

Mechanical and Surface Characterization of Sisal Fiber (Agave Sisalana) After Cold Glow Discharge Oxygen Plasma Treatment

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Abstract

Agave sisalana is a rosette-forming succulent plant farmed primarily for its fibers, Sisal is utilized in cordage primarily along with its toughness, longevity, stretchability, propensity towards particular colorants and saltwater tolerance Sisal fibers have just low compactness, are readily available, and are sustainable and environmentally friendly, although they have inherent drawbacks, including hydrophilicity with performance variability. Even though these composites have been natural fiber-reinforced, their composite structure including chemical characteristics attributable to mechanical characteristics has always been associated with a better. The application of cold glow discharge oxygen treatment on sisal fibers was investigated in this study. Unidirectional Sisal fibers had been surface treated with cold glow discharge plasma oxygen throughout this research, as well as the impact on mechanical properties would always be investigated. To increase the combination of sisal fiber with epoxy resin, oxygen plasma treatments were employed to enhance the characteristics of sisal fibers at varied cold glow discharge plasma energies 80W, as well as 120 W for 30 minutes. Oxygen plasma modification employing cold glow discharge once compared to an untreated sisal fiber-reinforced epoxy laminate, 80W(30min.) of sisal fiber offered the sisal fiber reinforced epoxy composite (SFREC) with well almost 76.92 % substantially greater interlaminar shear strength, 56.94 % comparatively stronger flexural strength, 76.41 % relatively improved larger elongation break, and 58.56 % tremendously greater tensile strength. The morphological characteristics of cold glow discharge were examined employing FT-IR spectroscopy and (XRD). Oxygen plasma-treated sisal fibers performed better than untreated sisal fibers in terms of surface structure. Sisal fibers have been exploited for engineering applications after becoming surface-treated, rendering them an appealing and competitive material that contributes to the world's objective of supporting self-sustaining and biodegradable natural materials.

Statement Of Novelty

Natural fiber as an intriguing option for reinforcing polymeric matrices has sparked a rising interest in creating sustainable and environmentally friendly lightweight structures. Natural fibers' are limited in their application due to deteriorating thermal properties, low compressive strengths, and strong attraction for water, which leads to poor crosslink density, which is a major hindrance to their use as reinforced composites. To improve the fiber-matrix functionality of polymer composites, several surface engineerings technologies, such as chemicals and plasma procedures, have been explored for natural fibers. The major goal of this study is to develop an experimental approach for assessing the influence of cold glow discharge oxygen plasma modification on the mechanical performance of sisal reinforced epoxy laminates along with sisal fiber surface characterizations owing to increased mechanical behavior and improve the cohesive forces between fiber and matrix. After being surface-treated, sisal fibers might be used in industrial applications, making them a highly appealing but viable resource that helps society achieve its goal of cultivating self-sustaining yet biodegradable natural resources.

1. Introduction

In recent years, long-term environmental development challenges have been a concern for several areas of business, as well as the subject of numerous research throughout the world. Environmental challenges such as soil and water pollution, garbage disposal, and efficiency gains for environmental sustainability are pushing the development of healthier materials [1]. Concerns about the sustainability and reprocessing of natural resources have rekindled enthusiasm with biopolymers, particularly renewable raw materials. Either as result, a wide range of plant fiber-based cultivars have lately recently been explored [2]. In this context, innovators focusing on fiber-reinforced polymer nanocomposites increasingly turning to bio-renewable bio-based fibers for manufacture and utilization [3]. Biomass resources fibers were some of the most researched as they provide high strength at reduced weight loads and are undoubtedly the most abundant fibers in nature. In addition, lignocellulosic fibers are mostly abrasion resistant with synthetic fibers, enabling composite manufacture easier and less expensive [4]. Furthermore, the production of sustaining biobased fibers can be less energy-intensive and polluting than the synthesis of various fibers [5]. Vegetable fibers, on the other hand, still have downsides, besides the fire resistance, wettability, very good heat conductivity. [6]. Natural fibers' hydrophilic character, on the other hand, is a major hindrance to their use as reinforced composites. Natural fibers' low moisture friction creates incompatibilities and poor permeability with hydrophobic polymers, which disrupts contact interactions at the fiber/matrix interfaces [7–8]. To improve the fiber-matrix functionality of polymer composites, many surface engineering technologies, such as chemicals and plasma procedures, have been explored for natural fibers. Whenever natural fibers are used to strengthen thermoplastics and thermosetting polymers, the most common chemical treatments are alkaline treatment [9–10], silane treatment [11–12], and acetylation treatment [9, 13]. Whereas chemically treated fiber surfaces have improved interfacial adhesion to some extent, there are still unsolved environmental issues linked to chemical recycling after treatment, as well as the high expense of chemical treatment. As a result, chemical changes need necessary to make them suitable for diverse applications [14]. Lignin, hemicellulose, lubricants, hydrocarbons, and other low molecular weight compounds components can be extracted using a range of different treatments [15]. Different chemical treatments have not been widely used in industry in recent years due to growing concerns about environmental contamination [16]. The low-temperature plasma method is one of the alternative technologies. This approach has also been used to alter and enhance the texture of fibers in the together mild but effective way [17]. Plasma treatment significantly increased fiber-matrix adhesion by having to introduce positively charged or energized clusters or even just a new polymer surface capable of forming a stable complex between the fiber and the matrix, as well as exfoliation the fiber surface to fiber surface to boost structural interlocks of both the fiber - matrix [17]. The application of the “methane cold ionization technique” to minimize the deterioration processes with “residual sisal fibers”. [18]. To treat jute fiber with polymerizing plasma gas, increasing the tensile strength including the plasma-treated “jute-polypropylene composite. [19] “Surface lignin forcibly removed alongside coir fibers” through modification of plasma massively increases thermoplastic starch composite cohesiveness. It boasts a tensile strength of over 300 percent as well as an elastic modulus of nearly 20 times, which is linked to greater fiber matrix cohesion [20]. The flexural strength for laminates developed utilizing plasma-modification fibers had been enhanced by about 14% even before compared to the raw composite material [21]. Adhesion can be enhanced by using chemical modification that

promotes “cohesive, electrostatic, or chemical bonding, as well as physical techniques” that significantly improve cohesive bonding between the reinforcement and resin. Plasma treatment, that either significantly improves the “mechanical, physical, and structural character traits of natural fibers,” is extremely crucial for using natural fibers in high-tech applications[22]. Woven-type basalt fibers incorporating cold oxygen plasma atmospheric to explore changes in water removal engendered with an oxygen plasma modification [23]. Plasma treatment of Ramie fiber risen the elastic modulus of the ramie fiber, culminating in a stiffer fiber[24]. Argon plasma through the use of a novel method identified as “pad dry plasma cure.” As per research, incorporating Argon plasma treatment towards the “bridging agent as well as cellulose molecules could help boost its bridging effect.” Argon plasma treatment significantly increased tensile properties as well as the percentage of elongation.[25]The application of cold glow discharge oxygen treatment on sisal fibers was investigated in this study. Unidirectional Sisal fibers had been surface treated with cold glow discharge plasma oxygen throughout this research, as well as the impact on mechanical properties would always be investigated. To increase the combination of sisal fiber with epoxy resin, oxygen plasma treatments were employed to change the surface of sisal fibers at varied plasma energies 80W, as well as 120 W for 30 minutes. The mechanical performance of pre-treated and cold glow discharge oxygen plasma modification sisal fibers had been investigated, and ILSS was used to evaluate the interfacial bonding of composites (interlaminar shear strength), as well as flexure testing analysis.FT-IR, XRD was used to explore surface characteristics and fiber surface chemistry, and single fiber tensile tests were utilized to examine the mechanical characteristics of pre-treated sisal fiber and cold glow discharge oxygen treated sisal fiber.

2. Materials And Methodology

2.1 Fiber and Matrix Materials

The raw resources used during work have also been unidirectional sisal fiber in its natural state and modified physically, as well as epoxy Lapox Metalam- B as reinforcement and matrix, respectively. Go green product Alwarthirunagar Chennai provided the sisal fiber. The epoxy Lapox Metalam - B was used as the matrix, with Lapox Metalam - B serving as the hardener and the resin binder. Lapox Metalam B (Viscosity at 25°C: 800–1,200 MPa, Density 1.00–1.20 g/cm³) and Lapox Metalam B Hardener (Viscosity at 25°C: 300–600 MPa, Density 0.95–1.00 g/cm³) were equipped by Atul Industries Gujrat in India.

2.2 Cold Glow Discharge Oxygen Plasma Treatment of Sisal Fibre

A system for producing Glow Discharge plasma utilizing RF and DC power has been developed, manufactured, and deployed at “Shri Vaishnav Vidyapeeth Vishwavidyalaya, Indore's Centre of Excellence for Plasma Research”. A cylindrical stainless steel vacuum chamber (stainless steel material (SS304)) is used in the plasma system. Figure 1 depicts a typical plasma treatment system, which has a “vacuum chamber” with a capacity of 30 liters, a height of 300 mm, and a diameter of 360 mm as well as a cathode-electrode unit (02 round electrodes, one of which has a movable displacement, which is referred

to as the "anode" and a fixed electrode which is referred to as the "cathode" An RF power supply (SEREN RF Generator) with 13.56MHz, 600 Watt, and an automatic matching network for RF electric discharge of gas and a high voltage DC-power supply (2 KV, 1 amp) for the DC electric discharge of gas. Incandescent discharge plasma is generated from gas. The device combines a rotary pump (CG COMMERCIAL MOTORS) and a turbo molecular pump to evacuate the container (Pfeiffer Vacuum, Germany). "A rotating pump (RP) was included to support the TurboMolecular Pump, it has a pumping speed of 60 L/s." When all outlets are bled, the reactor creates an ultimate vacuum of 1×10^{-6} mbar. Gas feeding connections are pumped across areas using a rotary pump. All cross-functional and cross lines to the gas cylinders, flow controllers, and vacuum chamber are separated by independent gas valves. An Ion gauge (PFEIFFER VACUUM) is used to measure vacuum within the chamber from 10^{-3} mbar to 10^{-9} mbar, and a digital Pirani gauge is often used to monitor vacuum from the ambient pressure. A mass flow controller (Dosing Valve EVN 116 metering valve) is a device that precisely regulates the gas flow.

As shown in Fig. 2, sisal fibers were treated using Cold Glow Discharge oxygen Plasma Treatment with RF-plasma systems at varying discharge power of 80 W (30 min) as well as 120 W (30 min). Compressed air was used to clean the inside of the chamber glass tube and the sample container. For both the "positive and ground electrodes," the substrates (40 grams of fibers) were weighed, treated, and placed in the vacuum chamber. The chamber was subsequently inflated to a pressure of 3×10^{-4} millibars using a mix of rotary and Turbomolecular pumps, and oxygen gas was supplied at a "pressure of 100 mbar lit/s inside the chamber through a Dosing valve." After achieving a vacuum of 3×10^{-4} millibars with a combination of rotary and Turbomolecular pumps, oxygen gas was supplied into the chamber via a "Dosing valve" at a flow rate of 100 mbar lit/s until the vacuum reached approximately " 1×10^{-1} millibars." After applying RF power to the electrodes and using an automatic matching network, oxygen plasma with zero watt reflection develops. We experiment with all of the factors, such as power, time, and flow rate, before reaching optimum. For sisal fibers, we got acceptable results after "30 minutes at 80 Watt and 30 minutes at 120 Watt." The gas flow was maintained for further 10 minutes after the treatment was completed to eliminate any undesirable reactive products. After being removed from the holder, the treated fibers were kept in a vacuum-sealed envelope for further analysis.

2.3. Composite preparation

Lapox Metalam B (Viscosity at 25°C: 800–1,200 MPa, Density 1.00–1.20 g/cm³) was chosen as the matrix material, with Lapox Metalam B Hardener (Viscosity at 25°C: 300–600 Mpa, density 0.95–1.00 g/cm³) as the resin binder. Epoxy resin was combined with a 2:1 ratio of hardener to create the composite. This solution was used to make the matrix. These laminates were created using an adapted hand lay-up process. The plastic sheet was coated with silicone spray, and the metal frame was used to cover the mold, allowing for simple withdrawal and an increased surface texture in the laminates. The epoxy was evenly distributed across the complete area of each surface and any air spaces were filled by brushing and pushing with the roller. The epoxy and fiber layers were stacked using ESESE. The fiber configuration was maintained at 0°. The laminates that had been expelled from the mold and cooled down to ambient temperature The casts of each composite were cured for 24 hours under a 60 kg load. The composites'

dimensions were cut with a diamond cutter in accordance with ASTM specifications. After the curing phase, the composites were hard and dry enough to cut.

2.4 Mechanical Characterisation of composite

The tensile specimen, which measures 165 mm x 19 mm x 3 mm and has a notch circle radius of 76 mm, was created using an ASTM D3039-00 computerized universal testing machine (UTM TUE-C-200/200 kN) with a cross-head speed of 5 mm/min. The flexural tests were performed in accordance with ASTM- D790 on a Digital Tensile Testing Machine (KMI-1.3 D)/2.5 kN with a sample size of 125 mm x 12.7 mm x 3 mm and a cross-head speed of 5 mm/min under a gradual load of 10 KN. To analyze ILSS values according to ASTM D-2344, a Digital Tensile Testing Machine (KMI-1.3 D)/2.5 kN with a cross-head speed of 5 mm/min Each specimen measured 6.4 mm in breadth and 26.3 mm in length. Three samples are taken for each measured reading, and the mean value of the analysis was recorded. For each measured reading, 03 specimens are collected, and the analyses' average results were recorded.

2.5 Surface Characterization of Composite

2.5.1 Mechanical Characterisation of Sisal fiber

The ASTM C1557-03 [26] standard is used to determine the mean diameter of samples, which was necessary for estimating the cross-sectional area of cold glow discharge plasma oxygen treated and untreated sisal fiber. A Polarising projection microscope (Innolab RPL-4,17204 with 10 X Magnification) was used to assess the mean diameter of samples. To examine the competence for the reinforcement, the tensile properties of the fiber have been determined employing a single fiber tensile test following ASTM C1557-03 [27]. The fibers were clamped inside the testing machine's jaws and the tensile test was done on a "Digital Tensile Testing Machine (KMI-1.3D/2.5kN) template"[28]. There were at least ten measurements taken. More than 30 fibers were tested for each cold glow to ensure that more than 10 precise measurements were taken.

2.5.2 Surface characterization of untreated and cold glow discharge oxygen treated sisal fiber by using FTIR

The alterations on the surface property with fiber as support for interfacial bonding, various physical treatments were investigated using FTIR analysis. In a twin beam "FTIR spectrophotometer (SHIMADZU FTIR-8900 spectrophotometer) with a resolution of 2 cm^{-1} in the wavenumber range of $4000 - 400\text{ cm}^{-1}$ ", the materials were examined using an attenuated total reflectance device. An average of 25 scans was recorded.

2.5.3 Surface characterization of untreated and cold glow discharge oxygen treated sisal fiber by using Surface characterization by using XRD

The X-ray diffractograms for cold glow discharge plasma oxygen treated sisal fiber, as well as pretreated sisal fiber, were recorded on the "image plate (Mar 345 dtb) area detector of Indus-2 Angle-Dispersive X-

ray Diffraction (ADXRD) Beamline (BL-12)" at calibrated photon energies (17.42111 keV) on the "image plate (Mar 345 dtb) area detector of Indus-2 Angle-Dispersive X"

3. Results And Discussion

3.1 Ultimate Tensile Strength and % Elongation Characterisation

As shown in Figs. 3 and 4, its ultimate tensile strength and % Elongation break within an untreated sisal fiber reinforced epoxy (SFREC) composite were 104.27 MPa and 3.18 mm, respectively. The experimental results demonstrated that ultimate tensile and % Elongation break increased consistently up to 10% volume of fiber, and the trend continued in 30% volume of fiber, while tensile strength and % Elongation break declined at 40% volume of fiber. The surface with sisal fibers was treated with cold glow discharge plasma oxygen at different power levels of 80 W (30 min) as well as 120 W (30 min), and the treated fibers were utilized to make composites. Tensile Strength and % Elongation of Cold Glow Discharge Plasma Oxygen treated SFREC of the various treated fibers is shown in Figs. 3 and 4. After cold glow discharge plasma oxygen treatment of sisal fiber with plasma powers of 80W(30 min) and 120 W,(30 min) its tensile strength as well as % elongation break for Sisal fiber-reinforced epoxy (SFREC) laminate increased to 165.34 MPa, 5.61 mm, 142.91 MPa, 5.21 mm respectively. This translates to increases of 58.26%, 76.41% and 37.05%, 61.0% respectively. Increasing the volume percentage increases the mechanical characteristics up to a point. Furthermore "the enhanced cohesion between matrix-fiber leads to a considerable increase in mechanical characteristics of the laminate reinforced by natural fiber"[29, 30]. The best mechanical characteristics, such as tensile strength and % elongation, were found in untreated sisal fiber-reinforced epoxy laminates with a volume percent of fiber of 30%. The greatest tensile strength and % elongation break were 104.27 MPa and 3.18 mm, respectively. Increasing the volume percentage enhances mechanical qualities up to a certain extent. Surface topography becomes rough as hemicellulose, lignin, waxes, and pectin are removed. In addition, the fiber bundles are broken down into smaller fibers, the fiber diameter is reduced, and the aspect ratio is enhanced. The tensile strength and % percentage elongation increase as a result of the increased surface area between the fiber and the matrix. The O-H and C-OOH groups on the fiber surface are exposed when waxy substances dissolve, resulting in enhanced polarity and decreased acidity of the surface topography of fiber. Surface functional groups are formed by interactions involving energized species in the plasma and the substrate surface, and further modifications, such as the bridge at the surface, can be produced by reactions among the stimulated surface species [31, 32]. When sisal fiber is exposed to cold plasma for 30 minutes at a power of 120 W, the fiber can deteriorate, lowering the compatibility between matrix and reinforcement, The mechanical properties of the fiber are affected by cold glow discharge plasma oxygen modification. This might be since "physical sputtering, as well as chemical etching, constitute two types of techniques involving plasma ablation of materials. "Physical sputtering has always been a high-energy ion knock-on method which involves direct sputtering of materials employing chemically non - oxidizing plasma." [33]

3.2 Flexural Test

Figure 5 depicts the flexural strength for different volumes of fiber % for untreated and cold glow discharge plasma oxygen treated sisal reinforced epoxy(SFREC) composites. With 30% volume of fiber untreated sisal reinforced epoxy composite has the maximum flexural strength (82.79 MPa), whereas 10% volume of fiber untreated sisal reinforced epoxy(SFREC) composite has the lowest (34.98 MPa). With increasing fiber loading, flexural strength usually increases. Because natural fibers have a high modulus, a higher concentration of fibers necessitates higher stress to achieve the same deformation. The natural fiber-reinforced laminate's mechanical strength is highly better due to improved matrix-fiber cohesion [33]. The flexural strength of the fiber-reinforced cold glow oxygen composite increased by nearly 56.94% and 50.76%, respectively, when compared to the untreated sisal fiber reinforced epoxy(SFREC) laminate at different power levels of 80 W (30 min) and 120 W (30 min), 30 volume of fibre % of sisal fiber loading. Plasma treatments can have a big impact by removing organic impurities from the surface, removing the poor boundary layer and increasing the contact area, strengthening the surface layer, and changing the chemical structure.[34, 35].However, flexural strength decreases as fiber volume percent fiber loading exceeds 40%, fiber aggregation, and all through the etching process, usually weight of the fiber surface is lost as well as the upper surface layer is removed. The weight loss rate is strongly influenced by laminate composition and "plasma energy level"[36].

3.3 Interlaminar Shear Strength (ILSS)

The interlaminar shear strength of sisal fiber-reinforced epoxy matrix treated using cold glow discharge plasma oxygen is shown in Fig. 6. According to the ILSS data, the ILSS of cold glow discharge plasma oxygen treated sisal fiber reinforced epoxy (SFREC)composites increases up to 80 W (30 min) at 30% volume of fiber. The ILSS then drops as the plasma intensity is raised at 120 W. (30 min). When compared to untreated Sisal fiber reinforced epoxy composites, ILSS values for cold plasma oxygen treated sisal fiber-reinforced epoxy laminates at plasma powers for 80 W and 120 W increase 76.92% and 70.99%, respectively. Plasma treatments chip away cellulose and hemicellulose more easily because they are more reactive to plasma, and boost cohesive adhesion [37]. The ILSS of the composite, however, decreases slightly after being oxygen plasma-treated at 120 W for 30 minutes. The mechanism of plasma surface treatment differs depending on the stage of plasma treatments, according to studies. Initially, the surface modification takes precedence, but later in the process, surface etching takes control [37–41].

3.4 Force- Displacement Curve

Tensile tests were used to plot force vs. displacement graphs for pretreated sisal fiber-reinforced epoxy laminate as well as cold glow discharge plasma oxygen modification sisal fiber-reinforced epoxy laminates 20%, 30%, as well as 40% vol. of fiber, as shown in Fig. 7(a-c), indicating that maximum force carrying capacity increases as fiber content in epoxy matrix increases. Figure 7 (a) shows how the load steadily grows until it approaches the maximum load-carrying capacity of the material, after which it declines. The influence of plasma power on Force – displacement graphs between sisal fiber and epoxy resin was investigated using composite force-displacement values. When sisal fiber is treated with cold plasma oxygen at 80 W for 30 minutes, the load progressively increases until it reaches the material's

maximum load-carrying capacity at 30% fiber volume and then gradually decreases. As shown in Fig. 7 (a), up to a load of 12789.37 N, the linear path of force-displacement graphs is followed. The ultimate point (ultimate load 17528.8 N) on the force-displacement curve reveals the entire full rupture of the polymer composite throughout the composite whenever the sisal fiber was treated using cold plasma oxygen at 120 W (30 min). When the sisal fiber being subjected to cold glow discharge plasma oxygen for 30 minutes with 80W as well as 120W, respectively, the force-displacement graph is followed linearly up to loads of 20279.8N and 17528.8 N. Rose force(N) values of 58.56% and 37.05% were reached utilizing plasma powers of 80 W as well as 120 W respectively, using cold plasma oxygen treated sisal reinforced epoxy laminates. The fibers of the material begin to break out as the bends decrease. The adhesion strength between the fiber and the epoxy is the most critical factor in determining the tensile strength of sisal and epoxy composites. The failure mode depicts the tensile test specimen breaking.

3.5 Single Fibre Pull Out Test

Table [1] compares untreated and cold glow discharge oxygen treated sisal single fibers in terms of average, fiber diameter, breaking load, ultimate tensile strength, and percent elongation. The ultimate tensile strengths for sisal fibers were determined using a tensile test, as seen in the table[1]. After cold glow discharge plasma oxygen treatment of sisal fiber with plasma intensities of 80W(30 min) and 120 W(60 min), the breaking load, tensile strength, and percent elongation break are 4.14 N,72.344 Mpa,12.1 mm, and 3.76N,46.775 Mpa,11.2 mm, respectively (30 min). After plasma treatment, the fiber's accessibility was improved as a result of the formation of fractures and grooves during the plasma treatment [40]. Plasma treatment has a positive impact on textile structures by altering the physical properties of yarns and textiles by forming micro pits on fiber surfaces [40–43].

Table 1

Outcome for cold glow discharge plasma oxygen modification on fiber diameter, breaking load, tensile strength and % elongation for untreated as well as cold glow discharge plasma oxygen modification sisal fibers

Samples	Fiber diameter (μm)	Breaking Load(N)	Tensile Streghth in (MPa)	% Elongation (mm) (50mm Gauge length)
Untreated Sisal fiber	450	3.14	19.753	9
Cold Glow Discharge Plasma Oxygen Treated Sisal fiber 80 W(30 min)	270	4.14	72.344	12.1
Cold Glow Discharge Plasma Oxygen Treated Sisal fiber 120 W (30 min)	320	3.76	46.775	11.2

3.6 Fourier Transform Infrared (FTIR) spectroscopy

"Fourier Transform Infrared (FTIR) spectroscopy" would be a technique for identifying organic components in physiological and industrial applications. Most materials' ability to perform a given function is determined by their chemical characteristics, as measured via Fourier Transform Infrared (FTIR) spectroscopy, enabling the investigation of surface composition and/or interactions at composite material interfaces. [44–47] The FTIR spectrum of untreated sisal fiber has been seen in Fig. 8(a). Inside this image, the characteristic peaks with absorbance bands spanning from 4500 to 400 cm^{-1} were illustrated. Its broad intensive peaks within $4000\text{--}3500\text{ cm}^{-1}$ as well as $2900\text{--}2350\text{ cm}^{-1}$ regions, as well as the medium at $3350\text{--}2900\text{ cm}^{-1}$, were indeed linked to the "valence vibrations for hydrogen-containing C–H and O–H functional groups" found in carbohydrate and alcohol compounds found in untreated sisal fibers, notably cellulose, hemicelluloses, and lignin.[44–47]. The "stretching vibrations inside the C = O and C = C groups of aromatic hydrocarbons," as well as ketone and aldehyde molecules, have been accompanied with strong bands spanning from 2350 to 1750 cm^{-1} , 1750 to 1650 cm^{-1} , and 1650 to 1450 cm^{-1} . Even though cellulose is ultimately accountable for aldehyde compound vibrations [44], all such peaks have been interconnected to ketone compounds found throughout hemicelluloses and aromatics located in lignin, which have higher reflectance intensities in these wave number areas than cellulose-related compounds [44–47]. Untreated sisal fiber has a bigger intensity peak with sections of $2300\text{--}1650\text{ cm}^{-1}$ as well as $1750\text{--}1650\text{ cm}^{-1}$ coupled towards the prevalence for additional hemicelluloses, lignin components, as well as other components. Although cellulose creates aldehyde compound vibrations [47], analogous peaks have been attributed to ketone compounds found within hemicelluloses and aromatics found in lignin, which have stronger reflectance intensities in these wave number domains with cellulose-related compounds [44–47]. Untreated Sisal fiber seems to have a greater intensity peak comprising areas of $2300\text{--}1650\text{ cm}^{-1}$ and $1750\text{--}1650\text{ cm}^{-1}$ due to the combination of more hemicelluloses, lignin components, and other components. The C–C and C–O looked in phenol, alcohol, and aromatic compounds induce the sequence of maxima registered with fingerprint region between 1450 and 400 cm^{-1} , with lignin having the highest intensities inside the range $1400\text{--}1200\text{ cm}^{-1}$ and cellulose and hemicellulose getting one of the highest intensities between 1200 and 900 cm^{-1} [44–47]. The FTIR spectra of sisal fiber throughout plasma treatment have been represented in Fig. 8(b),8 (c). The impact of cold plasma oxygen with 80 W for 30 minutes as well as 120 W for 30 minutes on sisal fibers was substantial. The "C-H bending vibration for hydrocarbon clusters," which seems to be a distinctive attribute among both lactic and glycolic acids, is represented by the spectra at 3050 and 2840 cm^{-1} . There is indeed a significant decline in intensity and peak heights between 3050 and 2840 cm^{-1} , "corresponding to CH₂ and CH₃ in lactic and glycolic acids." "Absorption bands of both the carbonyl (C = O) group, which seems to be a hallmark of cellulose and hemicellulose, create an amplitude of around $1675\text{--}1600\text{ cm}^{-1}$. Following cold glow discharge plasma oxygen treatment, the amplitude and apex height surrounding the" carbonyl group C = O" reduced, revealing that plasma treatment promoted cellulose crystallinity. Throughout all spectra, the absorption peaks at 1605 and 1600 cm^{-1} , $1515\text{--}1505\text{ cm}^{-1}$, as well as $1515\text{--}1505\text{ cm}^{-1}$ revealed entirely due to aromatic ring vibration, including the amplitude and apex height encircling completely diminishing during cold glow discharge oxygen plasma treatment.

In all absorption spectra, the absorption peak across $1430 - 1425\text{ cm}^{-1}$ seemed to be fully accountable for CH₂ deformation in lignin. Absorption spectra for the plasma treatment at 80 W (30 min) seemed to have slightly increased absorbance intensity, even as absorption spectra for the plasma treatment at 120 W would have slightly decreased absorbance intensity (30 min). The peaks at 1330 and 1325 cm^{-1} might be exacerbated via syringyl ring breathing in lignin. For celluloses and hemicelluloses, the C-O, C-C asymmetric vibration is responsible for the peak near $1230 - 1220\text{ cm}^{-1}$. Except for a maximum of approximately 1235 cm^{-1} adhering to C-O, C-C was most pronounced in cold glow discharge plasma oxygen treatment of sisal fiber attributable to cellulose oxidation. Once the characteristics of plasma treatment parameters were enhanced, the decrease in aromatic and ketone derivatives correlated with bulk lignocellulosic absorption through fibers was primarily attributable [45–46]. "Two distinctive bands for C-C, C-O stretch as well as C-H, C-O deformations vibration at $1230 - 1220\text{ cm}^{-1}$ as well as $1085 - 1030\text{ cm}^{-1}$, correspondingly, which appear to be typical of cellulose and lignin." Their lack of a noticeable peak around 1050 cm^{-1} represents whether cellulose had indeed been absorbed on the treated fiber's surface"[46]. The cellulose crystalline structural characteristics have been attributed to the band at $1420 - 1430\text{ cm}^{-1}$, whereas the amorphous region has already been linked to the spectrum around 898 cm^{-1} [45–46]. Alteration in this fingerprint area can be detected, revealing that the fiber composition has improved. It thus seemed to be strong evidence that plasma treatment alters fiber shape, as well as the XRD analysis corroborated this. Table 2 displays that sisal fiber 120 W (30 min) cold glow discharge plasma oxygen treated fibers had the greatest "Total Crystallinity Index and Lateral Order Index values" (0.918,1.246). Untreated sisal fiber had the lowest "Total Crystallinity Index and Lateral Order Index values" (1.008,0.983), implying that its cellulose is made up of more amorphous domains. It's possible to detect changes inside this fingerprint region, signaling that the fiber composition has improved.

Table 2

IR crystallinity ratio of untreated sisal fiber, Cold glow discharge plasma oxygen treated sisal fiber 80 W (30 Min), and cold glow discharge plasma oxygen treated sisal fiber 120 W (30 Min)

Sample	IR Crystallinity Ratio	
	Total Crystallinity Index [48–50]	Lateral order Index[47]
Untreated Sisal Fiber	1.008	0.983
Cold glow discharge plasma oxygen treated Sisal fiber 80 W (30 Min)	0.956	1.05
Cold glow discharge plasma oxygen treated Sisal fiber 120 W (30 Min)	0.966	1.031

3.7 X-ray diffractograms (XRD)

Figure 9 shows "X-ray powder diffraction patterns" for untreated sisal fibers and cold glow discharge plasma oxygen-treated sisal fibers at 80 W (30 min) and 120 W power levels (30 min). The spectra of untreated sisal fiber have a significant peak at 2θ values at 18.87° and a very strong peak at 22.638° ,

which correspond to 101 and 002 planes[51]. A little peak can be seen in the graph, which could be due to contamination or inorganic substance in the fiber. A strong peak at 2θ values at 18.710° , 18.725° , and a very strong peak at 22.609° , 22.624° respectively, which correspond to 101, and 002 planes, was seen after cold glow discharge plasma oxygen application for sisal fiber with plasma intensities of 80W(30 min), 120 W(30 min). The % Crystallinity, Crystallinity Index and amorphicity index values of sisal fiber 80 W (30 min) treated with cold plasma oxygen were the highest values(0.651,46.52% and 0.534) as shown in table [3] increased value of % Crystallinity, Crystallinity Index, and amorphicity index due to the surface of crystallites corresponding to shrank of amorphous cellulose sections with increasing size of crystallite, crystallinity index increased [52].

Table 3

XRD analysis of untreated sisal fiber, Cold glow discharge plasma oxygen treated sisal fiber 80 W (30 Min), and cold glow discharge plasma oxygen treated sisal fiber 120 W (30 Min)

Fibers	Crystallinity Index (%) Proposed by Segal et.al. [53–56]		
	% Crystallinity	Crystallinity Index Cr.I.(%)	Amorphicity Index
Untreated Sisal Fiber	0.6105	36.221%	0.6377
Cold glow discharge plasma oxygen treated sisal fiber 80 W (30 Min)	0.651	46.52%	0.534
Cold glow discharge plasma oxygen treated sisal fiber 120 W (30 Min)	0.643	44.56%	0.554

4. Conclusion

The tensile, % elongation, ILSS, flexural, force-extension curve, single fiber tensile test, FTIR, as well as XRD diffractogram attributes of sisal fiber reinforced epoxy laminates seemed to be significantly improved, implying that cold glow discharge oxygen plasma treatment with sisal fibers had always been responsible. The following seem to be the outcomes of the investigations:

- The ultimate tensile strength and % Elongation break for sisal fiber-reinforced epoxy laminate enhanced to 165.34 MPa,5.61 mm, and 142.91 MPa,5.12 mm, respectively, during cold glow discharge plasma oxygen treatment employing plasma intensities of 80 W (30 min) and 120 W.This corresponds to grows of 58.56%, 76.41%, and 37.05%, 61.0%, respectively.
- The flexural strength of the sisal fiber-reinforced epoxy laminate increased to 89.34 MPa at 30 percent fiber volume after 30 minutes of treatment with a cold glow discharge plasma power of 80 W.Cold glow discharge plasma oxygen treated sisal fiber-reinforced epoxy laminates with plasma intensities of 80 W(30 min) and 120 W(30 min) enhance flexural strength characteristics by 56.94% and 50.76%, respectively since compared to untreated sisal fiber reinforced epoxy.
- When compared to untreated sisal fiber reinforced epoxy composites, interlaminar shear strength values for cold glow discharge plasma oxygen modified sisal fiber reinforced epoxy composites

utilizing plasma intensities of 80 W(30 min) and 120 W(30 min) enhance significantly 76.92% and 70.99%, respectively.

- The force-extension behavior of the sisal fiber-reinforced epoxy laminate was significantly enhanced once it was treated using cold glow discharge plasma oxygen. Cold plasma oxygen treated sisal fiber reinforced epoxy composites had rising force(N) values of 58.56% and 37.05%, respectively, compared to untreated sisal fiber-reinforced epoxy laminate at plasma intensities of 80 W (30 min.) and 120 W (30 min). (30 min).
- To see how cold glow discharge oxygen plasma pretreatment of sisal fiber modifies the fiber strength stated. The breaking load, tensile strength, and percent elongation break are 4.14 N,72.344 Mpa,12.1 mm, and 3.76 N,46.775 Mpa,11.2 mm, respectively, during cold glow discharge plasma oxygen treatment for sisal fiber with plasma intensities of 80 W (30 min) as well as 120 W (30 min)
- The greatest values for the "Total Crystallinity Index" as well as "Lateral Order Index" for sisal fiber 80 W (30 min) treated with cold plasma oxygen were 0.956 and 1.05, respectively.
- The percent Crystallinity, Crystallinity Index, and Amorphicity Index values of sisal fiber 80 W (30 min) treated with cold plasma oxygen were 0.651, 46.52%, and 0.534, respectively, revealing one in which hemicellulose and lignin had been reduced significantly after cold glow discharge plasma oxygen treatment on sisal fiber.

Declarations

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Authors' Contributions Not applicable

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Data Availability

The datasets supporting the conclusions of this article are included within the article and its additional files(ie. Tables,Line Figures)

Compliance with Ethical Standards

Conflict of Interest

The authors whose names are listed have NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional

relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

Ethics Approval

Not applicable.

Consent to Participate

Not applicable.

Consent for Publication

Not applicable.

Code Availability

Not applicable.

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Figures



Figure 1

Experimental set up for Producing Glow Discharge Plasma

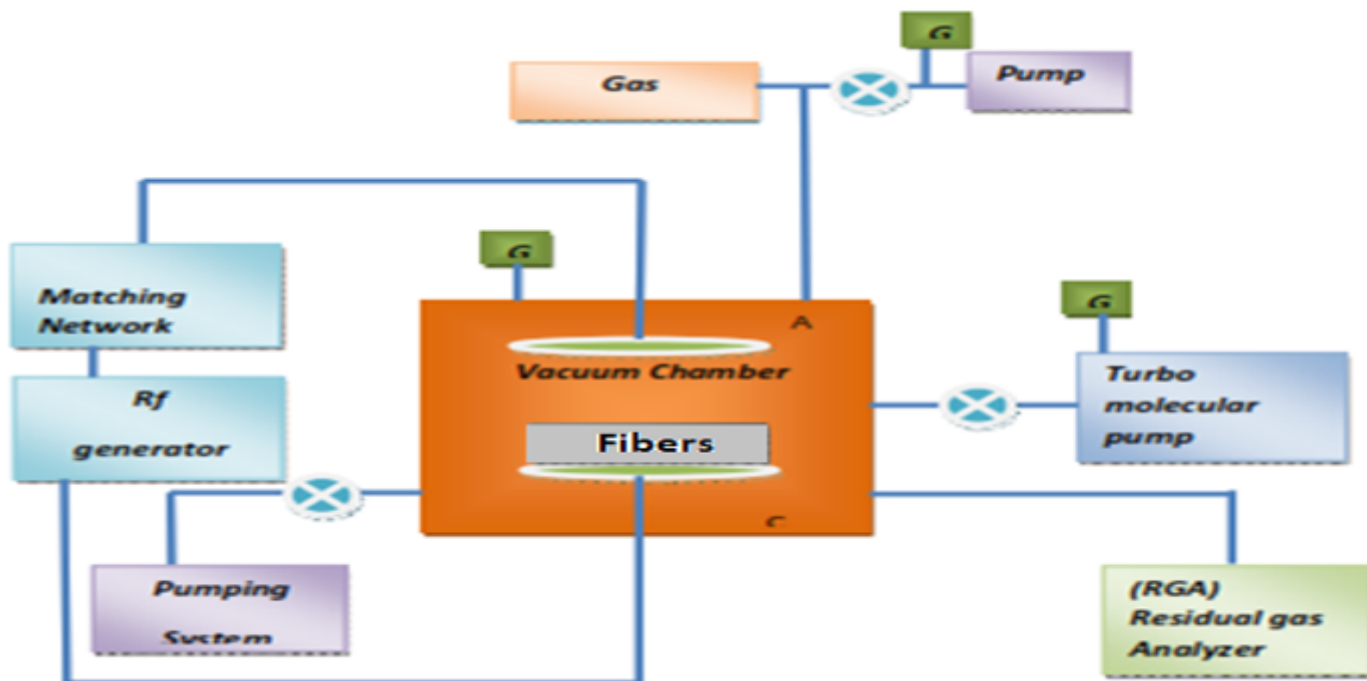


Figure 2

Flowchart for Producing Cold Glow Discharge Plasma Modification

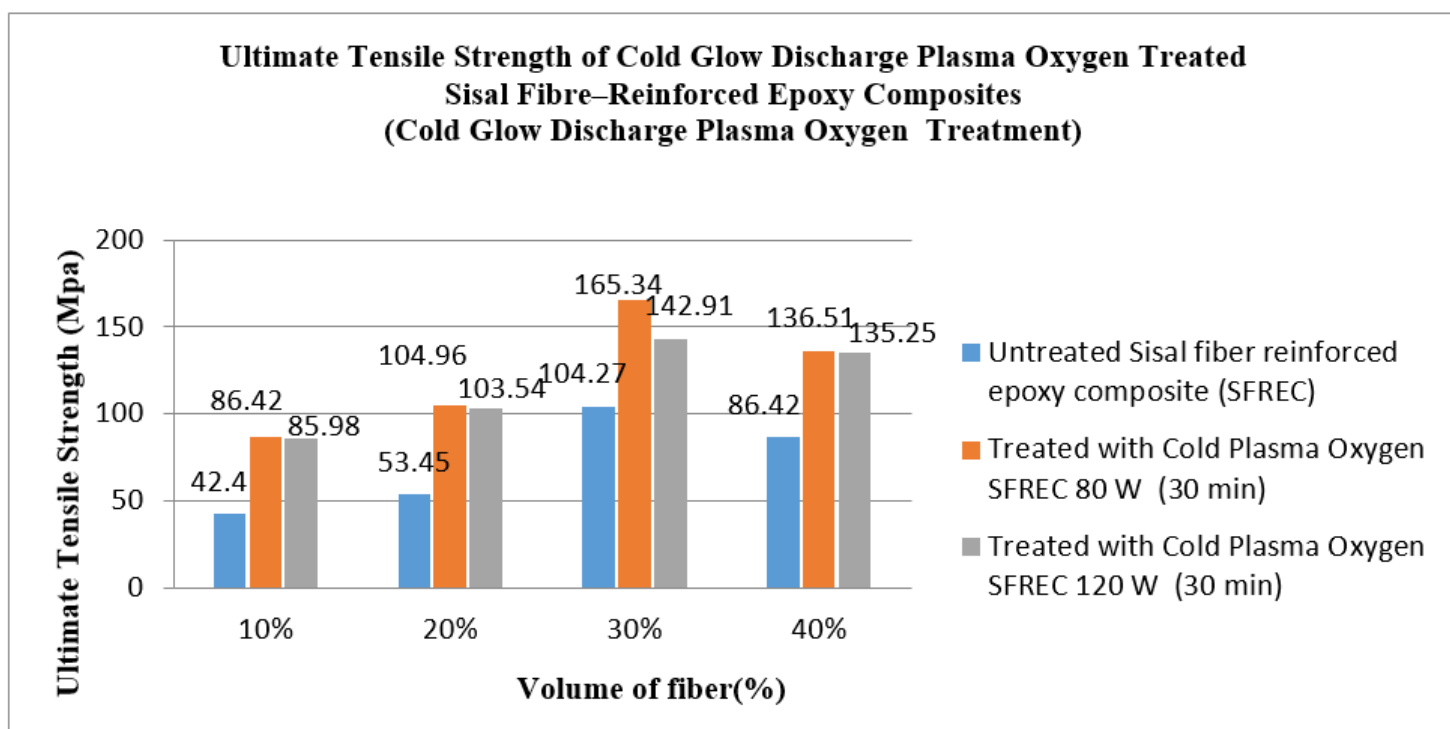


Figure 3

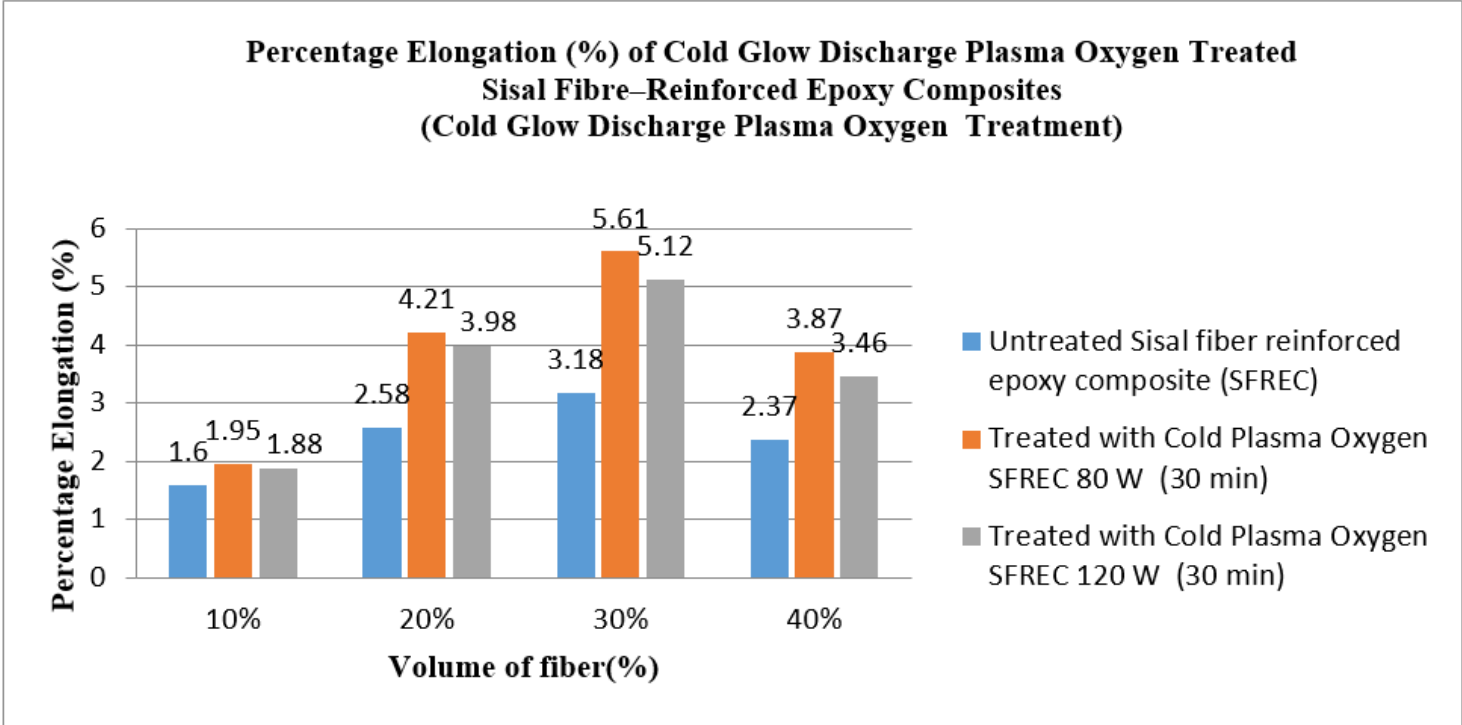


Figure 4

Impact of cold glow discharge plasma oxygen treatment on the % elongation sisal fiber-reinforced epoxy laminates

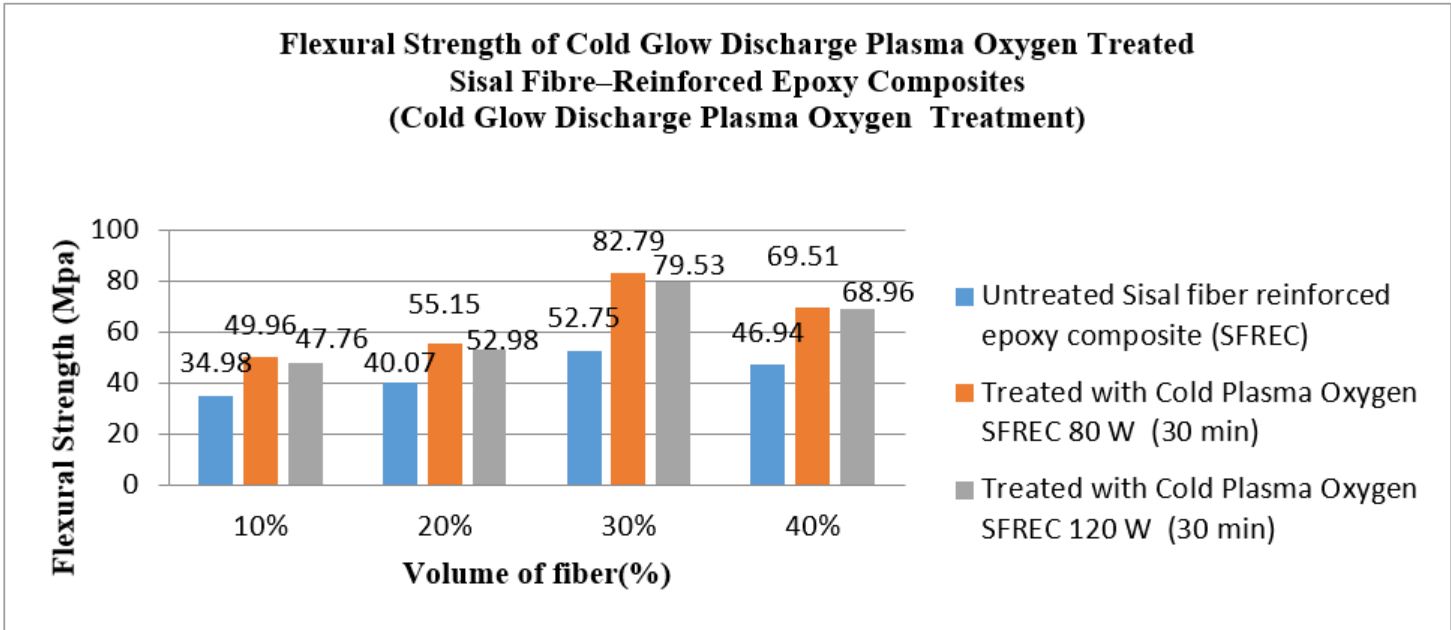


Figure 5

Impact of cold glow discharge plasma oxygen treatment on the flexural strengths of untreated as well as cold glow discharge of plasma-treated sisal fiber -reinforced epoxy composites

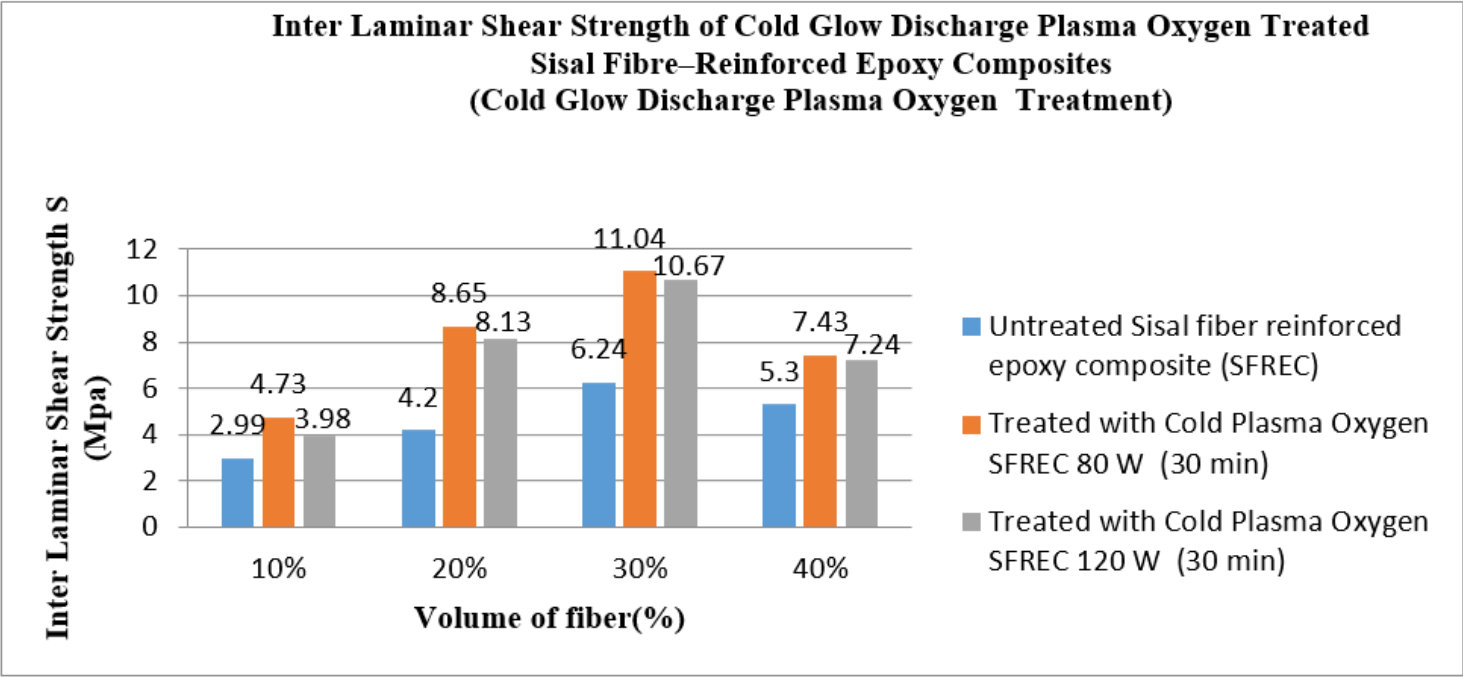


Figure 6

Impact of cold glow discharge plasma oxygen treatment on the ILSS strengths for untreated as well as cold glow discharge of plasma-modification sisal fiber -reinforced epoxy composites

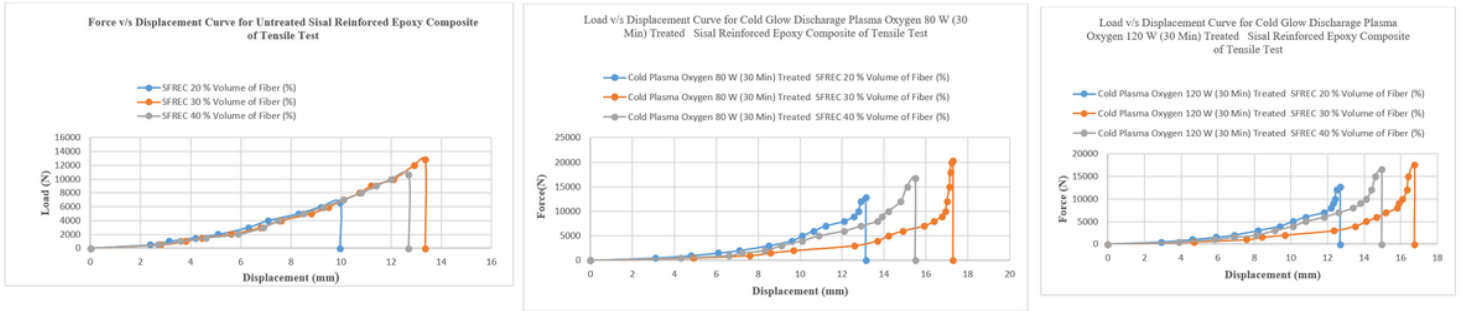


Figure 7

- (a).The force-displacement curve for Sisal fiber -reinforced epoxy composites
- (b). Impact of cold glow discharge plasma oxygen treatment on the Force- Displacement Curve for cold glow discharge of plasma oxygen treated sisal fiber -reinforced epoxy composites
- (c). Impact of cold glow discharge plasma oxygen treatment on the Force- Displacement Curve for cold glow discharge of plasma oxygen treated sisal fiber -reinforced epoxy composites

Figure 8

(a). FTIR spectra for untreated sisal fiber

(b). FTIR spectra for cold glow discharge plasma oxygen modification sisal fiber 80 W (30 min)

(c). FTIR spectra for cold glow discharge plasma oxygen modification sisal fiber 120 W (30 min)

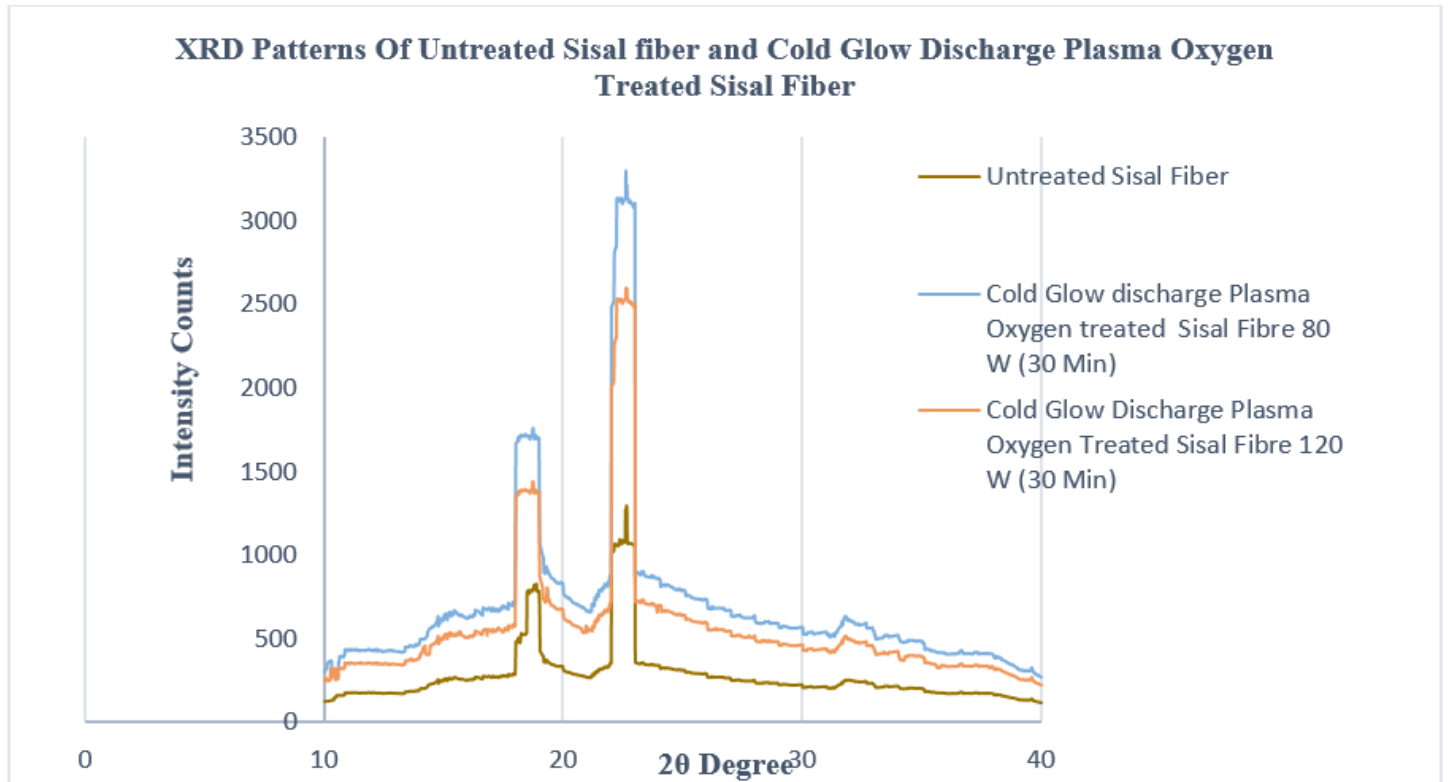


Figure 9

XRD diffractogram of untreated sisal fiber, Cold glow discharge plasma oxygen treated sisal fiber 80 W (30 Min), and cold glow discharge plasma oxygen treated sisal fiber 120 W (30 Min)