

SUSTAINABLE HYBRID COMPOSITES BASED ON AGRO-WASTE REINFORCEMENTS AND HIPS: MECHANICAL PERFORMANCE AND MICROSTRUCTURAL ANALYSIS

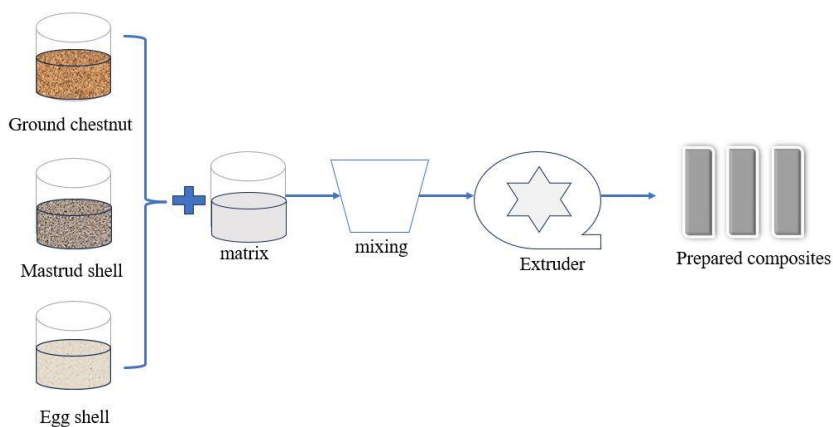
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Abstract

This study presents a novel approach to sustainable composites by hybridizing agricultural wastes—Ground Chestnut (GC), Mustard Shell (MS), and Eggshell (ES)—with a High-Impact Polystyrene (HIPS) matrix. Three hybrid systems (GC-ES-HIPS, MS-ES-HIPS, and GC-MS-ES-HIPS) were fabricated at 5–20 wt% total reinforcement. Optimal mechanical properties were achieved at 5 wt% loading, with the GC-ES-HIPS composite exhibiting the highest tensile strength (14.8 MPa), tensile modulus (909 MPa), elongation at break (3.8%), and impact strength (5.89 kJ/m²). Increasing reinforcement to 20 wt% reduced tensile strength by ~32% and increased water absorption eight-fold (0.067g to 0.536g) and burn rate (20 to 40.5 mm/min). SEM analysis confirmed that superior performance at 5 wt% GC loading resulted from uniform filler dispersion and strong interfacial bonding. The 5 wt% GC-ES-HIPS composite demonstrates an optimal balance of mechanical and physical properties for semi-structural applications.

Keywords: Ground chestnut, mustard shell, HIPS matrix, hybrid composites, and mechanical characteristics.



Graphical abstract

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Introduction

High-impact polystyrene (HIPS) is a flexible thermoplastic that is used in a lot of different things, such as consumer electronics, household goods, car interiors, and packaging. This is because it is very resistant to impact, stable in size, easy to work with, and cheap [1]. But even with these benefits, its mechanical and thermal performance—especially its tensile strength, stiffness, and wear resistance—is still not good enough for advanced structural and load-bearing uses [2–7]. As a result, modifying HIPS by adding fillers and reinforcements has become an important area of research to improve its performance.

Hybrid polymer composites, which mix two or more reinforcements in one matrix, have gotten a lot of attention because they can make things better in a way that works together. Compared to single-reinforcement composites, these systems often have better mechanical properties, thermal stability, and resistance to the environment [8–10]. Still, finding the right balance between performance, cost, and sustainability is a big problem.

Recent research has underscored the efficacy of both hybridisation and waste-derived reinforcements. Shuvo et al. [13] showed that jute nanocellulose and sugarcane bagasse greatly improve mechanical and thermal properties. However, their study only looked at non-hybrid systems. Likewise, Sheikh et al. [14] indicated enhanced strength in aluminum–bagasse hybrid composites, although the incorporation of metallic elements may undermine sustainability. Kocharla et al. [15] demonstrated that hybridisation with synthetic fibres improves performance, albeit at the cost of increased expense and diminished biodegradability. On the other hand, İnce [16] stressed the potential of biomass waste, like chestnut shells, to be used as sustainable fillers. However, their use in polymer composites is still not very common.

These results show that even though past studies have improved performance, there are still problems with sustainability, cost, and choosing the right materials. Consequently, a distinct research gap exists in the creation of fully sustainable, cost-effective hybrid composites utilising various agricultural wastes while ensuring balanced mechanical and thermal performance. Due to growing worries about the environment and the need for eco-friendly materials, natural reinforcements made from agricultural waste have become promising substitutes for synthetic fibres [11]. Groundnut shell (GC) and mustard straw (MS) powders are two of these. They are both very common lignocellulosic by-products that could be used to make things stronger. Eggshell (ES), which is mostly made of calcium carbonate, is also a good filler that makes things stiffer and more resistant to heat.

This study developed HIPS-based hybrid composites reinforced with GC, MS, and ES to examine their mechanical and thermal properties. We made three composite systems: GC–ES–HIPS, MS–ES–HIPS, and GC–MS–ES–HIPS. Each had a different amount of reinforcement (5%, 10%, 15%, or 20% by weight). The goal is to find out how different natural reinforcements and filler materials work together to affect the performance of composites. This study presents a fully waste-derived hybrid reinforcement approach, in contrast to numerous prior studies [17,18] that concentrated on single or partially hybrid systems. This method tries to find the best balance between cost, mechanical performance, and environmental sustainability. The results are expected to help make polymer composites that are lightweight, good for the environment, and work well in industry.

Materials and Methods

Raw Materials

High Impact Polystyrene (HIPS) pellets, Model PS 476L, are sourced from INEOS Styrolution in Korea. Ground chestnut fibers were collected from local fields in Rajshahi, Mustard shells were gathered from a nearby village, and Eggshells were collected from the local food industry.

Processing of Raw Materials

The as-received HIPS pellets were used. The ground chestnut and mustard seed husks were washed initially with fresh tap water to get rid of extraneous matter. The husks are sun-dried and further oven dried at 250 °C after washing. Followed by grinding into a thin powder using an electric blender. The white calcareous film covering the eggshells was removed, and the shells were cut into small fragments, which were dried in sunlight. These small pieces were further calcined at 900 °C in a muffle furnace to obtain the eggshell powder.

Preparation of Hybrid Composites [19]

The hybrid composites were prepared for all combinations of different proportions of raw powder materials (Table 1). The groundnut (GS) and mustard seed (MS) husk were used as a reinforcement, and egg shell powder (ES) was used as filler. High-impact polystyrene (HIPS) served as the polymer matrix for the preparation.

Table 1. The respective proportions of composite materials used

Raw Materials	Percentage of Raw Materials (%)				Composites
ES	10	10	10	10	ES-GC-HIPS
HIPS	85	80	75	70	
GC	5	10	15	20	
ES	10	10	10	10	ES-MS-HIPS
HIPS	85	80	75	70	
MS	5	10	10	20	
ES	10	10	10	10	ES-GC-MS-HIPS
HIPS	85	80	75	70	
GC	2.5	5	7.5	10	
MS	2.5	5	7.5	10	

The mixture was diced and transferred into the hopper of an extruder-type molding machine (310-330 °C), in which it formed a homogeneous paste while pulling it through the head. This soft pellet composition was thereafter cooled and formed into a die. The fabricated shapes were used to prepare samples of constant length, width and thickness on a hot press machine (Fig. 1). The mechanical properties of the samples were analyzed using a UTM.

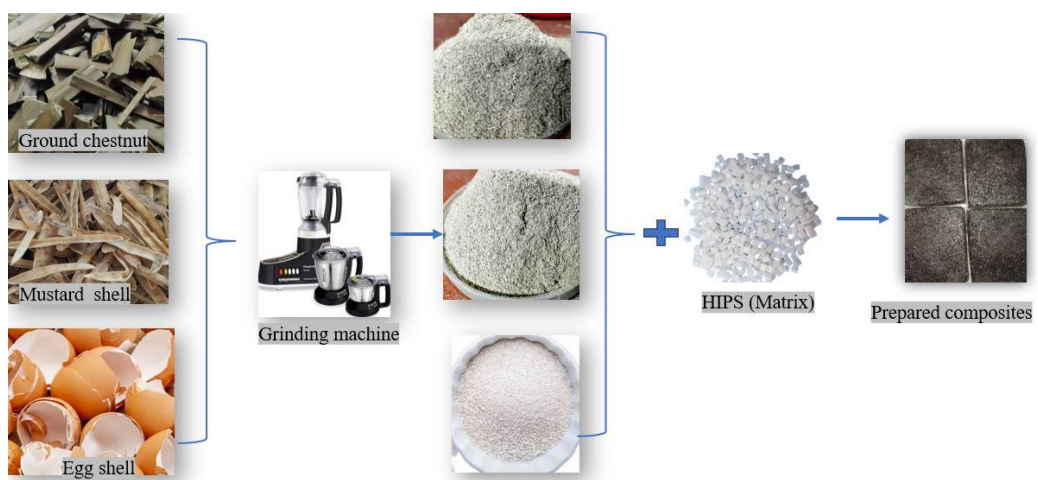


Fig. 1. Fabrication process of hybrid composites.

Mechanical Properties

Tensile Strength, Flexural Strength, and Compression Strength

A universal testing machine (UTM, H5OKS20Hounsfield, USA) was employed to determine tensile, flexural, and compression strength of the developed composites based on ASTM Type I. The composite samples were clamped into the grips of the machine together with an extensometer. During testing, the UTM registers any variations in the length of the surface to be tested. If no extensometer is used, the machine will instead measure the displacement between its crossheads to which the specimen is attached. However, while this technique measures the change in length of the specimen, it also senses everything else that is elastic on the testing machine or its drive systems, such as a slip that can occur between the specimen and grips. The machine is started and the load is now on the specimen. During the tests, the control and software values of load as well as extension or compression of the specimen are recorded continuously [19,20].

Impact Strength Testing

The impact strength of the fabricated composites was also evaluated by a Universal Impact Tester (model no. 7408, Hung Ta, Taiwan). This instrument evaluates the resistance of an object to high-rate loading, which is necessary when assessing the service life of a component or material. Impact resistance may be one of the more difficult-to-measure properties. It is important to know this property in evaluating product liability and safety [21].

Morphological Properties

Optical Microscopy

The morphology of the synthesized composite was observed with the aid of an optical microscope (OP) (ML-803, Taiwan). This method is the use of a lens to enlarge a sample that can be seen by means of visible light. An optical microscope, often referred to as a light microscope, uses one or several lenses to produce an enlarged image of a small object using visible light. A magnifying glass is placed between the viewer's eye and the sample, magnifying at a distance to enable viewing of fine gradations. The samples were polished with 120 and 1200 grit sandpaper, whereby acetone also further improved the polishing process for the metallographic analysis [22]. Once it was polished, the final buffing was with a velvet cloth. The microstructure of the samples was then analyzed with an optical microscope at different magnifications. It allowed the study of distribution of particles in the polymer matrix. The difference in weight content.

Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM) is one of the higher forms of a microscope, which creates an image of a sample by scanning it with a focused beam of electrons. The morphology of the composite surface is observed by a JEOL JSM-6480LV scanning electron microscope. The composite samples are carefully cleaned, air dried, and coated with a 100 Å Pt layer with a JEOL sputter ion coater before observation using SEM (accelerating voltage of 20 kV). Platinum thin film is vacuum evaporated onto the composite samples to improve their conductivity, prior to photographing micrographs. Furthermore, the SEM is utilized in observations of the fracture surface morphology of the composite sample.

Results and Discussion

Characterization of Hybrid Composite Materials

Tensile Strength of Prepared Composites

Effect of different reinforcement and percentage of reinforcement on the tensile strength of prepared hybrid composites.

Fig. 2 of tensile strength (MPa) for materials with different compositions of GC, MS, and GC+MS (referred to as ES-GC, ES-MS, and ES-GC-MS) is shown in Fig. 3, with concentrations [5%,10%,15% and %20]. The blue bars (ES-GC) are the tensile strength of the material with GC alone. The tensile strength of material composed only of MS is indicated as red bars (ES-MS).

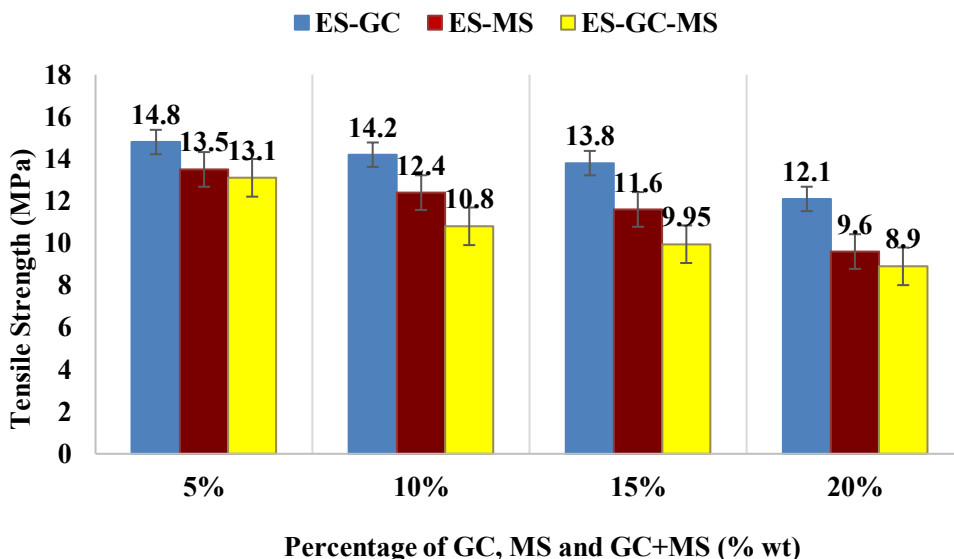


Fig. 2. Effect of GC, MS, and GC-MS reinforcement percentage loading on the tensile strength of prepared hybrid composites.

The yellow bars (ES-GC-MS) show the tensile strength for the GC and MS composite material, respectively, based on which it is obvious that among all compositions, ES-GC has much greater tensile strength, especially at 5% and 10%. On the other hand, composition ES-MS has the lowest tensile strength in all concentrations and a larger decrease at 20%. The composition of ES-GC-MS is weaker than that of ES-GC, which still shows a downward trend in tensile strength as the content of GC+MS increases to 15% and 20% contents predominantly [23,24].

Tensile Modulus of Prepared Composites

Effect of different reinforcement and percentage of reinforcement on the tensile modulus of prepared hybrid composites.

The graph in Fig. 3 shows the tensile modulus (MPa) comparison of MS-ES-HIPS composites with different weight percentages of Mustard Shell (MS). The plot presents three original compounds ES-GC, ES-MS, and ES-GC-MS depicted with blue, red, and gray bars, respectively, over 4 different MS fractions: 5%, 10%, 15% and 20%. It can be seen from the profile that the tensile modulus of the ES-GC composition increases with rising percentage. Tensile modulus values for the composites decreased with an increase in the percentage of ES-MS and ES-GC-MS. The highest tensile modulus is 909 MPa at 5% MS of ES-GC, as a series of ES-GC-MS peaks are observed at higher concentration, reaching the minimum value in the case of 20% (530 MPa). This decrease in tensile modulus at higher MS content may be explained by difficulties in obtaining a homogeneous dispersion of the MS reinforcement into the HIPS matrix. Above certain loading levels, agglomerated or poorly dispersed MS in the polymer matrix provides sites for stress concentrations, structural defects, and weak interfacial bonding, resulting in a decrease of the overall tensile modulus.

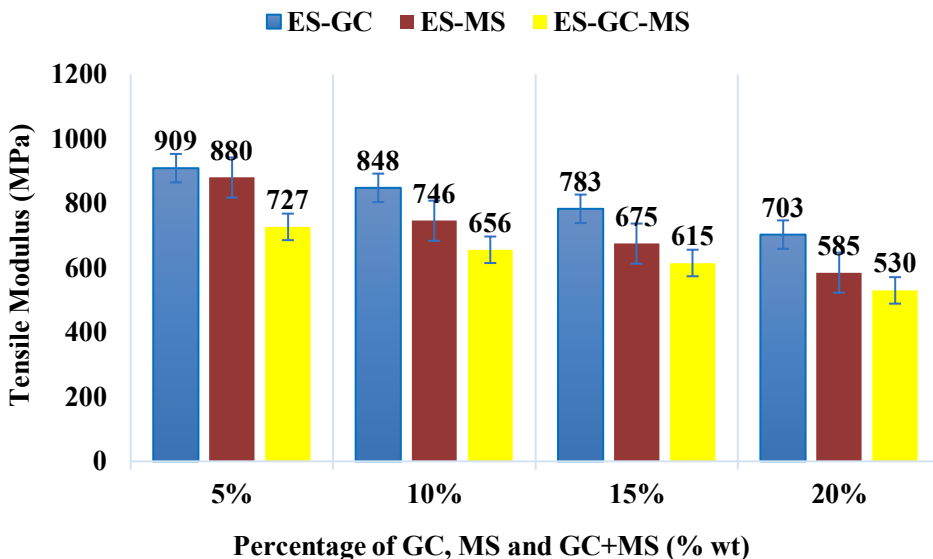


Fig. 3. Effect of GC, MS, and GC-MS reinforcement percentage loading on the tensile modulus of prepared hybrid composites

All these lead to a net decrease in the material performance at higher MS contents, and especially at 20%. The increasing trend is attributed to over-reinforcement, low filler-matrix compatibility, and reduced flexibility of the composite [25].

Elongation at break of Prepared Composites

Comparison of the elongation at break of prepared hybrid composites with the percentage of GC, MS, and GC-MS.

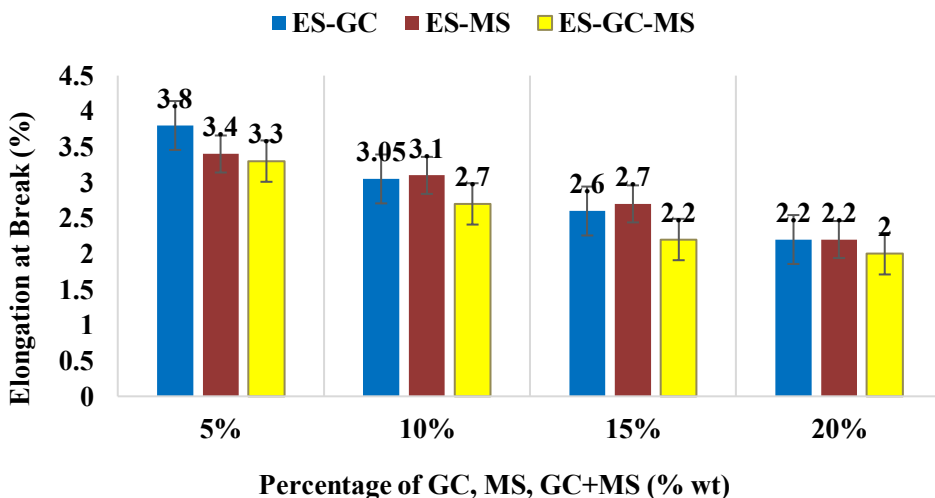


Fig. 4. Effect of GC, MS, and GC-MS reinforcement percentage loading on the elongation at break of the prepared hybrid composites

The Elongation at Break (%) of the sample with different mass ratios from 5 to 20%) was demonstrated in Fig. 4 S6 (ES-GC/MS, ES), which clearly reveals that the addition of MS or GC both improved the EAB value. The blue bars (ES-GC) are the elongation at break of material containing the only GC; the red bars (ES-MS) are the elongation of an only MS sample; and the brown squares (ES-GC-MS) are for material with both GC and MS. omnipresent behavior across all percentages, ES-GC is shown to have higher elongation at break than all others where up to 5%, it can reach up to 3.8%. On the other hand, ES-MS only slightly reduces the elongation with increasing MS content from 5% (3.3%) to 20% (2.2%). The ES-GC-MS composition exhibits a comparable behavior to GC-MS, with a 3.4% peak shift at the concentration level of 5%, whose value decreases to about 2% for the concentration level equal to 20%. Overall, the data suggest that ES-GC offers the highest flexibility, and increasing MS content decreases elongation at break. This indicates that an increase in MS concentration would lower the material ductility and flexibility, probably by encountering poor compatibility or dispersion of the MS inside the matrix [26].

Impact Strength of Prepared Composites

Effect of GC, MS, and GC-MS reinforcement percentage loading on the impact strength of the prepared hybrid composites.

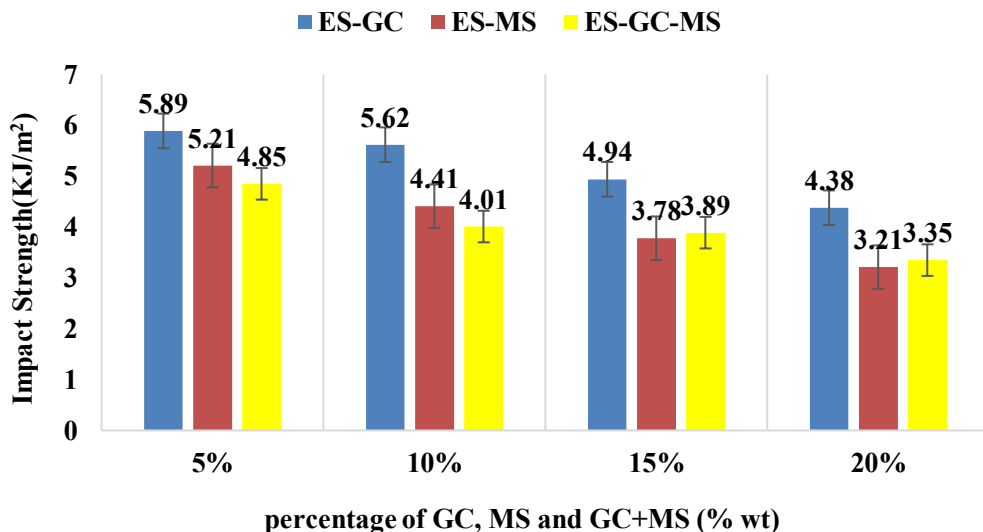


Fig. 5. Effect of GC, MS, and GC-MS reinforcement percentage loading on the impact Strength of the prepared hybrid composites

The graphical representation (Fig. 5) shows the Impact Strength (KJ/m²) concerning various proportions of GC, MS, and GC+MS materials (designated as ES-GC, ES-MS, and ES-GC-MS, respectively) at 5%, 10%, 15%, and 20% weight percentages. The blue bars (ES-GC) represent the impact strength of the material containing only GC, while the red bars (ES-MS) indicate the impact strength with only MS. The yellow bars (ES-GC-MS) show the impact strength of the composite with both GC and MS. These results evidently indicate that ES-GC had the best impact strength under all of the compositions tested and a maximum value 5.89 KJ/m² was achieved at 5% GC. This latter value goes up to normality, and falls regularly down to 4.38 KJ/m² at 20%. On the other hand, impact strength found for ES-MS was weaker than 5.21 KJ/m² and averagely decreases from 5% to 20% to a minimum of 3.35 KJ/m² respectively. In the case of ES-GC-MS, impact behavior was less clear; however, in most instances it resulted in the greatest reduction in impact strength (4.85 KJ/m² at 5% to 3.21 KJ/m² at 20%). These results suggest that

the impact performance is in favor of ES-GC, and increasing MS contents tends to decrease the impact strength. This reduction could be attributed to the agglomeration of MS and poor compatibility with the matrix at higher content (e.g., 20%).

Table 2. Water absorbency of prepared hybrid composites with percentage of GC, MS, and GC+MS

Percentage of GC, MS, and GC+MS	Water Absorbency (gm) of		
	ES-GC	ES-MS	ES-GC-MS
5%	0.067	0.188	0.113
10%	0.089	0.408	0.302
15%	0.129	0.623	0.487
20%	0.15	0.824	0.704

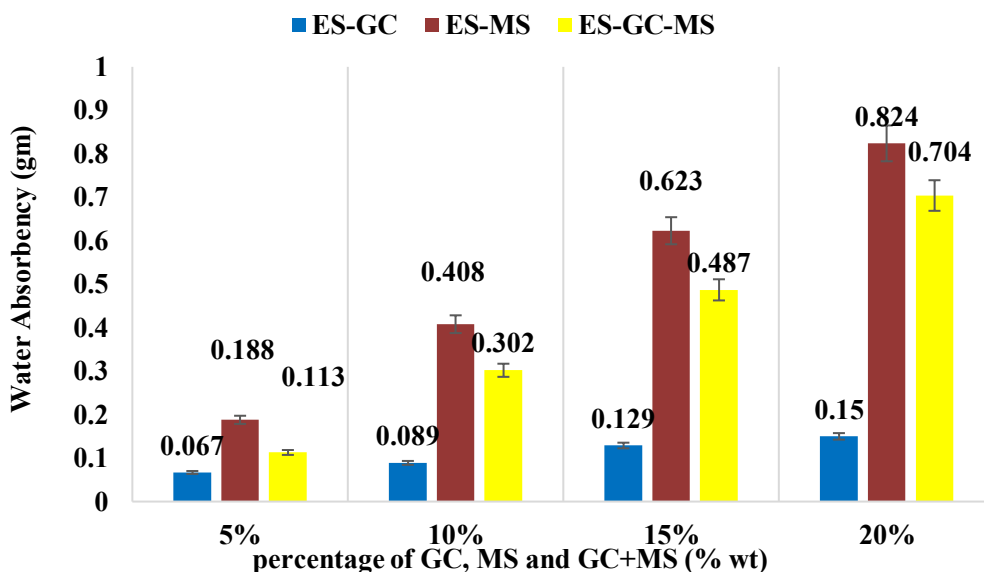
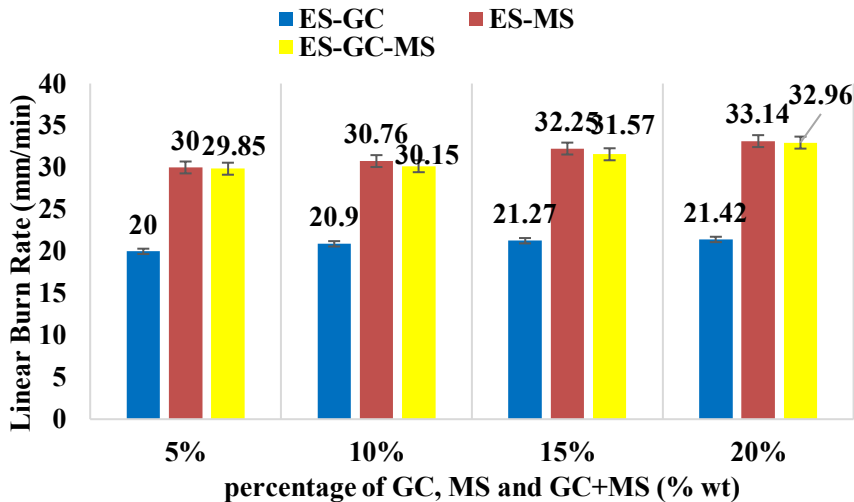


Fig. 6. Comparison of the water absorbency of prepared hybrid composites with the percentage of GC, MS, and GC-MS

The Fig. 6 shows the bar chart of Water Absorbency (gm) of GC, MS, and their composites, i.e., ES-GC, ES-MS, and ES-GC-MS, with different wt% (5 to 20 wt%) at each composition (5%, 10%, 15% and 20%). The blue bars (ES-GC) are for water absorbency of the material consists of only GC, red bars (ES-MS) is for that of only MS and yellow bars (ES-GC-MS) is that with both materials, from this chart it was observed ES-MS consistently exhibit highest water uptake at all percentages, it was also found to be peak value at 0.824 gm after 20%. In contrast, ES-GC has the least water absorbency, which is 0.067 g at a low loading of 5% and increases to only around 0.15 g when loaded with up to 20%. ES-GC-MS demonstrates a water absorbency of a moderate degree, about 0.113 gm, around 5% - 0.760 gm at 20%, and the change becomes significant when GC+MS percentage increased, as would be seen in Table 2. The data indicate that the water absorbency of the material is greatly enhanced by MS presence, especially at high concentrations. ES-GC (only GC) shows the minimum water absorption, meaning that it has the least moisture absorption tendency. There was a tendency for an increase in water absorbency with increasing MS content in the films, probably because of the hydrophilicity in the nature of the MS that facilitates its capacity to absorb water. Comparison of the fire retardant of prepared hybrid composites with the percentage of GC, MS, and GC-MS.

Table 3. Fire retardant of prepared hybrid composites with percentage of GC, MS, and GC+MS

Percentage of GC, MS, and GC+MS	Linear Burn Rate (mm/min) of		
	ES-GC	ES-MS	ES-GC
5%	20	5%	20
10%	20.9	10%	20.9
15%	21.27	15%	21.27
20%	21.42	20%	21.42

**Fig. 7.** Comparison of the fire retardant of prepared hybrid composites with percentage of GC, MS, and GC-MS

The Fig. 7 shows the Bar chart of Linear Burn Rate (mm/min) for materials with different GC, MS composition, i.e., UF as ES-GC, EG as ES-MS, and E+G as ES-GC-MS at 5%, 10%, 15% and 20% weight fraction. The blue (ES-GC) bars are the linear burn rates for the GC-only material, while the red (ES-MS) and yellow (ES-GC-MS) bars are for MS and GC+MS, respectively. From Fig. 8, it can be observed that ES-MS consistently shows a high linear burn rate throughout all concentrations, with maximum values up to 33.14 mm/min at 20%. The lowest LBR of the ES-GC is at all percentages, 20mm/min for 5%, and increased bit from 21.42 mm/min for 20%. The ES-GC-MS exhibits a medium burn rate, between 29.85 mm/min at 5% to 32.96 mm/min at 20%. The results indicate that the MS presence has a great effect on linear burning rate, especially at higher concentration values (Table 3). The ES-GC, solely loaded with GC, has lower combustion kinetic behavior, probably due to its more stable structure, and the addition of MS can increase its flammability. The ES-GC-MS burn ratio is intermediate to both, exhibiting higher than the GC burning speed, but lower than the ES-MS system; thus, it can be inferred that the combination of GC and MS provides a more moderate burn rate compared to MS alone.

Morphological Test of Prepared Composites

Morphological Observation of GC-ES-HIPS (5% GC) by Optical Microscope at 10X Magnification

In the case of HIPS thermoplastic polymer (Fig. 8a), this looks perfectly smooth and homogenous in a 100% state; you can not see it being filled or reinforced.

The ES-HIPS composite (Fig. 8b) shows that the eggshell powder is not well dispersed in the HIPS matrix and is largely agglomerated. However, there are some points with a good dispersion of the eggshell powder contributing positively to the strong interaction between matrix and filler [27].

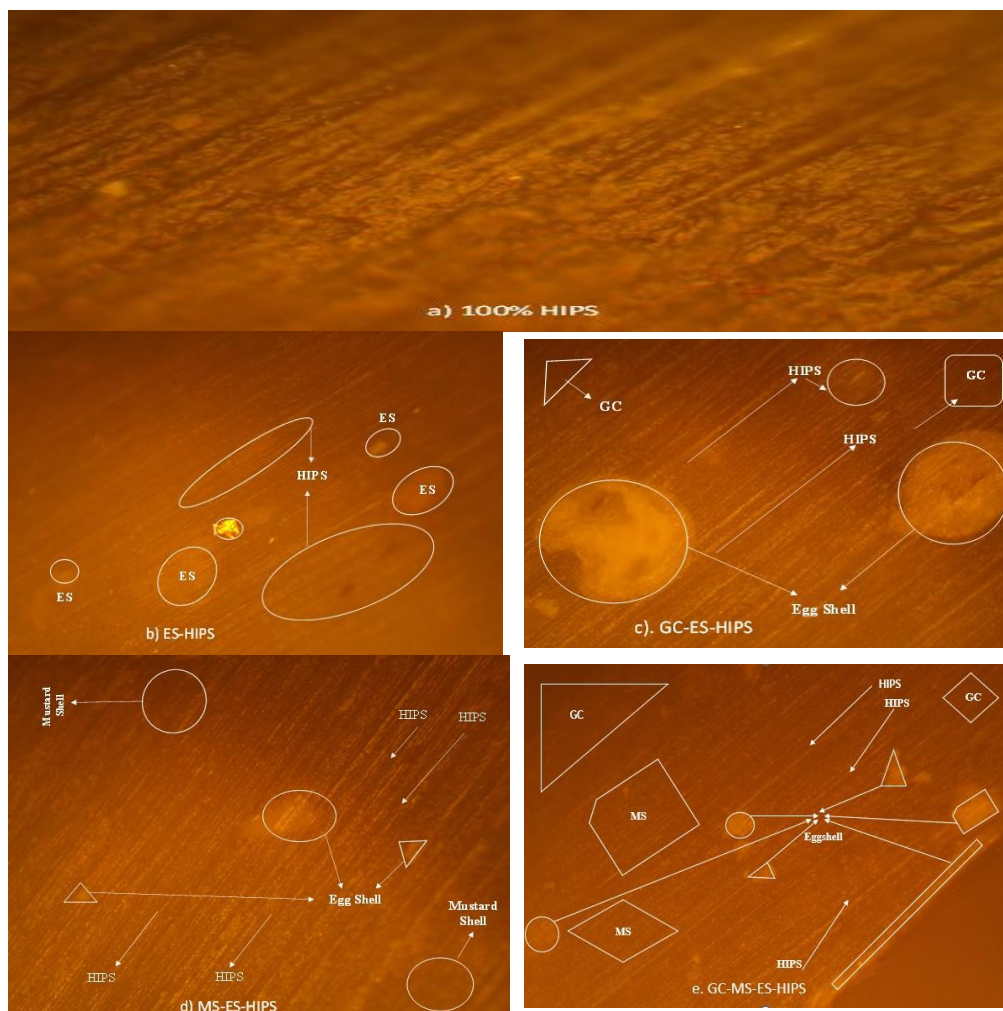


Fig. 8. a-e: Optical images of HIPS (100%), ES-HIPS (10% + 90%), GC-ES-HIPS (5% + 10% + 85%), MS-ES-HIPS (5% + 10% + 85%), and GC-MS-ES-HIPS (2.5% + 2.5% + 10% + 80%) composites

The ground chestnut particles are well-dispersed in the HIPS matrix (Fig. 8c). This distribution maintains the integrity, ductility, and stress concentration of the composite. Although the eggshell filler might somewhat compromise the adhesion quality between HIPS and chestnut powder, this still results in a relatively regular dispersion of these powders—especially with 5% chestnut content—that contributed to its better mechanical characteristics [28].

In Fig. 8d, the distribution of eggshell powder and reinforcement particles is not homogeneous in the MS-ES-HIPS composite. HIPS matrix is much visible on the surface and is most likely the cause of the poor mechanical properties of the MS composites, compared with GC composites. However, the diluted HIPS ratio (5% of mustard shell) still has an acceptable high volume of the HIPS to preserve ductility and cohesive density in the composite.

Finally, the GC-MS (HIPS-ES) composite image is shown in Fig. 9e, and HIPS are dominated on the surface with a moderate amount of eggshell powder and a small amount of ground chestnut shell and mustard shell powders. The eggshell powder is not homogeneous and shows some agglomeration. The uneven distribution of chestnut and mustard shell powders generates local points likely to concentrate stresses, leading to decreased adhesion with the matrix and therefore affecting the mechanical properties

of the composite material. Consequently, the mechanical property of the 5% GC + MS composite is weaker than that of the other two composites [29].

Morphological Observation of GC-ES-HIPS (5%) Composites by Scanning Electron Microscope (SEM) at 400X Magnification

SEM micrograph of the GC-ES-HIPS composite is presented in Fig. 9 showing the failure modes observed when tensile was done. These include fiber pullout holes, matrix failure holes, lined-up filler, or poor adhesion between the organic molecules and the fibers.

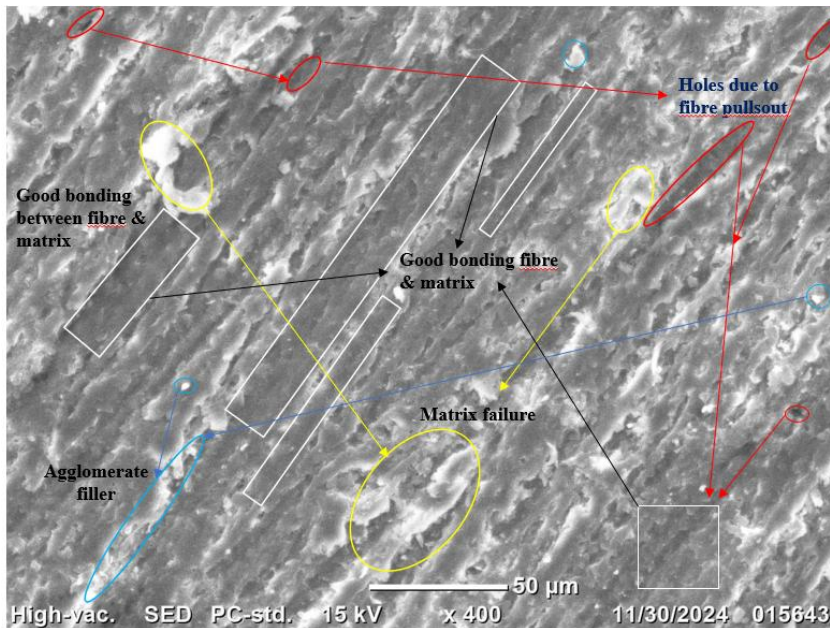


Fig. 9. GC-ES-HIPS (5%) Composites by Scanning Electron Microscope (SEM) at 400X Magnification.

Table 4. Comparison of ES, GC, and MS reinforced polymer composites from previous studies and the present work

Study	Matrix	Reinforcement	Key Findings	Limitation	Ref
Ing et al.	Epoxy	Jute + Eggshell (PES)	↑ Tensile, Flexural, Hardness	Single filler, not fully hybrid	[31]
Ramotra et al.	LLDPE/HDPE	Bagasse+ Eggshell	Mechanical ↑	Effects of individual fillers are not isolated	[32]
Jasim et al.	PP	Eggshell	Best mechanical at 5–7 wt%	Single filler only	[33]
Geeri et al.	Polymer	Eggshell+ Banana fiber	Mechanical ↑, Biodegradability ↑	Not GC/MS combination	[44]
Present Work	HIPS	ES + GC + MS	Optimal tensile, modulus, impact & water resistance at 5% GC; Fire retardancy ↑	Higher GC reduces mechanical and improves fire.	

The ground chestnut particles are highly dispersed in the matrix, and this enhances interfacial adhesion between the fibre and matrix. At 5% GC reinforcement level, the matrix integrity is maintained, guaranteeing enough ductility and a homogeneous stress distribution. Therefore, the 5% GC composite exhibits better mechanical performance than other compositions. However, locally, there is fiber pullout, agglomerated filler, and matrix failure on the surface of composites, which weakens the overall mechanical performance. If the matrix powder particles, filler, and

reinforcement were more uniformly dispersed, it would be beneficial to improve the mechanical properties of GC composites [30].

Conclusions

This research successfully developed and assessed HIPS-based hybrid composites reinforced with eggshell (ES), ground chestnut (GC), and mustard shell (MS) for their mechanical, physical, and fire-resistant properties. The results show that the amount of reinforcement is very important for how well a composite works. The composite with 5 wt.% GC (10 g ES + 5 g GC + 85 g HIPS) had the best overall performance of all the compositions. It had a maximum tensile strength of 14.8 MPa, a tensile modulus of 909 MPa, an elongation at break of 3.8%, and an impact strength of 5.89 kJ/m². This better performance is due to even particle dispersion, strong bonding between the interfaces, and good stress transfer within the matrix. The water absorption was also very low (0.067 g) at this composition, which shows that the HIPS matrix did a great job of encapsulating it. With more GC, fire retardancy got better, but with less GC, it didn't have as much of an effect because the matrix was more important. Higher GC levels (>5 wt.%) made the material less mechanically stable because the particles stuck together, didn't spread out well, and the matrix didn't stay together as well. Water absorption also went up, which hurt the mechanical integrity. Fire resistance got better with more GC content, but tensile and impact properties got worse.

A comparison with previous studies (Table 4) indicates that earlier research utilizing eggshell or other natural fillers enhanced mechanical properties; however, most concentrated on singular fillers or restricted hybrid combinations, lacking a systematic assessment of entirely waste-derived hybrid composites. In contrast, the current HIPS/ES/GC/MS composites show that a fully waste-derived hybrid system can have the best mechanical, thermal, and fire-resistant properties at 5 wt.% GC. This shows that it is both better for performance and better for the environment. In general, 5 wt.% GC content is the best combination for getting a balanced mix of strength, stiffness, flexibility, moisture resistance, and fire retardancy. This makes these hybrid composites good for industrial uses that are both eco-friendly and cost-effective.

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Credit author statement

Md. Rezaul Karim SHEIKH: Conceptualization, Supervision, Project administration, Data analysis; Asnaf Nihan SHUVO: Writing – original draft, Formal analysis, Literature review; A. Nayeem FARUQUI: Data curation, Validation, Methodology, Investigation.

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