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Development of High-performance Nanocoated Coir Fiber Cement Composite Using Surfactant Functionalized Multi-walled Carbon Nanotubes

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Abstract

Coir fibers have gained the interest of composite material reinforcement mainly because of their excellent mechanical properties in combination with their lightweight structure. Pure carbon nanotubes (CNTs) have hydrophobic surface and limited dispersion stability. CNTs may lack chemical affinities to these matrices and thus dispersion of CNTs into matrices poses the biggest practical challenge. The addition of a surfactant (sodium deoxycholate, NaDC) to a CNT improves its solubility in water, leading to anionic and cationic functional groups on the nanotubes surface. Epoxy resin acts as excellent component in binding CNTs to the coir fiber. After being treated with sulfuric acid and nitric acid in the ratio (3:1 v/v), multi-walled carbon nanotubes (MWCNTs) were functionalized with sodium deoxycholic acid to obtain functionalized-CNTs (f-CNTs). NaDC/CNT composites with different CNT loadings (i.e., 0.05, 0.1, 0.15, and 0.2 wt.%) are produced by initially dispersing the f-CNTs and sonicated. The main aim of this work was to investigate the impact of functionalizing MWCNTs as a coating material on coir fibers along the length of the beam of size 20 x 20 x 80 mm beam. The chemical interaction between the f-CNTs and coir fibers is established by Fourier transform infrared (FTIR) spectroscopy analysis. Scanning electron microscope (SEM) microstructural investigation demonstrates an improvement in the mechanical and fracture toughness of epoxy composites with 0.1 wt.%, owing to the proper MWCNT dispersion (with surfactant NaDC). Energy-dispersive X-ray spectroscopy (EDX) studies were carried out to validate the elemental composition of the sample. Consequently, it can be concluded that the CNT-coating proved effective in performing as a molecular coupling agent to make the composite structure compatible. Microscopic studies demonstrated coating of epoxy/f-CNTs and epoxy/non-functionalized-CNTs (n-CNTs) onto the fibers increased the contact through interlocking between the coir fiber, CNTs, and cement matrix.

Keywords

Carbon nanotubes, Coir fibers, Surfactant, Epoxy resin, Cement coir composites, Physical properties, Flexural test, Microstructural analysis

Introduction

The potential application of nanoparticles in the construction field has attracted a lot of interest. Based on its bonding, bridging, and mesh filling effects, studies demonstrated that the hybrid effect of CNTs and polymers boosted the compressive strength, decreased the conductivity abilities of the composites, and resisted crack formation by acting as a fiber in the cement aggregate interface. The addition of other materials to CNTs transforms them into the ideal and desirable for a broad spectrum of applications [1, 2]. The interfacial molecular engineering of CNTs, which aims to improve the state of interaction with other macromolecules and the eventual features of hybrid nanomaterials, has captured the interest of researchers and has become the topic of numerous processes [3, 4].

The procedures to develop hybrid composites have attracted considerable amount of attention, and to efficiently materialize most of these applications, CNTs are often functionalized with a variety of relevant polymers to enhance the characteristics of the finished product. Such assemblies have formerly shown that the functionalization of polymers to CNTs draws equal importance in the development of materials for future applications. Although CNTs and polymer matrix exhibit poor van der Waals interactions, their true potential as reinforcements has been severely restricted [4, 5]. Additionally, the commercialized CNTs supplied are in strongly entangled bundles which pose intrinsic challenges for dispersion. It has been focused on establishing approaches to alter the surface characteristics of CNTs to solve such challenges. Covalent and non-covalent functionalization can be distinguished between these approaches as they deal with interactions between active materials and CNTs [6, 7].

Although a variety of approaches have recently been used to improve the interaction between CNTs and polymers, the two primary approaches-covalent and non-covalent functionalization-remain the most prevalent. Depending on the platform, each approach offered merits. CNTs are typically subjected to a severe chemical treatment in covalent functionalization schemes to generate reactive compounds that improve aqueous solubility. This approach can damage the mechanical properties of nanotubes by additionally functionalizing their surfaces with carboxylic acid groups whilst leaving undesired structures behind. Non-covalent dispersion processes, on the contrary hand, employ the use of amphiphilic molecules that serve as surfactants. Their hydrophobic tail retains to the nanotube surface via non-covalent interactions that include - stacking, van der Waals, and charge-transferring interactions, while the hydrophilic half of the molecules confers aqueous solubility [3, 8, 9].

Surfactants are molecules that possess a hydrophilic portion, frequently referred to as the polar head group, and a tail that is hydrophobic. Due to their amphiphilicity, surfactants adsorb at surfaces and assemble themselves into supramolecular complexes. These characteristics have been employed to non-covalently disperse CNTs, whereby the surfactant physically adsorbs to the graphene sheet by hydrophobic, van der Waals and electrostatic attraction [10-12]. Based on the charge of their head group, surfactants are classified as anionic, cationic, non-ionic, or zwitterion. In the case of charged surfactants, electrostatic repulsions provide colloidal stability of the CNTs dispersion [13-15]. The substantial solvation shell produced by the hydrophilic moieties that surround the nanotube improves charge-neutral surfactant stability [16-18]. Ultrasonication, centrifugation, and filtration are widely employed in non-covalent functionalization to remove residual bundles and surplus surfactant molecules. The van der Waals forces interacts with CNTs are feeble, the huge surface area results in substantial interaction forces on the order of 1000 eV [9]. As a result of this intense contact, tight bundles form that are difficult to rupture. As a result, only prolonged and strong sonication can separate bundles [19-21].

Coir fibers offer excellent mechanical qualities, including the highest elongation of any existing natural fiber and the capacity to increase the durability of epoxy resin. Coir fibers, on the other hand, are hydrophilic, whilst epoxy resin remains hydrophobic. The hydroxyl groups on the outer layer of coir fibers absorb water molecules and establish hydrogen bonds, preventing mutual penetration and resulting in a fragile interfacial bonding capacity, which negatively impacts the mechanical characteristics of the composites [22-24]. By eliminating some non-cellulosic compounds from the fibers and increasing the surface toughness of the fibers, alkali treatment improves interfacial bonding strength [25-28]. Therefore, the current main research task is to improve the compatibility between coir fibers, CNTs and epoxy resin matrix, enhancing the interfacial bonding ability between them.

In the present investigation, f-CNTs and n-CNTs were used as reinforcement fibers. The composite/synergistic impact of coir fibers coated with CNTs along with epoxy resin reinforced across the length of the beam in cement mortar was formulated using various combinations of f-CNTs and n-CNT and the strength as well as durability efficiency was evaluated using mechanical properties. The elasticity of the mortar mix is evaluated using a universal testing machine. SEM is employed to characterize the hydrated products, and the outcomes were contrasted to the standard ordinary Portland cement paste.

Experimentation

Materials

Ordinary Portland cement

In the present study Ultratech cement of 43 grade is used matrix to develop the cement composite.

Coir fiber

Coir fibers are biodegradable materials and having various structural application. In the present study coir fiber use used as fiber, hence locally available coir fibers of length 10 cm are considered and then surface cleaned with sodium hydroxide (NaOH) to remove surface impurities. Ten numbers of coir fibers are taken and tied together in form of cluster and used as reinforcement at macro level. The properties of coir fiber are listed in table 1.

Table 1: Properties of coir fibers.

Coir fiber	Properties
Density	1200 kg/m^3
Pectin	3 - 4%
Lignin	41 - 45%
Hemicellulose	0.15 - 0.2%
Cellulose	32 - 43%
Ash	2%

MWCNTs

MWCNTs were obtained from UNITED NANOTECH (Bengaluru, Karnataka, India). At the nanoscale, it acts as reinforcement. The properties of MWCNTs are listed in table 2.

NaDC

NaDC were obtained from Research-Lab Fine Chem Industries, Mumbai (Maharashtra, India). This is chosen as they have been proven to enhance the dispersion of MWCNTs (Table 3).

Epoxy resin and hardener

L-12/K-6 were procured from LAPOX supplied by Atul Ltd. and is chosen for its distinctive ability to bond to fibers (Table 4).

Methodology

The proposed work involves the development of nanocoating for coir fiber to improves their mechanical properties and later these fibers used as reinforcement for cement composite. Two categories of nanocoating are developed by varying percentage of MWCNT with epoxy resin as binder. In former case, the CNTs are functionalized (f-CNT). with the sulfuric acid and nitric acid and in later case only MWCNT-epoxy resin n-CNTs as described in table 5. Epoxy resin and hardener, locally available coir fibers were taken and treated with NaOH to remove surface impurities. Cement composites are prepared by adding this coir fiber to evaluate the load carrying capacity and flexural strength of composite.

Table 2: Properties of MWCNTs as per suppliers' specifications.

MWCNTs	Description
Production method	Chemical vapor deposition (SLV)
Available form	Black powder
Diameter	Avg. outer diameter: 25 nm
Diameter	Avg. outer diameter: 25 nm
Length	Avg. 10 micron
Nanotubes purity	> 98%
Metal particles	< 1%
Amorphous carbon	< 1%
Specific surface area	220 m^2/g
Bulk density	0.14 g/cm^3

Table 3: Properties of NaDC as per supplier's specifications.

Character property	Standard	Observed
Description	White moist powder	White moist powder
Assay	(Min) 98%	91.52%
pH (10% water)	07 - 10	8

Table 4: Properties of epoxy and hardener as per supplier's specifications.

Character property	Inferences
Product type	Epoxy resin and hardener
Model No.	L-12 and K-6
Brand	Lapox
Pot life	1/2 to 1 h at 20 °C
Shear strength	1.4 kg mm/min
Temperature (°C)	100 °C
Viscosity	9000 to 12000 m Pa.s. at 25 °C
Curing time (minimum)	15 to 30 min at 100 °C

Table 5: Sample specification of various nanocoated coir fiber cement composition.

Sample constitution	Sample
PC - Plain cement	PC
PC + e-0.05f-CNT (Epoxy - 0.05% f-CNT coated coir fiber)	F1
PC + e-0.1f-CNT (Epoxy - 0.1% f-CNT coated coir fiber)	F2
PC + e-0.15f-CNT (Epoxy - 0.15 % f-CNT coated coir fiber	F3
PC + e-0.2f-CNT (Epoxy - 0.2% f-CNT coated coir fiber)	F4
PC + e-0.05n-CNT (Epoxy - 0.05% f-CNT coated coir fiber)	N1
PC + e-0.1n-CNT (Epoxy - 0.1% f-CNT coated coir fiber)	N2
PC + e-0.15n-CNT (Epoxy - 0.15% f-CNT coated coir fiber)	N3
PC + e-0.2n-CNT (Epoxy - 0.2 % f-CNT coated coir fiber)	N4

Functionalization of CNTs

Functionalization of CNT is carried by adding 2 g of MWCNT to 100 ml of sulfuric acid and nitric acid in the ratio (3:1) by volume. The mixture is heated in water bath at 30 °C for 3 h with stirring at regular intervals. MWCNTs are processed by filtering and washing using deionized water until the pH is neutral, then drying at 60 °C. Later it is modified by surfactant sodium deoxycholic acid with mass ratio (1:1). The processed mixture is ultrasonicated for 30 min and magnet stirred for 8 h, filtered and washed with ultrapure water and dried at 60 °C for 24 h. The product obtained is f-CNTs.

Coating of epoxy/f-CNTs and epoxy /n-CNTs on coir fiber

Coir fiber (10 cm long) is primarily treated with 5% of NaOH and kept in the solution for 72 h (3 days) and then washed and dried. Later immersed in epoxy resin first, and then f-CNTs (MWCNT/NaDC) are bonded to the coir fiber by hand lay-up method. Similarly, n-CNTs are coated on coir fiber. The coated coir fibers are now heated to 200 °C for 5 h to remove any contaminants.

Fabrication of composites

Coated coir fibers (10 cm long, 10 in number) are clustered together and tied at the ends on both sides, which is used as reinforcement in cement composite as shown in figure 1. The cement composites are casted by adding the various

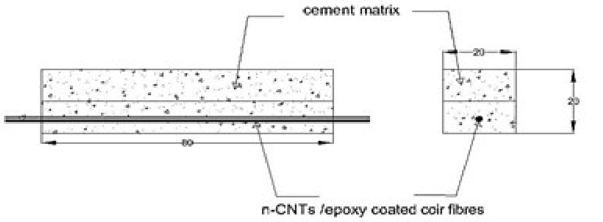

Figure 1: Schematic diagram of cement composite reinforced with coated coir fiber.

composition of above nanocoated coir fiber. Cement paste is prepared with a w/c ratio of 0.45 and poured into the mold of size 20 x 20 x 80 mm and cured for 28 days [29]. Flexural tests are conducted to evaluate the load carrying capacity of these composites.

Experimental testing

Tensile test on coir fiber

Tensile testing is performed using ASTM A370 specimen characteristics [30]. Tensile strength is determined on coated and uncoated coir fibers of 10 cm length. A load cell frame with a strain rate of 0.125 mm/min and a capacity of 10 kN was used to test the machine. The optimum proportion of coated coir fiber with epoxy/f-CNTs and epoxy/n-CNTs as reinforcement in beams was assessed based on the results of tensile tests. Figure 2 illustrates the experimental test setup for coir fiber.

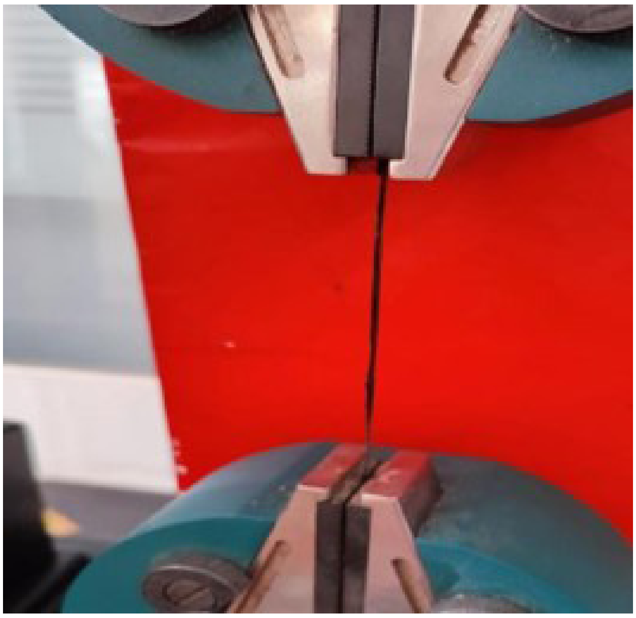

Figure 2: Tensile test of coir fiber.

Flexural test on nanocoated coir cement composite

The flexural properties of epoxy/f-CNTs and epoxy/n-CNTs coated coir fiber reinforced cement composite materials were computed using a three-point load accordance to ASTM C293 [31]. In a universal testing machine, a load cell frame with a capacity of 10 kN and a strain rate of 0.125 mm/min was implemented. Load-deflection graphs were constructed for each sample after obtaining deflections at regular load intervals. The loading equipment is shown in the figure 3 complying with ASTM requirements. Based on the outcomes of three-point loading tests, the appropriate proportion of coated coir fiber with epoxy/f-CNTs and epoxy/n-CNTs as reinforcement in beams was estimated. As a result, the flexural behavior of coated reinforced beams was investigated.

Figure 3: Flexural test of cement coir composite.

Results and Discussion

Tensile strength

Total of 11 samples were clustered and tested for tensile strength. The tensile load of tN2 sample (un-functionalized CNT with 0.1% wt.) is highest with maximum deflection of 0.058 mm. Due to functionalization there was increase in the tensile strength. C3 sample i.e., epoxy coated coir fiber has a comparatively increased tensile strength as epoxy has high cohesive force, excellent stability, and flexibility. The degree of activation of the fiber was improved due to the good wettability of individual CNT fibers by epoxy resin coating, which enhanced tensile and Young's modulus (Figure 4).

Flexural strength

It was performed using ASTM C293 specimen characteristics. There were in total 9 samples tested for flexure and each sample acronym is shown in table 5. Further, load vs deflection for each of the cases depicted in figure 5. For 975 N load the sample yielded 0.28 mm deflection. Till

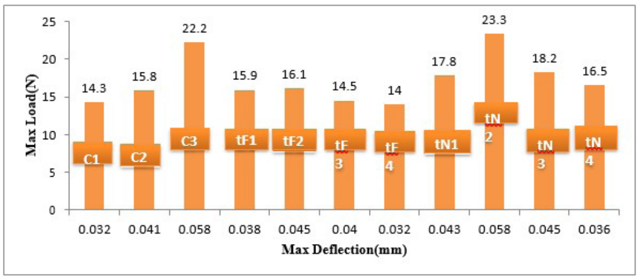

Figure 4: Tension test nanocoated coir cement composite specimen.

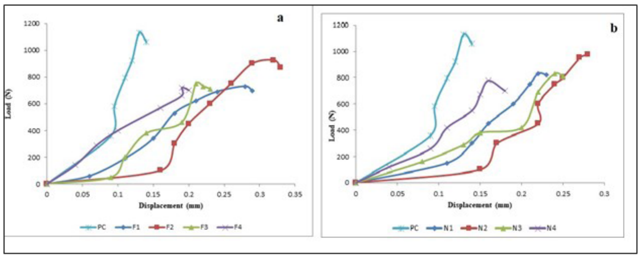

Figure 5: Load vs Displacement curve of **(a)** f-CNTs and **(b)** n-CNTs for various proportions coated with epoxy on coir fiber reinforced in cement composite.

0.1% n- CNT-epoxy coated coir fiber (N2) the deflection increased but in subsequent additions of n-CNTs resulted in reduction of load bearing capacity. This may be attributed to the agglomeration of the CNTs on the surface of the coir fiber. For f-CNTs- epoxy coated coir fiber (F2) the maximum load is 925 N which yielded 0.32 mm deflection. In comparison with the sample N2, though there is less load carrying capacity (5.4% lesser) but had increased deflection (14% more). This can be accounted to the functionalization of CNTs which increased the modulus of elasticity of the composite. Plain cement composite had high load carrying capacity but very low modulus of elasticity, owing to the fact of being a brittle material by itself. Thus, functionalization aided the increase in strength as well as overall increased the flexural strength of the composite [32, 33]. The composite beams containing n-CNT and f-CNT loadings of 0.05%, 0.1%, 0.15%, and 0.2% are tested for flexural strength. The beams with n-CNTs loading of 0.1% coated on coir fibers (N2) demonstrated improved load carrying capacity with higher deflections in comparison with the other proportions. This may be due to the proper dispersion of the CNTs in the epoxy resin. On the other hand, composite beams with f-CNTs loading of 0.1% coated on coir fibers (F2) had a slight lesser i.e., 5.4% lesser load carrying capacity in comparison with the n-CNTs.

Microstructural analysis

The microstructural analysis is performed to know the interaction between epoxy, f-CNTs and coir fiber. The effects on cement composites are assessed by as below.

SEM analysis

SEM images in figure 6a to figure 6d showed brittle fracture features and aggregations of NaDC/MWCNTs which explained its low yield strength. SEM images in figure 6e to figure 6h shows the epoxy coated n-CNT on coir fiber exhibits relatively smooth fracture surface as there is a proper dispersion of CNTs on the coir fibers. Figure 6a depicts the proper dispersion of f-CNTs/epoxy as a coating material on the coir fibers. In the figure 6c was established that NaDC/MWCNTs have high dispersion and possess strong interfacial interaction with epoxy. The primary objective was to locate the ITZ in SEM pictures. In figure 6e, since pure MWCNT samples do not easily break, transgranular failure behavior must commence at the nanotube's periphery. The overall lengths of these flakes are not consistent with peaks and valleys, allowing greater area for water and air particles to penetrate, resulting in early disintegration of the member. In figure 6g MWCNTs and epoxy in composites demonstrated greater load carrying capacity and deflection, which might be attributed to the reinforcement provided by the filler components at both the micro- and nano-levels. The epoxy resin infiltrated through the CNTs coated on coir fiber, generating a thick epoxy/CNT composite layer and relocating the fracture zone within the fiber.

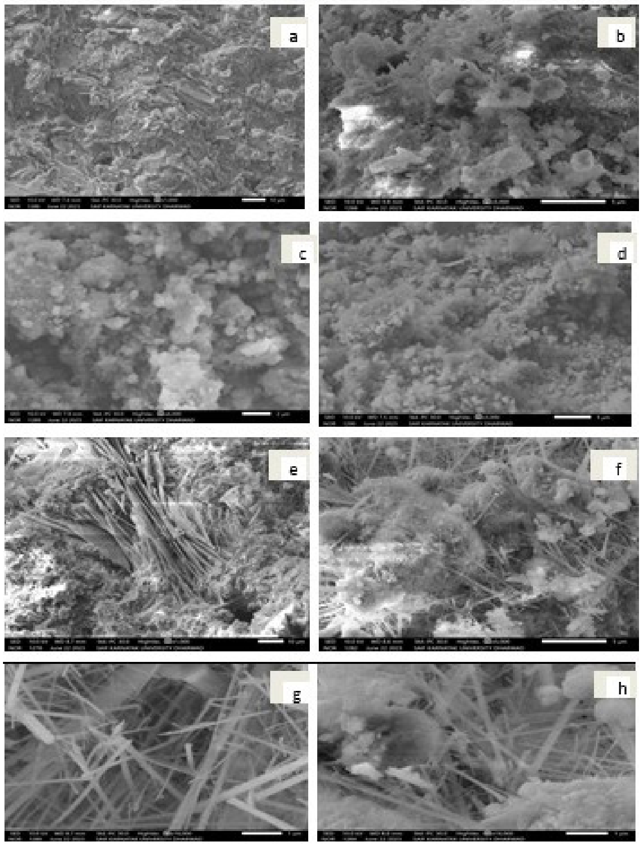

Figure 6: SEM images of **(a-d)** f -CNTs and **(e-h)** n-CNTs with epoxy coated coir fibers.

FTIR analysis

To investigate the interactions between the chemicals employed in the developed composite, a Spectrum two FTIR with diamond ATR [Perkin Elmer Singapore Pvt. Ltd.] is employed. FTIR analysis was performed in the region of 500 to 4000 cm^{-1} using the potassium bromide technique.

Figure 7 depicts the FTIR spectra of non-functionalized (CNT-Epoxy coated coir fiber) and functionalized MWCNTs to illustrate functionalization, along with plain cement powder, plain coir fiber, epoxy coated coir fiber. The focus is mainly on f-CNT and n-CNT with epoxy coated on coir fiber, to investigate its impact on the surface interfacial region and bonding between them. The peak at 1640 cm^{-1} corresponds to the stretching vibrations of C=C in the aromatic region

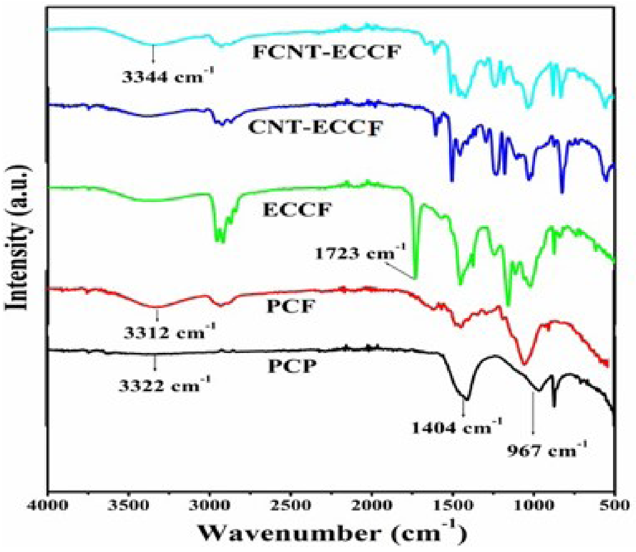

Figure 7: FTIR analysis of coir fiber nanocomposite.

of MWCNTs, which upshifted to a greater wave number when surfactants were adsorbed on the MWCNTs surface. It may be that this is due to a strong bond amongst CNTs and surfactants. The carboxyl peak (1740 cm^{-1}) was identified in the CNTs spectra, indicating that n-CNTs have carboxyl groups on substrates. Peaks with wave numbers of 2920 cm^{-1} and 2850 cm^{-1} occurred in all curves, correlating to the C-H stretching vibrations of -CH$_3$, -CH$_2$, and -CH$_3$. Furthermore, the new peaks 1552 cm^{-1} assigned to vibration of N-H of MWCNTs demonstrate their functionalization with NaDC. The peak at 3344 cm^{-1} proved a broad spectrum of stretching of -COOH. There was a sharp carboxylic peak at 1723 cm^{-1} which proves stretching of C=O attributed to -COOH. The broad range and peak of 3312 cm^{-1} are caused by the stretching vibrations of the -OH groups contained in both hemicellulose and cellulose of the coir fiber. Cement powder displayed a peak at wave number of 3322 cm^{-1}, which is associated with moisture in cement powder which could have been absorbed during sample processing. Si-OH wagging phase may be responsible for the low frequency band of 967 cm^{-1} [34].

EDX analysis

EDX examines were performed to confirm the elemental composition of a sample of nano-polymer-based composite, which influences the mechanical properties of the polymer composites under study. The cement composite samples employed in the EDX investigations were allowed to cure for 28 days before being analysed. Following that, after 28 days of curing, the bonded area between the combination of (i) epoxy/f-CNTs coated coir fiber and the cement matrix, (ii) epoxy/n-CNTs coated coir fiber and the cement matrix is the focus of microscope investigations on the cracked and flat surfaces of paste samples. Figure 8a and figure 8b indicates that the calcium carbonate and silica content, as well as the amount of wollastonite (CaK), grew considerably when the proportion of MWCNT inclusion increased from 0.05% to 2% in 0.05% steps. It reveals that the presence of MWCNTs may promote the growth of these minerals in the cement matrix. MWCNTs have the potential of acting as nucleation sites or enabling crystal formation, resulting in increased mineral growth. Components such as traces of oxygen, carbon, silica, and calcium have been found as important components in cement composites. As a result, it is apparent that the sample contains silica, calcium, alumina, and oxides. The EDX spectrum exhibits a slight sulfur peak, implying that the tiny crystals are calcium sulfo aluminate [35-37].

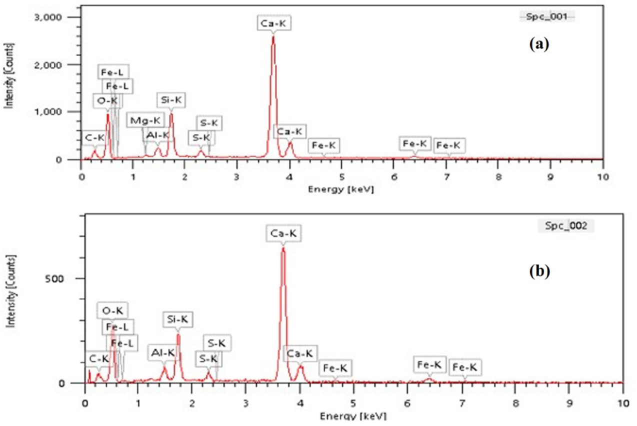

Figure 8: EDX of **(a)** n-CNTs and **(b)** n-CNTs with epoxy coated coir fiber.

Conclusion

Since coir fibers are hydrophilic and epoxy resins are hydrophobic, they are incorporated together as reinforcement for composite. By eliminating some non-cellulosic compounds from the fibers and enhancing the surface roughness of the fibers, alkali treatment strengthens interfacial bonding ability. The addition of a surfactant (NaDC) to a CNT improves its solubility in water, thus improving its dispersion. Coating epoxy/f-CNTs on the coir fiber increased the Young's modulus with f-CNTs loadings with 0.1 wt.%. Also, epoxy/f-CNT samples possessed 1.4 times higher in modulus as compared to epoxy/n-CNT. It could be due to strong van der Waals forces and hydrogen bonding which have strengthened interaction between CNTs and the fibers. Through π-π interactions or van der Waals forces, surfactant NaDC hydrophobic carbon chains interacted with MWCNTs. The water solubility, which is in contact with the solvents and epoxy matrix, is offered by their free opposite end, which was situated on the substrate. This can be accounted to the functionalization of CNTs which increased the modulus of elasticity of the composite. Plain cement composite had high load carrying capacity but very low modulus of elasticity, owing to the fact of being a brittle material by itself. Consequently, it can be concluded that the CNT-coating proved effective in performing as a molecular coupling agent to make the composite structure compatible. Microscopic studies demonstrated coating of epoxy/f-CNTs and epoxy/n-CNTs onto the fibers increased the contact through interlocking between the coir fiber, CNTs, and cement matrix.

Acknowledgments

None.

Conflict of Interest

None.

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