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Structural, Mechanical and Electrical Properties of Nelumbo Nucifera Fibers

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Abstract:

In view of exploring the structural and elasto-mechanical properties of the Nelumbo Nucifera (lotus) fiber cultivated in India, the Powder X-ray diffraction data (PXRD) of this fiber is utilized. The PXRD data was used along with Peakfit® software for the background data correction and to simulate the data. Checkcell® has been utilized to obtain the microstructural and lattice parameters. Our sample belong to monoclinic crystal system and lattice parameters are $a=7.784 \text{ \AA}$, $b=8.201 \text{ \AA}$, $C=10.380 \text{ \AA}$ and $\beta=96.28^\circ$. Using Debye Scherrer equation, the crystallite sizes along all the Bragg reflections were calculated and employing first principle calculations, the crystallite size has been plotted and concluded that, the size is not ellipsoidal as predicted by earlier researchers, in specific it is multidimensional in shape and depends on the existence of ordered region. The X-ray diffraction data is used along with Treloar's principle to obtain the 6×6 elastic tensor matrix, and the same has been plotted using Elastic Tensor Analysis tool. This result has shown that the lotus fiber is anisotropic in nature. The dependence of frequency and temperature on the dielectric properties of Nelumbo Nucifera fibres were studied in the frequency range 50 Hz to 5 MHz and in the temperature range 27°C to 100°C . Functional Data Analysis has been utilized to obtain the correlation among various microstructural parameters.

Keywords: Lotus fibre, XRD, ELATE, Crystallite size, FDA, Dielectric studies.

1. Introduction:

Natural plant fibres, acknowledged for their renewability and eco-friendly characteristics, have attracted substantial public interest with regard to health, biomedical and environmental concerns (Ramesh et al. 2017). Due to the escalating global environmental concerns, there is a substantial rise in demand for eco-friendly, fully biodegradable, sustainable composites across various industries, including packaging, textiles, bundling, medical sectors, and more (Andrew and Dhakal 2022). Composite materials that are entirely biodegradable, crafted from natural fibres and biodegradable resins, distinguish themselves as sustainable materials that depend on renewable resources. The use of natural fibre has been started in 6th and 7th centuries BCE (Rowell 2008; Andrew and Dhakal 2022). Hemp is also one of the natural fibres, and people started to cultivate and use this from 4500 BCE (Crini et al. 2020). The usage of cotton for the textile industry started

in 3000 BCE in India. The manufacture of silk and its products has originated around 2640 BCE in China (Chandan 2017). In 18th and 19th century the revolution in the industry has encouraged the further processing in the natural fibres which lead to tremendous upgrade in fibre cultivation (Chandan 2017).

Mengxi Wu, et al., (Wu et al. 2014) manufactured a green composite fibre employing green lotus fibre. The results on tensile strength showed almost comparable strength with the flax fibre and greater than cocoon and other natural fibres. Manju V V et al., (V V et al. 2022) has studied natural cotton fibre using its XRD data. The structural properties such as shape of crystallite size, crystal structure has been reported. The elasto-mechanical properties and its shape has been plotted using tensor analysis tool called ELATE. The functional data analysis (FDA) has employed to obtain the structure property relation. N V Bhat et al., (Bhat et al. 2011) have investigated the suitability of barometrical plasma treatment over crude cotton texture surfaces as an elective technique for supplanting the wet material pre-treatment processes. The results revealed that, hydrophilicity and the wickability has increased significantly after this treatment. Cheng et al., (Cheng et al. 2018) have studied the tensile strength and breaking elongation of lotus fibre after treating lotus fibers with sodium chloride. The result revealed that the concentration of NaCl is inversely proportional to their strength. The crystallinity index, crystallinity and regain of moisture content has improved after treating with NaCl. Qingyang Zhang et al., (Zhang et al. 2022) have studied the elastic properties of lotus fibre and revealed that, these can possess a high modulus upto 11.2 to 11.7 GPa and the hardness upto 0.41 to 0.57 GPa. All these studies are reported on improving the strength and applicability of fibres. Very few studies are reported on structural and elastic properties of fibres. Considering this, the X-ray diffraction data of lotus fibre has been employed in the course of the ensuing the following investigations.

- To compute the crystallite size of lotus fibre employing XRD data along with Debye Scherrer formula (Lele and Anantharaman 1966).
- Using first principle calculations, to obtain the shape of crystallite size and to plot this along crystallographic axes.
- Employing Treloar's principle [18], to compute the 6×6 tensor matrix for a monoclinic crystal system and utilize the same to obtain elastic moduli. These elastic moduli values will be incorporated into the Elastic Tensor Analysis tool (ELATE) to chart the spatial variation of elastic moduli. These three-dimensional variations in elastic moduli gives valuable insights into comprehending the inherent anisotropy of material.

- Examine the impact of frequency and temperature on the dielectric properties of Nelumbo Nucifera fibers within the frequency range of 50 Hz to 5 MHz and at temperatures ranging from 27°C to 100°C.
- Employing a novel technique called Functional Data Analysis (FDA) (Urs et al. 2016), the correlation among microstructural parameter along with various crystal reflections were identified and plotted.

2. Collection of Sample

The naturally cultivated lotus fibre is plucked directly from the nearby lake of south Asia. Petiole of the lotus is cleaned physically using mineral water. Then petiole has been cut into small bundles and minuscule amount of thin sticky fibres were extracted. These extracted fibres were rolled together and dried before taking it to the characterization. This lotus fibre has been employed for the study without adding any extra chemical ingredients.

3. Experimental Technique

3.1 X-ray Diffraction Studies

Utilizing X-ray diffraction, a sophisticated technique for studying material structural properties (Ali et al. 2022), small bundles of lotus fiber were extracted for this investigation. These fibers were compressed using a pellet maker and affixed to a sample holder, which was then connected to a goniometer to ensure the X-ray beam paralleled the detector. Employing a Cu target with a wavelength of 1.54 Å, the XRD machine operated at 30 mA and 45 kV. Scanning occurred from 10° to 50° at a rate of 0.02° per minute. The obtained Weissenberg geometry (Fields et al. 1992) is depicted in Figure 1. Adjustments for X-ray intensities (I_0) were made using polarization (P) and Lorentz (L) factors, following the relation proposed by (Buerger 1940).

$$LP = \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} - (1)$$

Lotus fibre, comprising a significant 68% cellulose (Chen et al. 2012), was analysed using X-ray diffraction (XRD). Six reflections were identified, and their accurate and precise resolution was achieved through the residual peak fitting procedure facilitated by the Peakfit® software (Singh et al. 2011). The obtained XRD data is as shown in Figure 1.

3.2 Dielectric Studies

Lotus fibers were compacted into cylindrical pellets (3 mm radius, ~1 mm thickness) using a 10-ton load. An NF LCR meter (Model: ZM-2376, Make: Japan) analysed AC conductivity,

dielectric properties, impedance, and electric modulus. Testing spanned temperatures of 27°C, 50°C, 75°C, and 100°C, and frequencies from 50 Hz to 5 MHz.

4. Results and Discussion

4.1 Computation of shape of crystallite size

The Powder XRD data, comprising 2θ and intensity information, was processed using Peakfit® and the Gaussian deconvolution strategy to simulate the obtained profile. This software effectively isolates overlapping peaks and provides advanced data manipulation features, enabling the separation of six reflections for the considered fiber. The Checkcell® software (Manju et al. 2023b), utilizing the cell parameters and 2θ values from Peakfit®, calculated Miller indices (hkl) for all observed reflections. XRD data facilitated the evaluation of peak broadening concerning crystallite size and lattice strain due to inherent dislocation.

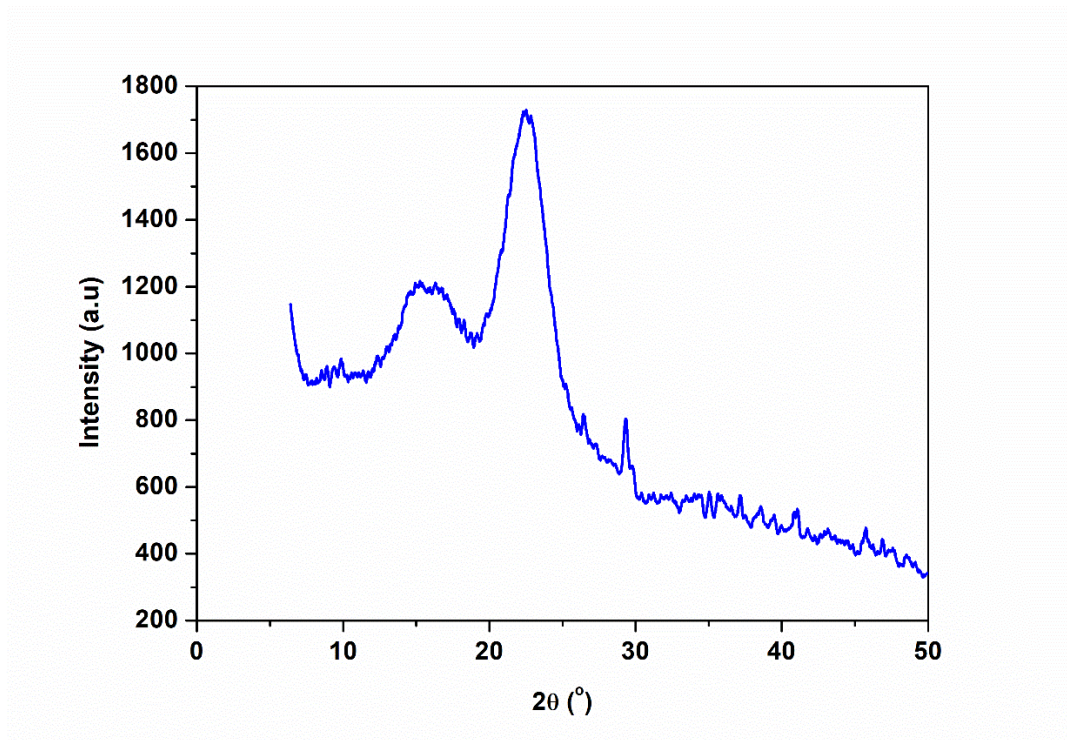


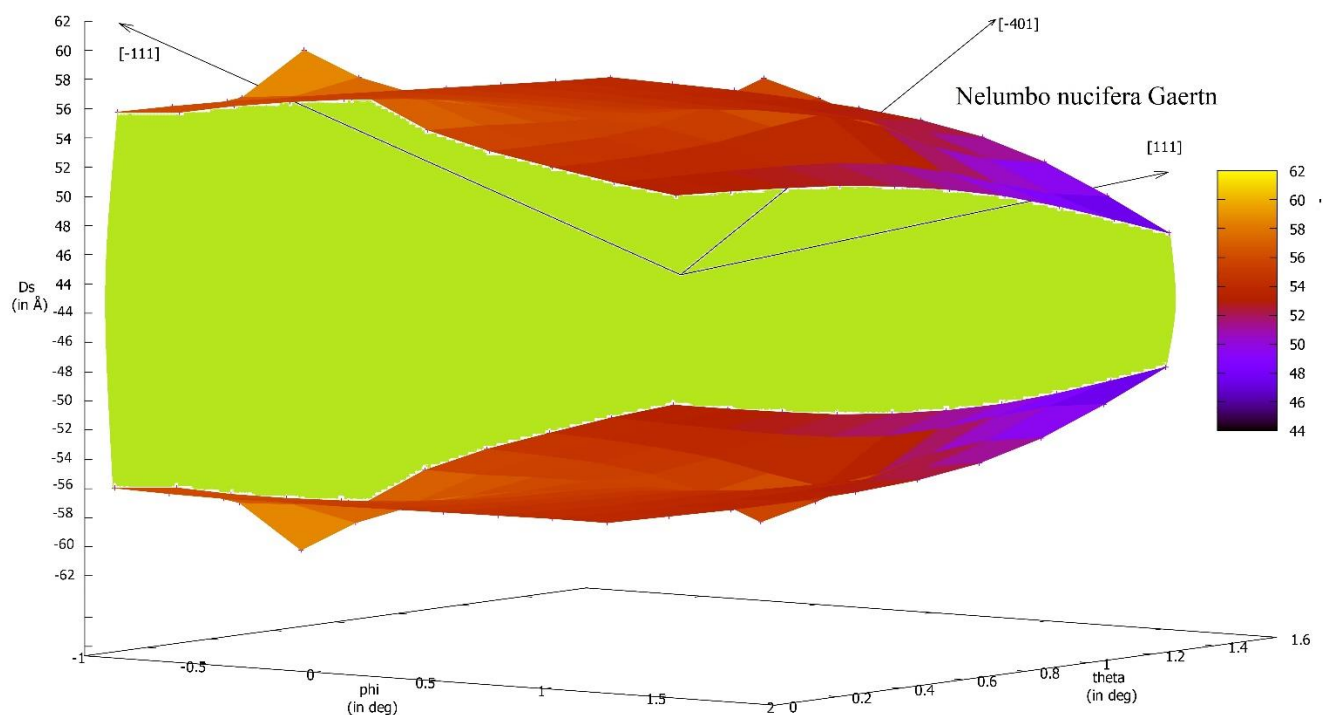
Figure 1: The XRD data 2θ vs Intensity

Crystallite size for all reflections was calculated using the Scherrer formula,

$$D_s = \frac{k\lambda}{\beta \cos\theta} \text{Å} \quad (2)$$

where D_s is the crystallite size in Å, k is the Scherrer constant (taken as 0.9 for spherical crystals), β is the peak width at half maximum intensity, and θ is the peak position.

The obtained average crystallite size values for the considered sample are detailed in Table 1. Using the obtained cell parameters, crystallite size (D_s) values and simple spherical coordinate properties (θ and ϕ) were determined for Bragg's reflection directions. A FORTRAN program, operating on a Linux-based system, was employed to assess spherical coordinate representation for a given set of reflection (hkl) and size values. Surface plots for the parameters θ , ϕ , and D_s were created using Gnuplot (Janert 2009), showcasing the obtained crystallite size shape for Lotus fibre. The variation in crystallite size values along crystallographic axes reveal the presence of anisotropy in the fiber. This characteristic is influenced by factors such as the location of fibre growth, the composition of minerals, and the surrounding environment.



The microstructural parameters obtained, including crystallite size, interplanar spacing, and Miller indices, are presented in the table 1.

Table 1: Microstructural Parameters of Lotus fibre.

2θ (in deg)	Miller indices (hkl)	Crystallite size (D_s in Å)	Lattice strain (G in %)	Interplanar spacing (d in Å)
13.6	-101	28.1	42.6	6.6
15.4	-110	55.9	18.9	5.8
17.6	101	69.6	13.3	5.1
20.4	111	60.3	13.3	4.4
22.6	020	62.3	11.6	4.0
24.7	-201	39.7	16.6	3.6

4.2 Elasto-Mechanical Characterization: Treloar's Principle and Elastic Tensor Analysis tool (ELATE)

Strength, a fundamental quality crucial for yarn in the textile industry, is a direct indicator of its overall quality (Wang et al. 2020). The mechanical response of fiber materials to applied forces and deformations plays a pivotal role in their performance. Understanding the elasto-mechanical characteristics of fiber materials is essential for their successful utilization in industrial applications. The elastic tensor, encompassing components like XX, YY, ZZ, XY, XZ, ZY, exhibits symmetry (XY=YX, XZ=ZX, YZ=ZY), providing three-dimensional insights into the fiber's strength. This tensor, with components like modulus of elasticity, contributes to a comprehensive understanding of the material's response to external stresses within the elastic limit. Consequently, Elastic Tensor Analysis is employed to calculate these elastic properties. The modulus of elasticity significantly influences the structure and morphology of fiber materials (Alves Fidelis et al. 2013). The number of elastic constants is contingent on the system's symmetry, with monoclinic unit cells possessing 13 independent elastic constants. These constants are expressible as:

$$C_{ij} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & - & C_{15} & - \\ C_{21} & C_{22} & C_{23} & - & C_{25} & - \\ C_{31} & C_{32} & C_{33} & - & C_{35} & - \\ - & - & - & C_{44} & - & C_{46} \\ C_{51} & C_{52} & C_{53} & - & C_{55} & - \\ - & - & - & C_{64} & - & C_{66} \end{bmatrix}$$

Our earlier study (Manju et al. 2023a) extensively outlines the procedure of calculating elastic constants for a monoclinic unit cell. In this context, the elastic constant matrix $|C_{ij}|$ is derived by reducing 81 independent constants to 21, and subsequently to 13, specifically for a monoclinic unit cell. Treloar's principle (Treloar 1960) is employed in computing these elastic constants, guided by the following relations:

$$C_{22} = C'_{22} + C''_{22} + C'''_{22} \quad (3)$$

where C'_{22} is the primary bond, C''_{22} is the inter-chain bond and C'''_{22} is the intra-chain bond. According to Treloar (Treloar 1960),

$$C'_{22} = CL^2/V \quad (4)$$

The determined elastic constants for a monoclinic crystal system were integrated into the ELATE program (Gaillac et al. 2016). Utilizing the Voigt principle, elastic moduli such as Young's modulus (E), shear modulus (G), Poisson's ratio (ν), and linear compressibility (β) were computed. These

elastic moduli were then graphed in 2 and 3 dimensions, illustrating their spatial directions in a mechanically stable system. ELATE requires a 2nd order 6×6 symmetric matrix (C_{ij}) in GPa as input. Through the open-source Elastic Tensor Analysis tool (ELATE), mechanical properties were calculated for all considered samples, and the results are detailed in Table 2. The 3-dimensional elastic moduli obtained along their spatial axes are depicted in the Figure 2 and Figure 3. Observing the variation in Young's modulus, rigidity modulus, Poisson's ratio, and linear compressibility, one can discern changes in values, indicating the material exhibits an inhomogeneous behaviour. This, in turn, signifies the presence of anisotropy in the material.

Table 2: Elastic properties of Lotus fibre

Parameters	Young's modulus (in GPa)		Linear Compressibility (in TPa ⁻¹)		Shear modulus (in GPa)		Poisson's ratio	
	$E_{\min}$	$E_{\max}$	$\beta_{\min}$	$\beta_{\max}$	$G_{\min}$	$G_{\max}$	$\nu_{\min}$	$\nu_{\max}$
Variation	3.9	127.6	8.7	260.2	2.3	39.9	-0.42	0.72
Anisotropy	32.18		29.74		17.07		∞	

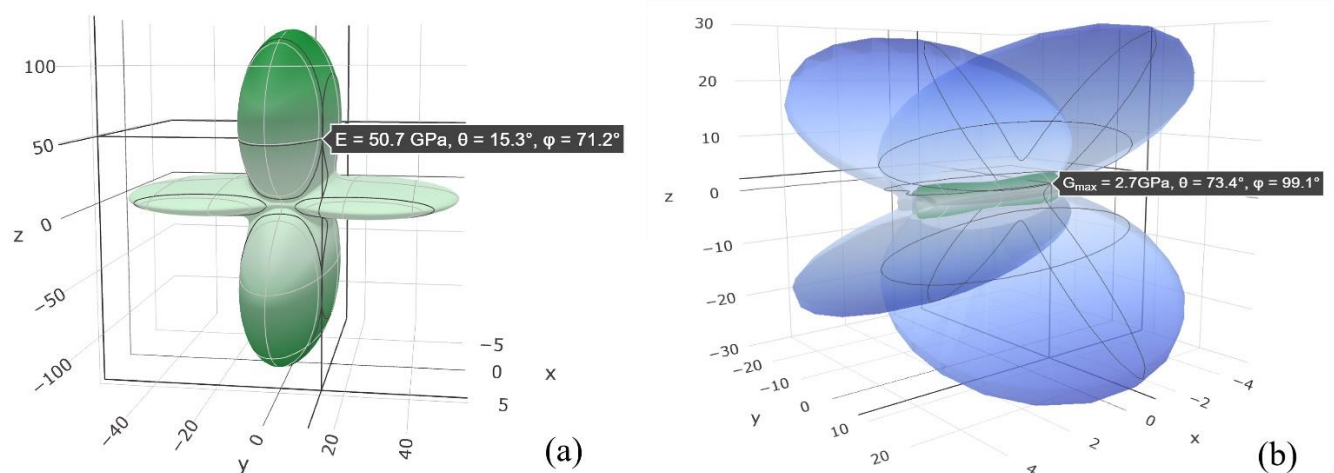


Figure 2: Spatial Distribution of (a) Young's Modulus and Rigidity Modulus (b)

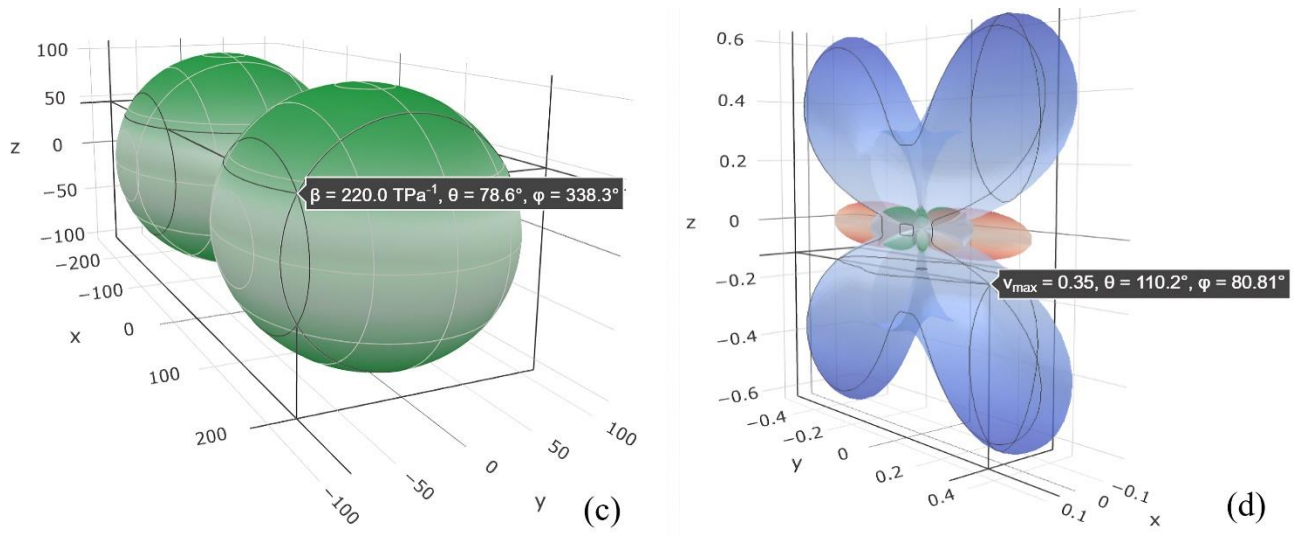


Figure 3: Spatial Distribution of (c) Linear Compressibility (d) and Poisson's ratio

Figure 2 (a) indicates the orientation-dependent behaviour of Young's modulus. Figure 2 (b) depicts the variation of rigidity modulus along their crystallographic axes, it shows the deviation of its shape from spherical, indicating the anisotropic behaviour. This phenomenon of anisotropy involves distinct physical characteristics appearing in different directions and is widely applied in diverse applications of the textile industry (Tokarska and Gniotek 2015). Figure 3 (c) and (d) depicts the variation of linear compressibility and Poisson's ratio in their spatial axes. The linear compressibility is the of material's susceptibility to linear compression or contraction when subjected to external forces and it is angular dependent parameter. The (B/G) ratio gives 2.11 which is higher than the index value (Hegde et al. 2022) to define ductile in nature, this brittle property favours in retaining typical higher mechanical strength and maintains stability at higher temperature.

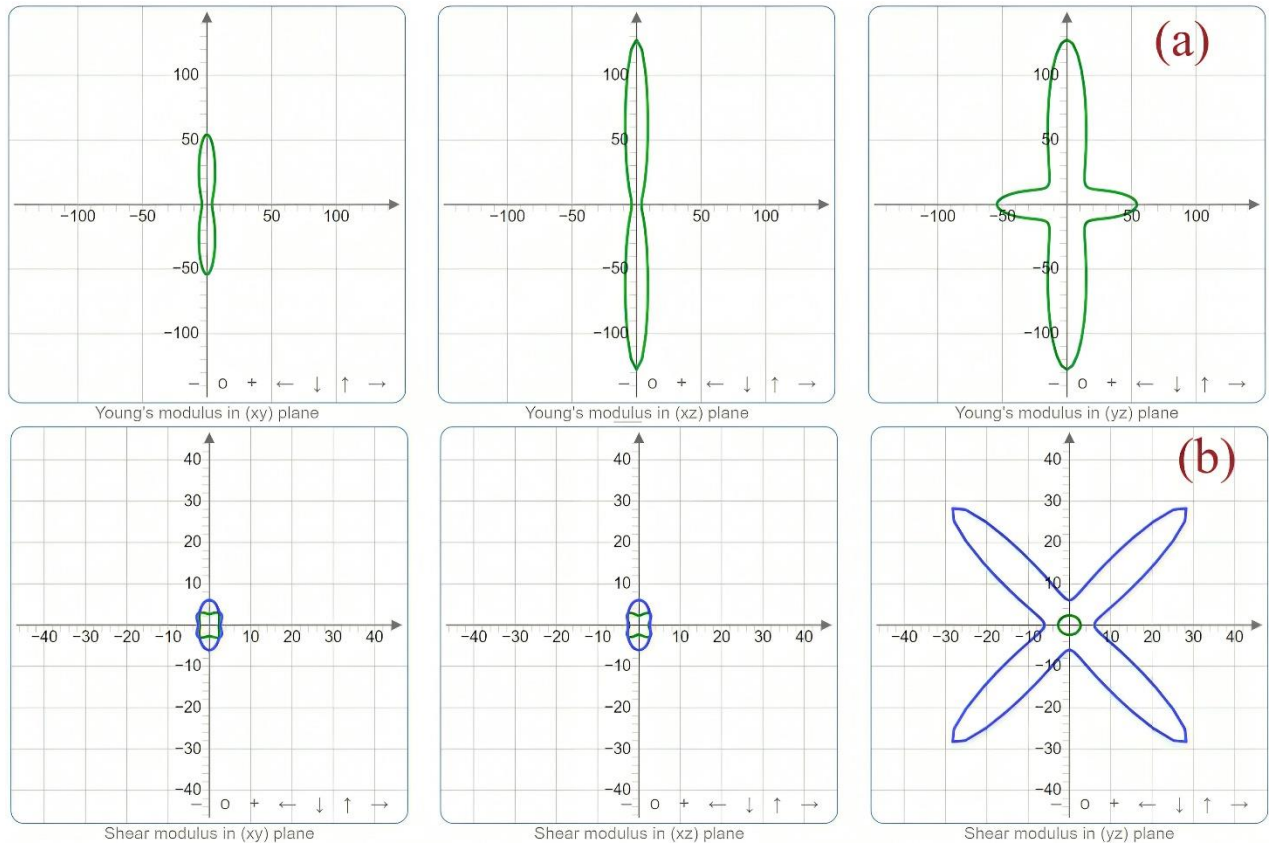


Figure 4: Variation of (a) Young's modulus and (b) shear modulus in 2D.

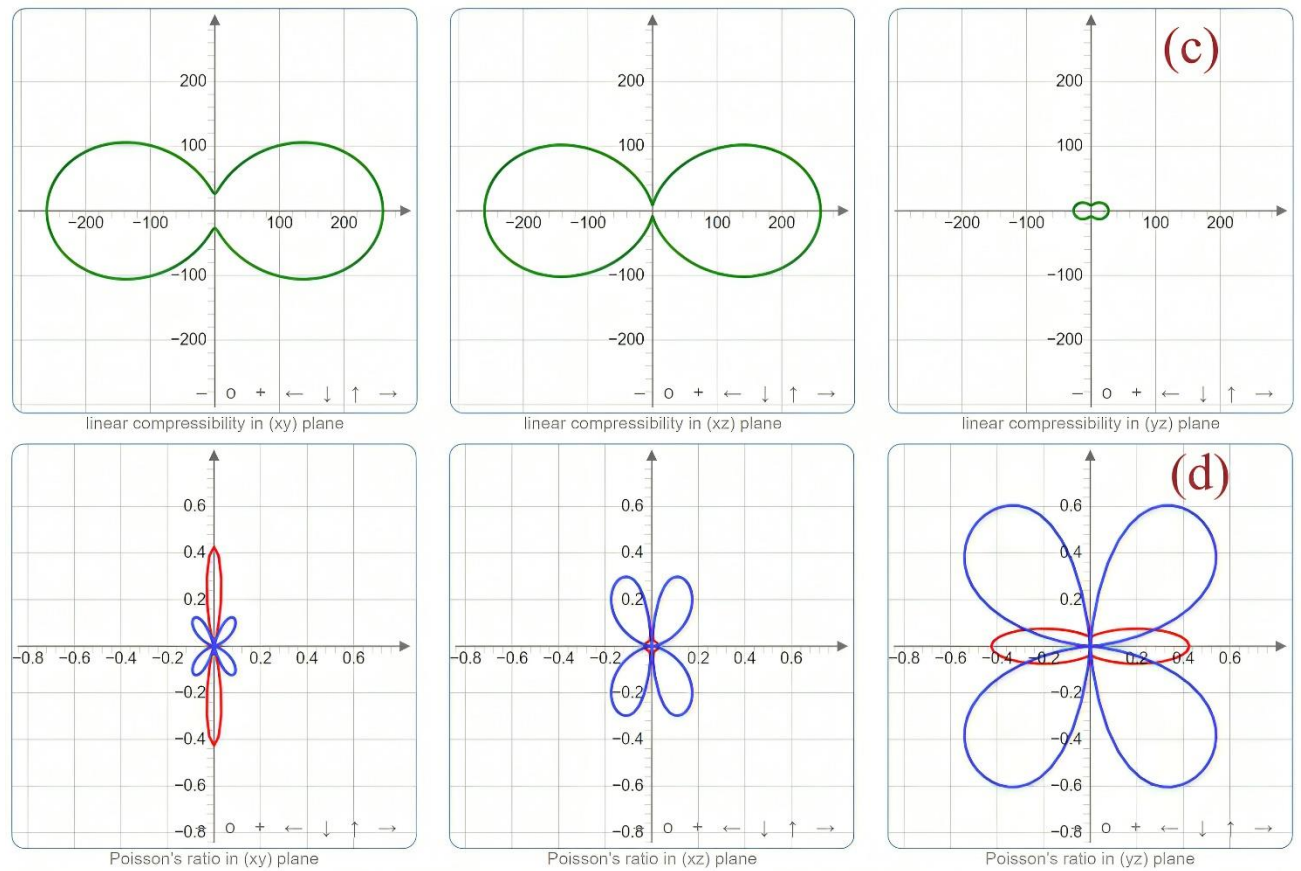


Figure 5: Variation of (c) linear compressibility and (d) Poisson's ratio

This indicates that, the lotus fibre is the main constituent of cellulose, has a fibrous and brittle in nature. Although it has the capacity to absorb moisture and undergo a degree of stretching, this fibre lacks the plasticity and malleability commonly found in ductile materials. The Figure 2 (d) is the variation of Poisson's ration along spatial direction. This shows that Poisson's ration is also anisotropic in nature. The predicted anisotropic values are tabulated in Table 2. Figure 4 and Figure 5 indicates the variation of elastic moduli in 2 dimensions X and Y.

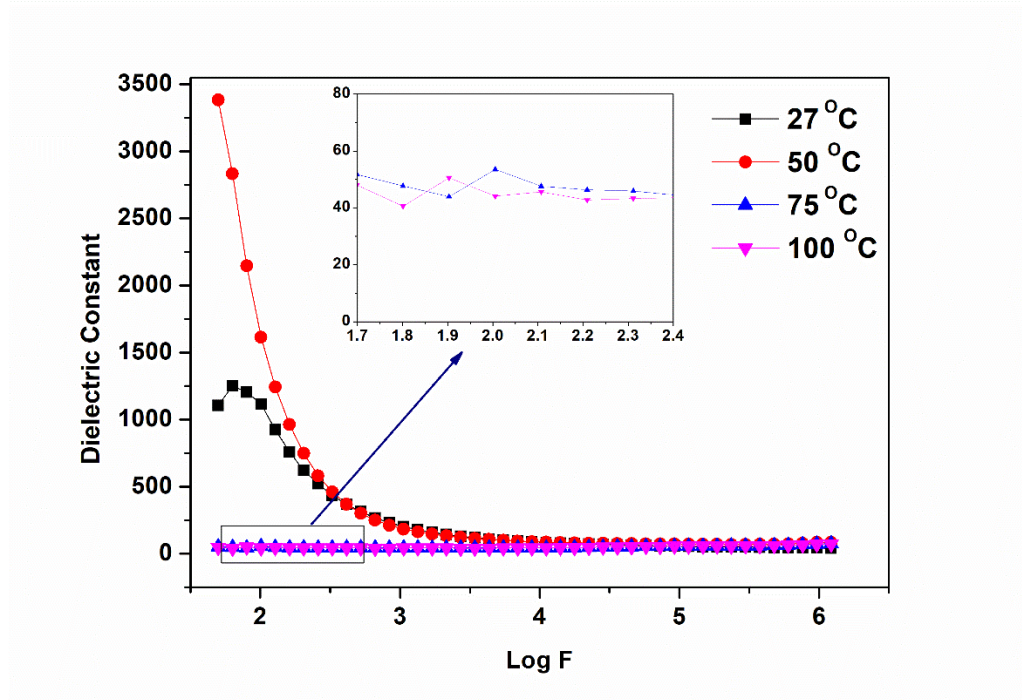


Figure 6: Dielectric Constant as a function of frequency.

Dielectric constant, it is also known as permittivity, is a measure of an ability of material to store electrical energy in an electric field. In many materials, especially biological materials like plant fibres, the dielectric constant can exhibit frequency and temperature dependence due to molecular or structural relaxation processes. Figure 6 depicts the variation of dielectric constant with frequency. It is observed that the dielectric constant found to decrease with increase in frequency and temperature at lower frequency region. At lower frequencies, molecules or dipoles within the material may not be able to respond quickly to the changing electric field, leading to a lower effective dielectric constant. As the frequency increases, the molecules or dipoles have more difficulty keeping up with the field changes, causing the dielectric constant to decrease. At higher frequencies, the relaxation processes may become less significant, and the dielectric constant tends to reach a plateau or become independent of frequency. This behaviour is often observed in dielectric spectroscopy studies, where the dielectric constant is measured across a range of frequencies. It is also noted that the dielectric constant become independent of temperature and

frequency at higher frequencies. This can be related to the fact that at these frequencies, thermal motion of molecules or dipoles may have less impact on the overall dielectric response. Figure 7 shows the dielectric loss as a function of frequency and temperature. It is evident from the figure that the dielectric loss is more at lower frequencies and at lower temperatures. As the frequency and temperature increases dielectric loss tend to decrease. At lower frequencies, where the electric field oscillates relatively slowly, the molecules or dipoles have sufficient time to align with the field, and the dielectric loss is relatively low. As the frequency increases, the ability of the material to respond to the rapidly changing electric field reduces, resulting in a decrease in dielectric loss.

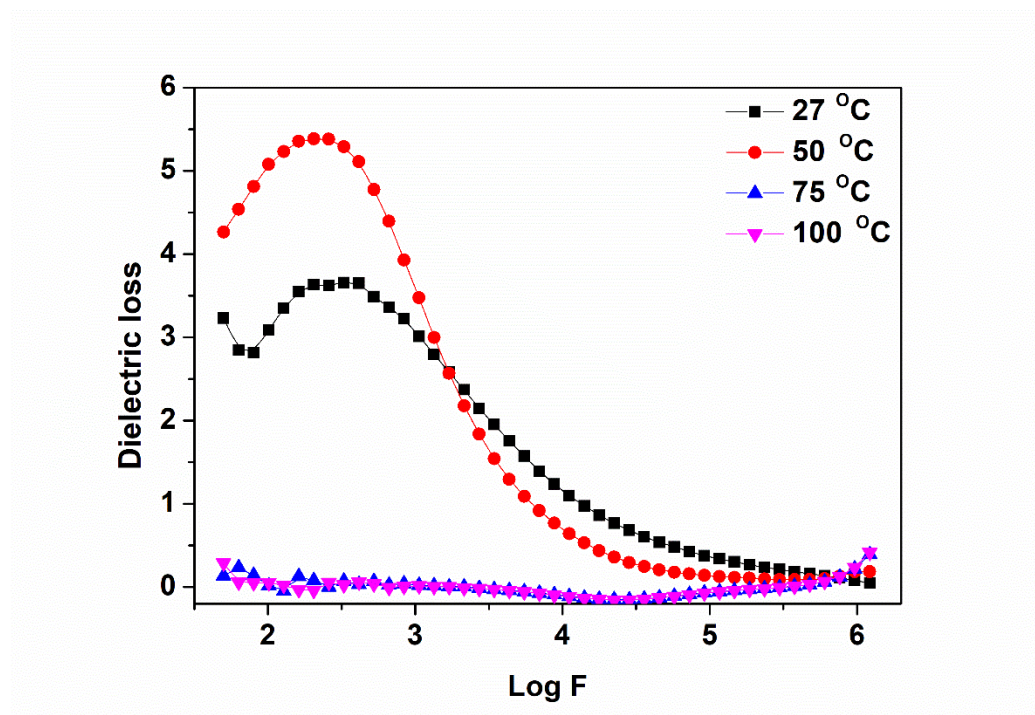


Figure 7: Dielectric loss as a function of frequency.

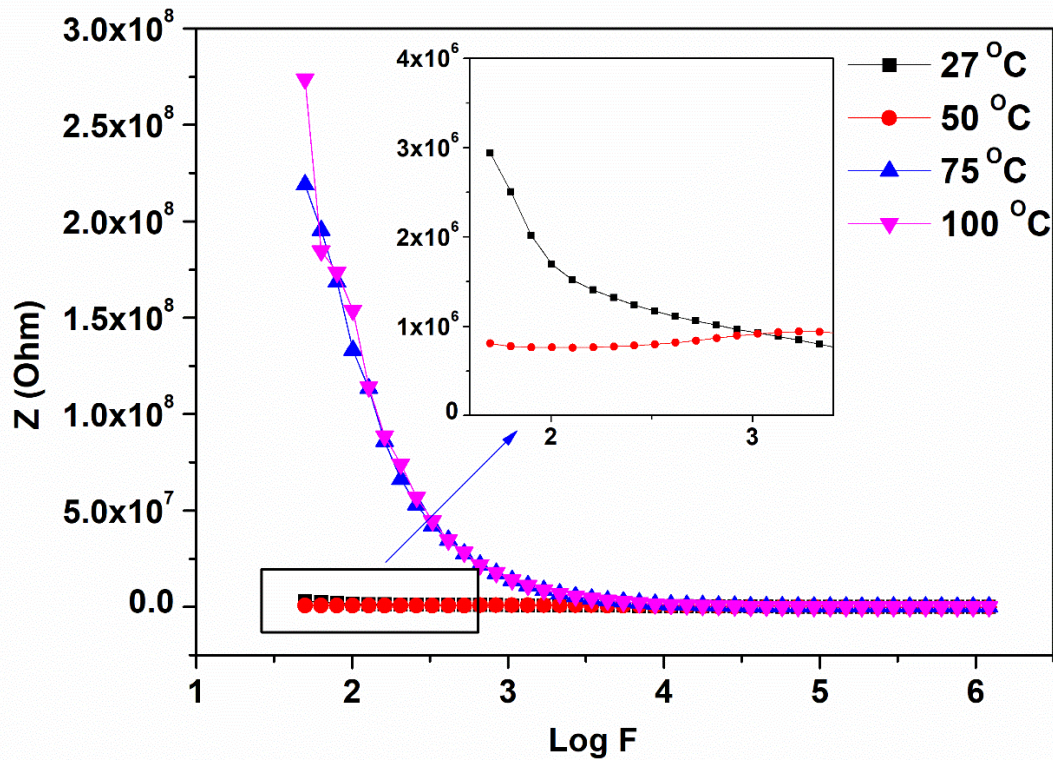


Figure 8: Impedance as a function of frequency.

The figure 8 shows the variation of impedance with respect to frequency and temperature. The Impedance was found to decrease with increase in frequency and increase with the increase in temperature. The impedance of a material is often dominated by its resistance at lower frequencies. As the frequency increases, the reactive components (inductive or capacitive) of the impedance become more significant. The impedance may decrease with frequency due to the reduced impact of resistance compared to the reactive components.

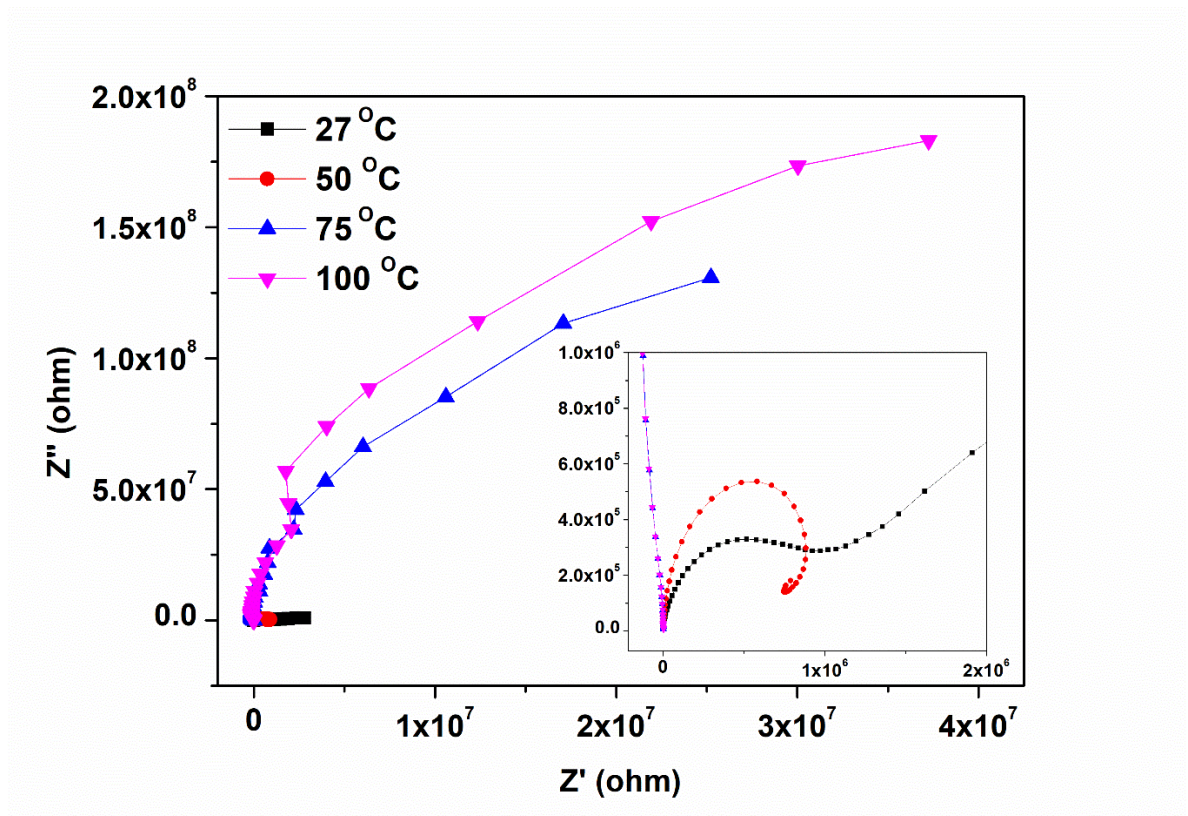


Figure 9: Cole-Cole Plot.

The Cole-Cole plot (Salman et al. 2014) shown in figure 9 also known as a complex impedance plot, is used to analyse the electrical response of materials. In a Cole-Cole plot, if the material exhibits a single relaxation process, the data points tend to form a semicircle. The shape of the semicircle is characterized by its radius, which can be related to the relaxation time of the material. However, deviations from a perfect semicircle can occur due to various factors, such as distribution of relaxation times or the presence of multiple relaxation processes. From figure 9 it can be seen that the complete semicircle was not observed for any temperatures. Also, as the temperature increases radius found to increase. An increase in the radius of the semicircle with temperature may be due to the changes in the relaxation behaviour of the material. This could be due to alterations in the molecular or structural dynamics of the material as temperature increases. The absence of a clear semicircle may be due to the presence of multiple relaxation processes, phase transitions, presence of impurities and broad distribution of relaxation times (Singh et al. 2023).

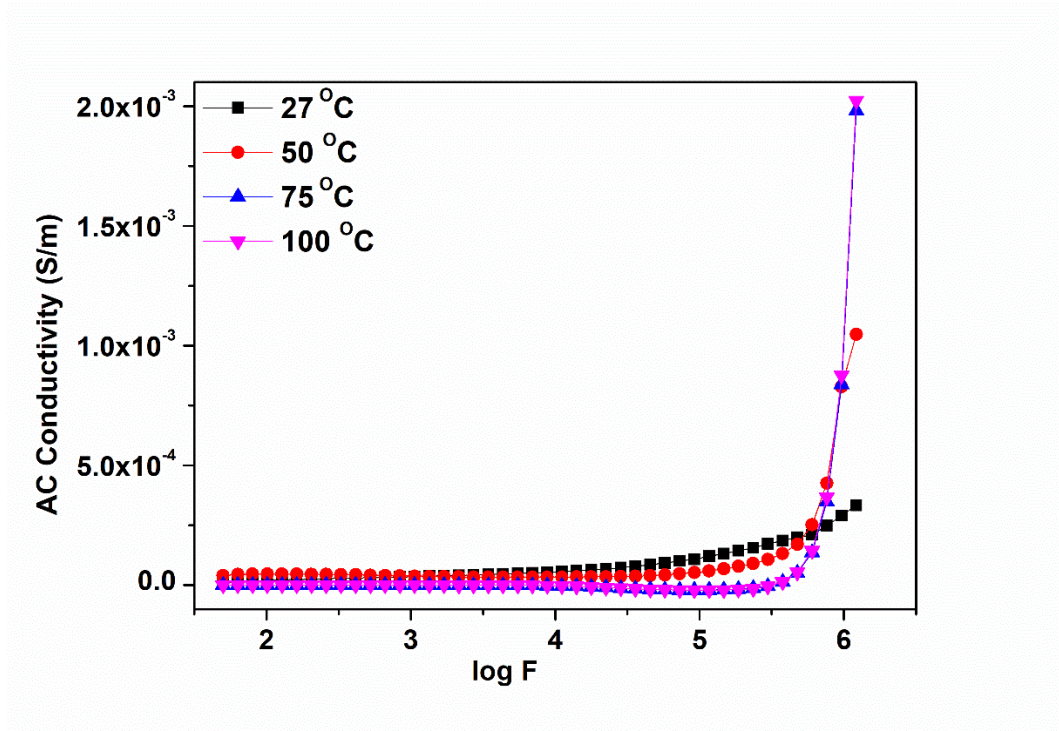


Figure 10: AC conductivity as a function of frequency.

AC conductivity is an important parameter to understand the ionic transport properties of materials. It's determined using the following relation.

$$\sigma_{AC} = 2\pi f \varepsilon_0 \varepsilon' \tan \delta \quad (5)$$

where, f is the frequency of the applied field, ε' is the imaginary part of the dielectric constant and ε_0 is the permittivity of free space. The variation of the AC conductivity with frequency and temperature is shown in Figure 10. From the figures it can be observed that the conductivity is found to be constant in the lower frequency region which defines the DC part of conductivity which is independent of frequency and increase with the increase in frequency which represents the AC part. This phenomenon of the conductivity dispersion is usually analysed by the Jonscher's universal power law (Greenhoe et al. 2016) and is given by,

$$\sigma(\omega) = \sigma_{DC} + A\omega^n \quad (6)$$

where, σ_{DC} is the DC conductivity, A is the temperature dependent factor, ω is angular frequency and n is the frequency exponent of the mobile ions. It is evident that the sample obey this law at all temperatures. It is noted that the conductivity is independent of temperature at low frequencies. Hence, no variation in conductivity is observed DC plateau. However, at higher frequencies, conductivity found to increase with temperature and is due to the occurrence of hopping type of conduction mechanism.

4.2 Functional Data Analysis

Functional data analysis is technique to obtain the correlation between various parameters established in 1991 by Dalzell and Ramsay (Urs et al. 2016). The distinctive aspect of the FDA lies in its capacity to leverage information regarding the rates of change of curve derivatives. By harnessing slopes, curves, and other inherent characteristics of smooth curves, we can explore and utilize this data from various perspectives. The FDA is increasingly employed for the analysis, modelling, and prediction of time series data. Employing the equation of FDA (Urs et al. 2016)

$$\hat{\mu}(t): \operatorname{argmin}_{f \in F} \sum_{i=1}^n \sum_{j=1}^{n_i} [\theta_{ij} - f(t_{ij})]^2 + \lambda \int_{R \geq 0} [f''(t)]^2 dt \quad (7)$$

In this equation, F represents the square integral function possessing at least two derivatives. Functional Principal Component Analysis (FPCA) has been applied to fulfill this objective (Urs et al. 2016). Here the crystallite size values computed six times is treated as θ , along with variable reflections are considered as 't. The observed value of θ for a sample (j^{th}) in the i^{th} trial can be expressed as equation (7).

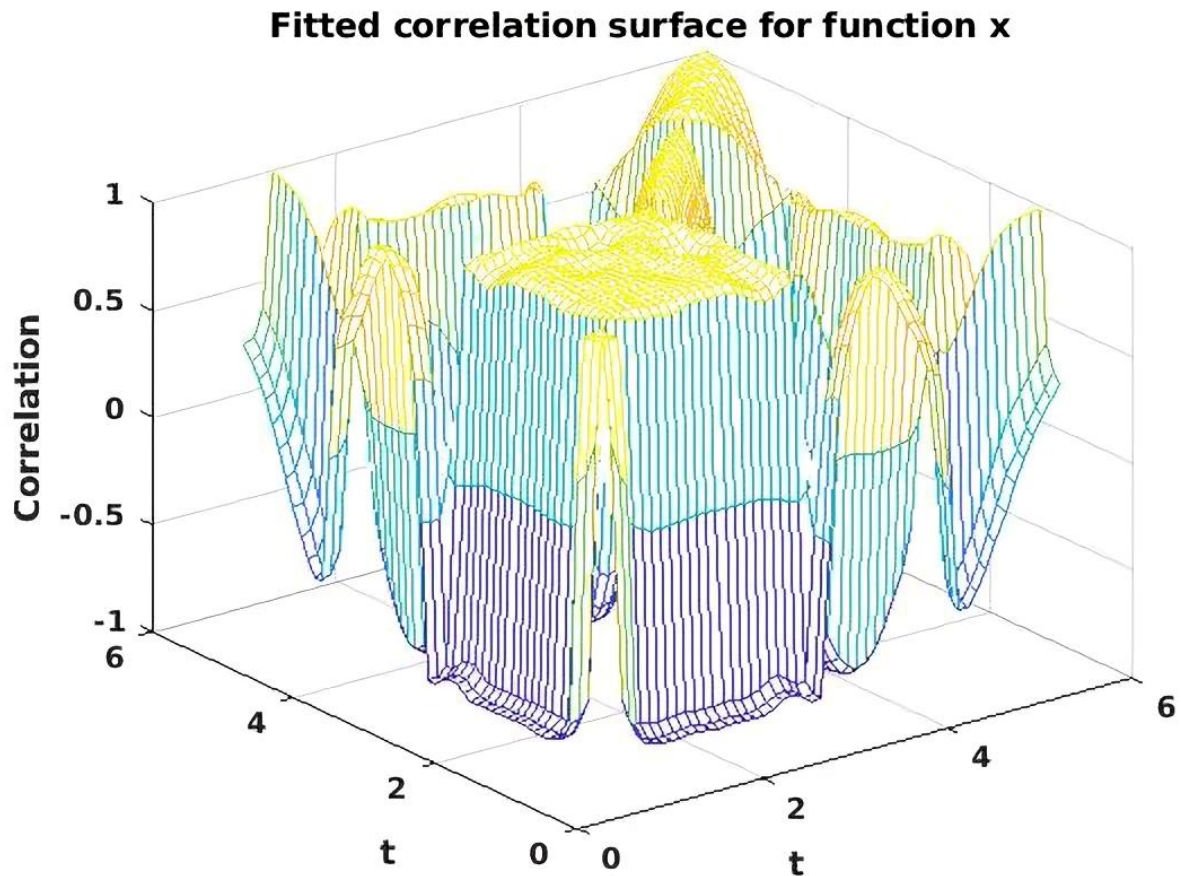


Figure 11: Correlation surface obtained utilizing functional data analysis

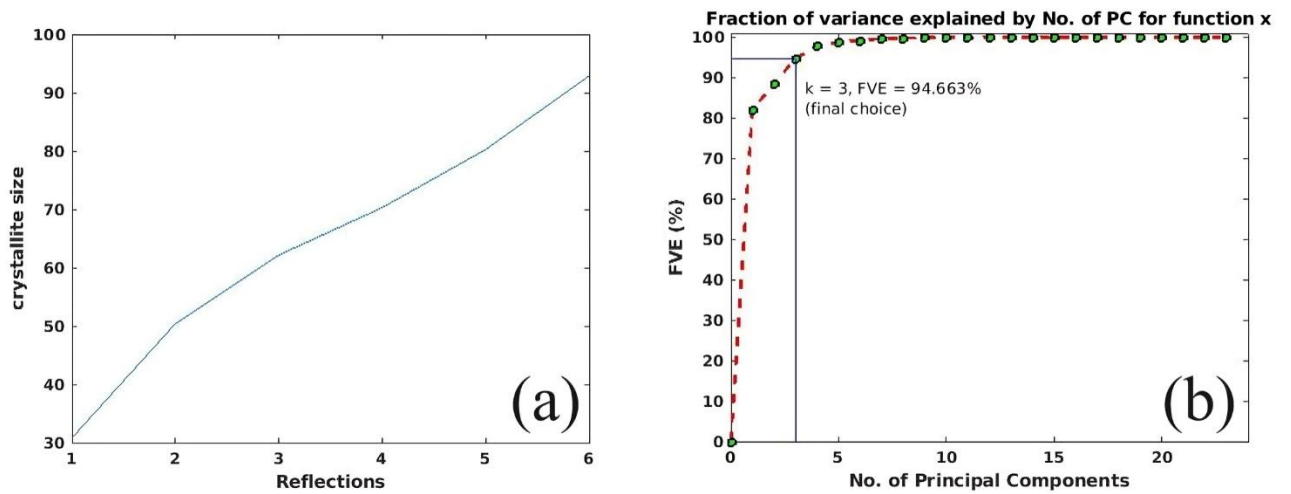


Figure 12: Mean variation and variation principal components

Figure 6 interprets the correlation surface of crystallite size obtained for six reflections. It shows positive correlation for the reflections 1, 3 and 5. Correlation decreases for the reflection 6 due to the wider sharpness of intensity. Figure 7 (a) is the variation of crystallite size values as a function of reflections, this mean function shows that, crystallite size values increase steeply at the reflections 2, 3 and gradually reaches highest values. Figure 7 (b) Illustrates the scree plot, indicating that two principal components adequately capture 94.663 % of the variation in measured values from the mean values for crystallite sizes.

5. Conclusion

The structural, mechanical, and elastic properties of lotus fibre is examined using powder X-ray diffraction data. The discerned ordered regions in this lotus fibre showcase varied topographies along their spatial crystallographic directions. This irregular variation is attributed to factors like the lack of a defined form and a leveling-off degree of polymerization (DP), the minerals present in the soil, the overall environment where the fibre is grown. Applying Treloar's principle, the elastic constants for a monoclinic crystal system were computed and shown by utilizing the elastic tensor analysis tool (ELATE). The deviation of elastic moduli from their spherical shape was evident, indicating the presence of anisotropy in the fiber. Anisotropic materials display varying mechanical properties in different directions, thereby influencing the overall mechanical stability of the fiber. These investigations clearly indicate that both the fiber's growth and mechanical stability are susceptible to environmental influences. Dielectric studies have shown that, dielectric properties vary with frequency and temperature. Lower frequencies show decreased responses, reaching a plateau at higher frequencies. Impedance shifts from resistance dominance to reactive components. Cole-Cole plot deviations suggest diverse relaxation processes. AC conductivity follows Jonscher's law, with a constant DC part at low frequencies and increased temperature-dependent AC part at

higher frequencies, indicating hopping conduction. Also, employing the FDA techniques, the correlation among crystallite size versus six reflections were obtained. These studies can be extended to any material of interest to delve into the structural and physico-mechanical properties of nascent materials.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical Approval

Not Applicable

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Availability of data and materials

Data can be made available on request.

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