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Effect of Hybridization on the Hardness Properties of Nanoclay/S-glass Fibre/Epoxy Hybrid Nanocomposites

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Abstract : *This study evaluates the effects of nanoclay and S-glass fibre loadings, both individually and in combination, on the hardness of epoxy composites. Three composite groups were fabricated: epoxy/S-glass fibre, epoxy/nanoclay, and epoxy/S-glass fibre/nanoclay hybrids. Hardness values ranged from 31 to 93 HRA for epoxy/S-glass fibre composites, 31 to 109.6 HRA for epoxy/nanoclay composites, and 31 to 121.8 HRA for hybrid composites. The hybrid system with 36 wt% S-glass fibre, 4 wt% nanoclay, and 60 wt% epoxy exhibited the highest hardness (121.8 HRA), representing a 78% increase over neat epoxy and 31% and 13% improvements compared to the best epoxy/S-glass fibre (93 HRA) and epoxy/nanoclay (109.6 HRA) composites, respectively. Rockwell hardness testing confirmed progressive improvement with increasing filler content, while scanning electron microscopy (SEM) revealed uniform filler dispersion at optimal loading and interfacial defects at higher concentrations. The results demonstrate that hybridization of S-glass fibre and nanoclay provides synergistic reinforcement, achieving superior hardness compared to single-filler composites. However, beyond the saturation point, excessive filler addition resulted in diminished hardness due to agglomeration and weak matrix–reinforcement interfaces.*

Keywords: *Nanotechnology, Nanocomposite, S-glassfibre, Epoxy, Nanoclay, Hybrid composite, Hardness and SEM*

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1.0 Introduction

Hardness is one of the most important mechanical properties of engineering materials, reflecting their ability to resist permanent deformation when subjected to applied forces. Materials with high hardness are particularly valued for applications requiring wear resistance and durability, such as automotive components, gears, brake pads, construction frames, and structural parts (Abdullah *et al*., 2021; Abdullahi & Ansari, 2014). In modern material design, the reinforcement of polymer matrices with fibres and nanofillers has emerged as a vital strategy to enhance hardness and other mechanical properties (Abral *et al*., 2019; Almeida *et al*., 2018).

Nanotechnology plays a critical role in advancing polymer composites, as nanofillers such as nanoclays can significantly improve thermal, mechanical, and surface properties when dispersed within a polymer matrix (Kamel, 2019; Nikalje, 2024). Epoxy resins, in particular, are widely used in coatings, adhesives, aerospace, and structural applications due to their excellent adhesion and chemical resistance. However, their relatively low hardness and brittleness necessitate reinforcement for more demanding applications (Lam, 2020; Kusmono *et al*., 2023). Reinforcement with fibres such as glass fibre has been shown to improve stiffness, strength, and hardness of epoxy composites (Adekomaya & Adama, 2017; Avci *et al*., 2024; Aktas *et al*., 2023).

Research has demonstrated the positive impact of both glass fibres and nanoclays when used independently in epoxy composites. For example, glass fibre-reinforced epoxy composites exhibit significant improvements in hardness and impact resistance depending on fibre loading and orientation (Aramide *et al*., 2022; Awan *et al*., 2019). Similarly, nanoclay-reinforced epoxy composites enhance hardness, modulus, and dimensional stability due to the barrier effect and interfacial interactions of the nanoclay platelets (Man-Wai *et al*., 2020; Majid *et al*., 2021). Despite these advances, challenges remain regarding the optimization of reinforcement ratios and the interfacial compatibility between epoxy, glass fibres, and nanoclays (Al-Mosawi & Tabil, 2022; Marquis *et al*., 2022).

A key knowledge gap exists in understanding the synergistic effects of combining nanoclay and glass fibre in epoxy matrices. While previous studies have reported on epoxy/glass fibre composites and epoxy/nanoclay composites separately, fewer studies have systematically explored how hybridization of these reinforcements influences hardness properties and microstructural characteristics (Njuguna & Alcock, 2023; Njuguna *et al*., 2021). Addressing this gap is important for advancing the design of hybrid nanocomposites with tailored properties for industrial applications.

The present study, therefore, aims to investigate the effect of hybridization on the hardness properties of nanoclay/S-glass fibre/epoxy hybrid nanocomposites. Specifically, it compares the hardness performance of epoxy/glass fibre composites, epoxy/nanoclay composites, and epoxy/glass fibre/nanoclay hybrid composites. The findings of this research are significant because they provide insights into the potential of hybrid composites to surpass the limitations of single-reinforcement systems, thereby broadening their applicability in high-performance engineering, automotive, and structural applications.

2.0 Materials and Methods

2.1 Materials

The materials used for this research work and their specifications are: Montmorillonite Nanoclay (≥20 nm, 10- 400 GPa, 1.72 gm/cm^3 and 682608-500G), E-glass Fibre (300 GSM Specific gravity 2.6 gm/cc), Epoxy Resin (Araldite LY 506 Specific gravity 1.15-1.20 gm/cc), Epoxy Resin Hardner (Aradur HY 951 Specific gravity 0.97-0.99 gm/cc), Mould Releasing Agent (Poly vinyl alcohol PVA). Whereas the equipment/apparatus used for this research work, their model/standard, capacity and manufacturer/source are listed in Table 1.

Methods

2.2.1 Mould Design and Fabrication Method

The moulds used for fabricating all the composites were made of silicate glass and have the same dimensions of 200 x 200 x 3 mm dimension were fabricated locally using inert glass material. The glass sheets were marked and diamond glasscutter used to cut out the glass block from the sheet whereas the edges of the moulds were held together with adhesives.

2.2.2 Composites Preparation Method

The formulated mixtures of nano clay/epoxy of 99, 98, 97, 96 and 95-wt % epoxy with 1, 2, 3, 4 and 5-wt % nano clay respectively were prepared. The ratios were mixed using high-speed motorised stirrer. Mould release agent PVA was applied on mould plates in order to have smooth removal of moulded composites after curing. Secondly, epoxy/E-glass fibre composites consisting mixtures of 90, 80, 70, 60 and 50-wt % epoxy with 10, 20, 30, 40, and 50-wt % E-glass fibre respectively were also fabricated. Finally, hybrid nanocomposites were fabricated with a constant 60 wt % of epoxy while glass fibre and nano clay ratios were varied thus: 39, 38, 37, 36, 35 wt % glass fabric and 1, 2, 3, 4 and 5 wt % nano clay, respectively.

Table 1: List of Equipment

S/N	**Instrument**	**Model/standard**	**Capacity**
1.	Rockwell Hardness Tester (Scale A)	5019	53505 EN ISO 868
2.	Open Glass Moulds	-	-
3.	Atomic Force Microscope (AFM)	-	-
4.	Motorized Stirrer	-	1000 rev/min

The glass fibre (in fabric form) was placed on the mould after the initial application of the uniformly mixed formulation, the nano clay and the matrix system, which contains epoxy and hardener were mixed and spread uniformly. A roller was used to remove some of the entrapped air bubbles in order to reduce the development of voids. Hand Layup method was used to prepare glass fibre/epoxy and glass fibre/nano clay/epoxy composites by placing one glass fabric (mat) over another and applying the epoxy matrix between the glass fabric alone and the glass fabric with the nano clay (lamination) respectively while maintaining the thickness of the composite. Casting method was employed for nano clay/epoxy composites of various ratios. The moulds were left undisturbed for 24 hours and the samples were removed and cut into circular, dumbbell, square & rectangular shapes with diamond cutter according to ASTM

2.2.3 Glass fibre/ Epoxy Composites Fabrication Method

Hand layup method was employed in the preparation of glass fibre/epoxy composites. The following weight ratio 50:50, 60:40, 70:30, 80:20, 90:10 and 100:0 (control) of compositions were prepared see Table 3.3. Glass fabric was weighed,

Table 2: The Compositions of Epoxy and Glass fibre in the Composites

S/No	**Composite Name**	**Composite Code**	**Epoxy (wt %)**	**Nanoclay (wt %)**	**Glass Fibre (wt %)**
1	EP100GF0NC0	A	100	0.00	0.00
2	EP90GF10NC0	B	90	0.00	10.0
3	EP80GF20NC0	C	80	0.00	20.0
4	EP70GF30NC0	D	70	0.00	30.0
5	EP60GF40NC0	E	60	0.00	40.0
6	EP50GF50NC0	F	50	0.00	50.0

Epoxy was measured out and poured into a beaker and epoxy hardner was measured and poured into the same beaker in the ratio of 2:1. The contents of the beaker were subjected to mechanical agitation using a high-speed motorized stirrer at 1000 rev/min. for a period of five minutes for proper mixing. Releasing agent (PVA) was applied on the mould, a little of the epoxy and hardner homogenous mixture were poured in, an E-glass fabric placed, matrix mixture applied again, E-glass fabric placed, matrix mixture poured in, this continued until the glass fabric was exhausted and the remaining mixture was poured, to form the outer covering of the composite. This method was used to fabricate all the varieties of E-glass/epoxy fabric composites produced in this research work.

2.2.4 Nano clay/Epoxy Composites Fabrication Method

The nano clay/epoxy composites were prepared by varying the weight percentage of nanoclay from 1 wt% to 5 wt% while keeping the epoxy content as the primary matrix. A neat epoxy sample (without nanoclay) was also prepared as the control. The detailed compositions of the fabricated composites are presented in Table 3.

Table 3: The Compositions of Epoxy and Nano clay in the Composites

S/N	Composite Name	Composite Code	Epoxy (wt %)	Nanoclay (wt %)	S-glass Fibre (wt %)
1	EP100GF0NC0	A	100	0.00	0.00
2	EP99GF0NC01	B	99	1.00	0.00
3	EP98GF0NC02	C	98	2.00	0.00
4	EP97GF0NC03	D	97	3.00	0.00
5	EP96GF0NC04	E	76	4.00	0.00
6	EP95GF0NC05	F	75	5.00	0.00

Nano clay/epoxy composites with percentage weight composition of 99:1, 98:2, 97:3, 96:4, 95:5, and 100:0 (control) were fabricated with open moulds see Table 3.4. The moulds were cleaned and mould release agent applied on them. Proper weight percent of epoxy resin was measured and poured into a beaker and proper weight nano clay was measured out and added to the beaker. The contents were mechanically agitated using high-speed motorized stirrer at 1000 rev/min, for five minutes for proper mixing, then Epoxy hardener were measured and poured into the same beaker and the agitation continued for another about five minutes for homogenous mixing. The uniform mixture was then poured into the moulds and allowed to cure overnight. The composites were removed from the mould and the process repeated using with the various compositions as listed above.

2.2.5 Glassfibre/Nano clay/ Epoxy Hybrid Nanocomposites Fabrication

To investigate the synergistic effect of glass fibre and nanoclay on the hardness properties of epoxy composites, hybrid nanocomposites were developed by varying the proportions of nanoclay and glass fibre while keeping the epoxy content constant at 60 wt%. Six different formulations, including a neat epoxy control sample, were prepared. The detailed weight compositions of epoxy, nanoclay, and glass fibre used in fabricating the hybrid composites are presented in Table 4**.**

10 g grams of S-glass fibre/nano clay/epoxy hybrid composites with percentage weight composition of 60:39:1, 60:38:2, 60:37:3, 60:36:4, 60:35:5, and 100:0:0 (control) were fabricated with open mould, see Table 3.5. For the first composite, 3.9 g of glass fibre of stable length 200 mm was measured out and five even mats of dimension 200 x 200 mm were woven from it, then the moulds were cleaned and mould release agent applied. 6.0 g matrix system containing epoxy resin and epoxy hardener in the ratio of 2:1 i.e. 4.0 g epoxy and 2.0 g hardener were measured

separately. The epoxy alone was then poured into a beaker and 0.1 g nano clay was measured out and added to the beaker.

The content was mechanically agitated using high speed motorized stirrer at 1000 rev/min, for five minutes for proper mixing then epoxy hardener was measured and poured into the same beaker and the agitation continued for another five minutes for homogenous mixing. A portion of the uniform mixture was poured into the mould and an E-glass fabric layed up the mixture is poured again and spread with a brush on the mat, another mat placed and the mixture poured in this continues until all the five the E-glass fabric were exhausted, then the remaining mixture poured. The moulds were allowed overnight to cure properly. The composites were removed from the moulds and the processes repeated with the other compositions.

Table 4: The Compositions of Epoxy, Glassfibre and Nano clay in the Composites

S/No	Composite Name	Composite Code	Epoxy (wt %)	Nanoclay (wt %)	Glass Fibre (wt %)
1	EP100GF0NC0	A	100.0	0.00	00.0
2	EP60GF39NC01	B	60.0	1.00	39.0
3	EP60GF38NC02	C	60.0	2.00	38.0
4	EP60GF37NC03	D	60.0	3.00	37.0
5	EP60GF36NC04	E	60.0	4.00	36.0
6	EP60GF35NC05	F	60.0	5.00	35.0

2.3 Hardness Property Testing Method

The hardness of the various composites; epoxy/glass fibre, epoxy/nano clay and epoxy/glass fibre/nano clay hybrid composites were tested experimentally using Rockwell hardness tester scale A, Model Number: 5019, Serial Number: 01554, DIN: 53505EN ISO 868, ASTM: D2240 ISO 7619. Rockwell tests is manually operated with the following features: preliminary test force: 98.07 N (10 kgF), additional test force: 490.3, 882.6, 1373 N (50.90, 140 kgF) and total test force: 588.4, 980.7, 1471 N (60, 100, 150 kg F). The test samples were square-shaped.

3.0 Results and Discussion

The hardness properties of the three groups of composites—glass fibre/epoxy, nanoclay/epoxy, and glass fibre/nanoclay/epoxy hybrid nanocomposites—are presented in *Fig.* 1. The results showed that hardness values varied from 31 to 93 HRA for glass fibre/epoxy composites, 31 to 109.6 HRA for nanoclay/epoxy composites, and 31 to 121.8 HRA for hybrid glass fibre/nanoclay/epoxy composites. The neat epoxy served as the control sample with the lowest hardness value of 31 HRA.

Among the fabricated composites, the maximum hardness value of 121.8 HRA was recorded for the hybrid composite (Composite E) with a composition of 60 wt% epoxy, 36 wt% glass fibre, and 4 wt% nanoclay. This represents a significant improvement compared to both single-reinforcement composites and the neat epoxy matrix, indicating that hybridization provided a synergistic effect. The progressive increase in hardness values observed from composites B through E was attributed to the increased filler content and the strong interfacial bonding achieved between nanoclay platelets, glass fibres, and the epoxy matrix. Good interfacial adhesion enhances stress transfer efficiency from the matrix to the reinforcements, which directly improves the hardness and overall resistance to deformation (Kusmono *et al*., 2023; Man-Wai *et al*., 2020).

However, further increase in filler content, as observed in composite F, led to a slight decrease in hardness. This reduction may be due to poor dispersion and the onset of filler agglomeration or clustering, which created stress concentration zones within the epoxy matrix. Excessive filler loading can also cause fibre–matrix debonding and non-uniform distribution of nanoclay platelets, thereby reducing the interfacial cooperation between the matrix and reinforcements (Etmimi, 2022; Almeida *et al*., 2018; Al-Mosawi & Tabil, 2022). This trend confirms that while moderate reinforcement levels significantly enhance hardness, excessive loading beyond the saturation limit can compromise the mechanical performance of the composites (Majid *et al*., 2021). These findings are in agreement with previous studies. For instance, Almeida *et al*. (2018) reported that the mechanical properties of coir/polyester composites improved with increasing reinforcement content up to an optimal threshold, beyond which deterioration occurred due to clustering. Similarly, Kusmono *et al*. (2023) and Man-Wai *et al*. (2020) observed that nanoclay-reinforced epoxy nanocomposites exhibited enhanced mechanical properties only at moderate filler concentrations, while excessive nanoclay loading led to agglomeration and poor stress transfer. Etmimi (2022) also highlighted that the synthesis and dispersion methods for polymer nanocomposites critically affect their hardness and other mechanical properties.

The results of this study demonstrate that hybridization of glass fibre and nanoclay in an epoxy matrix can provide superior hardness compared to single-filler composites, but optimization of filler loading is crucial to avoid clustering and interfacial incompatibility.

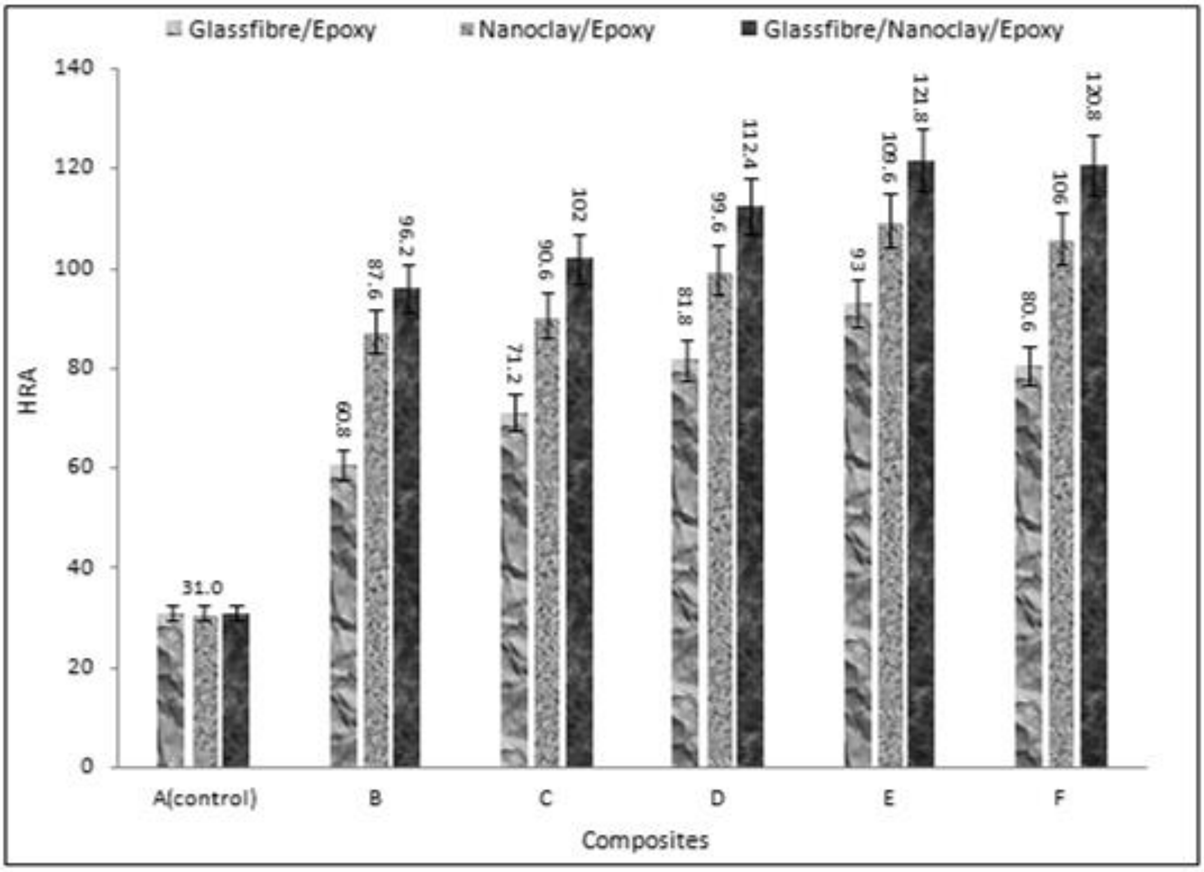


Fig. 1; Comparison of Hardness Properties of the Three Composites Fabricated

The hardness of glass fibre/epoxy composites varied from 31 to 93 HRA. The maximum hardness value of 93 HRA was obtained for Composite E, which contained 40 wt% glass fibre and 60 wt% epoxy. The increase in hardness from Composites B to E can be attributed to the increment in glass fibre content and the strong interfacial adhesion between the glass fibre and epoxy matrix. Effective wetting of the glass fibres by the epoxy matrix promotes uniform stress transfer across the fibre–matrix interface, thereby improving hardness performance (Adekomaya & Adama, 2017; Avci *et al*., 2024).

However, beyond 40 wt% glass fibre, as in Composite F, a reduction in hardness was observed. This reduction can be attributed to weak cohesion and poor wetting of the fibres by the epoxy, which led to void formation and interfacial debonding. At high fibre loading, fibre–fibre contact increases, preventing the matrix from sufficiently penetrating and bonding with all the fibres, which reduces the efficiency of load transfer (Parida *et al*., 2023; Awan *et al*., 2019). Similar results have been reported in epoxy/carbon fibre composites, where hardness and strength decreased at higher reinforcement loadings due to poor matrix infiltration (Ratiu, 2016).

The hardness of nanoclay/epoxy composites ranged from 31 to 109.6 HRA, with the maximum hardness observed in Composite E containing 4 wt% nanoclay. The improvement in hardness with increasing nanoclay content up to 4 wt% was due to the strong interfacial cohesion between the nanoclay platelets and the epoxy matrix, as well as the barrier effect created by uniformly dispersed nanoclay particles (Man-Wai *et al*., 2020; Majid *et al*., 2021). Proper exfoliation of nanoclay platelets allows for efficient load transfer, improved stiffness, and enhanced hardness.

Further increment in nanoclay beyond 4 wt% (Composite F with 5 wt%) resulted in a decline in hardness. This decline was due to poor dispersion and agglomeration of nanoclay platelets, which generated weak zones and hindered effective stress transfer. These clusters create micro-voids and reduce interfacial adhesion, ultimately compromising hardness (Asasutjarit *et al*., 2018; Kamel, 2019; Nikalje, 2024). Previous studies have confirmed that nanoparticles like nanoclay significantly enhance mechanical properties only at low concentrations, typically below 5 wt%, beyond which agglomeration reduces their reinforcing efficiency (Aramide *et al*., 2022; Kusmono *et al*., 2023).

The hybrid composites showed superior hardness compared to single-reinforcement systems, with values ranging from 31 to 121.8 HRA. The maximum hardness value of 121.8 HRA was achieved in Composite E with 60 wt% epoxy, 36 wt% glass fibre, and 4 wt% nanoclay. The synergistic effect of combining glass fibre and nanoclay produced enhanced interfacial bonding, better stress transfer, and minimized matrix deformation under load (Aktas *et al*., 2023; Njuguna & Alcock, 2023).

As with the single-filler systems, an excessive increase in nanoclay content (Composite F with 5 wt%) led to a decline in hardness. The decrease was linked to poor dispersion and cluster formation of nanoclay, which disrupted the fibre–matrix interaction. This supports earlier reports that nanoparticles, including nanoclays, significantly enhance mechanical properties only in minute quantities, and that excessive content results in clustering and poor interfacial cooperation (Parida *et al*., 2023; Marquis *et al*., 2022; Njuguna *et al*., 2021).

Scanning electron microscopy (SEM) images provided further insights into the microstructural reasons behind the observed hardness trends. Plate A shows Composite E of the glass fibre/epoxy group, where a homogenous distribution of fibres and effective matrix wetting resulted in a strong interface and high hardness. In contrast, Plate B (Composite F with >40 wt% glass fibre) revealed partial interaction between the matrix and fibres, with voids and fibre pull-outs, confirming that excessive fibre loading prevents proper wetting and reduces hardness. Similar findings have been reported by Adekomaya and Adama (2017), Awan *et al*. (2019), and Njuguna *et al*. (2021).

Plates C and D represent Composites E and F of the nanoclay/epoxy group, with 4 wt% and 5 wt% nanoclay, respectively. Plate C displayed exfoliated structures with uniform distribution and minimal clustering, corresponding to the highest hardness (109.6 HRA). Conversely, Plate D showed more agglomeration, leading to decreased hardness (106 HRA). Such cluster formation aligns with previous observations that agglomerated clay structures act as stress concentrators and reduce mechanical strength (Etmimi, 2022; Majid *et al*., 2021).

Plates E and F, representing hybrid composites with 4 wt% and 5 wt% nanoclay alongside glass fibre, revealed both uniform and clustered distributions. Plate E showed exfoliated structures with voids caused by entrapped air bubbles during mixing, while Plate F revealed larger clusters and slight agglomeration. Despite these imperfections, Composite E outperformed all others due to an optimal balance between fibre reinforcement and nanoclay distribution (Avci *et al*., 2024; Al-Mosawi & Tabil, 2022).

AFM imaging (Plate VIII) of the hybrid Composite F indicated an intercalated nanoclay structure, with orderly arrangement and limited voids. This suggested partial penetration of polymer chains into the nanoclay interlayer spacing, contrasting with agglomerated structures where no penetration occurs. Such intercalated and exfoliated structures are characteristic of high-performance nanocomposites (Kusmono *et al*., 2023; Marquis *et al*., 2022; Nikalje, 2024).

Finally, the control sample (Plate G) with 100 wt% epoxy showed a clear surface with minor voids, attributed to entrapped air bubbles during curing (Njuguna *et al*., 2021).

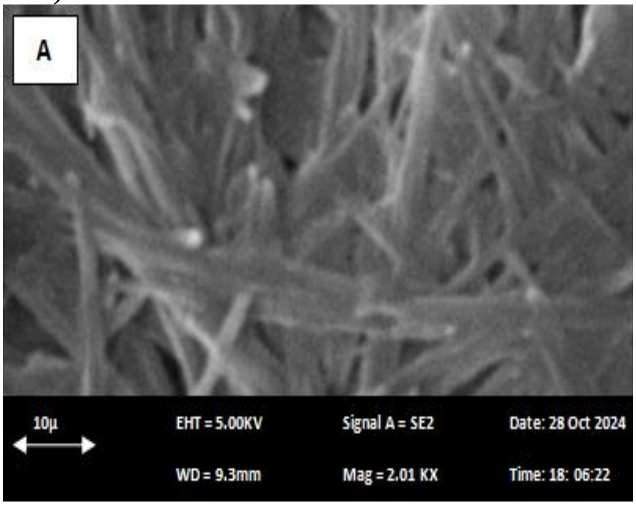


Plate A: SEM image of Composite E (60:40 wt % epoxy/s-glassfibre content)

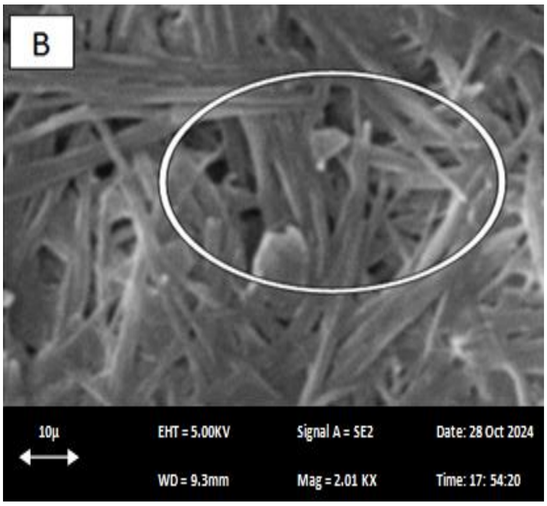
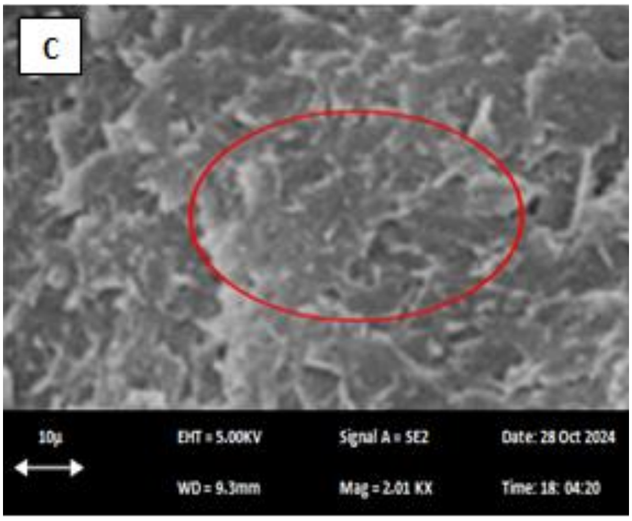


Plate B: SEM image of Composite F (50:50 wt % epoxy: S-glass fibre content)



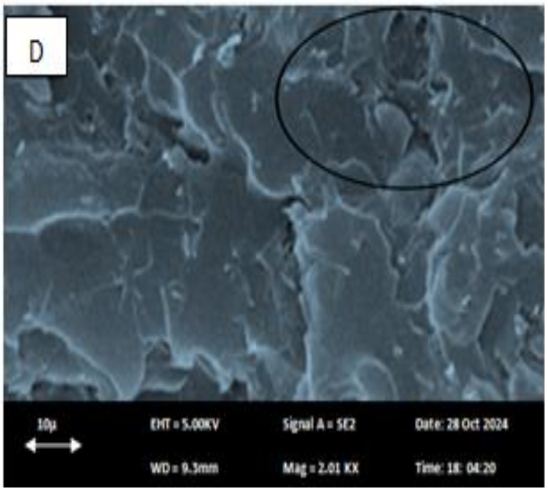


Plate C: SEM image of Composite E (96:4 wt % epoxy: nano clay content)

Plate D: SEM image of Composite F (94:6 wt % epoxy: nano clay content)

Fig. 2 : SEM micrographs of composites A to D at various compositions

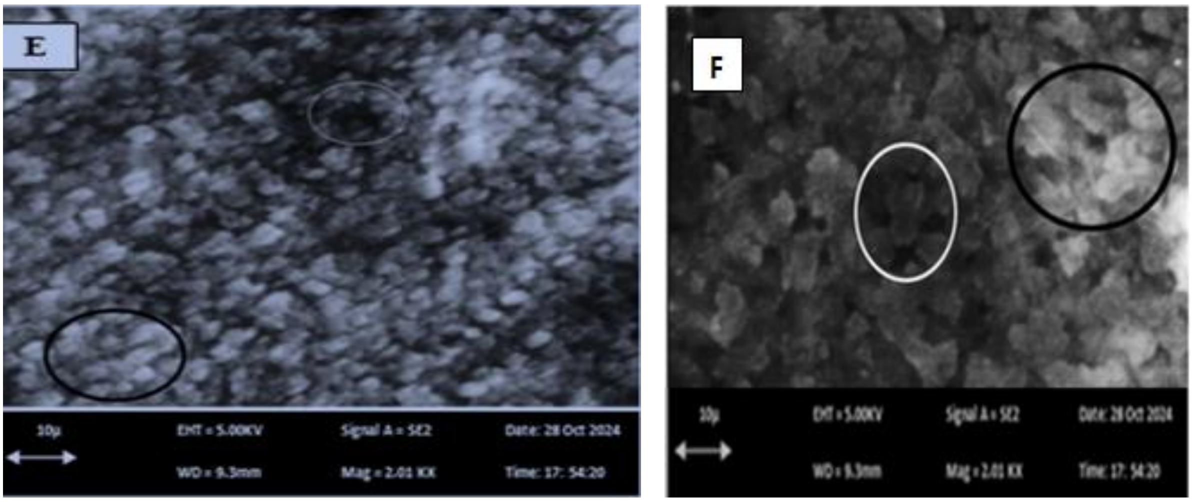


Plate E: SEM image of Composite E (4:36:60 wt % nano clay: S-glass fibre: epoxy content)

Plate F: SEM image of Composite F (5:35:60 wt % nanoclay: S-glassfibre: epoxy content)

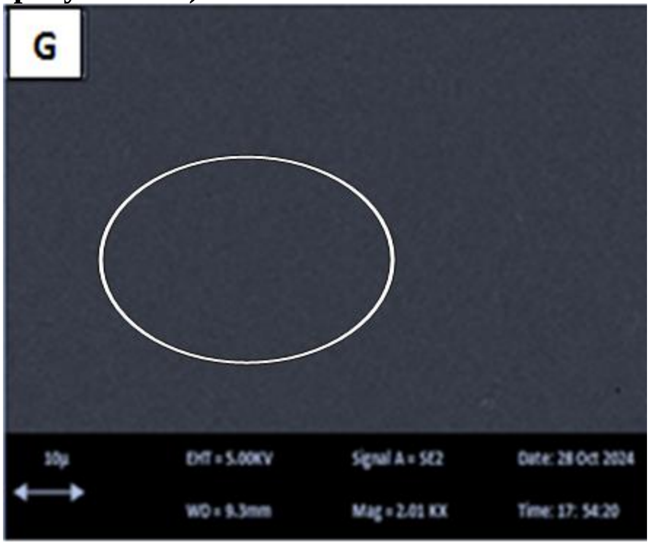


Plate G: SEM image of Composite 1 (control sample cured epoxy material)

Fig. 3 : SEM micrographs of Composites E, F and G

4.0 Conclusion

This study comprehensively investigated the hardness properties of epoxy/glass fibre/nanoclay composites. The results showed that the hybrid composite containing 60 wt% epoxy, 36 wt% glass fibre, and 4 wt% nanoclay (Composite E) exhibited the highest hardness value of 121.8 HRA. This was followed by the nanoclay/epoxy composite, with a maximum hardness of 109.6 HRA at 4 wt% nanoclay, while the glass fibre/epoxy composite recorded the lowest maximum hardness of 93 HRA at 40 wt% glass fibre. These findings confirm that both E-glass fibre and nanoclay improve the hardness of epoxy composites individually, but their combined reinforcement produces superior performance.

Overall, the hybrid composites demonstrated the best hardness properties, followed by nanoclay/epoxy composites, while glass fibre/epoxy composites performed least. SEM analysis further revealed that these differences in performance were due to variations in interfacial bonding, cohesion, and filler distribution within the epoxy matrix, with optimal interactions achieved in composites containing 4 wt% nanoclay and 36 wt% E-glass fibre.

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Consent for publication

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Availability of data

Data shall be made available on demand.

Competing interests

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Not applicale

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Authors' Contributions

Uche Ibeneme conceived the study, supervised fabrication, and drafted the manuscript. Bisike Chidiebere Egere guided methodology and data validation. Ejiogu Ibe Kevin managed research coordination. Amoke Austin conducted material characterization. Catherine Kigbo Oseshi analyzed results. Philip Abubakar handled experimental design. Yusuf Lawal Omeiza assisted fabrication. Sunday Adaogoshi Eya revised manuscript.