Research Article

Processing and Characterization of Carbon Nanofibre Composites for Automotive Applications

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1. Introduction

Nanocomposites provide a new class of material having combined properties of matrix and filler [1]. Nanocomposites using different fillers such as carbon nanotubes, nanofibres, silicates, clays, and metal nanoparticles can be prepared and applied in different fields like biomedical engineering, environmental applications, surface science, and the pharmaceutical field [2]. High-performance engineering materials with innovative properties were prepared through nanocomposite fabrication [3]. From the past few decades, the potential of carbon nanofibres (CNFs) and carbon nanotube (CNT) has been expanding [4]. Researchers around the globe are working to utilise the superior properties that these nanocomponents possess for various applications. The applications range from biosensors to new-age batteries [5]. The high surface area with less volume of CNF is suitable to suppress the defects that can be raised [6]. For micro-mechanical interlocking, the CNTs should exhibit some surface defects. This may include bonds in the CNT structure due to nonhexagonal defects and variation in diameter [7]. This kind of adhesion is very poor in CNT reinforced polymer composites because CNTs possess an almost smooth surface [8]. Chemical bonding includes ionic or covalent bonding capable of making changes in the smooth surface structure of CNTs. This helps to improve the effective stress transfer between the filler and matrix [9].

Depending on the carbon atom layer orientation in the CNF, the properties have varied. Carbon sp² filaments are stacked, and the CNFs are formed [10]. Depending on the stacking of the graphite planes, the CNF has different shapes [11]. Magesh et al. developed MWCNT incorporated...
semitransparent composites from PVDF and studied its electrical properties [12]. The distinctive arrangements of the graphene layers depend on the geometric aspects of the metallic nanoparticle catalyst and the feedstock of the gaseous carbon (CO or hydrocarbon gas) introduced during the synthesis processing [13]. For the manufacturing of VGCFNs, catalytic chemical vapour deposition (CVD) in combination with heat- and plasma-assisted vapour deposition is the most often utilised approach. CNFs are grown via chemical vapour deposition using gaseous hydrocarbon precursors and metal catalysts at high temperatures [14]. CNF can be made using a mix of organic polymer electrospinning and thermal aftertreatment in an inert environment [15]. Merneedi et al. studied the effect of MWCNT/graphite nanoplate in polystyrene. The developed nanocomposite which has application in the EMI shielding area showed graphite nanoplate-MWCNT-graphite nanoplate network-ing [16]. The creation of electrospun polymer nanofibres is usually the first stage in the electrospinning process, followed by stabilisation and carbonization treatments, the latter of which is done in an inert atmosphere [17]. Avinash et al. developed epoxy/MWCNT composites. They studied the effect of the change in diameter, aspect ratio, and X-band microwave absorption using three different commercially available MWCNTs [18]. CNF features the intrinsic qualities of traditional carbon fibres and huge surface-to-volume ratios, making them particularly suited for applications involving environmental contact [19]. Surface features of multifunctional CNFs must be changed depending on the application [20]. The excellent intrinsic properties and high aspect ratio make MWCNT favourable in the research and industrial field [21]. The π-σ interaction between MWCNT and PTT makes the importance of MWCNT-based PTT nanocomposites in the engineering application field [22].
Based on the assumption that this extended \(\pi-\pi\) interaction will greatly influence the electrical properties and EMI shielding capability of PTT/MWCNT nanocomposites, a suitable mixing method was opted to prepare PTT/MWCNT nanocomposites [23].

The present study is aimed at determining the best percentage of the CNF in the CNF-based epoxy composites for obtaining optimal mechanical properties. The content of CNF in the composite is varied from 1 to 4% concerning the weight of the phenolic resin. The developed samples are then tested for their tensile strength and toughness, and micrographs are obtained using SEM.

2. Methodology

In this study, billets of CNF composites with phenolic resin are prepared with dimensions 5 mm diameter and 10 mm length [24]. CNF is procured with diameter dimensions of 10 nm and a length of 100 nm [25]. CNFs with large aspect ratios (length/diameter (L/D) ratios) agglomerate easily in general [26]. An overview of the properties of CNF and applications is shown in Figure 1.

Their aggregation decreases as the aspect ratios decrease. Vibration milling was employed to dissolve the agglomeration of CNFs and disseminate them uniformly in the matrix [27]. The different shapes such as platelet type CNF, tubular type CNF, and fishbone CNF type are shown in Figures 2(a)–2(c). The CNFs were milled to lower their aspect ratios throughout this operation [28]. The vibration-milled CNFs were agitated using ultrasonication in distilled water with polycarbon acid amine as a surfactant to improve their dispersion [29].

The distributed CNFs were sensitised and activated in SnCl\(_2\) and PdCl\(_2\) solutions at room temperature, respectively [30]. The pretreated CNFs were then filtered and thoroughly washed in distilled water [31]. CNF powder was combined

| Sample  | Trial 1 | Trial 2 | Trial 3 |
|---------|---------|---------|---------|
| CNF 1%  | 27.6    | 29.1    | 25.4    |
| CNF 2%  | 31.5    | 30.1    | 31.2    |
| CNF 3%  | 35.2    | 36.1    | 35.7    |
| CNF 4%  | 36.2    | 37.8    | 36.6    |

Figure 3: SEM micrographs of (a) 1% CNF, (b) 2% CNF, (c) 3% CNF, and (d) 4% CNF composites.

Figure 4: XRD of CNF composites (JCPDS card no. 96-101-106).

Table 1: Microhardness (HV) of CNF composites.
with polyglycol-dissolved phenolic resin before making the CNF composite [32]. The phenolic resin was a thermosetting polymer made from phenol and formaldehyde with ammonia as a catalyst [33]. The billets are prepared such that the weight ratio of CNF varies between 1%, 2%, 3%, and 4%. For each percentage of CNF, multiple billets are prepared without changing the parameters. The mixed CNF and phenolic resin are then cured in an oven at 160°C to obtain the solidified billet sample [34]. For the developed samples, microhardness is tested with microindenter, tensile strength is calculated using UTM, and SEM micrographs are obtained by FE-SEM [35]. The chemical analysis of the CNF is done by using XRD analysis.

### 3. Results and Discussion

**3.1. SEM Micrographs.** The developed CNF composites are tested under the FE-SEM setup for identifying the distribution of CNF in the composite. The developed SEM micrographs are given below. It indicates that the CNF have been dispersed uniformly in the matrix. All the CNF content percentages are dissolved uniformly in the composite due to ultrasonic mixing before adding the resin [36]. The CNF ratio has been reduced by milling before the mixing with resin and improved the wettability of the fibres. No agglomeration of CNF in the matrix has been found for any of the developed composites. The four samples analysed for the SEM micrographs are given in Figure 3 and have no voids or crack defects, and also, agglomeration of fibres is absent.

**3.2. XRD Analysis.** Figure 4 exposes the peaks of graphitic-like carbon which can be seen in the CNF composite XRD diffraction pattern conforming JCPDS card no. 96-101-106. In terms of XRD patterns, the CNF composite closely mimics CNFs, implying that the graphite-like structure is preserved after the CNF powder is formed with polymer binder by pressing and then carbonized at high temperatures [37]. In the XRD diffraction spectrum, the contribution of polymer-derived carbon in the composite is undetectable. This could be caused by the surface graphitization of thermosetting resin during carbonization, resulting in a graphite-like coating that is distinct from the bulk glassy carbon. Therefore, it can be said that the composite is unaffected by any other contaminant or the curing medium or gasses. And the properties obtained by further investigation give the values of the CNF composite [38].

**3.3. Mechanical Properties**

**3.3.1. Hardness.** When a load is applied on a softer material, the substrates elastically deform, and the hardness measured is underreported. As a result, the results achieved in this case are the matrix and CNF collective hardness, which considerably reduced the composite attributes. Every sample has been subjected to many trials, with an average hardness value derived. The hardness of the composite is tested with the microindenter with a 100 g load for each indentation. Vickers indentation by Shimadzu has been used for the assessment. The hardness of the composites is increasing with an increase in the percentage of CNF in the composite.

| Sample | Trial 1 | Trial 2 | Trial 3 |
|--------|---------|---------|---------|
| CNF 1% | 31.5    | 30.8    | 32.6    |
| CNF 2% | 35.5    | 37.8    | 35.3    |
| CNF 3% | 39.2    | 39.8    | 41.3    |
| CNF 4% | 40.5    | 42.3    | 45.8    |

![Figure 5: Hardness results of CNF composite.](image)
1% CNF composite has a lesser hardness, and 4% has a higher hardness than the other composites. The values of the hardness obtained for each sample are given in Table 1. All values are recorded for an indentation period of 15 seconds. Figure 5 shows the hardness of CNF composites. The variation in the hardness values can be explained as the distribution of CNF at the surface layers of the composite for the higher CNF percentage composites is high; hardness is higher for the 4% CNF composites and gradually reduced for the lesser CNF percentage composites.

3.3.2. Tensile Strength. The tensile strength of the composites measured by UTM is given in Table 2. The composite with higher CNF content has a higher tensile strength value compared to other fabricated composites. The higher CNF in the composite absorbs the load as the bonding between matrix and CNF is sufficient without any voids. Figure 6 shows the tensile strength of CNF composites. There is a 48% increment in tensile strength for the composite when the CNF content is increased from 1% to 4% in the composite composition. The increment percentage is 22% when CNF is increased from 1 to 2% in the composite [39]. The percentage is reduced to 5% for tensile strength increment when CNF is bumped up to 3% from 2%. And from 3% CNF to 4% CNF, the tensile strength is even lesser than 5%. This shows that the increment in CNF content increases the tensile strength, but the increment level reduces gradually.

4. Conclusion

Phenolic resin with different wt% of carbon nanofibre composites was fabricated successfully. Among the composites, the one with higher CNF content has superior properties in terms of mechanical properties. The superior mechanical properties of CNF have influenced greatly testing composites.

(1) The XRD analysis revealed that there are no contaminants and other elements in the composite sample. XRD peaks obtained that CNF and resin

(2) The increment in CNF content increases the tensile strength, but the level of increment reduces gradually

(3) CNFs with 4 wt% composite tensile strength results were increased 48% compared to 1% of CNF composite

(4) The gradual increase in the CNF content in the composite will increase the strength of the composite and hence is useful in the automotive industry

Data Availability

The data used to support the findings of this study are included within the article. Should further data or information be required, these are available from the corresponding author upon request.

Disclosure

The study was performed as part of the employment of Jimma Institute of Technology, Jimma University, Ethiopia.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.
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