MICROSTRUCTURE AND MECHANICAL PROPERTIES OF VACUUM INFUSED RHA REINFORCED POLYMER COMPOSITES

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The synthesis and characterization of epoxy-based rice husk ash (RHA) polymer composites with enhanced mechanical properties have been discussed. RHA was used as a filler material (10, 20, 30, 40 and 50%) in Epoxy resin and hardener and then using vacuum infusion the composites are fabricated. The composites were then tested according to ASTM-D3039 standard for mechanical properties evaluation. The microstructure was observed by using an optical microscope. The 5 composites were produced successfully without any macro level defects. As the quantity of the RHA in the composites increased the percent elongation has reduced. This means that by increasing the percentage of RHA, brittleness of the composites also increases. The maximum tensile strength was found for the 20% of RHA composite i.e. 17.78 MPa. This composite has great potential for various applications in our day-to-day life usage. The dispersion of RHA micro particles within epoxy resin has controlled the extent of porosity, particle bonding with epoxy resin and tensile strength in resultant composites.

Key words: Polymer Composites; RHA Reinforcement; Epoxy Resin; Grain Structure; Mechanical Properties

1. Introduction

The epoxy-based composites are synthesized by reinforcing certain particulate form of material inside the matrix. The hybrid reinforcement approach is used to increase the mechanical properties such as high strength and stiffness. Composites have exceptionally unique applications in industries such as aviation, car manufacturing and construction industries [1-5]. Reinforcement in the context of composite materials refers to the element that gives the composite its strength, stiffness, and other desired mechanical properties. Usually, it is in the form of fibers, particles, sheets, or other structural components. The specific needs of the composite determine the choice of reinforcement material. Carbon fibers, glass fibers, aramid fibers, and natural fibers like hemp or bamboo are examples of common reinforcement materials. These fibers have a high tensile strength and stiffness, which improves the composite's overall mechanical performance. In this

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instance, rice husk ash (RHA) serves as the reinforcement, and strength is increased throughout when it is coupled with the matrix material, epoxy resin. The byproduct of burning the rice husk, which is the outer protective coverings of rice grains is known as rice husk ash. It is beneficial in a variety of applications due to its silica content and pozzolanic properties. It is used in the synthesis of silica-based nanoparticles, the manufacture of ceramics, insulation materials, and other fields. The RHA is a cost-effective material that aids in the long-term use of agricultural waste [6].

Epoxy resin is a common matrix material in composite materials. It is a type of thermosetting polymer. It is renowned for its exceptional mechanical, chemical, and adhesion properties. Epoxy monomers and curing agents react to form epoxy resins, which go through a cross linking process to create a rigid and long-lasting material. Epoxies are known for their adaptability and high-performance qualities, and these resins are widely used in sectors like construction, aerospace, automotive, and electronics [7-10]. For composite materials, the mechanical properties under tension are measured using tensile testing. Tensile testing helps to determine characteristics like tensile strength, elastic modulus, and elongation at break, offering important insights into the functionality and behavior of the material. The grain structure of a composite material pertains to the arrangement and alignment of its grains or particles. It has a significant impact on the mechanical and physical characteristics of the composite, including strength, stiffness, and resistance to crack propagation. The understanding of properties enhancement through optimizing the grain structure is essential for tailoring the performance of composite materials [12-16].

However, despite the potential of RHA as a reinforcement material in composites, there is a lack of comprehensive understanding of the physical, mechanical, and thermal properties of RHA composites. There is a need to investigate the properties of RHA composites and provide insights for their development and application in various fields. In this study, synthesis of RHA reinforced epoxy composites inheriting the consistent composition and performance is emphasized. Important goals of the study include characterizing the microstructure of the composites, evaluating their mechanical properties and understanding the effect of RHA content variation on mechanical properties. Finally, the aim of the utilization of RHA as a sustainable reinforcement in composite materials is to be evaluated.

2. Experimental Procedure

The mould fabrication, mixing of RHA, resin and hardener, vacuum infusion and curing of the composites are the important experimental stages for composites fabrication. For the composite's fabrication, initially a mould of dimension (283 mm x 45 mm x 11 mm) with a cavity of (250 mm x 25 mm x 3 mm) has been printed using 3D printing machine. The mould cavity is shown in fig. 1. The raw RHA was processed in the blender to convert the mixture into fine powdered grains. The ratio of epoxy resin and hardener was kept constant and RHA particles volume percentage was changed in each specimen. The composition for each specimen is mentioned in Table 1. The mixing of the RHA, resin and hardener was carried out through stirring for 3 minutes, with no bubble formations during the stirring process. Finally, the homogenous mixture of all these constituents was formed and ready for further process.

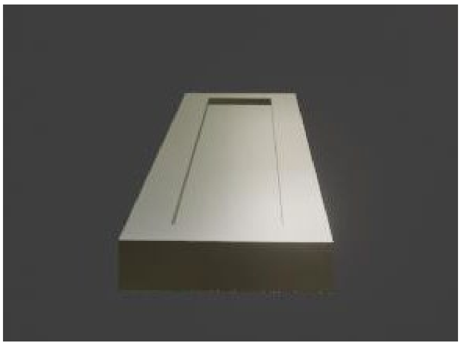

Fig. 1: 3D CAD model of Mould

Table 1

Composition of RHA, Epoxy Resin and Hardener

Sr. No	Composite specimen	Rice Husk Ash Composition (%Volume)	Epoxy Resin & Hardener, Composition (%Volume)
1)	D-1	10	90
2)	D-2	20	80
3)	D-3	30	70
4)	D-4	40	60
5)	D-5	50	50

The PVA release agent was applied on the mould to prevent sticking of the resin into the mould. The vacuum bag was applied on the top of the mould and sealed such that there was no air gap between mould and bag.

The resin-RHA solution was placed in a beaker, one tube is connected to the solution and other end is connected to vacuum bag such that resin can flow into the mould. The vacuum pump is connected to the mould via another tube connected to vacuum bag so that resin can flow through this tube. Then the vacuum pump was turned on, and due to pressure difference, the epoxy-RHA solution started to flow through the tube and eventually to the mould. The cavity in the mould is filled up with the RHA solution in expected shape. This procedure was done one at a time for each specimen. The vacuum bag and tube were removed afterwards, and the solution started to cure. To accelerate the curing process, it is placed into the curing furnace. The furnace temperature was set to 80°C. The curing took time of 3 hours. After curing, the specimen produced was removed from the mould. Thus, by this experimental method, the epoxy-based RHA reinforced five composites of size (250 mm x 25 mm x 3 mm) were produced.

After the fabrication of the specimens, it was placed under the optical microscope to study its microstructure. The inverted metallurgical optical microscope was used to study the granular structure of the material as well as its porosity. All specimens were tested for tensile testing on the universal testing machine under normal environmental conditions i.e. temperature 23°C and humidity of 55%. The specifications followed during tensile tests are indicated in Table 2. The vacuum infused epoxy-based RHA composites synthesized are shown in fig. 2. The composite manufacturing is successful as there are no macro level defects present on the surface of the composites.

Table 2

Specification of the Tensile Test

Parameter	Value
Stress Range	5000 MPA
Strain Range	500 %
Speed	50mm/min
Gauge length	150 mm
Elongation 1	50%
Elongation 2	100 %
Elongation 3	150 %
Elongation 4	200 %
Elongation 5	300 %
Approach Speed	1 mm/min
Preload	0 N

Fig. 2: Epoxy-RHA composite specimens

3. Results and Discussion

Five epoxy-based RHA reinforced composites D1 to D5 were successfully fabricated using vacuum infusion and no macro level defects were observed. As a part of investigation, the composites were cut and tensile specimens were tested for mechanical properties evaluation, and then the tensile results observed are correlated with composition and microstructural observations of the composites.

3.1 Tensile Tests

The composites were tested according to ASTM-D3039 standard [17] for mechanical properties evaluation. The stress vs strain curves were obtained for composite samples after doing the tensile tests, and the curves are shown in fig. 3-7, respectively. The D-2 composite with 20% RHA content demonstrates the maximum tensile strength of 17.78 MPa and elongation of 3.18 % amongst all the composites. The increase in the volume% of RHA reinforcement has reduced the percent elongation in the resultant composites, which indicates more brittleness induced with RHA content inside the epoxy matrix. However, the D-1 and D-2 composites showed their strength and elongation values comparable with slight increase in case of D-2.

The RHA content beyond 20% i.e. in case of D-3 and D-5 has led to the brittle failure without much elongation in early loading conditions. Thus, the composite ductility was reduced with RHA particles percentage increase. This means that by increasing the percentage of RHA, brittleness of the composites also increases. Amongst all the composites, the D-5 composite specimen inhibited least tensile strength of 3.734 MPa. Thus, D-5 with maximum RHA content i.e. 50% is the most brittle of all the specimens.

The tensile strength and percentage elongation values for all the five composites are enlisted in Table 3. Amongst all the composites, D-2 composite has good potential for utilization in various applications since its good strength and elasticity. The rice husk ash is readily available and affordable, making the composites suitable for mass production. In conclusion, the RHA composite with 20% RHA content is recommended to produce any epoxy-based product, as it also possesses desirable properties such as water resistance and insect resistance due to the presence of epoxy resin.

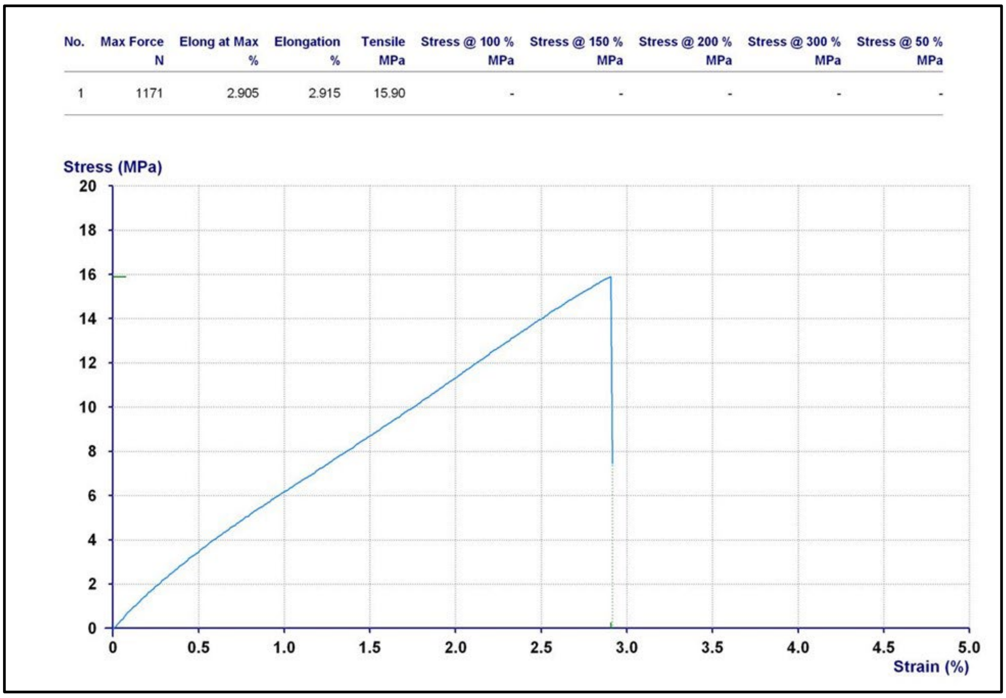

Fig. 3: Stress v/s strain curve for D-1 composite

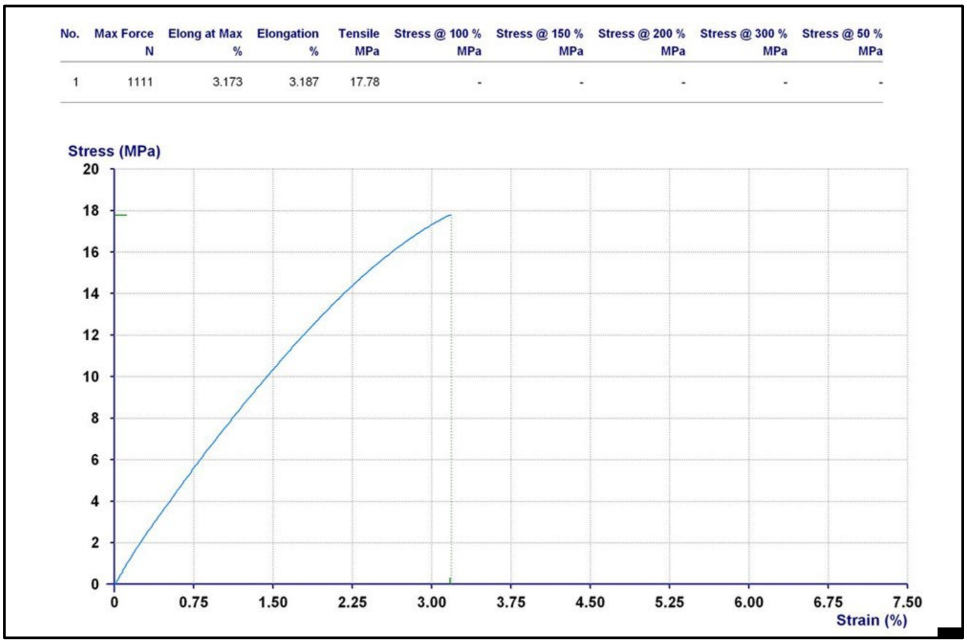

Fig. 4: Stress v/s strain curve for D-2 composite

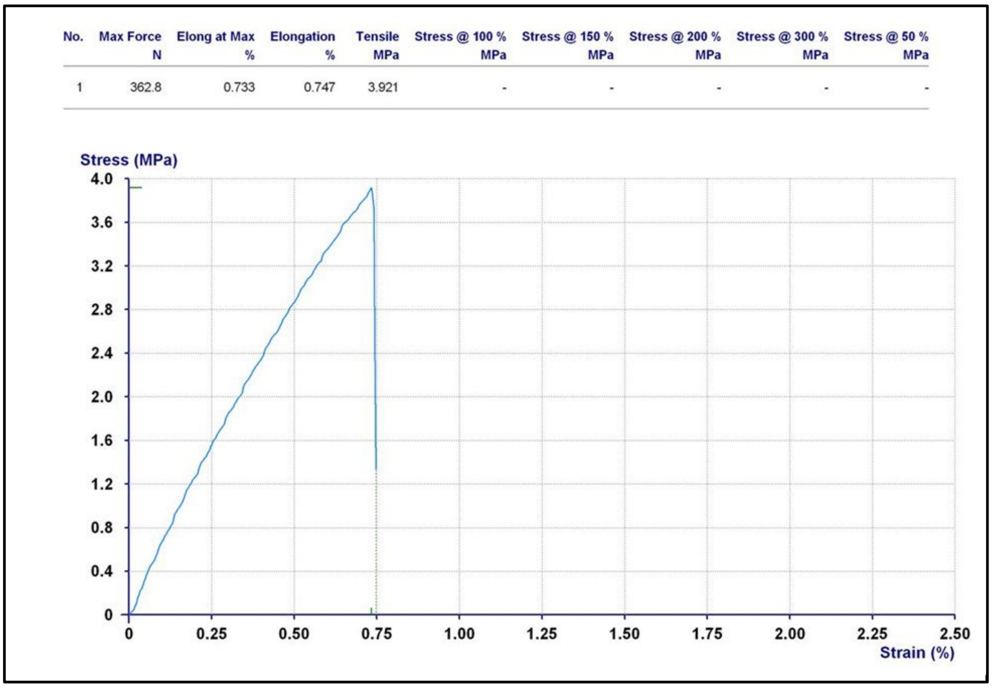

Fig. 5: Stress v/s strain curve for D-3 composite

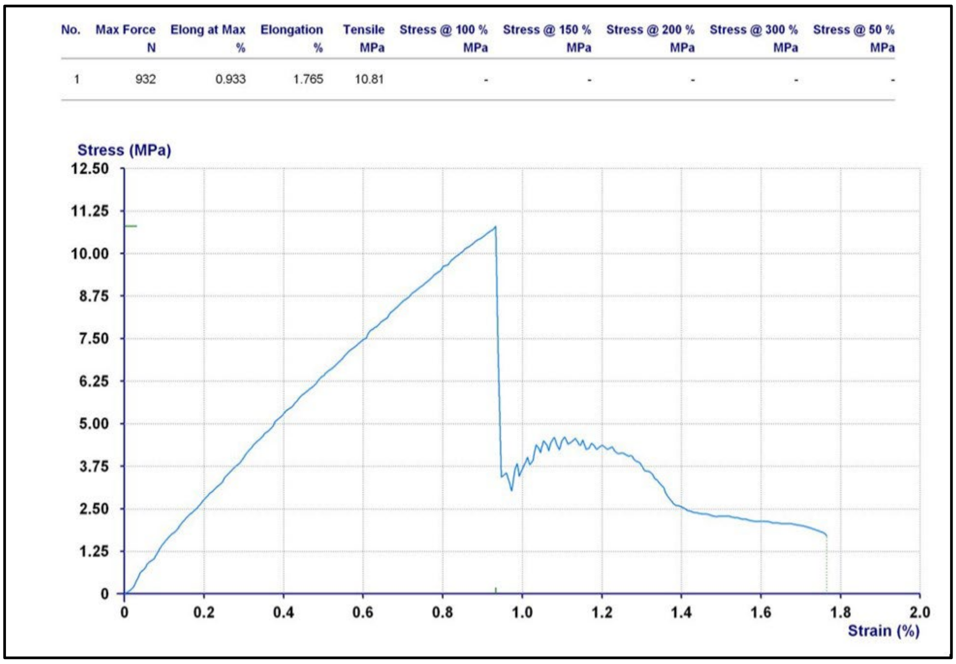

Fig. 6: Stress v/s strain curve for D-4 composite

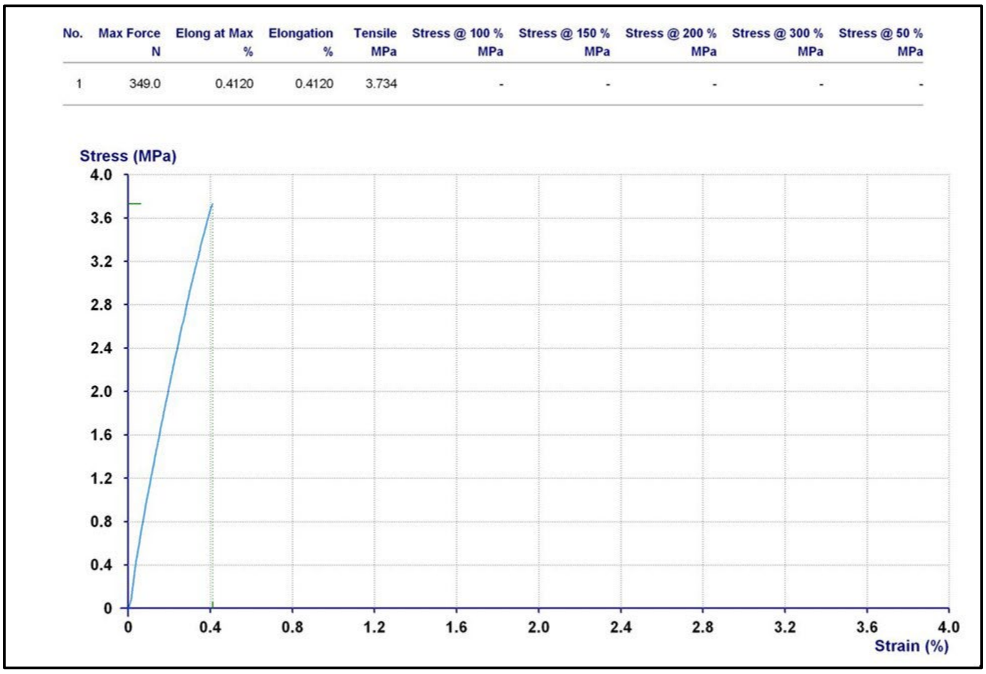

Fig. 7: Stress v/s strain for D-5 composite

Table 3

Tensile Test and Elongation Test Result

Specimen composition	Tensile strength (MPa)	Elongation (%)
10%	15.90	2.915
20%	17.78	3.187
30%	3.92	0.747
40%	10.81	1.765
50%	3.734	0.4120

3.2 Microstructure

The optical microscopic analysis of the epoxy composite surfaces was conducted using Dewinter DMI-PRIME optical microscope. The micrographs of the composites are as shown in fig. 8-12. All micrographs are taken for magnification of 100 μm. The RHA particles dispersion varies in different composition of the composites. The microstructure of each composite surface is as shown in fig. 8 to fig. 12, respectively. The D-1 and D-3 sample exhibits few dark small sized RHA particle agglomerates. Moreover, the dark agglomerated zones are more prominent for the D-4 to D-5 composites because of large quantity of the RHA powder added in the epoxy resin matrix. The agglomerate inside the composites leads to more porosity and loose bonding of RHA particles with the epoxy matrix. These agglomerates regions are susceptible to brittle failure during early loading conditions. The strength is gained by the grains size refinement in the composites due to dispersion of RHA particles. However, the loose bonded RHA particles agglomerates act adversely and deteriorate the composite's resultant mechanical properties.

As per the tensile test results, the content of RHA particles was limited to 20% for better ultimate tensile strength and elongation. The RHA particles content till 20% was able to disperse in a way that particles bonding with epoxy resin was good enough and agglomerates formation was not prominent. However, in case of the RHA content more than 20%, i.e. for D-3 to D-5 samples, the microstructure exhibits the dark agglomerates more prominently visible. For these samples, the benefits of grain size refinement are overcome by increased porosity at the agglomerates due to loose particles bonding. These agglomerates zones are prone to brittle failure during tensile tests. The optical micrographs of fig. 13 to fig. 17 indicate that the grains sizes are different in the resultant composites. The particles bonding between the matrix and the reinforcement i.e. between RHA and Epoxy resin in case of D-4 and D-5 sample observed to be weaker compared to other composite samples, as the surface micrograph indicate loose particles agglomerated on the surface. The high percentage of RHA was not homogenously distributed and led to the weak bonding between particles and epoxy resin.

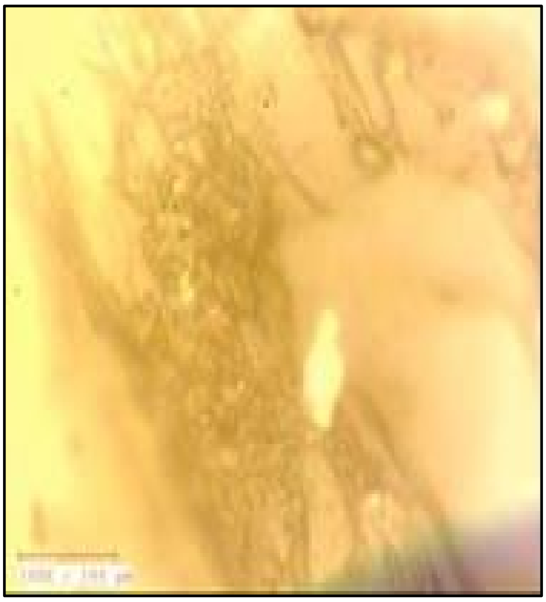

Fig. 8 D-1(10%RHA) surface micrograph

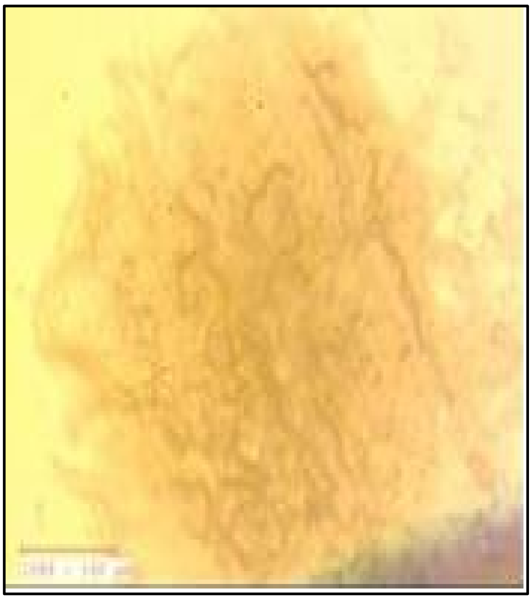

Fig. 9 D-2 (20% RHA) surface micrograph

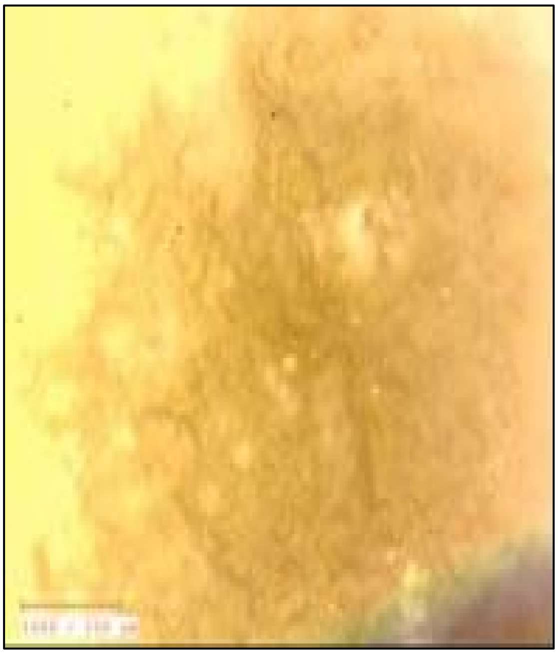

Fig. 10 D-3 (30% RHA) surface micrograph

Fig. 11 D-4 (40% RHA) surface micrograph

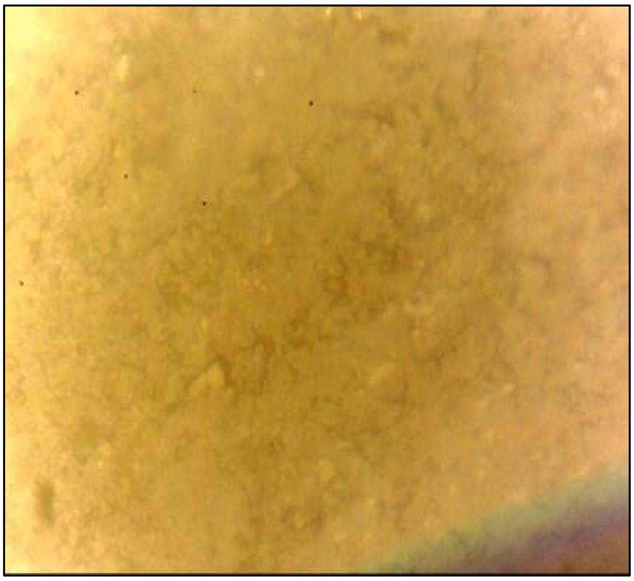

Fig. 12 D-5 composite (50% RHA) surface micrograph

The microscopic images of the composites indicated in fig. 8 to fig. 12 are analyzed in Dewinter microscope's image analyzer for understanding each composite's grain size distribution. The grain structures of the composites D-1 to D-5 are shown in analyzed images indicated in fig. 13 to fig. 17, respectively. The optical micrographs are mapped on the grid by image analyzer software and the different sizes of the grains are traced for each composite sample. It is observed that specimens having 20% to 50% RHA content have a mixture of coarse and refined grain structure. The coarse grains at the center and surrounded by fine grains. The maximum coarse grains are observed for 10% composite as shown in fig. 13. There are very few fine grains observed on D-1 composite surface. Then for increasing the reinforcement percentage, the extent of the grain's refinement increased because of grain pinning by reinforcement particles.

In conclusion, the microstructural observations indicate that there is tradeoff between two phenomena for controlling the mechanical properties. One of them is optimum amount of the RHA particles dispersion inside the epoxy with good bonding, which exhibits enhanced strengthening through grain size refinement. The second phenomenon of loose particles bonding with matrix due to exceeding RHA content inside matrix leading to agglomerates, which deteriorates the strengthening through increased porosity. In this study, the optimum RHA content that can be encapsulated for enhanced strength is up to 20% volume of the resultant composite.

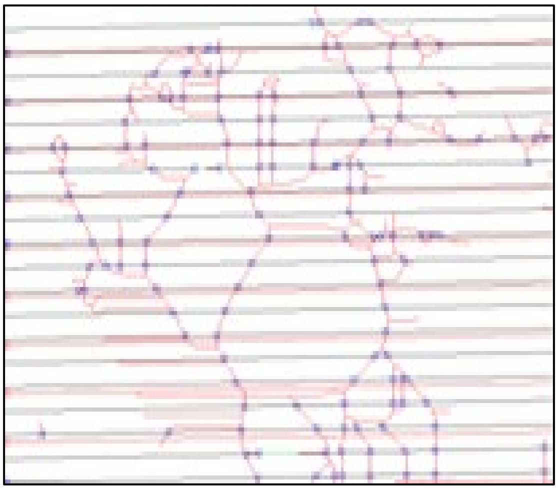

Fig. 13 Grain structure of D-1 composite

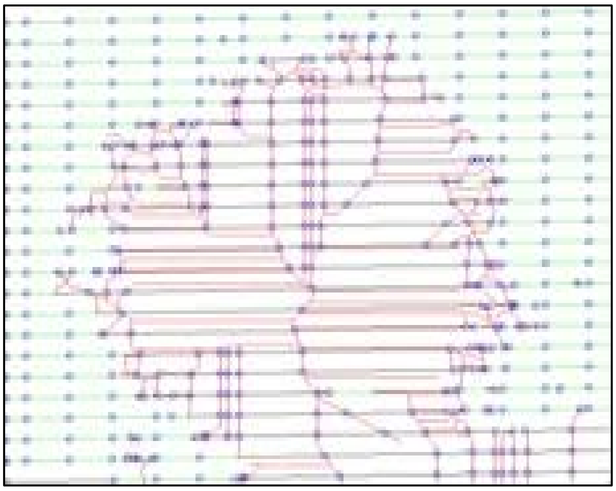

Fig. 14 Grain structure of D-2 composite

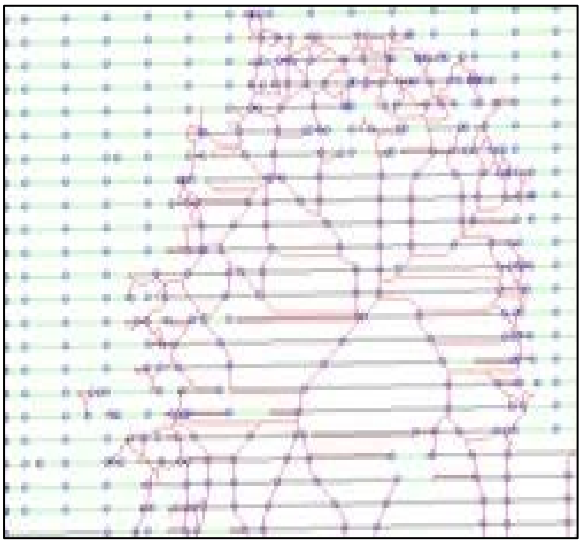

Fig. 15 Grain structure of D-3 composite

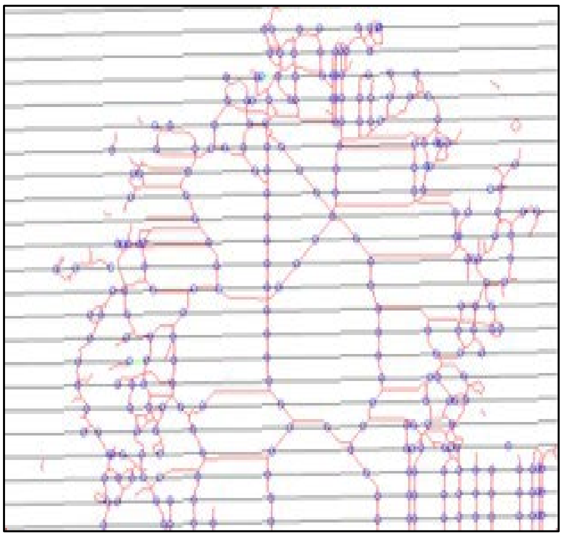

Fig. 16 Grain structure of D-4 composite

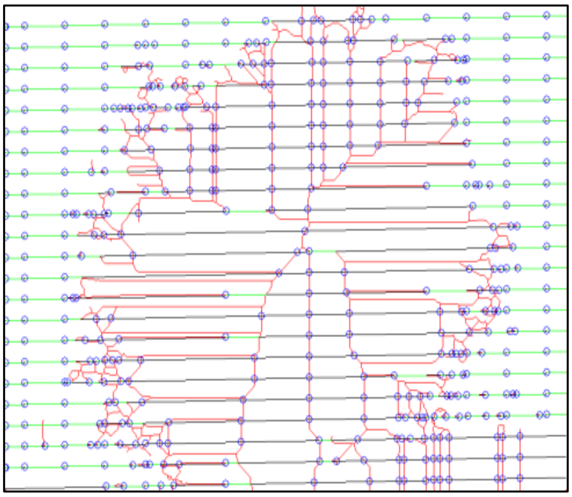

Fig. 17 Grain structure of D-5 composite (50% RHA)

4. Conclusions

The agricultural waste rice-husk ash has been successfully utilized for enhancing the mechanical properties of the vacuum infused epoxy-based polymer composites. The RHA content must be restricted to 20% volume of resultant composite, to exhibit mechanical properties enhancement through strengthening by grain size refinement. The agglomeration and loose particles bonding are observed prominent inside the composites with more than 20% RHA volume content. The maximum strength of 17.78 MPa and elongation of 3.18% obtained for D-2 composite with 20% RHA content. Thus, this study demonstrates feasibility and potential of utilizing epoxy-based RHA composites in practical applications, offering an environment friendly and cost-effective alternative to traditional materials.

REFERENCES

[1] *Egbo, M.K.,* A fundamental review on composite materials and some of their applications in biomedical engineering. Journal of King Saud University - Engineering Sciences, vol.33, no.8, pp.557-568, 2021. https://doi.org/10.1016/j.jksues.2020.07.007

[2] *Barbero, E.J.,* Introduction to Composite Materials Design. CRC Press, Third Edition, 2017. https://doi.org/10.1201/9781315296494

[3] *Hsissou, R., Seghiri, R., Benzekri, Z., Hilali, M., Rafik, M., Elharfi, A.,* Polymer composite materials: A comprehensive review. Composite Structures, vol.262, pp. 113640, 2021. https://doi.org/10.1016/j.compstruct.2021.113640

[4] *Singh, P., Raghavender, V., Joshi, S., Nikale, P.V., Awasthi, A., Nagpal, A., Al-saheb, A.J.A.,* Composite material: A review over current development and automotive application.

Materials Today: Proceedings, 2023. https://doi.org/10.1016/j.matpr.2023.11.012

[5] *Shubham, Ray, B.C.,* Introduction to Composite Materials. In: Fiber Reinforced Polymer (FRP) Composites in Ballistic Protection. Engineering Materials, Springer, 2024. https://doi.org/10.1007/978-981-99-9746-6_1

[6] *Ahmed, S., Jones, F.R.,* A review of particulate reinforcement theories for polymer composites. Journal of Material Science, vol.25, pp.4933–4942, 1990. https://doi.org/10.1007/BF00580110

[7] *Raju, A., Shanmugaraja, M.,* Recent researches in fiber reinforced composite materials: A review. Materials Today: Proceedings, vol.46, no.19, pp.9291-9296, 2021. https://doi.org/10.1016/j.matpr.2020.02.141

[8] *Peters, S. T.,* Handbook of composites. Springer New York, ISBN 978-1-4615-6389-1, 1998. https://doi.org/10.1007/978-1-4615-6389-1

[9] *Amran, M., Fediuk, R., Murali, G., Vatin, N., Karelina, M., Ozbakkaloglu, T., Krishna, R.S., Sahoo, A.K., Das, S.K., Mishra, J.,* Rice Husk Ash-Based Concrete Composites: A Critical Review of their Properties and Applications. Crystals, vol.11, no.2, 2021. https://doi.org/10.3390/cryst11020168

[10] *May, C.,* Epoxy Resins: Chemistry and Technology, Routledge, Second Edition, 1998. https://doi.org/10.1201/9780203756713

[11] *Jin, F.L., Li, X., Park, S.J.,* Synthesis and application of epoxy resins: A review. Journal of Industrial and Engineering Chemistry, vol.29, pp.1-11,2015. https://doi.org/10.1016/j.jiec.2015.03.026

[12] *Zhang, C., Cui, J., Sui, W., Gong, Y., Liu, H., Ao, Y., Shang, L.,* High heat Resistance, Strength, and toughness of epoxy resin with cellulose nanofibers and structurally designed ionic liquid. Chemical Engineering Journal, vol.478, 2023. https://doi.org/10.1016/j.cej.2023.147063

[13] *Panda, H.,* Epoxy Resins Technology Handbook (Manufacturing Process, Synthesis, Epoxy Resin Adhesives and Epoxy Coatings). Asia Pacific Business Press Inc, Second Revised Edition, 2019. ISBN 8178331829, 9788178331829

[14] *Karle, A.H., Patil, N.A., Arakerimath, R.,* Modelling of Density and Tensile Strength of Wollastonite-Filled Epoxy Composites. In: ICREEM-2022, Lecture Notes in Mechanical Engineering. Springer, 2024. https://doi.org/10.1007/978-981-99-5946-4_12

[15] *Sadeq, N.S., Mohammad Salih, Z.G., Mohammed, R.H.,* Effect of grain size on the structure and properties of coir epoxy composites. SN Applied Sciences, vol.2, no.1191, 2020. https://doi.org/10.1007/s42452-020-2991-x

[16] *Liu, H., Chai, M., Wu, Y., Zhang, P., Liu, M., Liu, L., Huang, Y.,* Highly flame retardant, strength, and tough epoxy resins and CFRPs via introducing a self-assembly novel ionic bond, rigid-flexible structure PPI. Chemical Engineering Journal, vol.584, no.152415, 2024. https://doi.org/10.1016/j.cej.2024.152415

[17] ASTM D3039/D3039M-17, Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials. DOI: 10.1520/D3039_D3039M-17