Filler loading (wt%)

3.7 Micro-Hardness

One of its most crucial qualities, the hardness test, demonstrates how well composite with hybrid filler can tolerate being indented. Micro hardness levels were assessed for the current study project. **Figure 12** shows the specimen's results as a graph. The rigidity of the composite increases from 0.268 to 0.298 GPa when the ratio is 1:1 (CE7 & CE8). It proves it because the adhesive strength of the resin and filler has increased. Think about the 1:3 ratio (CE9 & CE10), where the hardness much improved over the earlier ratio. The findings imply that adding RH filler to AIN in a 1:3 composition increases the stiffness of the composite made of hybrid fillers. **Figure 12** illustrates how adding RH & AIN filler to the 3:1 composition increases hardness in a manner similar to the two previous ratios. At the end of the day, it demonstrates that rice husk is far harder than aluminium nitride. **Figure 12** displays RH and AlN at different weight-to-proportion ratios and demonstrates a steady rise in stiffness values with the addition of fillers.

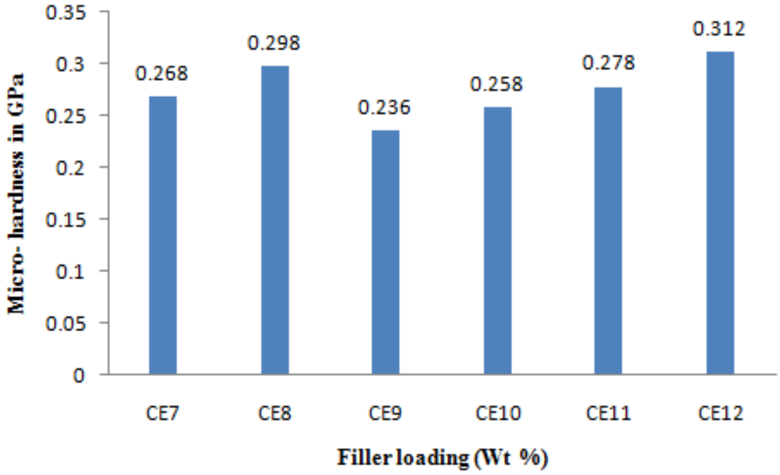

Figure.12 Micro-hardness (GPa) v/s Filler loading (wt %)

Conclusion

An extensive experimental examination has been done to assess the effect of hybrid particles on the mechanical and physical characteristics of hybrid polymer composites. RH and AlN are used in different weight ratios to create the specimens. The following is a list of the findings from the current research effort.

1. The volume fraction of voids is determined by the difference between theoretical and experimental density values. It has been observed that the composite weight ratio of 1:3 considerably increases both density and void fraction when compared to the other two weight ratios.
2. The interface between the RH and AlN particles and the epoxy resin is ineffective in carrying load, the tensile and flexural strength exhibit a decreasing trend as a result. The strength of the composites also decreases with increasing particle loading.
3. Micro-hardness values of hybrid composites filled with RH and AlN in varying weight ratios, which demonstrate a steady increase in hardness values with increasing filler content.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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