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Research Article

Some Features of Doping of Nano–Graphite in Natural Coir Fibre Epoxy–Composites

Mohammad Ayaz Ahmad[1], Gülşen Güven[2*], Nursabah Sarıkavaklı[2]
[1] Physics Department, Faculty of Sciences, P.O. Box 741,University of Tabuk, 71491, Saudi Arabia
[2] Department of Chemistry, Faculty of Sciences, and Arts, Aydın Adnan Menderes University, Aydın, Turkey

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Abstract

An attempt has been made to find some direct or indirect uses of nano-composite polymers in various sectors such as; biomedical engineering, electronics, structural engineering and many more due new advancement of science and technology. In the present research work, we used the doping of nano-graphite in the natural coir fibre reinforced polymer composites. The used samples of composites were 45 % of natural coir fibre and 3.2 % of nano-graphite (N-G) in an epoxy resin matrix by weight fraction. We find that on doping the above % weight of nano-graphite in the natural coir fibre, the tensile strength roughly increased by 45-50 % and the rest of the mechanical properties by 2-3 times on the average. And finally, the SEM analysis revealed a good dispersion of nano-graphite in the present samples of composite of coir fibre.

Keywords: Nanocomposites, coir natural fibre, characterization, reinforced epoxy composites, SEM analysis.

* Corresponding Author: Department of Chemistry, Faculty of Sciences, and Arts, Aydın Adnan Menderes University, Aydın, Turkey, ORCID: 0000-0002-2162-2576, gguven@adu.edu.tr

1. Introduction

The natural fibre composites are very much important materials due to their relatively high biodegradability levels. The inadequacy in some mechanical properties of these materials remains a serious hindrance to their progress. The coir is the natural fibre of the coconut husk where it is a thick and coarse but durable fibre. It is relatively water-proof and has resistant to spoil by salt water and microbial degradation [1, 2]. The use of coir fibre reinforced composites is essential in the industrial automotive where it used to make seat cushions for Mercedes automobiles. Even though it has profitable properties, the coir fibre composites still have some undesirable properties such as dimensional instability, flammability which are not suitable for high temperature application and degradability with humidity, ultraviolet lights, acids and bases [3-6]. A lot of efforts have been carried out to improve the performance of coir fibre reinforced composites and the potential of this fibre to be a good reinforcement for polymer matrices was brought into light by a few experiments in the recent past. This fibre is composed of cellulose 61-71.5%, hemicelluloses 13.6-20.4%, moisture content 12.6%, lignin 12-13%, and others 0.70%, which contribute to its reinforcing potential in polymer matrices [7-11]. In a study on coir fibre reinforced matrix, the matrix showed 50-60% increase in tensile strength after reinforcement by esterified coir fibres [9-11]. Besides, the procurement and processing of coir fibres are simple and very inexpensive. The processing of this fibre does not involve polluting techniques like those involved in synthetic fibre composites. Therefore, a combination of natural fibres and nano-fillers in a polymer matrix can enable the development of a high performance material with a relatively good level of biodegradability. In the present experimental work, composites consisting of nano-modified epoxy (containing 3.2%wt of NG) as the matrix phase and coir fibre (40% and 50%wt) and as the reinforcement phase were fabricated and their mechanical and microstructure properties were studied and compared with those of pure epoxy-coir composites without nano-fillers.

Since last few decades, the nano-technology started scaling new heights as its applications extended into various field of natural science from medicine to metallurgy / modern age of engineering and technology. The improvement in mechanical properties of metallic and polymeric materials by doping of nano-particle produced new interesting outcomes. A few research ventures in the past have demonstrated the ability of nano-fillers to improve the mechanical properties of polymers. Addition of nano clay by 1.5 in an epoxy matrix resulted in an increase of fracture toughness by 46% on the average [12-16]. The epoxy LY564 reinforced with MWCNTs (multi walled carbon nano tubes) showed 70% increase in tensile impact strength [17]. The Inter laminar shear stress of epoxy reinforced by glass fibre, showed an increase by 20% upon the addition of 0.30 wt% of CNT (carbon nano tubes) [13-16, 18]. In a very few attempts nano-graphite (NG) was used a filler for polymer matrices. After the successful fabrication of monolayer grapheme with excellent mechanical properties in 2004 [19], the development polymeric composites with graphitic fillers started gathering steam. In a study of mechanical properties of polyetherimide (PEI) reinforced with various nano fillers like MWCNT (multi walled carbon nano-tubes (CNT)), CNF (carbon nano fibre), and Graphite nano platelets (GNP), GNP reinforced PEI nano-composites show highest strength and modulus compared to the other two [13-16, 20]. In a comparative study of the impact toughness between HDPE (high density polyethylene) / GNP composites and HDPE / carbon black (CB) composites, the former recorded much greater values [21].

In the present article, the used composite was manufactured by hand-layup technique in our laboratory. The epoxy-coir composite without nano fillers, a maximum tensile strength of 20-25 MPa, compressive strength of 100-110 MPa, flexural strength of 140-160 MPa and impact energy of 1.65 Joules were obtained for the sample with 40-50% fibre content. After that the addition of 3.2% weight fraction of nano-graphite to the same increased the tensile strength roughly by 50-60% and the rest of the mechanical properties by two times on the average. And finally, the Scanning Electron Microscopy (SEM) analysis revealed good dispersion of nano-graphite in the composite.

2. Materials and Methods

2.1. Raw Samples Collection

The coir fibre was sourced locally in Cochin, India¸ State of Kerala and the epoxy resin purchased from a chemical store [22]. The natural Coir from coconut husk has been depicted in Figure 1, and was extracted by retting process. This involves soaking the coconut husks in water and extracting the fibres manually from the husks. The coir fibres were treated with alkaline treatment using 0.5% Sodium hydroxide (NaOH) salt. This was to remove wax, lignin, oils and other fibre constituents that may reduce adhesion between the matrix and fibres thereby constituting a weak boundary layer.

Figure 1. The Coir from coconut husk [This Fig. taken from Ref. 23].

2.2. Fabrication Procedure of Composite Samples

The fabricated epoxy resin composites samples produced from the natural Coir fibre have been shown in Figure 2. The hand lay-up technique was used for the fabrication of composite samples in our laboratory by using coir fibre and epoxy resin. The epoxy resin and hardeners were mixed using the ratio of 1.25:1 parts by weight, further the epoxy resin and hardener were weighed separately and magnetic stirred till the mixture became warm (exothermic reaction). A portion of the mixture was poured into the glass bottle of diameter 10 mm and allowed to set for 24 hours. The tests samples were cut from the cylindrical bottle in the form of circular discs of 2 mm thickness and 10 mm diameter. Uniformity of the surface of test samples was obtained by polishing the sample using polishing cloth. The test samples were then kept in between the electrodes of the impedance analyzer for various measurements.

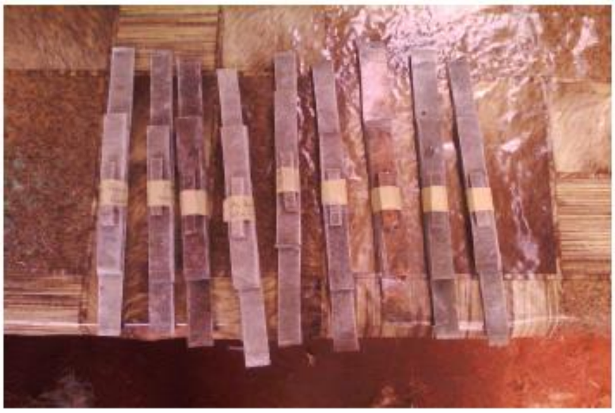

Figure 2. The fabricated epoxy resin composites samples produced from the natural Coir fibre.

2.3. Chemical Treatment and doping nano-graphite (N-G) of Composite Samples

A chemical treatment by using the salt of Sodium Hydroxide (NaOH) was carried out on the above obtained samples of coir fibres after isolation, to increase the fibre-polymer matrix compatibility. This analysis was used to produce the effective fibre surface area which leads to increase the fibre-matrix adhesion [5-11 and also references therein], and also in FRCs good fibre-matrix adhesion is a requirement for good mechanical properties. The chemical treatment removes significant amounts of hemi-cellulose and pectin from the coir fibres leading to increased thermal stability [5-11, 13]. The treatment was done by soaking the coir fibres in the solution of 1N 10-20% NaOH salt for 3-5 hours followed by thorough washing in distilled water and drying.
Further, as it is well know that the graphite is inorganic, it is chemically incompatible with the organic epoxy matrix and to increase the compatibility it was pre-treated using a surfactant called [3-(2-Aminoethylamino)-propyl]-trimethoxysilane (a reactive silane coupling agent). The used nano-graphite (N-G) was a form of graphite with a particle size of less than 50nm in the present experimental work. The chemical surfactant was capable of forming covalent bonds with both the epoxy resin and reinforcing nano-particle [24]. The treatment was carried out by mixing N-G and the surfactant in the ratio 20:1 manually and leaving the mixture for 24 hours to facilitate the chemical reaction. Then, the treated N-G was mixed with epoxy in the mass ratio 1:40 and the mixture was ultrasonicated up to 2-3 hours.

3. Results and Discussions

The mechanical properties were studied through standard tensile, compressive and flexural tests. A perfect standard, named as ASTM D3039 tensile testing standard [25] was used to measure the tensile and compressive strengths for the present samples. All the samples were prepared as per an International Organization for Standardization (ISO); ASTM standard EN ISO 14125 (1998)/ (subcommittee SC 13, *Composites and reinforcement fibres*) [26] to determine flexure property using three-point bending. We selected 5-7, samples / specimens from the same sample were used for all the tests and the average values were recorded in the form of circular discs of 2 mm thickness and 10 mm diameter. In Figure 3 (a-d), we draw the characteristics for tensile strength, compressive strength, flexural strength and impact strength respectively. These characteristics were in term of behavior / dependency of weight fraction of fibres as a function of (3.a) Tensile strength in MPa, (3.b) Compressive strength in MPa, (3.c) Flexural strength in N/mm^2, and (3.d) impact strength in Joules. From these figures one can conclude that that the mechanical properties i.e. tensile, compressive and flexural behaviours of chemically treated coir fibre reinforced epoxy composite is found to be less than pure epoxy composite. These results were found within good agreement with some of our earlier work as well some other researchers working in the same field [4-7, 9, 10 & 13-16]. It is also well recognized for reinforced polymer composites, that the interfacial zone governs an important role in transferring the load between the fibre and matrix which subsequently affect the mechanical properties such as strength. As we know that flexural failure [9-10] depends mostly on the fibre and matrix adhesion, an increase the value of flexural strength in chemically treated composite may be due to the increase in the efficient surface area available for contact with the matrix [9, 10].

Based on our above mentioned experimental technique, the results of the tensile tests showed that addition of nano-graphite (N-G) to the composite increased the tensile strength nearly 2.0 times. A maximum value of tensile strength of 25.0 MPa was obtained for the nano-graphite (N-G) reinforced composite with 45-50% fibre content. This was an outcome of improved polymer-filler interaction caused by the high surface area of nano-graphite (N-G) particles and the surface treatment. This was more effective than other strengthening techniques because the polymer-filler interaction takes place through chemical bonds [13-16, 27], and strengthening takes place at a molecular level. The compressive strength showed a drastic improvement and in case of the composite with 45-50% fibres content the value increased by 2.5 times. Apart from facilitating good dispersion, compatibility between the phases increases stress transfer due to high interfacial adhesion, resulting in improved composite modulus [13-16, 28], and thereby good resistance to compression.

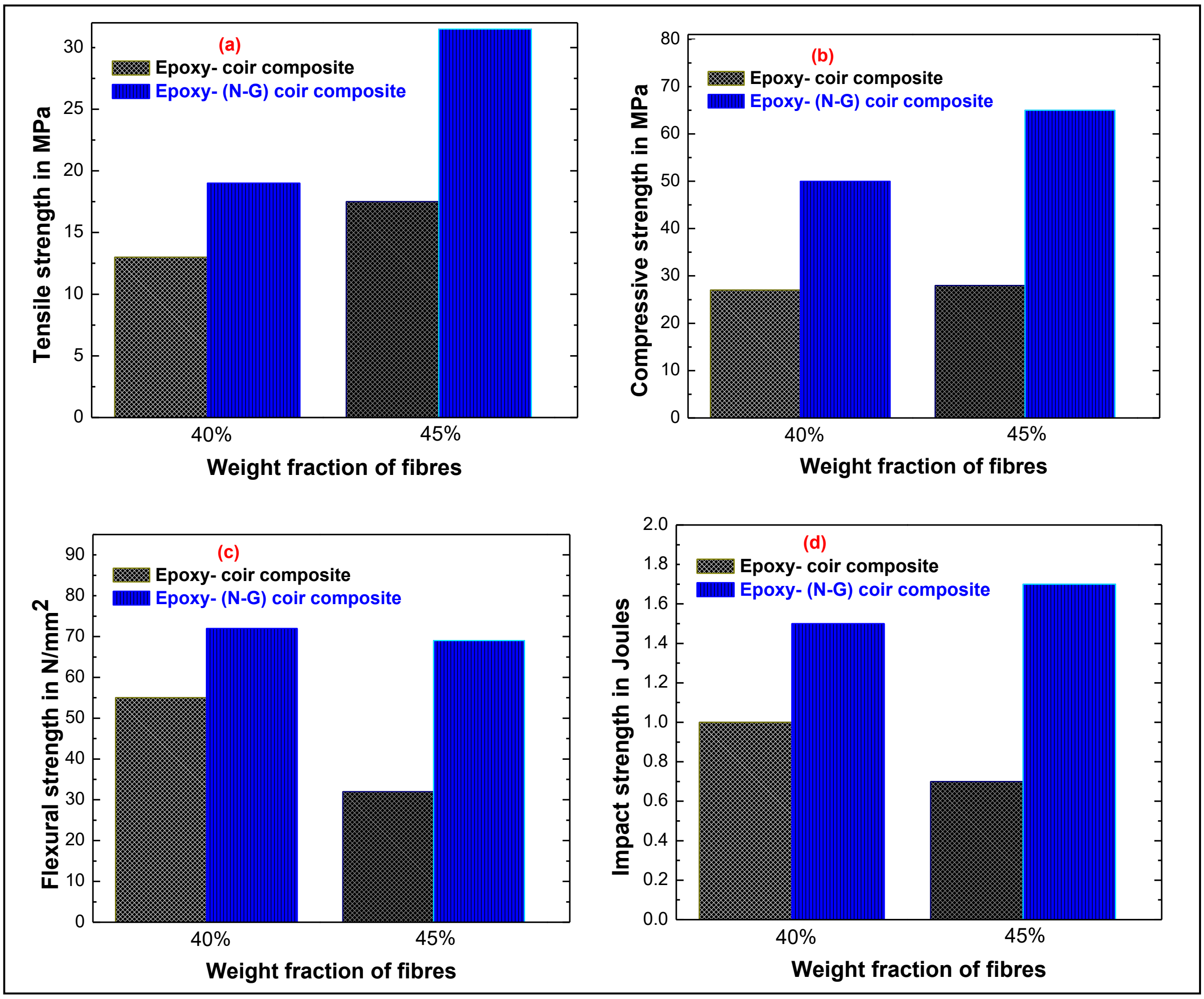

Figure 3 (a-d). The behavior of weight fraction of fibres as a function of (a) Tensile strength in MPa, (b) Compressive strength in MPa, (c) Flexural strength in N/mm^2, and (d) Impact strength in Joules.

The impact strength almost doubled in case of the composite with 45% fibre content but a lower increase was observed in the composite with 35% fibre content. A similar trend was seen in the case of flexural strength. But in contrast to all the above properties, flexural strength values were higher for the composites with 35% fibre content than those with 45% fibre content and this can be attributed to the style of reinforcement (chopped random fibre). The higher concentration of randomly oriented fibre (which is used in this work) results in non-uniform distribution of stress which is a major contributor to poor flexural strength. Therefore, it is obvious that addition of nano-graphite (N-G) of imparts excellent mechanical properties to natural fibre composites and matches their performance to that of synthetic fibre composites.

Furthermore, the scanning electron microscopy analysis of the test samples were done by JSM 6390A (JEOL Japan) at various magnifications. This was done for the micrographs expose sensible to low agglomeration of nano-graphite particles which is an indication of the advantage of nano-graphite (N-G) over graphene which agglomerates very easily due to high aspect ratio [16]. The images of the prepared samples were taken at the plane polished surface. Figure 4 (a) shows the scanning electron microscopy (SEM) image of micrograph of pure epoxy- coir composite, Figure 4 (b) shows the SEM analysis of the micrograph that is for the dispersion of nano-graphite (N-G) into the NG-epoxy coir composite with 40% fibre while in Figure 4 (c) the micrograph shows the dispersion of nano-graphite (N-G) into the NG-epoxy-coir composite with 45% fibre content. In the pure epoxy-coir fibre composite (Figure 4(a)), the fibre strands appear very distinct which makes them more vulnerable to breakage. The presence of intact nano-graphite (N-G) particles in the micrographs (as it has been shown in Figure 4(b & c), indirectly reveals that the covalent bonding of NG with the epoxy is not achieved to a desired level, thereby recommending a scrutiny into the pre-treatment process.

In fibre reinforced composites, mechanical interlocking is an easily achievable and effective mechanism for fibre-matrix bonding [17, 18]. In the micrographs (depicted in the Figure 4(c)), agglomerated nano-graphite (N-G) particles can be seen in between two fibre strands The presence of these particles causes a kind of interlocking in between the fibre strands leading to a synergistic effect of the fibres and (N-G) particles for resisting deformation, which incorporates improved mechanical properties to the composite.

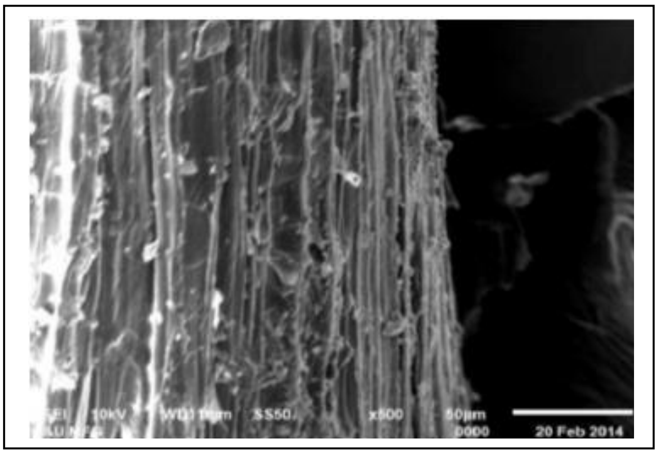

Figure 4 (a). The SEM image for the micrograph of pure epoxy- coir composite.

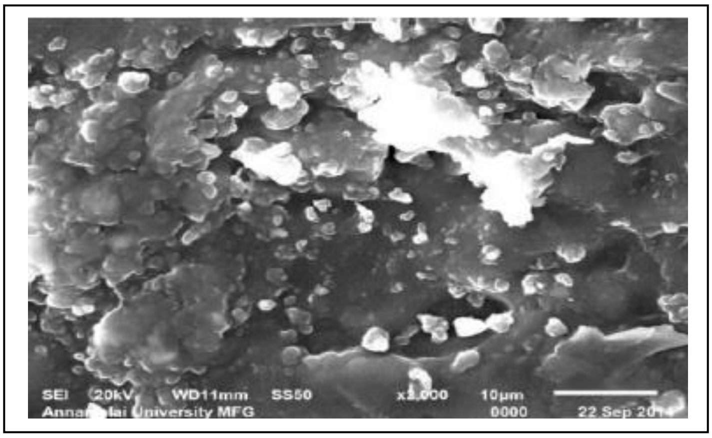

Figure 4 (b). The SEM image of micrograph for the dispersion of nano-graphite (N-G) into the NG-epoxy coir composite with 40% fibre.

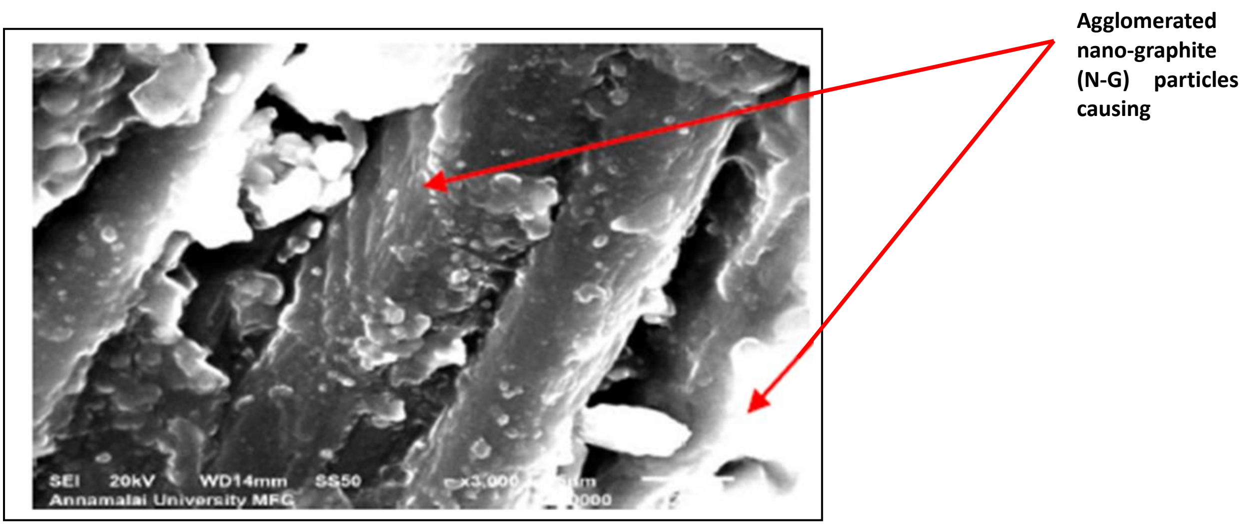

Figure 4 (c). The SEM micrograph for the dispersion of nano-graphite (N-G) into the NG-epoxy-coir composite with 45% fibre content.

The micro-filling of the voids in the coir fibres by nano-particles is also partly visible in the micrographs and this contributes towards the enhancement of compressive properties has been depicted in Figure 5. This is very less in case of synthetic fibres due to the presence of very less pores. As illustrated above, the contribution of the voids to the compressive failure is nullified by the micro-filling of the voids in the fibre by NG particles. These particles move into the fibre voids and cause delay in their rupture.

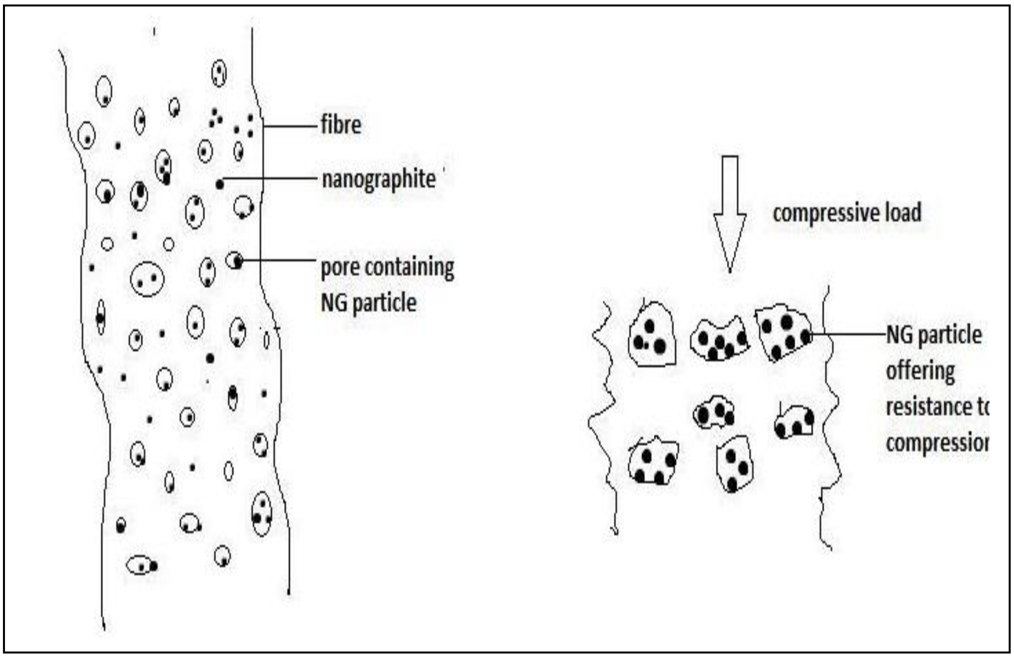

Figure 5. The Micro-filling of voids phenomenon

4. Conclusions

The present experimental work has been carried out, with an inspiration to explore the potential of the Coir fibre polymer composites and to find some interesting features of doping of nano–graphite (N-G) in Natural Coir Fibre Epoxy–Composites. This study also leads to boost up some extensive knowledge for the further research in the field of nano-composite polymers in a simplified manner. This work is also an implication that, a partly bio-degradable natural coir fibre composite can be made a high performing material too. Hence, the present article has shown the feasibility of presenting degradability and good performance in the same material i.e. it can be a better economic effort (e.g. the high nano-particles cost).

Based on the above analysis one can get the following out comes:

(i) The tensile, compressive and flexural tests have been done between the tensile strength (in MPa) and weight fraction of coir fibres.

(ii) The studied mechanical properties have shown geometric increase which describes the feasibility of boosting the performance levels of natural fibre composites to those of their synthetic counterparts.

(iii) The reinforcing effect of nano-graphite (N-G) particles shows great credibility for the enhancement of mechanical properties of natural coir fibre reinforced polymer composite.

(iv) The chemical treatment improves the morphology of natural coir fibre. One can understand from the evaluation of micrographs in present experimental work that the maximum property values achieved in this experiment are below threshold level, thereby indicating the scope for further improvement through adoption of different pre-treatment methods.

(v) The large increase examined in the impact strength is an indication of the importance of the nano-filler accumulation in the production of impact energy absorbing polymeric materials.

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