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Corresponding Author	FamilyName	**M. S.**
	Particle	
	Given Name	**Sreekala**
	Suffix	
	Division	Postgraduate & Research Department of Chemistry
	Organization	Sree Sankara College
	Address	Kalady, Kerala, India, 683574
	Division	School of Chemical Sciences
	Organization	Mahatma Gandhi University
	Address	Priyadarshini Hills P.O, Kottayam, Kerala, India, 686560
	Division	
	Organization	International and Inter University Centre for Nanoscience and Nanotechnology (IIUCNN), Mahatma Gandhi University
	Address	Kottayam, Kerala, India
	Phone	
	Fax	
	Email	sreekalams@yahoo.co.in
	URL	
	ORCID	http://orcid.org/0000-0002-9357-0947
Author	FamilyName	**Ravindran**
	Particle	
	Given Name	**Lakshmipriya**
	Suffix	
	Division	Postgraduate & Research Department of Chemistry
	Organization	N.S.S Hindu College
	Address	Changanacherry, Kerala, India, 686102
	Division	Postgraduate & Research Department of Chemistry
	Organization	Sree Sankara College
	Address	Kalady, Kerala, India, 683574
	Phone	
	Fax	
	Email	
	URL	
	ORCID	
Author	FamilyName	**Anilkumar**
	Particle	
	Given Name	**S.**
	Suffix	
	Division	Postgraduate & Research Department of Chemistry
	Organization	N.S.S Hindu College
	Address	Changanacherry, Kerala, India, 686102

	Phone	
	Fax	
	Email	
	URL	
	ORCID	
Author	FamilyName	**Ponnan**
	Particle	
	Given Name	**Sathiyanathan**
	Suffix	
	Division	
	Organization	International and Inter University Centre for Nanoscience and Nanotechnology (IIUCNN), Mahatma Gandhi University
	Address	Kottayam, Kerala, India
	Phone	
	Fax	
	Email	
	URL	
	ORCID	
Author	FamilyName	**Kalarikkal**
	Particle	
	Given Name	**Nandakumar**
	Suffix	
	Division	
	Organization	International and Inter University Centre for Nanoscience and Nanotechnology (IIUCNN), Mahatma Gandhi University
	Address	Kottayam, Kerala, India
	Division	School of Pure and Applied Physics
	Organization	Mahatma Gandhi University
	Address	Kottayam, Kerala, India
	Phone	
	Fax	
	Email	
	URL	
	ORCID	
Author	FamilyName	**Thomas**
	Particle	
	Given Name	**Sabu**
	Suffix	
	Division	School of Chemical Sciences
	Organization	Mahatma Gandhi University
	Address	Priyadarshini Hills P.O, Kottayam, Kerala, India, 686560
	Division	
	Organization	International and Inter University Centre for Nanoscience and Nanotechnology (IIUCNN), Mahatma Gandhi University
	Address	Kottayam, Kerala, India
	Phone	
	Fax	
	Email	
	URL	
	ORCID	

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Abstract

Biomaterials have become an integral part of our lives as a result of the current focus on renewable

sustainability. In the realm of composites, a diverse variety of polymers coupled with multiple natural fibres enables the achievement of varied functional property criteria. Bio- composite materials have several benefits, in addition to the use of renewables, including minimal impact on the environment, light weight and biodegradability. Here we have isolated nano-silica from rice husk and fabricated woven flax fabric coated with nano-silica and phenol formaldehyde composite with varying weight percentages of nano-silica. The extracted nano-silica was confirmed from XRD, TGA and FESEM. Five types of composites (PF-F, 1 NS, 2 NS, 3 NS, 4 NS) prepared keeping constant weight of PF resin and flax fabric. Mechanical, morphological and electrical behaviour of the prepared composites were examined. From the obtained results, tensile strength was found to be maximum for 1 NS loading. In particular, as compared to neat composites (PF-F), the tensile strength of prepared specimens with nano-silica (1 NS, 2 NS, 3 NS, 4 NS) improved by 93.92%, 99.55%, 49.85% and 26.43% respectively. The FESEM pictures of fracture surfaces demonstrated that the inclusion of nano-silica boosted fibre's interfacial strength, strengthened both fibre and matrix, and improved resin adhesion to fibre, therefore enhancing the composite tensile characteristics. Due to polarisation processes mediated by nano-silica inclusion, the dielectric constant in PF hybrid composites increases as the loading of nano-silica increases. Electronic polarisation causes an increase in AC conductivity at high frequencies (3 NS). The graph shows that as the amount of nano-silica loaded rises, the AC conductivity increases. The dielectric constant values in 2 NS are the highest.
Graphical Abstract:

Keywords (separated by '-')	Nano-silica - Phenol–formaldehyde resin - Flax fabric - Hybrid composite - Mechanical - Fracture morphology - Electrical properties
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ORIGINAL PAPER

Mechanical, Morphological Behaviour and Electrical Conductivity of Phenol Formaldehyde-Flax Fabric (PF-F) Hybrid Composites Reinforced with Rice Husk Derived Nano-silica

Lakshmipriya Ravindran[1,2] · Sreekala M. S.[2,3,4] · S. Anilkumar[1] · Sathiyanathan Ponnan[4] · Nandakumar Kalarikkal[4,5] · Sabu Thomas[3,4]

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Abstract
Biomaterials have become an integral part of our lives as a result of the current focus on renewable sustainability. In the realm of composites, a diverse variety of polymers coupled with multiple natural fibres enables the achievement of varied functional property criteria. Bio- composite materials have several benefits, in addition to the use of renewables, including minimal impact on the environment, light weight and biodegradability. Here we have isolated nano-silica from rice husk and fabricated woven flax fabric coated with nano-silica and phenol formaldehyde composite with varying weight percentages of nano-silica. The extracted nano-silica was confirmed from XRD, TGA and FESEM. Five types of composites (PF-F, 1 NS, 2 NS, 3 NS, 4 NS) prepared keeping constant weight of PF resin and flax fabric. Mechanical, morphological and electrical behaviour of the prepared composites were examined. From the obtained results, tensile strength was found to be maximum for 1 NS loading. In particular, as compared to neat composites (PF-F), the tensile strength of prepared specimens with nano-silica (1 NS, 2 NS, 3 NS, 4 NS) improved by 93.92%, 99.55%, 49.85% and 26.43% respectively. The FESEM pictures of fracture surfaces demonstrated that the inclusion of nano-silica boosted fibre's interfacial strength, strengthened both fibre and matrix, and improved resin adhesion to fibre, therefore enhancing the composite tensile characteristics. Due to polarisation processes mediated by nano-silica inclusion, the dielectric constant in PF hybrid composites increases as the loading of nano-silica increases. Electronic polarisation causes an increase in AC conductivity at high frequencies (3 NS). The graph shows that as the amount of nano-silica loaded rises, the AC conductivity increases. The dielectric constant values in 2 NS are the highest.

Keywords Nano-silica · Phenol–formaldehyde resin · Flax fabric · Hybrid composite · Mechanical · Fracture morphology · Electrical properties

✉ Sreekala M. S.
sreekalams@yahoo.co.in

[1] Postgraduate & Research Department of Chemistry, N.S.S Hindu College, Changanacherry, Kerala, India 686102

[2] Postgraduate & Research Department of Chemistry, Sree Sankara College, Kalady, Kerala, India 683574

[3] School of Chemical Sciences, Mahatma Gandhi University, Priyadarshini Hills P.O, Kottayam, Kerala, India 686560

[4] International and Inter University Centre for Nanoscience and Nanotechnology (IIUCNN), Mahatma Gandhi University, Kottayam, Kerala, India

[5] School of Pure and Applied Physics, Mahatma Gandhi University, Kottayam, Kerala, India

1 Introduction

Polymers have a numerous benefit which allows polymeric materials and composite materials, to make friction parts like bearings, wheels, piston rings, and soft seals. These are easy to fabricate, ultra-lightweight and resistant to chemical and environmental effects. Fabric reinforced polymer composites, as compared to other polymer composites, provide increased mechanical properties in both the horizontal and vertical directions of the fabric, as well as the ability to adhere to curve surfaces without tangling [1, 2] Fabric reinforced polymer composites, as a result of their large use in the fields of aircraft, aviation, high-speed railways, automotive, and other fields, have recently sparked a broad range of financial and research interest.

The polymer used is the commanding factor which plays the key role in determining the properties of the hybrid composite. If fire, smoke, and toxicity (FST) conditions must be taken into consideration, phenolic resin have a considerable market share compared to epoxy resin. They're also employed as ablative composites for thermal protection in aerospace applications like solid rocket motor nozzles and heat shields for space vehicle atmospheric re-entry. There are several prepreg conditions that may be discovered here. Because of the phenolic resin's chemistry, phenolics have a substantially longer storage life than most epoxy prepregs. This is due to the nature of the phenolic resin's chemistry [3, 4].

Natural fibres have recently received much attention as a replacement for synthetic fibres in FRP (fibre reinforced polymer) composites, attributed to high environmental issues and a high demand for eco-friendly materials [5]. Flax is one of the natural fibres that has desirable material properties and could be used to replace glass fibres in fibre reinforced polymer (FRP) composites. Flax fibre provides the best possible combination of low cost, light weight, and high tensile strength for structural applications [6–8]. According to Yan et al. [9] flax, hemp, and jute are the three most excellent materials for substituting glass fibres in terms of cost, mechanical efficiency, and production yielding. Flax fabric/epoxy composite has a tensile strength of 300 MPa, which is comparable to GFRP composite, according to Assarar et al. [10] automotive engineering is currently a wide demand market for natural FRP composites. The advantage associated with natural FRP composites are it reduce the energy required for production by 80% and minimises component mass [11]. Natural FRP composites have strength and durability, therefore it can be used as crashworthy structures for the manufacture of vehicles.

From the perspective of energy absorption, Yan et al. [12] recently investigated the crash safety of flax fabric reinforced epoxy composites. Flax fabric/epoxy composite were discovered to have the capability to be applied as energy absorber devices. Comparing with traditional metallic or G/CFRP composite tubes flax fabric/ epoxy composite tubes with foam-filler absorbed more energy during axial and lateral crushing [13, 14]. After comparing the energy absorption capacities of composites reinforced by unwoven hemp, woven flax, and jute, Meredith et al. [15] suggested flax/epoxy composites to be used as energy absorbers for vehicle construction. The most important application of natural FRP composites is probably as construction materials. These renewable products would help to lower the costs and improve energy consumption, addressing urgent infrastructure needs while also encouraging sustainability. Natural fibres/fabric composites may be used in construction as a hybrid framework in combination with traditional construction materials, such as a flax fibre reinforced polymer (FFRP) tube encapsulated coir fibre reinforced concrete (CFRC) structure (FFRP-CFRC) [16].

Natural fibres, known as cellulosic biomass, such as banana, curauá, coconut, flax, hemp, jute, pineapple, ramie, sisal, and sugar cane bagasse, are often used as filler in composite material since they provide numerous advantages over synthetic fibres, including: relatively low mass; easy handling; protection thermal, electric, and acoustic; architectural features; non-toxic; and low specific mass [17–19]. Within the automotive sector, it is thought to have a bright future being used in the interior coating of vehicles, buses, and trucks; in construction works to reinforce cement; as a woven fabric; in aerospace, sport, and packaging, indicating a booming market [20, 21]. For applications in engineering sector the durability issues should be rectified primarily. In practical use, these bio-composites would be subjected to a variety of extreme conditions, including atmospheric and weather conditions, resulting in composite material deterioration and improving safety issues. As a result, learning about the stability of flax FRP composites may be useful for applying in practical uses [22]. But nevertheless, technological problems prevent widespread use of these composites [23].

The most significant are fibre cultivation inconsistencies, composite manufacturing inconsistencies, and a lack of clear information of their behaviour. Due to a variety of factors such as temperature, water, radiation, fungus/bacteria, and mechanical stress using such products in the indoor and outdoor applications causes particular ageing issues leading to total deterioration. Joseph et al. [24] studied the mechanical properties of sisal fibre-polymer composites subjected to water sorption and UV degradation to investigate the performance of natural FRP composites. Many researchers investigated the properties of natural fibre reinforced polymer composites [25–27].

An increasing tendency in the fabrication of hybrid nanocomposites, which has sparked widespread interest and awareness nowadays. Nano clay layers, nanotubes, nano-silica and spherical particles (metal nanoparticles) are examples of inorganic materials used in nanocomposites. The uniqueness of nanomaterials are they have the potential to change the polymer's properties by altering the mobility of the polymer chains. One of the promising nanomaterial is inorganic silica nanoparticles which can enhance the thermo-mechanical properties of a polymer [28, 29]. The peculiar properties of nano-silica are due to the surface chemistry characterised by the presence of silanol groups in silica nanoparticles.

Dinesh et al. [30] investigated mechanical, thermal and morphological behaviour of nano-silica treated pineapple woven fabric in epoxy resin. From the mechanical tests they found that 1 vol% nano-silica and 30 vol% pineapple fibre gave maximum properties. The thermal degradation was slowed down by the addition of 1 vol% nano-silica. Same was the trend

Journal : Large 12633 | Article No : 2193 | Pages : 13 | MS Code : 2193 | Dispatch : 1-12-2022

for tribological behaviour. The fracture toughness was also improved by the nano-silica addition which was confirmed from the SEM images. Despite the fact that silica has many applications in polymers, the agglomeration and low compatibility of SiO_2 in the rubber matrix leads to its restricted applications. The surface treatment of sisal fibres improved the interface adhesion between fibres and resin by adding nano-silica to the phenol formaldehyde resin [31]. Under various temperatures, the borax-treated fibres demonstrated the highest heat resistance, constant friction coefficient, and low wear rate. The best fibre content was 15 wt.%. The impact of nano-silica and nano-clay on glass fibre reinforced composite have been studied [32]. It is for this reason that adding nano-silica and/or nano-clay to GFRP composites may help them perform better in corrosive conditions.

As a result of their higher biocompatibility, reusability, and degradability compared to synthetic fibres, natural fibres (such as coconut fibres, silk, flax fibres, and flax seed fibres) and synthetic fibres (such as glass fibres) are often used to reinforce polymer or hybrid or natural resin matrix composites [33]. Epoxy resin is blended with Lannea coromandelica (LC, Anacardiaceae plant gum) to create hybrid Lannea Coromandelica Blender Epoxy matrix composites (LCE) that are biodegradable and environmentally friendly. Hybrid Composites (Pineapple/Silk/Flax fibre mats with 2%, 4%, and 6% volume fraction of Bentonite nanoclay in each composition reinforcement in hybrid LCE resins prepared by compression hand moulding process) and hybrid LCE resin are made using this approach. Hybrid composites' mechanical properties have improved by three to four times when compared to hybrid LCE resin. DMA findings show that untreated and treated hybrid composites have the highest storage modulus and lowest damping factor compared to hybrid LCE resin. LCE resin composites reinforced by P/G/P fibre mats can be biodegraded at a 4% volume fraction of nano filler, according to biodegradability studies. In light of the increasing environmental, economic, and application issues, hybrid composites are expected to garner substantial attention in the future [34, 35]. As an alternative to synthetic fibres, researchers developed novel composites that included more than one reinforcement from natural resources, such as natural fiber/natural fibre or natural fiber/nanofiller from organic sources [36]. It is important to use natural fibres and fillers instead of synthetic ones in order to reduce the environmental impact of our products. There has been a rise in the use of natural filler/fibers as a reinforcing mediator because of the abundance of these materials [37].

So far, there have been very few studies on the property studies (mechanical, morphology and electrical properties) of flax fabric polymer composites. According to a survey of the literature, no particular research has been done to evaluate the influence of a PF matrix containing nano-silica (extracted from rice husk) on the mechanical, morphological and electrical behaviour of Flax fabric/PF hybrid composites at the same time. Natural fibres and natural fillers that are environmentally friendly are becoming more popular, and they should be used instead of synthetic fibres and fillers to meet the growing demand for green products. Because natural filler/fibers are readily available, the use of natural filler/fiber as a reinforcing mediator has evolved in the application of science and technology. The combined effect of nano-silica on the mechanical, morphological behaviour and electrical properties of flax fabric-phenol formaldehyde hybrid composites are examined in this study. The prepared hybrid composites have the potential for large-scale applications because to their commercial availability, inexpensive raw material, and simple production procedure. The method we have developed is an easy, low-cost, and ecologically friendly method. This paper describes the fabrication of an environmentally friendly PF-flax fabric hybrid composites using compression moulding. Nano-silica extracted from rice husk were utilised to increase the hydrophobicity of the materials and to improve their dimensional stability. When the nano-silica is added, the properties of the products were greatly enhanced. This research was able to increase the mechanical, and electrical properties of the PF hybrid composite, and it has implications for the industrial manufacturing as well as applications in high-humid environments.

2 Materials and Methods

2.1 Materials

Rice husk was obtained from local sources (rice mill, Kalady, Kerala, India). Rice husk contains impurities such as alkali metals, and organic constituents. Sodium Hydroxide, Hydrochloric acid, Ethanol of analytical grades (AR) were purchased from Merk, India. Plain woven flax fabric was obtained from W.F.B. Baird & Company, Kochi, Kerala, India (Table 1). Phenol formaldehyde resin (PF) was purchased from Polyformalin, Ernakulam, Kerala, India. The pH of PF is 9–10, specific gravity at 30 °C is 1.252 and total solid content is 49.68% w/w.

2.2 Extraction of Nano-silica from Rice Husk

The raw material rice husk was obtained from rice mill at Kalady, Ernakulam, Kerala. All reagents used were of analytical grade, and their solutions were made up in distilled

Table 1 Basic parameters of flax fabrics

Parameters	Flax fabric
Woven style	Plain
Density (gm/cc)	1.3
Thickness (mm)	0.45
Weight (gsm)	140

Journal : Large 12633 | Article No : 2193 | Pages : 13 | MS Code : 2193 | Dispatch : 1-12-2022

deionized water. The rice husk was thoroughly cleansed so as to eradicate sandy and dust particles. The extraction process was adopted from published literature, with a few minor alterations, to meet our needs [38, 39]. Later it was subjected to heat treatment to obtain the ash. Samples were burned inside a programmable muffle furnace at 700 °C for 2 h. Followed by the addition of 1.5 M sodium hydroxide solution. Then the filtrate was treated with conc. HCl to form the silica sol. The suspension was filtered and washed several times and kept in oven for 24 h at 70 ℃. The dried powder was calcined at 700 °C for 2 h and used for further studies.

2.3 Preparation of Nano-silica Coated Flax Fabric/Phenol–Formaldehyde Composites

PF-flax fabric hybrid composites were fabricated by the compression moulding technique. The weight of nano-silica for the preparation of PF hybrid composites was taken based on the total weight of PF and flax fabric (Table 2.). Ethanol is used as a dispersing agent for nanomaterials. To obtain a uniform dispersion, the solution was homogenised for half an hour. Flax fabric was cut into a square piece (15*15 cm). The nanomaterial dispersion was sprayed on the fabric and kept in the oven at 70 ℃ for the removal of ethanol. On the dried fabric, previously weighed PF resin was poured and spread using a roller. After that, the sample is pressed in a pre-heated compression moulding machine at 100 ℃ for 30 min. The sheets of hybrid composites were produced and cut into the desired size after cooling, and were used for further research.

2.4 Characterisation of Nano-silica Coated Flax Fabric/Phenol–Formaldehyde Composites

2.4.1 X-ray Diffraction Analysis (XRD)

The crystallinity of the nano-silica and PF hybrid composites were recorded on Bruker AXS D8 Advance with Cu Kα radiation with an angle range 5°-80° (2θ angle range) at a wavelength of 1.541 Å and an operating voltage of 45 kV and a current of 35 mA.

Table 2 Designation and formulation details of prepared PF-F hybrid composites

Sample code	PF resin (g)	Flax fabric (g)	Nanofiller loading (g)	Weight percentage of composites
PF-F	10	10	0.0	0.00
1 NS	10	10	0.1	0.02
2 NS	10	10	0.2	0.04
3 NS	10	10	0.3	0.06
4 NS	10	10	0.4	0.08

2.4.2 Field Emission Scanning Electron Microscopy (FESEM)

The morphology of nano-silica and fracture surface of PF hybrid composites were analysed using Hitachi SU6600 Variable Pressure Field Emission Scanning Electron Microscope (FESEM) at acceleration voltage of 30 kV and Probe current of 1pA ~ 200nA. All the samples were sputter-coated with gold to avoid charging.

2.4.3 Thermogravimetric Analysis

The thermal properties of the samples were analysed using Perkin Elmer Diamond TG/DSC. In a nitrogen atmosphere, 10 mg of the sample was placed on the alumina cup and heated at a rate of 20 ℃ /min. The temperature range of measurement is from 25 ℃ to 700 ℃.

2.4.4 Atomic Force Microscopy (AFM)

The surface morphology of pristine nano materials and composites were characterised with Atomic Force Microscopy (WITec GmbH, Ulm, Germany) in contact mode at room temperature. The suspension of CNT and CNT-COOH was sonicated well and then analysed.

2.4.5 Mechanical Characterisation

The mechanical properties of the samples were tested using Universal Testing Machine (Tinius Olsen) according to ASTMD 638. The samples were cut into rectangular strips and the testing were conducted at room temperature with a gauge length of 60 mm and speed rate 2 mm/min.

2.4.6 Electrical Conductivity

The dielectric properties of the samples were measured using Wayne Kerr 600B precision LCR meter with frequency ranging from 20 Hz to 30 MHz.

3 Results and Discussion

3.1 X-ray Diffraction (XRD) of Nano-silica

The XRD pattern of nano-silica isolated from rice husk (Fig. 1) showed a wide-angle at 2θ = 22°, confirming nano-silica formation from rice husk. This unique broad peak at 2θ = 22° indicated the presence of silica in the amorphous form [40–42]. The obtained broad diffraction peak clearly

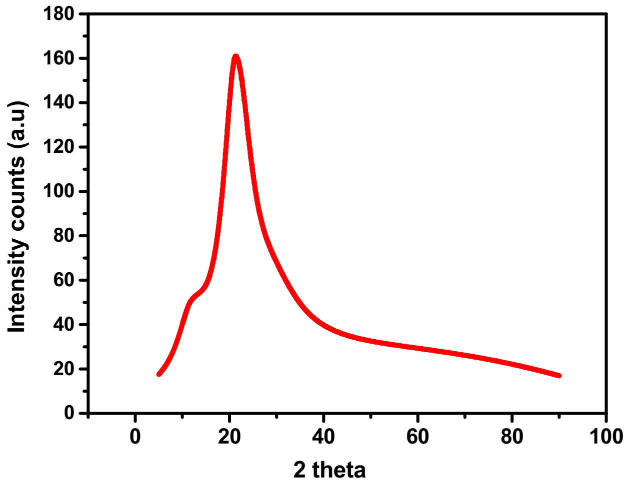


Fig. 1 XRD curve of nano-silica from rice husk

shows the existence amorphous nano-silica. If the peak was sharp then the silica obtained should be crystalline. The pure nano-silica, on the other hand, was sintered at 1373 K to reach the crystalline phase and quantify the crystallite size.

By treating with sodium hydroxide, sodium silicate is formed. When concentrated HCl is added pure nano-silica is formed.

$$SiO_{2(ASH)} + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$
$$Na_2SiO_3 + 2HCl \rightarrow SiO_2 + 2NaCl + H_2O$$

The presence of only one peak confirms the removal organic constituents and alkali metals associated with silica [43]. Chidambaram et al. [44] obtained similar diffraction peaks for nano-silica derived from groundnut shell, coconut husk, banana peel, walnut shell and orange peel. Previous findings demonstrated by other researchers, in which biogenic nano-silica was produced using rice husk as a precursor [45, 46]. The XRD peak of the produced biogenic silica revealed that it was amorphous in form, which is closely related to the current study.

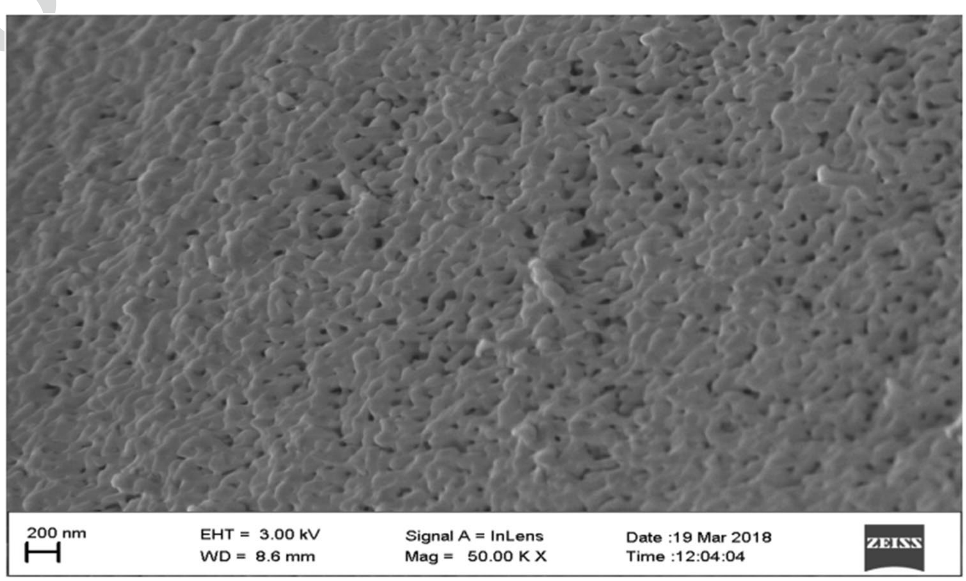
3.2 Field Emission Scanning Electron Microscopy (FESEM): Nano-silica

From the FESEM analysis (Fig. 2) it was observed that rice husk-derived nano-silica exhibited agglomerations. The hydrogen bonding between silanol groups on the surface of nano-silica was determined to be the cause of aggregation. The particles were found to be spherical and aggregation of silica-silica was found to be minimal. This result is in agreement with other literature reports [47]. Jen et al. [48] have isolated silica nanoparticles from palm kernel shell ash and analysed the morphology. SEM images showed porous nature of silica nanoparticles similar to our result. These bio-derived nanoclusters can be used as carriers in biomedical and nutraceutical applications [44].

3.3 Thermogravimetric Analysis (TGA): Nano-silica

Here we can see that weight loss was little with an increase in temperature (Fig. 3). Initial weight loss was due to the removal of residual moisture [42, 49, 50]. The very slow weight loss can be attributed to the porous nature of nano-silica. As a result, nano-silica required more time to heat up before water molecules could be released from the nano-silica.

Fig. 2 FESEM image of nano-silica obtained from rice husk



Journal : Large 12633 | Article No : 2193 | Pages : 13 | MS Code : 2193 | Dispatch : 1-12-2022

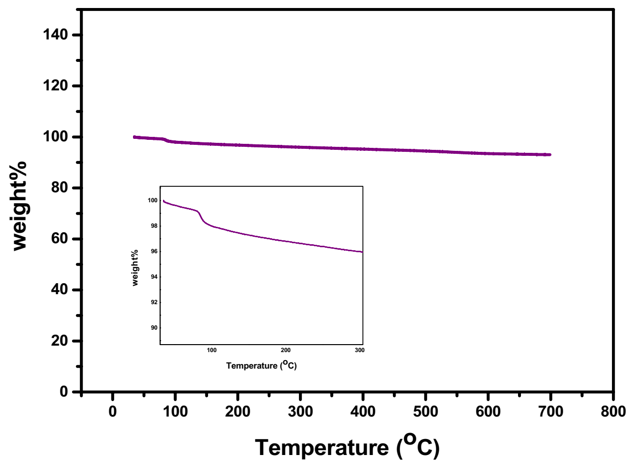


Fig. 3 TGA of nano-silica

3.4 X-ray Diffractogram (XRD) Analysis

The XRD data is used to determine the structural properties of PF hybrid composites with nano-silica. The XRD of PF hybrid composites with different loadings of nano-silica is shown in Fig. 4. The major peaks are observed at 18° and 22°. Pure PF does not show any strong peaks. The peak of nano-silica is at 22°. All PF hybrid composites have identical XRD patterns. The XRD results further show that the interfacial interaction of nano-silica with the PF-F matrix caused distinctive variations in crystallinity. The interfacial interaction of nano-silica with the PF-F matrix produced a characteristic improvement in the crystallinity of the hybrid composites.

3.5 Atomic Force Microscopy (AFM)

The surface topographies of PF-F and 2 NS is shown in Figs. 4 and 5. Atomic force microscopy is used to look at

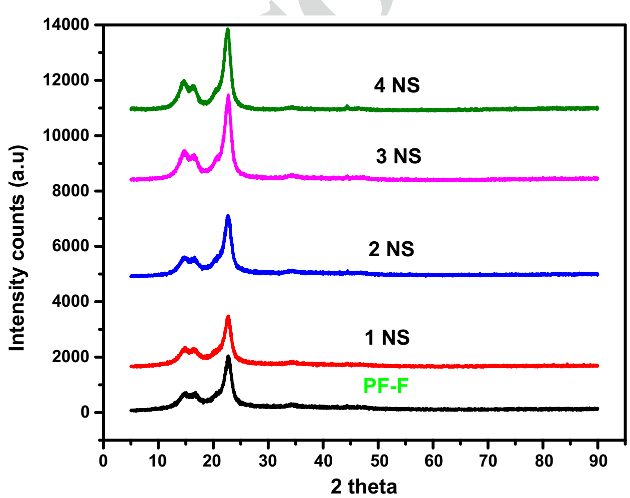


Fig. 4 XRD spectrum of PF-F hybrid composite with nano-silica

the surface roughness of composites that have been made. Figure 5A-C shows the AFM images of PF-F composites. This shows slightly rough surface. Figure 6A-C shows the AFM images of 2 NS composites. This loading was selected because it provides superior features in terms of sorption properties, hydrophobicity, thermal stability, and other characteristics. According to the enhanced properties of PF-F hybrid composites, as demonstrated in the experiment, the figure shows that the nano-silica were spherical, with minor agglomerations. One of the most important factors contributing to the better performance of produced composites is the fine dispersion of nanomaterials in the matrix. It has been demonstrated that the roughness of the hybrid composites prepared decreases as a result of the addition of nano-silica from 8.58 nm (PF-F) to 7.31 nm (2 NS). It is possible that the enhanced roughness of the surface of PF-F's is responsible for the lower contact angle and better wettability. Hence decreased roughness will lead to less wettability [51].

3.6 Mechanical Properties of PF-F Hybrid Composites Reinforced With Nano-silica

The stress–strain behaviour of woven flax fabric–PF composites and nano-silica coated composites is shown in Table 3. From the stress–strain curve, it is observed that the stress increases steadily with strain until all specimens break. It is obvious from the table that, in the case of brittle material, the stress–strain curve approaches linearity almost all the way to failure. At low strains, the stress–strain curves of the composites exhibit linear behaviour; nevertheless, when the composites fail completely, a considerable shift in slope is detected, indicating that the composites are exhibiting nonlinear behaviour. The stress–strain behaviour becomes more pronounced with the addition of nano-silica (2 NS). Due to the excellent load transmission between the nano-silica and the PF matrix and flax fabric, the composite 2 NS exhibits the highest tensile stress value. With the addition of nano-silica, the stiffness of the PF hybrid composites improved. The composites became more brittle by the addition of nano-silica, and before the composite ruptured, the elasticity of the matrix had been significantly enhanced. Here, the maximum energy needed for rupturing is 3 NS > 4 NS > 1 NS > 2 NS > PF-F.

The tensile strength of the PF hybrid composite was the least, while nano-silica inclusion increased the composite mechanical properties. Because 2 NS has good tensile strength, it will experience less deformation and degradation during testing and can withstand higher stress, which will improve its tensile strength. It should be emphasised that all samples showed brittle failure characteristics. The nano-silica content plays a crucial role in improving mechanical properties. The addition of fibres, reinforcements, and additives in the matrix is known to improve

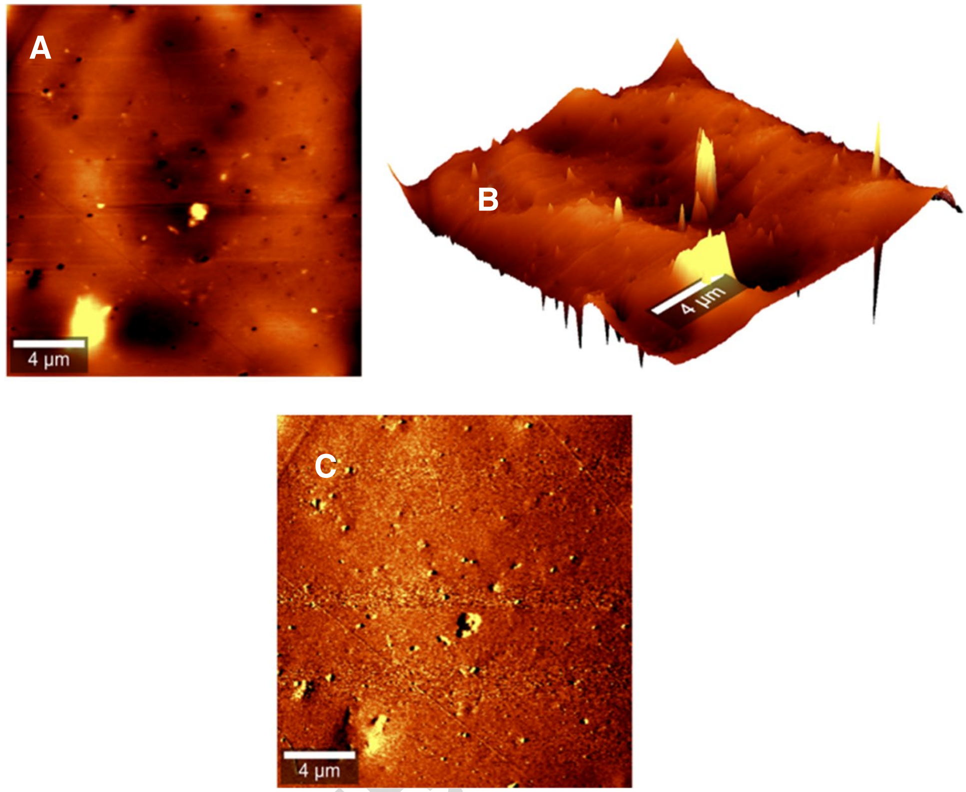


Fig. 5 **A-C** AFM image of PF-F composite **B** 3D image of PF-F composite

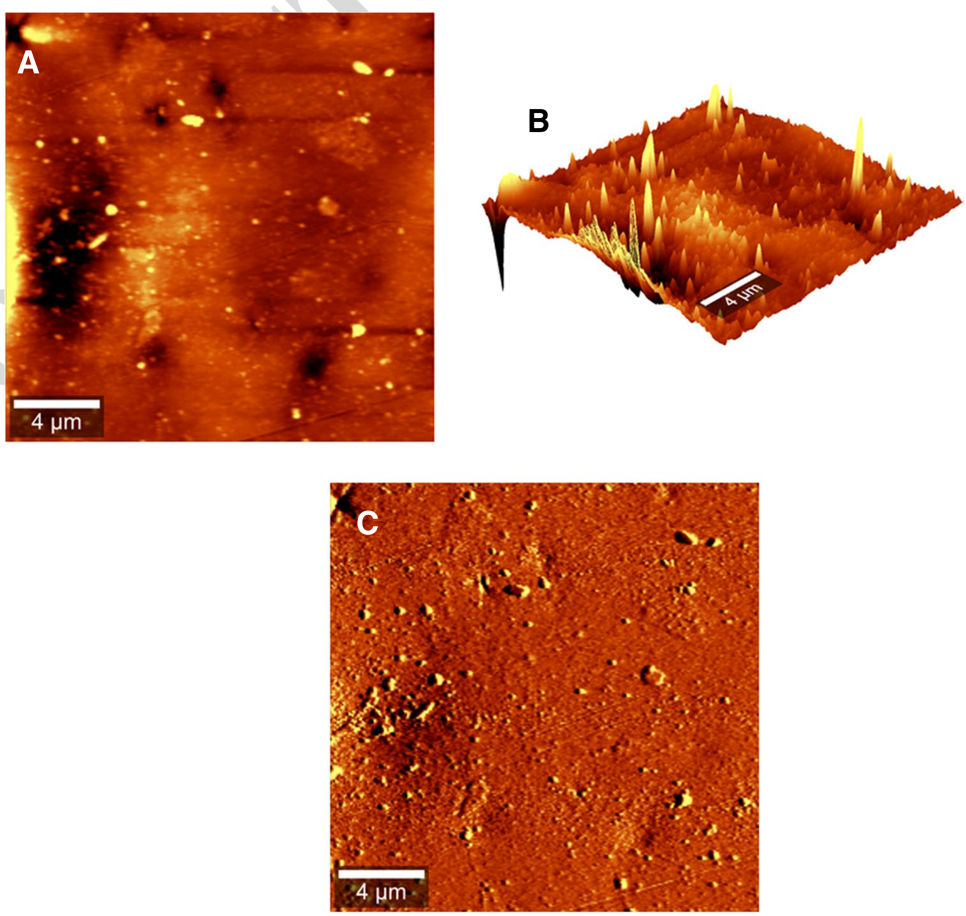


Fig. 6 **A-C** AFM image of 2 NS **B** 3D image of 2 NS

Journal : **Large 12633**	Article No : **2193**	Pages : **13**	MS Code : **2193**	Dispatch : **1-12-2022**

Table 3 Mechanical properties of PF-F hybrid composites with nano-silica

Sample code	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
PF-F	31 ± 1	1010 ± 2	8 ± 1
1 NS	61 ± 2	1095 ± 7	11 ± 1
2 NS	63 ± 2	1217 ± 4	10 ± 1
3 NS	47 ± 1	1051 ± 4	22 ± 1
4 NS	39 ± 1	1025 ± 6	20 ± 1

composite mechanical properties. By increasing nano-silica concentration, the tensile strength of composites improves. The tensile strength of the flax/PF composite is initially 31.59 MPa, but the addition of 1 NS raises the tensile strength to 61.26 MPa. That is, it shows an increase of 93.92%. The tensile strength is at its maximum observed for 2 NS, which is 99.55% higher than the PF-F composite. The tensile strength of 3 NS and 4 NS is 47.34 MPa (49.85%) and 39.94 MPa (26.43%), respectively.

The even distribution of nano silica enhances the weight carrying capabilities of composites. The nano-silica in nano-silica-coated flax/PF composites works as a deformation barrier, increasing composite strength. The tensile strength of composites is increased as a result of the formation of a strong interface between PF, nano-silica, and flax fabric [52–54]. At higher loading, the reduction in tensile strength was observed due to agglomeration, resulting in ineffective stress transfer. A tremendous improvement in tensile strength was observed due to the nano silica addition since an effective stress transfer is possible from nano-silica coated flax fabric to the PF matrix. The primary elements of flax fibre are cellulose, hemicellulose, wax, lignin, and pectin, in different ratios. The mechanical properties of the composite are determined by the cellulose, the toughest and strongest chemical ingredient in the fibre [9]. In all natural fibres, the cellulose is wrapped in non-cellulosic ingredients. The inclusion of silica nanoparticles into the structure of the fibre improves the fibre's load bearing capability of PF-Flax fabric composites. At higher loadings of 3 NS and 4 NS, the tensile strength decreased gradually due to the ineffective load transfer. At higher loading there is a chance of agglomeration, which results in the formation of localised stress points. This results in a lowering of mechanical properties.

The higher amount of nano-silica in the composite could operate as a stress concentration zone, resulting in localised cracks that inhibit the composite from improving its properties. A better particle dispersion order efficiently restrains the load flow [55]. The aggregation of particles causes poor dispersion quality at higher loading of nano-silica. The tensile strength reduces as a result [56–59]. Because of the improved fibre-matrix interaction caused by good adhesion, the tensile strength and modulus of a fibrous composite system were boosted. The reduction in free volume contributes to the improved tensile characteristics of prepared composites. The nano-silica loading improved the elastic nature PF-flax fabric composites.

The elastic behaviour of the composites is determined from the percentage of elongation. The highest elongation at break was observed for 3 NS and at higher loading of nano-silica (4 NS) the elongation at break slightly decreased than 3NS. The 1 NS and 2 NS composite have toughening behaviour compared to other loadings which is evident from less elongation at break. The addition of nano-silica at lower loading improves the stiffness of the entire composite thereby reducing less plastic deformation. From the results we can conclude nano-silica addition improved mechanical properties. The tendency for elongation at break (%) to decline and eventually break at higher filler loading, matrix deformation is not only influenced by the nature of the interface but also by the dispersion of the fillers. Due to the addition of mechanical constraints by the nearly indeformable nano-silica particles, the modest fall in ductility implies a reduction in matrix deformation. Nano-silica act as stress concentrators due to various elastic characteristics of the material's constituent parts. Particle agglomeration encourages increased cavitation, higher stress concentration, and faster breaking [60]. Many researchers got similar trend with the addition of nano-silica in other polymer matrices [61, 62]. Feli et al. [63] got improvement in mechanical properties with nano-silica loading in epoxy composites. Zhou et al. [64] found enhancement in elongation with the addition of 2 wt.% nano-silica in epoxy-carbon fibre composites. In corelation with stress–strain curve the toughness was calculated and found that nano-silica addition improved the same. The mechanism of interaction is schematically shown in Fig. 7.

3.7 Fracture Mechanism by Morphology Analysis (FESEM)

The fracture surface morphology of PF-flax fabric composites and nano-silica coated flax fabric/PF composites were evaluated under a scanning electron microscope (Fig. 8). When a polymer is used, it transmits the load to the fabric and functions as structural support for the entire composite. As a result of the loading, the composite is pulled and the composite's elongation occurs. Elongation causes cracks to appear in the material, and the brittleness of the PF will tend to spread the locally formed crack while the material is being stretched. As a result, a piece of the polymer surrounding the fibres is removed throughout the procedure. This is responsible for the debonding of the fibres. As the fibre de-bonds,

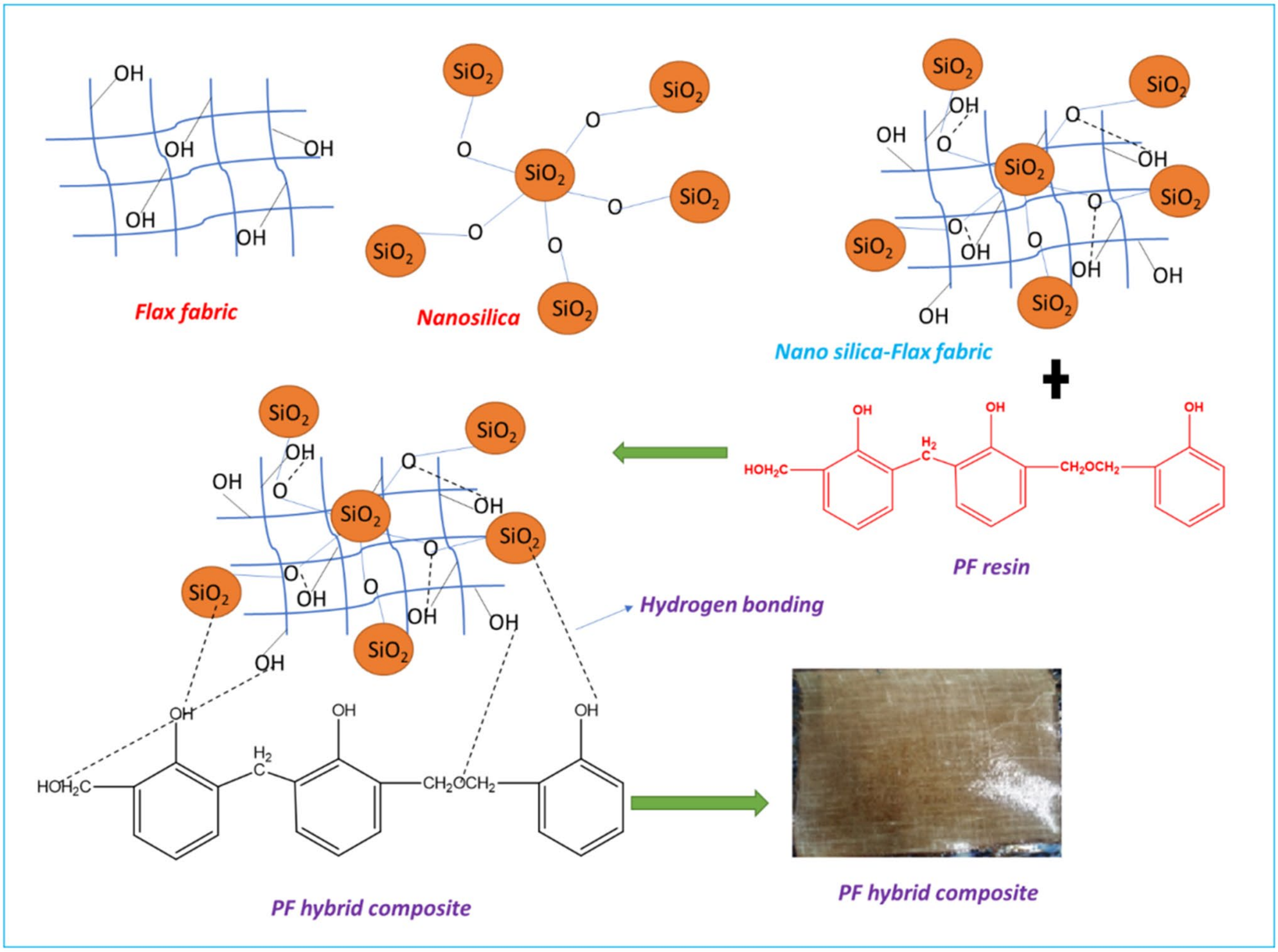


Fig. 7 Plausible mechanism of interactions present in PF-F hybrid composite

it progressively loses its capacity to bear weight, and, as a result, the material's strength diminishes with time.

Figure 8a.1 & a.2 shows fibre pulling out from the fabric and debonding from the PF-F matrix. The presence of nano-silica can limit the growth of microcracks caused by stress concentration by having good interaction and a strong bond with the PF matrix and flax fabric (Fig. 8b.1 & b.2). Therefore, the toughness of hybrid composites is improved. It's critical to have effective stress transmission between the PF, flax fabric, and nano-silica in order to obtain maximum mechanical properties. The existence of interfacial interaction enhances the passage of stress within the composite [65]. Composites reinforced with nano-silica reinforcement, on the other hand, show evidence of better fabric-matrix bonding [66]. On closer inspection, it can be observed that the nano-silica reinforcement ensures better load-bearing efficiency. There are no apparent fibre pull-outs, indicating that the de-bonding of fibres has been greatly decreased. As a result, increasing the load-bearing capability of nano-silica can be advantageous.

3.8 Electrical Properties of PF-F Hybrid Composites Reinforced With Nano-silica

The polarizability of a substance has an effect on the dielectric constant of that material. The dielectric constant of a polymeric material is affected by polarisation at the interface, at the dipole, at the electronic level, and at the atomic level [67]. Due to the presence of polar groups in PF resin, it has a dielectric constant. The interfacial polarisation is also present in hybrid composites since they are heterogeneous. Because the molecule may be fully oriented at low frequencies, the dielectric constant has a frequency dependency. Medium-frequency transmissions provide little time for orienting. At extremely high frequencies, molecular orientation is impossible. Here we can see that due to the presence of nano-silica, the dielectric properties have been improved. This is because of the polar groups (nano-silica) present in the hybrid composite. 2 NS has highest AC conductivity [Fig. 9a, dielectric conductivity Fig. 9b and dielectric loss Fig. 9c]. This was most likely due to a higher population of nano-silica in the backbone, as well as phase separation. The

Journal : Large 12633	Article No : 2193	Pages : 13	MS Code : 2193	Dispatch : 1-12-2022

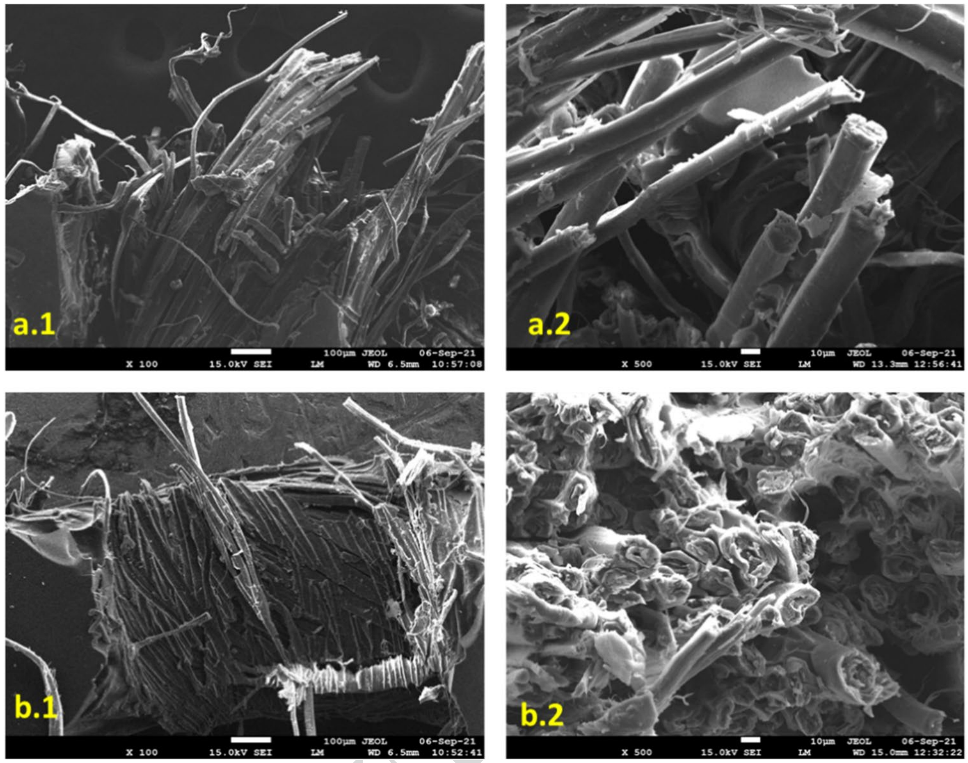


Fig. 8 FESEM images of **a.1** & **a.2** PF-F and (lower and higher magnification) **b.1** & **b.2** nano-silica coated PF-F hybrid composites (2 NS) (lower and higher magnification)

total dielectric constant of the PF hybrid composite was greater than the PF-F composite because nano-silica has a higher dielectric constant than pristine composite [68]. It is apparent that when the frequency of application is increased, the ac conductivity rises. This might be attributed to an increase in absorbed energy, which in turn leads to an increase in the number of charge carriers involved in the conduction process. These findings corroborate previous findings for various polymer composites [69–71].

Interfacial, orientation, atomic, and electronic polarizations all have a role in determining a composite's dielectric constant (E). The matrix and filler conductivities, or polarizations, vary, resulting in interfacial polarisation in composites [72]. When polymers with polar groups are put in an electric field, orientation polarisation occurs. Filler concentration affects both the composite's orientation and interfacial polarisation. At low frequency, nano-silica loading improved the dielectric constant as seen in Fig. 9b. Natural fibres include polar groups of cellulose, which contribute to an increase in orientation and interfacial polarisation with increasing fibre loading. The E exhibits greater values at lower frequencies for a given fibre loading. Orientation polarisation diminishes as frequency increases, and this may be attributed to this phenomenon. Orientation polarisation takes longer to attain equilibrium than electronic and atomic polarisation, and lower frequencies are required for full orientation of the molecules. Due to the lag in orientation polarisation, as frequency rises, the E decreases.

4 Conclusions

Phenolic-based hybrid composites were prepared with flax fabric and nanofillers. With the addition of nanofillers significant improvements were found. The following conclusions may be drawn from the data:

- Nano-silica was successfully isolated from rice husk. The diffraction peak shows $2\theta = 22°$ which is specific for nano-silica. Higher thermal stability was observed from TGA. From the FESEM images it shows clusters of nano-silica particles.
- The XRD data revealed that the incorporation of nano-silica into the PF-flax composite provided efficient reinforcement.
- The XRD data revealed that the incorporation of nano-silica into the PF-flax composite provided efficient reinforcement. From the mechanical properties, the addition of nano-silica improved the tensile strength, modulus, toughness, and ultimate stress. The improved mechanical characteristics of composites containing 2 NS were attributed to the uniform dispersion of nano-silica in the PF-flax fabric composites. In the presence of 2 NS, an increase in tensile strength of approximately 61% has been recorded. From the fracture surface morphology, it is clear that the absence of visible fibre pull-outs and the debonding of fibres has been significantly reduced by the addition of nano-silica (2 NS).
- The dielectric constant in PF hybrid composites grows as the loading of nano-silica increases due to polarisation processes

Journal : Large 12633	Article No : 2193	Pages : 13	MS Code : 2193	Dispatch : 1-12-2022

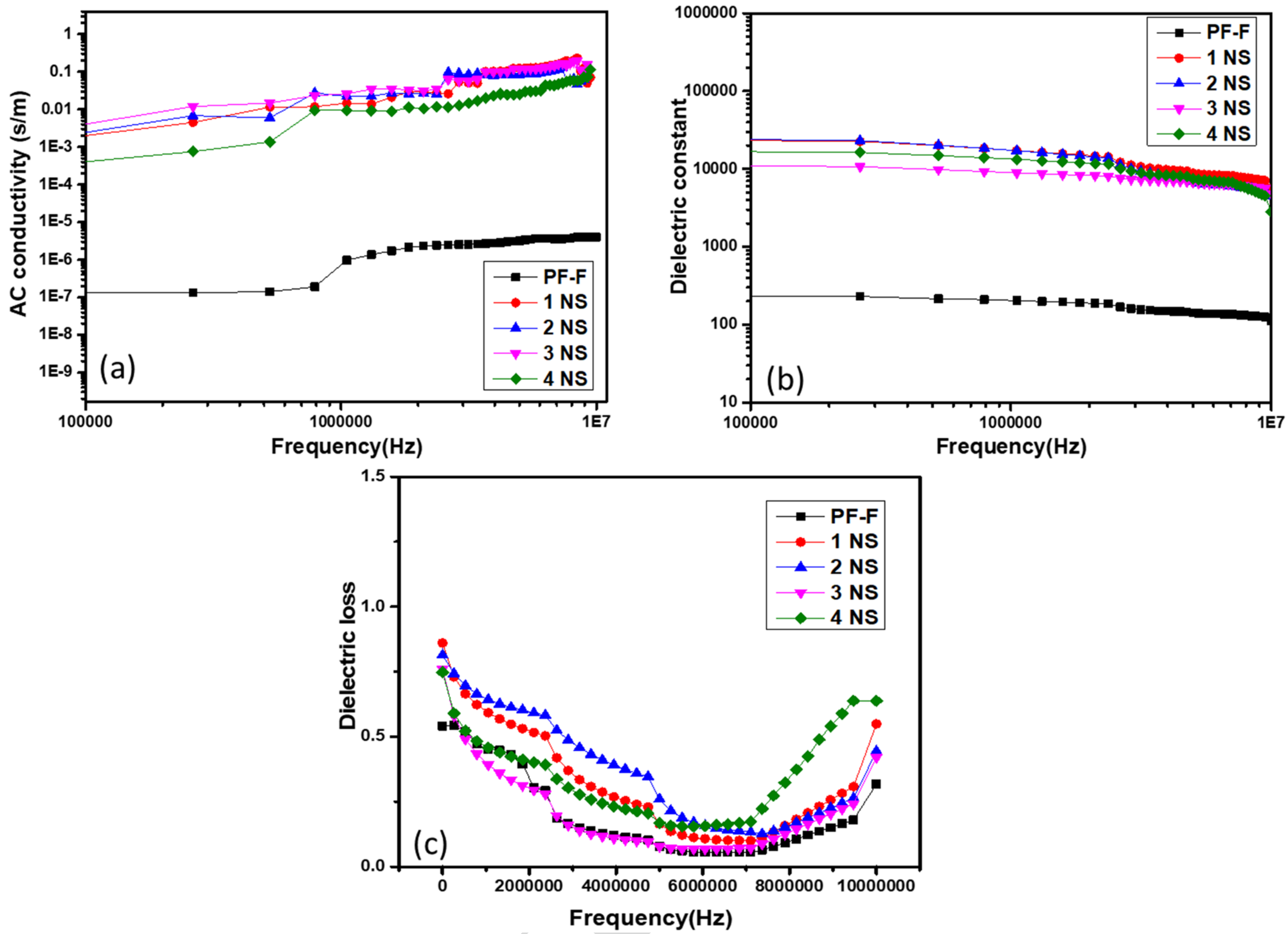


Fig. 9 Dielectric properties[**a**-AC conductivity, **b**- dielectric constant, **c** dielectric loss] of PF hybrid composites with nano-silica

mediated by nano-silica inclusion. At high frequencies, electronic polarisation induces an increase in AC conductivity (3 NS). The graph demonstrates that the AC conductivity increases as the loading of nano-silica increases. The highest dielectric constant values are found in 2 NS.

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Authors' Contributions All the authors contributed equally to this work.

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Data Availability The data that support the findings of this study are available from the corresponding author, [Dr. Sreekala M.S.], upon reasonable request.

Declarations

Ethics Approval and Consent to Participate Not Applicable.

Consent for Publication Not Applicable.

Research involving Human Participants and/ or Animals Not Applicable.

Informed Consent Not Applicable.

Competing Interests The authors declare that they have no competing interests.

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