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Exploring seashell and rice husk waste for lightweight hybrid biocomposites: synthesis, microstructure, and mechanical performance

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Abstract

Hybrid composites are made by fusing together, typically using resin, a matrix material (typically metal), a fiber, and a filler component. Fibers and particles are encased in a matrix of another material to create modern composites. Natural fiber composites are becoming increasingly popular due to rising awareness of their many practical applications. The debris produced by the seashell farming becomes serious environmental threat. Recent research has centered on the potential applications of this seashell waste. The purpose is to reduce seashell waste that pollutes the coast near Kanyakumari. Agricultural waste, such rice husk, is more accessible than other types of biomass. Conventional materials are weighed more, so lightweight materials can be used as alternatives for the structural components of an automobile. This swatch is made from combination of biocomposite and repurposed seashells. Mechanical tests, including tensile, flexural, impact, and hardness testing, were performed on the prepared samples. The morphological analysis shows good laminar and interfacial connections throughout the structure. The EDAX spectrum shows the presence of elements like silicon, sulfur, and zinc. The EDAX spectrum of C5 hybrid biocomposites (40% rice husk + 10% seashell + 50% polyester resin) has more zinc than silicon. The C2 (10% rice husk + 40% seashell + 50% polyester resin) hybrid composite outperforms other composites in tensile strength (51.47 MPa), Brinell hardness (132BHN), Rockwell hardness (62RHN), impact energy (51.4 J), flexural strength (203.03Mpa), and water absorption (1%). Based on research investigations, hybrid biocomposites made of bio seashell and bio rice husk are superior than standard biocomposites without sacrificing the eco-friendliness of the automobile.

Keywords Bio seashell \cdot Tensile \cdot Impact \cdot Hardness \cdot Rice husk \cdot SEM

1 Introduction

Rotator cuff tears are commonly repaired using biodegradable suture anchors; however, these devices have a number of drawbacks, including weak mechanical strength, poor osteon integration, and the generation of acidic breakdown byproducts [1]. Composites are defined macroscopically as a heterogeneous combination of two or more materials with distinct chemical, morphological, and physical properties. The goal is to produce materials having novel characteristics that cannot be manufactured using conventional methods alone [2]. There is a need for materials with better mechanical, tribological, and biological qualities [3, 4] to replace polyethylene in artificial joint applications. There has been a rise in interest in the usage of sustainable biocomposites in recent years [4] as a result of the environmental burden caused by the use of non-renewable carbon or glass fiber composites. Natural fibers have been utilized in various architectural and structural applications. In recent times, there has been a growing utilization of cellulosic products and wastes as fillers in polymers, mostly driven by the objective of achieving cost savings and introducing favorable characteristics. The utilization of numerous natural fiber reinforcements in hybridization has been acknowledged as a promising method for the production of composites.

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Furthermore, it has the potential to improve specific mechanical characteristics [5].

Because of their low density, good specific mechanical properties, global availability, low cost, and favorable environmental profile [6], natural fibers are being evaluated as a possible replacement for conventional composite reinforcements. Biodegradable polymers polystyrene (PS) has been increasingly acknowledged as effective materials for preventing environmental pollution problems, especially when compared to traditional petrochemical-based polymers [7]. To combat this worldwide environmental problem, material scientists are honing in on the creation of eco-sustainable materials [8]. Rising environmental concerns have pushed the development and application of biodegradable composites to the forefront of scientific inquiry. Researchers need to know how the composition of biocomposites affects their performance. Benefits of biodegradable composites include longevity, light weight, and outstanding mechanical performance [9]. These composites are fabricated using sustainable biomass and high-performance polymers.

When combined with the right eco-friendly and/or renewable biopolymers, they allow the development of fascinating totally sustainable biocomposites that are compostable or biodegradable [10]. It was looked into that Brewers spent grain (BSG) might be used as a biofiller in biocomposites using a polycaprolactone (PCL) matrix. The biocomposite strongly filled with PCL/BSG was fabricated by meltcompounding method, gives significant performance for mechanical (tensile strength, elongation at break, hardness) and thermal properties and water absorption properties [11]. Because of their advantageous unique mechanical properties, fascinating viscoelastic and acoustic damping performances, and lower environmental impact during processing and use, as well as at the end of their life cycle [12], natural fibers are a promising alternative to synthetic fibers in technical textiles and composites applications.

Therefore, innovative, sustainable, and renewable materials that can swiftly and safely disintegrate in the atmosphere are needed as substitutes for compounds generated from petroleum [13]. The use of physical modification techniques for the processing of plant fibers is becoming increasingly prevalent. In these procedures, electrical discharge results in fibrillation of the bamboo fibers [14]. Over the past few decades, there has been a rise in the use of biomaterials for the restoration and replacement of damaged or diseased body components in an effort to increase human lifespans. Implants, tissue engineering, drug delivery, bone regeneration, gene therapy, and biosensors are just a few examples of the many ways in which biomaterials have been put to use [15]. Researchers are incorporating nanofillers to biopolymer-based composites in modest amounts. As additions, these nanofillers improve the mechanical, thermal, flame retardancy, and water absorption properties of nano composite materials and preserve their optimal density [16].

An increasing focus on environmental sustainability has prompted a paradigm shift toward biofiber-reinforced biocomposites in the field of fiber-reinforced composites. The biodegradability, renewability of precursor materials, greenhouse gas emissions, and low manufacturing energy of biofiber-reinforced composites outweigh those of conventional synthetic fiber-reinforced composites [17]. The fabricated specimen is subjected to SEM analysis and reveals that it has smooth surface, absence of porosity, and excellent binding characteristics [18]. It has been found that a novel hybrid composite can help keep the environment healthy while also being useful in a variety of nature-based applications [19].

The objective of this work is to use bio seashell and rice husk to fabricate the ceramic composites for automobile structures. The automobile structures were made up of new materials and the cost is high. To reduce this cost, discarded materials can be recycled to fabricate the composite materials. Seashell and rice husk can be obtained abundantly in seashore as well as in agricultural farm. Natural seashell and rick husk are corrosion resistance in nature. The utilization of these discarded biomaterials in composite material will increase the stiffness and strength of the material. The incorporation of bio seashell and rice husk into ceramic hybrid composites improves mechanical strength, adhesion, corrosion resistance, and the life span of automobile structures.

2 Materials and experiments

This section addresses the production process for hybrid composites and the experimental techniques used to characterize their mechanical properties and morphological behavior. Raw materials included waste products like seashells, rice husks, polyester resin and hardener, molds, and paraffin wax. The size of the mold used in this fabrication is $300 \text{ mm} \times 225 \text{ mm} \times 6 \text{ mm}$. Figures 1 and 2 show the image of raw bio seashell and the bio rice husk.

A sea creature's shell is a more robust, protective shell and found abundance in seashore. After 3 days of drying in the sun, the shells were ready to be ground. A hand sieve of varying micron diameters was used to sort the shells. The shells are reduced to a powder by being crushed. The raw rice species obtained from the agricultural land is processed and rick husk is obtained. Because of the difficulty of disposing it, it poses a threat to air quality. Table 1 shows the chemical composition of ground seashell. The particle size is measured using the laser diffraction (LD) method. Laser diffraction analyzers are used for particles in a material within





Fig. 3 Particle size distribution

Table 2Chemical compositionof natural fiber	Fiber (%)	Rice husk
	Cellulose (wt)	35–45
	Hemicellulose (wt)	19–25
	Lignin (wt)	20

Fig. 1 Bio seashell



Fig. 2 Bio rice husk

of ground seashells

Table 1 Chemical composition

Oxide	Percentage (%)
SiO ₂	1.60
Al_2O_3	0.92
CaO	51.56
MgO	1.43
Na ₂ O	0.08
K ₂ O	0.06
H ₂ O	0.31
LOI	41.84

the range of 10 to 3 mm. The size of the rice husk particle and seashell particle is 82 μ m and 70 μ m respectively as shown in Fig. 3.

Table 3 Chemical composition of seashell powder

Elements	Percentage (%)
Calcium carbonate	95
Chitin	2.8
Amino acid	1.3
Polysaccharides	0.9

2.1 Synthesis of bio seashell and bio rice husk

Seashells were used in the synthesis of bio seashell nanoparticles via grinding and heavy ball milling. To convert calcium carbonate (CaCO₃) to calcium oxide (CaO), carbon-di-oxide (CO₂) to evolve, and other organic chemicals to decay, the resulting shell powder was sintered and heat treated in a muffle furnace at 700 °C for 2 h. The release of calcium carbonate results in the creation of calcium oxide, as shown by the following equation [20]:

$$CaCO_3 \rightarrow CaO + CO_2$$
 (1)

By grinding the raw husk, seashells can be used in the synthesis of bio rice husk nanoparticles. Rice husk and seashell powder are heat treated in a muffle furnace at 700 $^{\circ}$ C for 2 h. In agricultural sector, we can obtain the rice husk abundantly when compared with other biomass products [21]. Tables 2 and 3 show the chemical composition of the powdered rice husk and seashell.

The composite material under study is made up of polyester resins and filler. Unsaturated polyester was used to

 Table 4
 Designation of composites compositions in weight percentage

Composites	Polyester resin	Seashell	Rice husk
C1	50%	50%	-
C2	50%	40%	10%
C3	50%	30%	20%
C4	50%	20%	30%
C5	50%	10%	40%

create the samples, and methyl ethyl ketone peroxide and cobalt nephthenate were added as a catalyst and an accelerator, respectively; ground up waste seashells were also used as filler. Consumption waste, such as discarded seashells, is common along the coast. The substance is firm, hard, and fragile. A polymeric ester is the product of the reaction between a dihydric alcohol like ethylene glycol and an aromatic acid like phthalic acid. Methyl ethyl ketone peroxide (MEKP) is an organic peroxide with explosive qualities similar to those of acetone peroxide. MEKP is a colorless, oily liquid. MEKP has improved stability in storage and is more resistant to stress and temperature changes. It is something that, when added in the right amounts, causes polyester resin to cure and harden. Extreme caution was exercised when combining the MEKP and the resin so that the latter would not fly out of the mixing jar. Instead of using a syringe, which may potentially fly back out of the bottle and into the patient's eyes, a measuring cup was utilized. To ensure appropriate curing, it was extensively included into the resin. Without it, some areas of the resin would not be catalyzed. The MEKP utilized was readily accessible on the market. Cobalt II ethyl hexanoate(acid) was used as a polyester initiator and accelerator in the composite's production. Paraffin wax served as the release agent for the mold. The mold is made up of four galvanized metal sheets that measure $300 \text{ mm} \times 225 \text{ mm} \times 6 \text{ mm}$. Table 4 shows the different combinations of fabricated hybrid biocomposites with various proportions.

3 Results and discussion

3.1 Tensile test

Figure 3 displays the experimental tensile strength data. The tensile strength of composite C2 is greater than that of other composites. Figure 4 displays the minimal tensile strengths that can be achieved with composites. The procedure follows the guidelines laid out in ASTM D638. Figure 5 illustrates the sized specimen of the fabricated hybrid biocomposites for tensile test.



Fig. 4 Tensile strength of the bio hybrid composites



Fig. 5 Tensile specimen

Table 5	Tensile	strength	of the	bio	hybrid	composite
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Composites	Tensile strength (MPa)				
	Trial 1	Trial 2	Trial 3	Average	
C1	45.13	46.48	45.86	45.82	
C2	50.64	52.42	51.35	51.47	
C3	41.4	41.82	42.08	41.77	
C4	40.03	41.52	40.46	40.67	
C5	38.84	39.47	39.81	39.37	

The dimensions of the sample used in the test are 210 mm in length, 15 mm in breadth, and 6 mm in thickness. The tensile test is carried out with the aid of the Universal Testing Machine (UTM). Table 5 represents the different trials of tensile test carried out and its average. Tensile strengths of 40.03 MPa, 50.6 MPa, 39.8 MPa, 41.4 MPa, and 45.13 MPa are recorded for specimens C1, C2, C3, C4, and C5, respectively. The results show that specimen C2 is the superior one and the material is strengthened because of the incorporation of bio seashell (50%) nanoparticle and bio rice husk (10%) in the hybrid biocomposites [22].

3.2 Hardness test

In order to evaluate the inherent characteristics of a material that occurs during the deformation under the influence of an externally applied load, mechanical properties were examined.



Fig. 6 Hardness specimen



Fig. 7 Brinell and Rockwell number for various compositions

In general, the material's strength, hardness, impact, roughness, ductility, and fracture were examined. Hardness measurements were obtained using Brinell and Rockwell scale. The sized specimen used for this performance is shown in Fig. 6.

3.3 Brinell hardness test

The Brinell method, as specified by the ASTM standard, was used to determine the hardness of a material having an abnormally coarse or rough surface. Figure 7 displays the hardness values of specimens C1, C2, C3, C4, and C5 to

be 125HB, 132HB, 120HB, 127HB, and 130HB, respectively. The results show that specimen C2 is the superior one. Avoiding vacuum is a priority for it. The material is strengthened because of the incorporation of bio seashell nanoparticle and bio rice husk in the hybrid biocomposites.

3.4 Rockwell hardness test

Indender penetration depth was used to calculate Rockwell hardness under light and heavy loads. The procedure follows the standards set by the ASTM. The Rockwell hardness method was used to measure the hardness of the fabricated hybrid composite material. Rockwell hardness values of 56HR, 62HR, 53HR, 58HR, and 61HR are recorded for specimens C1, C2, C3, C4, and C5, respectively shown in Fig. 7. The results show that specimen C2 is the superior one. The material is strengthened because of the incorporation of bio seashell nanoparticle and bio rice husk in the hybrid biocomposites.

3.5 Impact test

The impact resistance of seashell biocomposites performance is shown in Fig. 8. For this impact analysis, we employ the Izod impact measurement unit. The sized specimen for impact resistance is shown in Fig. 9. Impact strengths range from 45.9 to 51.4 J to 42.6 J to 47.8 J to 50.3 J for specimens C1, C2, C3, C4, and C5, respectively. When comparing these results, specimen C2 stands out as having greater intensity due to the small particle size. The hybrid biocomposites exhibit enhanced strength as a result of the integration of bio seashell nanoparticles and bio rice husk into the composite material.

3.6 Flexural test

Figure 10 depicts the flexural strength values of the fabricated hybrid biocomposites. The procedure follows the standards set out by ASTM D790. The flexural strengths of specimens C1, C2, C3, C4, and C5 range 139.93, 203.03, 129.9, 138.86, and 158.74 MPa respectively. When comparing these results, specimen C2 stands out as having greater intensity due to the small particle size. Avoiding vacuum is a priority for it. The material is strengthened because of the incorporation of bio seashell nanoparticle and bio rice husk in the hybrid biocomposites. The flexural test was conducted experimentally, as shown in Fig. 11.

3.7 Water absorption capacity in sea water

The formula used to calculate the water absorption experiment is

$$W_{Absorption} = \frac{\text{Intial Weight} - \text{Final Weight}}{\text{Initial Weight}} \times 100$$
(2)







Fig. 9 Impact test specimen



Fig. 10 Flexural strength for various compositions

The results of the weight absorption experiment are displayed in Fig. 12. It shows that the specimens C2 and C5 absorb less sea water (0.434%) than the others do. The C2 and C5 show the incorporation of 40% of bio seashell and 40% bio rice husk respectively.



Fig. 11 Flexural test specimen



Fig. 12 Water absorption experimental result

3.8 Characterization techniques

3.8.1 Morphological structure—scanning electron microscope (SEM)

The SEM micrographs of the polished C2 specimen are displayed in Fig. 13, split evenly between polyester resin (50%) and bio seashell (40%) and bio rice husk (10%). The results show that the nanoparticle size of the bio seashells integrates smoothly with the polyester resin. It is indicative of the fact

Fig. 13 SEM image of specimen C2





Fig. 14 SEM image of specimen C5

that the hybrid composite's resin, seashell, and negligible amount of rice husk form excellent interfacial bonds. Morphological analysis reveals a vanishingly small number of voids. The mixing capacity and sintering of the bio seashell nanoparticles contribute greatly to the outstanding laminar interface between the seashell and resin.

The SEM micrographs of the polished C5 specimen are shown in Fig. 14. The particle size distribution of the polyester resin and bio rice husk shows that the two work well together. It is indicative of the fact that the hybrid composite's resin, seashell, and negligible amount of rice husk form excellent interfacial bonds. After being bleached, cellulose's surface took on the appearance of a regular porous nanostructure with uniformly distributed individual crystalline particles. Morphological analysis revealed that, compared to C2, the nanoscale particle size of C5 hybrid composites results in the occurrence of tiny voids. The bond between the rice husk and resin, which is Laminar, is quite strong. The hybrid composite's smooth surface was dotted with white specks that looked like rice husk and nano bio seashell.

Table 6	EDAX	of specimen	C_2
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Element	Net counts	Weight %	Atom %
Si	33,190	49.60	65.82
S	4687	9.20	10.69
Zn	2121	41.20	23.49
Total		100.00	100.00

3.8.2 Energy-dispersive X-ray (EDAX) spectroscopy

The EDAX analysis reveals the incorporation of inorganic materials into the manufactured hybrid composites. There are fifty percent polyester resins, fourty percent bio seashell, and ten percent bio rice husk in the C2 hybrid composite. The composites include the inorganic elements Si (49.60 wt%), S (9.20 wt%), and Zn (41.20 wt%), as shown in Table 6. The EDAX studies performed to determine the inorganic components of the hybrid composites yielded the spectrum depicted in Fig. 15.



 Table 7
 EDAX of specimen C5

Fig. 15 EDAX image of speci-

men C2

Element	Net counts	Weight %	Atom %	
Si	8370	18.92	30.93	
S	7114	16.63	23.81	
Zn	2676	64.46	45.26	
Total		100.00	100.00	

The EDAX analysis reveals the incorporation of inorganic materials into the manufactured hybrid composites. Polyester resin accounts for 50%, bio seashell for 10%, and bio rice husk for the remaining 40% in the C5 hybrid composite. Table 7 reveals that the composites contain the inorganic elements Si (18.92 wt%), S (16.63 wt%), and Zn (64.46 wt%). The EDAX studies performed to determine the inorganic components of the hybrid composites yielded the spectrum depicted in Fig. 16.

4 Conclusion

The researchers have looked at how adding different amounts of rice husk filler and different sized nanoparticles of bio seashells affects hybrid biocomposites. The aforementioned findings and discussion lead to the following inferences. The tensile and flexural strengths of the material are improved when bio seashell particles are included into the polymer matrix as a reinforcement and filler. The tensile and flexural strength of the bioactive seashell hybrid composite has obtained the maximum value of 52.42 MPa and 203.92 MPa respectively for the C2 specimen. Brinell hardness of 120 to 132, Rockwell hardness of 53.66 to 62, and impact strength of 42.26 to 51.46 J are all within the range of acceptability [23]. The morphological analysis shows that the structure is very well bonded together both laminar and at its interfaces.



Fig. 16 EDAX image of specimen C5

Elements such as silicon, sulfur, and zinc can be seen in the EDAX spectrum. The C2 specimen has a high silicon content, while the C5 hybrid composites have a higher zinc content. These two inorganic substances boost the durability of structural uses. According to the results of this study, bio active composites can be used in the creation of both indoor and outdoor applications.

Author contribution Amala Mithin Minther Singh A: conceptualization (lead); fabrication (lead); testing (equal); Arul Franco P: formal analysis (equal); testing (equal); validation (lead); writing—review and editing (equal). Azhagesan N: testing (equal); investigation (supporting); writing—original draft of manuscript (equal). Sharun V: testing (equal); supervision (equal); writing—review and editing (equal).

Data availability It is an ongoing work and so research data cannot be shared at this moment.

Declarations

Ethical approval No human/animals were involved in this work.

Competing interests The authors declare no competing interests.

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