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Grey Relational Analysis of Cuttlefish Bone Particles–Glass Fiber Reinforced Epoxy Composites

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Abstract

This research examines the mechanical characteristics, water absorption performance, and thermal stability of epoxy composites reinforced with Cuttlefish Bone Particles (CFBP) and glass fibers. Composites were fabricated with varying CFBP contents (0%, 3%, 6%, 9%, and 12%) using an epoxy resin as the matrix material and glass fiber as the primary reinforcement. The experimental results revealed that the CFBP-0 composite (neat epoxy with glass fiber) exhibited the highest tensile strength, whereas the CFBP-3 composite demonstrated superior impact resistance owing to its improved energy absorption capability. The hardness of the composites increased with the addition of CFBP, reaching its maximum value at CFBP-6, which also exhibited the highest flexural strength. Thermogravimetric analysis (TGA) indicated that the residual char content increased with higher filler percentages, particularly at CFBP-12, confirming an enhanced thermal stability. In contrast, water absorption also gradually increased with increasing CFBP content, which can be attributed to the hydrophilic nature of the bio-filler. Gray Relational Analysis (GRA) revealed that CFBP-0 achieved the highest Gray Relational Grade (GRG), representing the best overall performance among all tested composites. Scanning Electron Microscopy (SEM) observations confirmed fiber–matrix debonding, microvoids, and surface cracks, which influenced the fracture mechanisms. Overall, the study highlights that while increasing CFBP content enhances thermal resistance, it slightly compromises mechanical strength, suggesting an optimal composition for a balanced performance.

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Keywords: Cuttlefish bone particles, epoxy, glass fiber, gray relational analysis, mechanical properties, thermal stability, water absorption behavior

INTRODUCTION

The composite materials are biodegradable, with cuttlebone/natural rubber degrading within months under certain conditions, and cuttlebone particles enhance the mechanical properties of natural rubber, rivaling commercial calcium carbonate fillers [1,2]. Cuttlebone also effectively enhances tensile strength and structural integrity in epoxy composites [3], while in chitosan/aragonite biocomposites, it shows bioactivity, suitable for tissue engineering applications [4]. Utilizing waste cuttlebone promotes sustainability in composites by reducing costs and environmental impact, with organic components forming bound rubber layers that improve filler–matrix interactions and boost mechanical properties like modulus under stress [5,6]. Cuttlebone offers superior reinforcement in natural rubber over synthetic isoprene due to better adhesion and non-rubber components [7], and also improves tensile properties in composites, boosting montmorillonite intercalation and mechanical performance in PVOH composites [8]. Incorporating graphene nanoplatelets (GNPs) into UHMWPE boosts flexural strength by 25% and compression strength by 40% at optimal content [9], while hybrid composites with casuarina and graphite fillers show a 72.77% increase in flexural strength over those with only natural fillers [10]. Natural fibers, being biodegradable and renewable, provide eco-friendly alternatives to synthetic fillers [11], although sunflower seed husk filler may cause composites to display brittle behavior [12]. Using agricultural byproducts like agave fiber bagasse reduces waste and promotes recycling [13], and natural fillers like date palm seeds enhance wear resistance and toughness in glass-epoxy composites, increasing toughness by 80% with 10% filler [14]. Adding Al2O3 improves thermal conductivity and mechanical properties, with optimal performance at 2% filler content [15], and incorporating nano and micro fillers boosts fracture toughness by 20–26% [16]. Graphene oxide improves heat transfer and stress distribution, though thermal stress may cause fiber detachment [17]. Combining natural and glass fibers achieves tensile strengths up to 23.8 MPa, much higher than untreated fibers [18], and hybrid composites with natural fillers and glass fibers outperform glass-only composites, especially under impact and flexural loads [19]. Glass fiber epoxy composites offer strong mechanical properties, ideal for aerospace and civil engineering applications [20], with the addition of glass fibers boosting impact strength but potentially reducing hardness compared to pure epoxy [21]. Adding silicone resin to glass fiber/epoxy composites reduces compressive strength, with the highest recorded at 240.63 MPa without modification [22]. Enhanced glass fiber epoxy composites are increasingly suitable for aeronautics and automotive applications due to their improved mechanical and thermal properties [23], and incorporating 0.4 wt% graphene oxide (GO) into glass fiber epoxy composites boosts tensile strength by 20% and impact strength by 41% [24]. GRA has been employed to optimize hybrid composites with hexagonal Boron Nitride (hBN) and natural fibers, improving thermal conductivity (1.03 W/m.K) and electrical resistance (279.88 GΩ) [25], and has also optimized processing parameters in 3D-printed PLA/wood dust composites, achieving a tensile strength of 20.71 MPa [26]. GRA optimized dual-curing polyurethane adhesives, improving peel and shear strength for textile applications [27], and enhanced the mechanical properties of biopolymer blends, identifying the best formulations for improved performance [28]. Additionally, GRA optimized natural fiber composites, combining 40% sisal and 5% glass fiber to reduce weight and fuel consumption [29], and with the DMAIC approach, helped select polyethylene as the optimal thermoplastic matrix based on performance and environmental factors [30]. GRA also identified the best processing parameters for optimizing tensile and flexural strengths with specific chemical treatments and fiber content [31], optimized a pineapple leaf and glass fiber hybrid composite, improving water absorption and mechanical reliability [32], and identified optimal abrasive parameters in PTFE-based composites, greatly improving wear performance [33]. The current study aims to investigate the mechanical properties, TGA, and water absorption properties of cuttlefish bone particle (CFBP)-glass fiber reinforced epoxy composites, using Gray Relational Analysis (GRA).

MATERIALS AND METHODS

Materials

Cuttlefish bone sourced from a local market was used to prepare composites with Araldite LY556 epoxy resin, HY951 hardener, and E-glass woven fabric as reinforcement. The bones were cleaned, air-dried for a week, ground into 5–30 μm particles, pre-dried at 100°C for an hour, cooled to room temperature, and stored in a desiccator to prevent moisture absorption before use as reinforcing filler.

Composite Preparation and Specimen Testing

The epoxy resin was weighed and mixed with cuttlefish bone particles according to the specified composition. The hardener, maintaining a 10:1 epoxy-to-hardener ratio, was added to the mixture and stirred for 10 minutes. The prepared mixture was poured into a wax-coated mild steel mold (210 × 170 × 3 mm) containing two layers of woven glass fiber mat for reinforcement. A 1000 kg load was applied using a compression molding setup for 24 hours to cure the composite. After curing, specimens were fabricated as per ASTM standards for tensile (D638), flexural (D790), impact (D256), and hardness (D2240) tests. Scanning Electron Microscopy (SEM) was used to analyze the tensile-tested specimens. Table 1 details the sample IDs and compositions of the CFBP/glass fiber epoxy composites.

Table 1. Composition and sample IDs of CFBP-glass fiber epoxy composites.

S n.	Sample ID	Composition		
		Filler - cuttlefish bone particle (v_f%)	***Reinforcement - glass fibre (v_f%)***	***Matrix - epoxy resin + hardener (v_f%)***
1	CFBP-0	0	6	94
2	CFBP-3	3	6	91
3	CFBP-6	6	6	88
4	CFBP-9	9	6	85
5	CFBP-12	12	6	82

Table 2. Steps and equations used in GRA.

Steps in GRA	**Equation**	**Nomenclature**
Data Pre-Processing and Normalizing	Expected data sequence is of the form "Higher-the-better"$x_i^*(k) = \frac{x_i^0(k)-\min x_i^0\ (k)}{\max\ x_i^0(k)-\min x_i^0\ (k)}$ Expected data sequence is of the form "smaller-the-better" $x_i^*(k) = \frac{\max x_i^0(k)-x_i^0(k)}{\max\ x_i^0(k)-\min x_i^0\ (k)}$	$x_i^0(k)$is the original sequence $x_i^*(k)$ the sequence after the data preprocessing max $x_i^0\ (k)$ the largest value of $x_i^0(k)$ min $x_i^0\ (k)$ simply the smallest value of $x_i^0\ (k)$
Grey Relational Coefficient (GRC)	$\xi_i\ (k) = \frac{\Delta_{min} +\ \zeta.\Delta_{max}}{\Delta_{0i}(k) +\ \zeta.\Delta_{max}}$	$\Delta_{0i}(k)$ is the deviation sequence of the reference sequence $\Delta_{0i}(k) = \parallel\ x_0^*(k) -\ x_i^*\ (k) \parallel$ $\Delta_{max} = \max\limits_{\forall j\epsilon i}\ \max\limits_{\forall k} \parallel x_0^*(k) -\ x_j^*(k) \parallel \Delta_{min} =$ $\min\limits_{\forall j\epsilon i}\ \min\limits_{\forall k} \parallel x_0^*(k) -\ x_j^*(k) \parallel \zeta$ is distinguishing or identification coefficient $[0,1]\zeta = 0.5$ is generally used
Grey Relational Grade (GRG)	$\gamma_i = \frac{1}{n}\sum_{k=1}^{n} w_i(k)\xi_i\ (k)$	$\xi_i\ (k)$ = Grey relational coefficient $w_i(k)$ = weight (1) n = Number of alternatives

Gray Relational Analysis (GRA)

Gray Relational Analysis (GRA), introduced in the 1980s, evaluates the correlation between multiple factors within a system. Widely used in engineering, economics, and management, GRA addresses complex problems by calculating deviation sequences to compare reference and comparative sequences. The Gray Relational Coefficient (GRC) quantifies the correlation, and its average, the Gray Relational Grade (GRG), indicates overall performance. GRG values range from 0–1, with higher values reflecting stronger alignment and better outcomes. Table 2 clearly outlines the steps and equations used in GRA. In this study, equal weighting (w = 1) was assigned to all performance parameters in the Gray Relational Analysis to ensure uniform contribution of mechanical, thermal, and moisture-related responses. This approach follows conventional GRA procedures for exploratory composite optimization where parametric significance is not pre-established. Although advanced weighting methods such as entropy or Analytic Hierarchy Process (AHP) could provide data-driven or expert-based weighting, the equal-weight model was adopted to avoid subjective bias. A preliminary sensitivity assessment indicated that small deviations in weights (<10%) did not significantly alter the GRG ranking, confirming the robustness of the uniform weighting assumption.

RESULTS AND DISCUSSION

Gray Relational Analysis (GRA)

This current research study evaluated the mechanical properties, water absorption behavior, and thermal stability of cuttlefish bone particle (CFBP)–glass fiber reinforced epoxy composites, as illustrated in Table 3. The mechanical properties, water absorption behavior, and residual char values obtained through TGA analysis. CFBP-0 (pure epoxy with glass fiber) exhibited the highest tensile strength (63 MPa) and flexural strength (133 MPa), serving as the baseline for comparison. CFBP-6 marginally exceeded this with the highest flexural strength (134 MPa), a 0.75% improvement over CFBP-0, attributed to optimal filler dispersion. The hardness values increased with higher filler content, with CFBP-6 showing the maximum hardness (62.66 Shore D), marking a 35.25% improvement over CFBP-0. The higher hardness and flexural strength observed for the CFBP-6 composite arise from uniform filler distribution and effective interfacial bonding between cuttlefish bone particles and the epoxy matrix. Although SEM images in this study were obtained from tensile-tested specimens, the micrographs consistently showed well-embedded fillers and limited void formation at this composition, reflecting improved stress transfer capability. Such morphological uniformity enhances resistance to localized deformation, which is consistent with the measured improvement in flexural and hardness properties. At higher filler loadings, SEM evidence of particle agglomeration and microvoids supports the corresponding decline in these properties. Impact strength peaked at CFBP-3 (60 kJ/m²), a 100% improvement compared to CFBP-0, due to enhanced energy absorption at lower filler concentrations. Conversely, tensile strength showed a significant reduction with filler addition, with CFBP-3 (33 MPa) demonstrating a 47.62% decrease compared to CFBP-0. Water absorption increased sharply with higher filler content, from 0.0067% in CFBP-0 to 0.6281% in CFBP-12, due to the hydrophilic nature of the filler. Similarly, residual char improved with filler content, with CFBP-12 exhibiting the highest value (22.0036%), representing a 200.7% increase over CFBP-0, indicating enhanced thermal stability from the inorganic nature of the filler. The TGA results demonstrated a steady increase in residual char content with higher CFBP loading, confirming enhanced thermal stability. However, the present work focused on comparative thermal performance rather than kinetic modeling. Future studies could employ Kissinger or Flynn–Wall–Ozawa (FWO) methods to estimate the apparent activation energy (E_a) and reaction order (*n*) of the degradation process. Such kinetic analysis would offer deeper insight into the thermal decomposition mechanisms of CFBP–epoxy systems.

The experimental values in Table 3 were normalized to a 0–1 range for uniform comparison (refer to Table 4). CFBP-0, with the highest tensile strength and lowest water absorption, was normalized to 1.000 for those attributes. CFBP-3 achieved a normalized value of 1.000 for impact strength, while CFBP-6 reached 1.000 for flexural strength and hardness, reflecting its superior performance in these categories.

Table 5 presents the Gray Relational Coefficients (GRC) and Gray Relational Grades (GRG), which quantify overall performance. CFBP-0 recorded the highest GRG (0.667), attributed to its superior tensile and flexural strengths and minimal water absorption. CFBP-3 ranked second with a GRG of 0.655 due to its exceptional impact strength and balanced properties. CFBP-6, which had the highest flexural strength (134 MPa) and hardness (62.66 Shore D), achieved a GRG of 0.579. CFBP-9 demonstrated moderate performance with a GRG of 0.456, while CFBP-12, with the highest residual char (22.0036%) and enhanced thermal stability, secured a GRG of 0.553.

Scanning Electron Microscopy (SEM)

SEM images of tensile tested specimens of fabricated composites are shown in Figure 1. SEM images reveal debonding, cracks, voids, and serrations in the composite structure. The observed debonding between the glass fibers and the matrix suggests the need for improved surface treatment or coupling agents to enhance adhesion. The presence of voids or air pockets within the composite can weaken the structure, compromising its overall integrity. Poor bonding between the fibers and the matrix can result in intermittent load transfer, contributing to the formation of serrations. The matrix material may yield or deform non-uniformly under stress, further promoting serrated fracture surfaces. The initiation and propagation of micro-cracks can lead to sudden drops in stress, manifesting as serrated patterns. Serrations are typically associated with areas of stress concentration and structural weakness, which can reduce the overall tensile strength of the composite. While in some cases, serrations might indicate localized ductile behavior that could improve toughness; they more commonly suggest brittle behavior. This is due to the increased ease of crack initiation and propagation, which ultimately lowers the composite's toughness. The uneven breakage of fibers during failure can also create serrated fracture surfaces, indicating a weak fiber–matrix interface, which diminishes the composite's load-bearing capacity. The SEM observations support the hypothesis of interfacial weakening through fiber–matrix debonding and microvoid evolution. While quantitative pull-out or work-of-adhesion tests were not performed in this study, these techniques, along with cohesive zone modeling, are recommended for future investigations to precisely quantify interfacial adhesion energy.

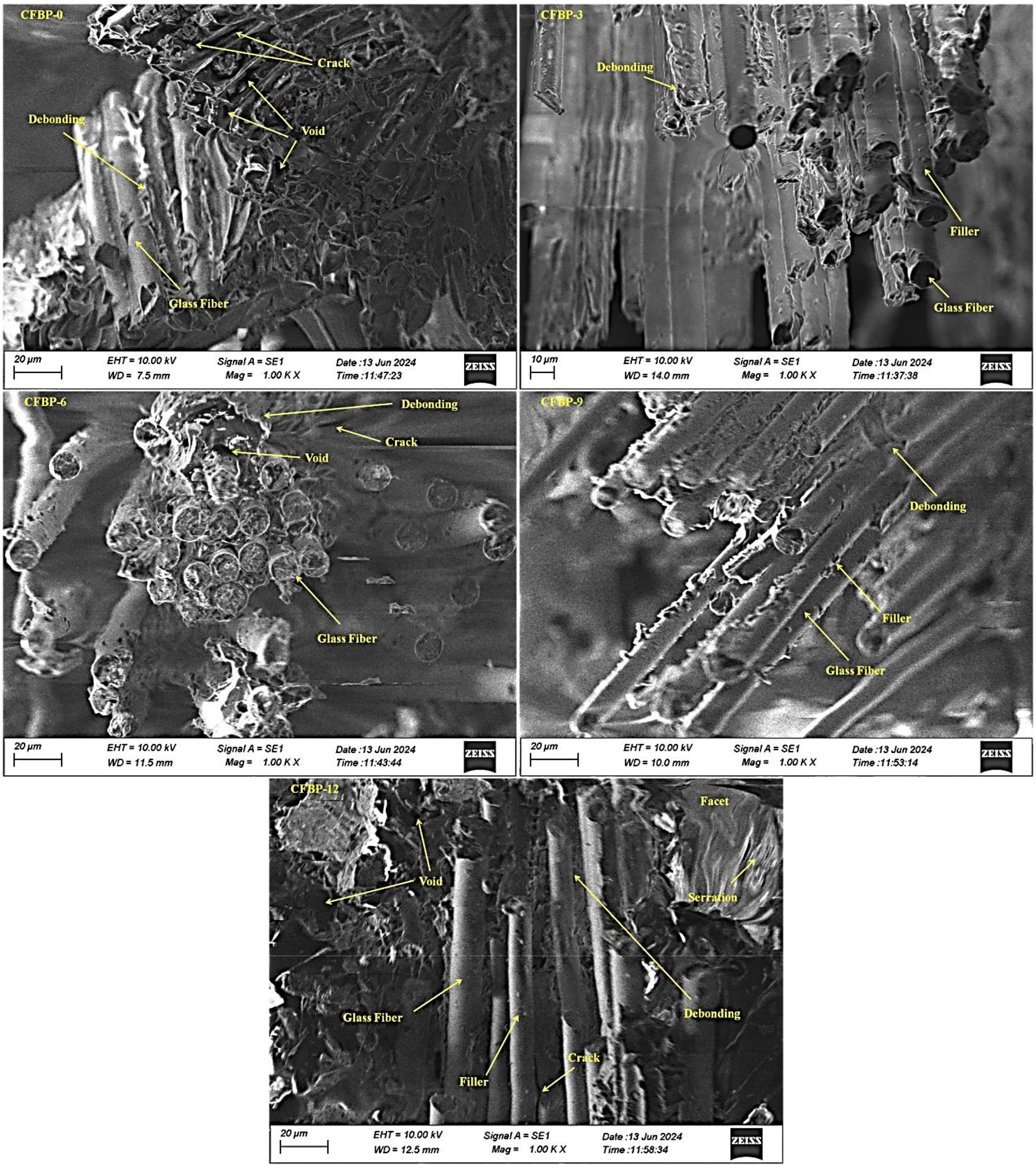

Figure 1. Tensile tested specimens of CFBP-glass fiber epoxy composites (CFBP-0 to CFBP-12) showing fracture morphology.

Table 3. Mechanical properties, water absorption, and TGA residual char of composites.

Sample ID	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m^2)	Hardness (shore D)	Water absorption (%) 24 Hours	TGA-residual char (wt. %)
CFBP-0	63	133	30	46.33	0.0067	7.3193
CFBP-3	33	110	60	60	0.5338	22.0036
CFBP-6	45	134	30	62.66	0.5763	6.2342
CFBP-9	38	102	30	62	0.5778	7.3193
CFBP-12	36	91	30	60.66	0.6281	22.0036

Table 4. Normalized data of mechanical properties, water absorption behavior, and residual char (TGA) of composites.

Sample ID	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m^2)	Hardness (shore D)	Water absorption (%) 24 hours	Residual char (wt. %)
CFBP-0	1.000	0.977	0.000	0.000	1.000	0.069
CFBP-3	0.000	0.442	1.000	0.837	0.152	1.000
CFBP-6	0.400	1.000	0.000	1.000	0.083	0.000
CFBP-9	0.167	0.256	0.000	0.960	0.081	0.069
CFBP-12	0.100	0.000	0.000	0.878	0.000	1.000

Table 5. Gray relational coefficients & gray relational grade.

Sample ID	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m^2)	Hardness (shore D)	Water absorption (%) 24 Hours	Residual char (wt. %)	GRG
CFBP-0	1.000	0.977	0.333	0.333	1.000	0.349	0.667
CFBP-3	0.333	0.473	1.000	0.754	0.370	1.000	0.655
CFBP-6	0.455	1.000	0.333	1.000	0.352	0.333	0.579
CFBP-9	0.375	0.403	0.333	0.927	0.351	0.349	0.456
CFBP-12	0.357	0.333	0.333	0.804	0.333	1.000	0.553

CONCLUSIONS

This study evaluated the mechanical properties, water absorption behaviour, and thermal stability of cuttlefish bone particle (CFBP)–glass fiber reinforced epoxy composites using Grey Relational Analysis (GRA) and Scanning Electron Microscopy (SEM). The following conclusions are drawn:

- CFBP-6 exhibited the highest flexural strength and hardness, while CFBP-3 excelled in impact strength. However, tensile strength decreased with filler addition
- Higher filler content increased water absorption and residual char, with CFBP-12 showing the highest values, indicating enhanced thermal stability
- CFBP-0 achieved the highest Gray Relational Grade (GRG) of 0.667 due to its superior tensile and flexural strengths and low water absorption, followed by CFBP-3 (GRG = 0.655), which excelled in impact strength and balanced performance
- Utilizing waste cuttlefish bone as a filler not only improves composite performance but also promotes eco-friendly and cost-effective material development

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