Exploring The Physical and Mechanical Properties of Rice Husk-Aluminium Nitride Hybrid Filler Composites

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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.**

***Index Terms*—Aluminium nitride, hybrid filler, mechanical and physical properties**

I. 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 [1-3]. 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 [3-6]. 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 [7-10]. 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 [11-14]. 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 [15-18]. 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 [19-21]. 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 [23]. 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 [24]. 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 number of voids in the hybrid composites was assessed.

II. EXPERIMENTAL

A. Materials

The composite was mostly made of epoxy, aluminium nitride, and rice husks. Rice husks and 25 micron-sized aluminium nitrides 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.

B. Treatment for 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.

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C. Sample preparation

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.%)

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.

D. Investigation Technique

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 employs 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.

III. RESULTS AND DISCUSSION

A. 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

B. 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 distribution 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 Fig. 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.

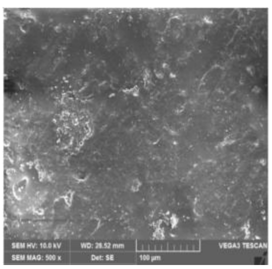
Fig 1 (CE08)

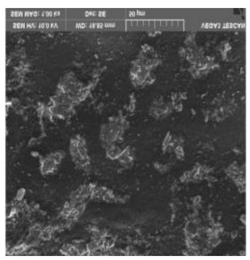
Fig 2 (CE10)

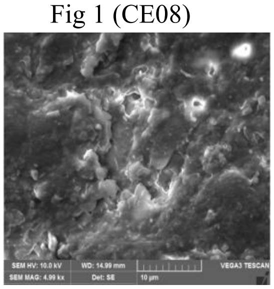
Fig 3 (CE12)

C. 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 Scherrer equation.

$$D = \frac{k.\lambda}{\beta \cos\theta} \qquad (1)$$

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.

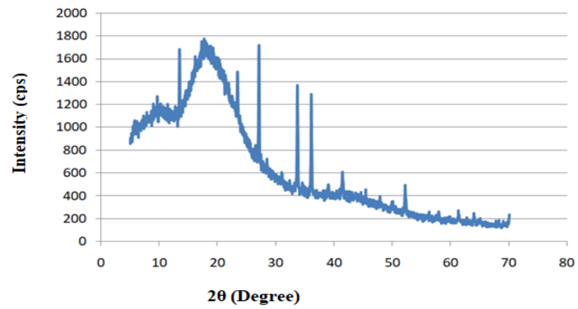

Figure. 7 (CE8)

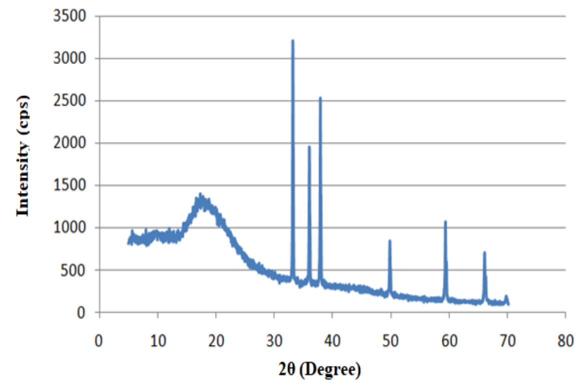

Figure. 8 (CE10)

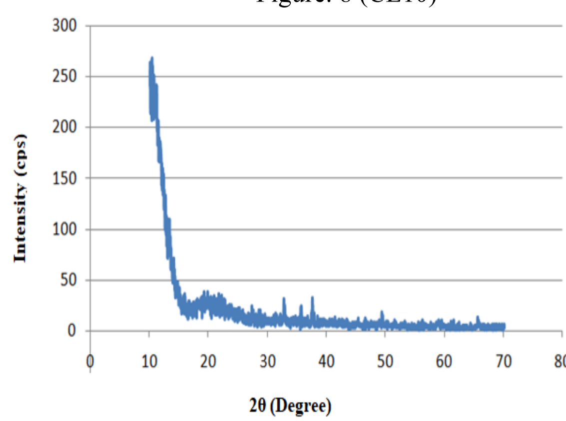

Figure.9 (C12)

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 composite that included AlN (20% weight), RH (20% weight), and epoxy resin (60% weight) in an amorphous form. This composite is depicted in Fig. 7. As shown in Fig. 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 Fig. 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.

D. Tensile Strength

Six hybrid composite specimens for the ratios of 1:1 (CE7 & CE8), 1:3 (CE9 & CE10), and 3:1 (CE11 & CE12) are shown in Figure 10's tensile strength test results. It depicts how the tensile strength steadily diminishes as the amount of filler material increases. The specimen CE11 composite with both fillers added in a weight ratio of 3:1 has the highest tensile strength when compared to the remaining composition. When RH and AIN were introduced to CE10 composites at a weight ratio of 1:3, the tensile strength fell. As can be seen in the accompanying Fig. 10, CE7 and CE8 composites perform less well than CE11 and CE12 but have higher tensile strength than CE9 and CE10.In comparison to AlN with epoxy resin, RH offers better adhesive properties, with the fillers acting as stress raisers in tensile loading situations

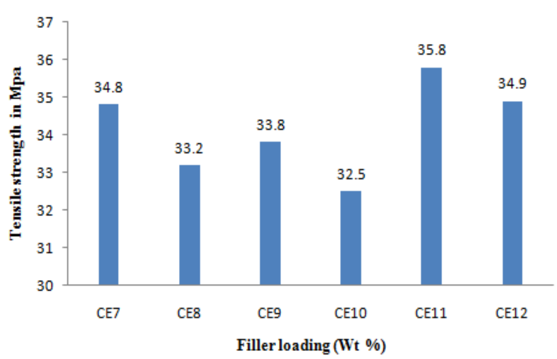

Figure.10 Tensile (MPa) v/s Filler loading (wt %)

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E. Flexural Strength

The current study examines the flexural resistance of epoxy-based hybrid composites reinforced with rice husk and aluminium nitride particles. The hybrid filler composite specimen is subjected to compression, tension, and shear stress under flexural loading [25]. The effects of filler loading and weight ratios on the flexural values of resin-based composites are shown in Figure 11 with examples. It has been demonstrated that including RH & AIN particles increases the flexural strength of composites by up to 20% weight of the fillers. Increased particle-to-particle contact, an increased risk of void formation, and decreased resin and particle adhesion are all possible effects of increasing the AlN filler loading, and they could all be factors in the fall in flexural resistance at the weight ratio of 1:3(CE9 & CE10). Due to an increase in RH content in the resin, which helps to reduce particle-to-particle contact and enhances the adhesive force between AlN and resin, flexural resistance has increased with a ratio of 3:1 (CE11). In comparison to other ratios, the 1:1 ratio offers the best composition

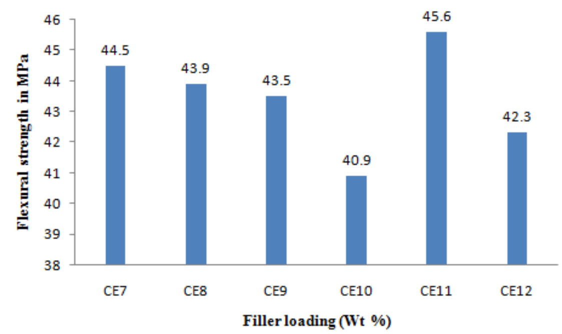

Figure.11 Flexural (MPa) v/s Filler loading (wt%)

E. Micro-hardness

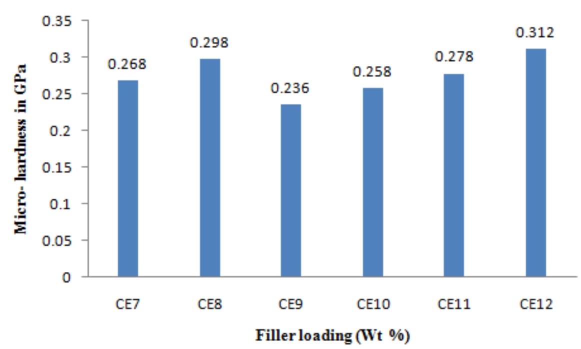

Figure.12 Micro-hardness (GPa) v/s Filler loading (wt %)

The hardness test, one of its most important characteristics, shows how well composite with hybrid filler can withstand being indented. In the current study project, micro hardness values were evaluated. The specimen's results are depicted in Fig. 12 as a graph. When the ratio is 1:1 (CE7 & CE8), the composite's stiffness rises from 0.268 to 0.298 GPa. It demonstrates that due to an increase in the resin and filler's adhesive strength. Consider the ratio of 1:3 (CE9 & CE10), where the hardness dramatically increased from the previous ratio. The results imply that the stiffness of the composite constructed of hybrid fillers is increased by adding RH filler to AIN in a 1:3 composition. As seen in fig. 12, the inclusion of RH & AIN filler in the 3:1 composition increases the hardness similarly to the previous two ratios. Ultimately, it shows that rice husk is far harder than aluminium nitride. Figure 12 shows RH and AlN in various weight-to-proportion ratios and shows a progressive increase in stiffness values as fillers are added.

IV. 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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