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EXTRACTION AND CHARACTERIZATION OF CELLULOSIC FIBRE FROM THE STEM OF *HIBISCUS SURATTENSIS*

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ABSTRACT

The modern world makes the use of natural fibre for the manufacturing of biomaterials. The natural fibres are long, thin and flexible thread like material abundant with continuous supply. Now a day researches explores the natural fibre for reinforcement applications with promising physic-chemical properties. *Hibiscus surattensis* is an annual plant from which the natural fibre can be extracted (HS stem fibre). Fourier Transform Infrared Spectroscopy test confirmed the presence of cellulose, hemicelluloses, lignin, with wax and moisture content. The crystallinity index (58.24%) of HS stem fibre was computed from X-ray diffractogram. The texture and structure of fibre were explained through SEM analysis showed that these fibres are rough and contain wax and mucous content. The carbohydrates, flavanoids, alkaloids, tannins, and phenol were found form the phytochemical analysis. The physicochemical properties of *Hibiscus surattensis* stem fibre (HS stem fibre) were revealed and compared to other natural fibres.

Keywords **:** *Hibiscus surattensis* fibre, FT-IR studies, Crystallinity index, Morphological characters, Phytochemical analysis.

Introduction

The natural fibres are nontoxic have a lower density, are easy to handle, compostable and abundant with continuous supply. Rather than the synthetic fibres, natural fibres are also biodegradable, renewable and eco-friendly they make it more convenient for human beings. All these have economic as well as commercial value. In recent years reinforcing polymers with fibre has been an active method to substitute materials in industrial applications (Pickering *et al*., 2016). A variety of synthetic fibres have specific features which improves the commercial value of fibre in the industry. Unlike most synthetic fibres, all natural fibres are non-thermoplastic, and susceptible to microbial decomposition (Shao *et al*., 2010). The properties such as length, strength, pliability, elasticity, abrasion resistance, absorbance and various surface properties define the commercial use of fibres (Legland *et al*., 2013). Based on the economic and environmental consideration the usefulness of natural fibre can be determined by its physical, chemical and mechanical properties. Cellulose, hemicelluloses, and lignin are the major chemical components of natural fibre. The cellulose structure, smaller fibre diameter, length and roughness in the surface are the advantageous characters of natural fibre. The chemical composition varies with the type of natural fibre. The phyto-constituents provide pharmacological properties to the plant (Loganathan *et al*., 2020). The natural fibres are plant origin derived from the stem, leaf, root and seed. A wide variety of plants including cotton, jute, flax, ramie, and hemp are used to produce plant fibre. Also the unknown traditional wild plants also contain fibre. Among these the stem fibres are long with thick cell wall and shows higher reinforcement properties (Placet *et al*., 2018).

From *Hibiscus surattensis* L. (HS) stem a fibre with greater mechanical properties can be extracted. *Hibiscus surattensis* is an annual climbing plant in family Malvaceae, genus *Hibiscus*. The plant commonly called Bush sorrel, Assam susor, or Chemmeenpuli, covered with soft hairs and prickles.

The leaf is palmate, lobed with toothed margin. Flower is solitary yellow in colour with dark centre have false sepals (Gamble, J. S., 1915) as shown in Figure 1. It is native to the seasonally dry tropical and subtropical old world and introduced to the islands of Indian Ocean. It is common in roadsides, abandoned lands, vacant plots, landslides and construction sites. It also considered as a weed of grassland and marshy field (Tiko *et al*., 2020). The investigation is to understand the morphological and phytochemical properties of *Hibiscus surattensis* fibre by Fourier transform-infrared spectroscopy (FT-IR), X-ray diffraction studies, Scanning electron microscopic studies and phytochemical analysis.

Materials and Methods

Extraction of HS fibre

Hibiscus surattensis plant found from the town of Marathakkara-Thrissur, Kerala India was taken for the present research. Initially, the stem of the plant was cut carefully and were washed thoroughly in running water to clean and remove dirt particles. Then the stems were immersed in a tray containing water for the degradation of surface tissue to separate the fibre from the plant part for 10 to 15 days respectively as shown in Figure 2a. The separated fibre was washes in tap water to remove debris and dried in sunlight. The extracted fibres are pale yellow in colour, thin and uniform as seen in Figure 2b.

Fig. 1 : *Hibiscus surattensis* plant

2a Soaked stem 2b Extracted fibre

Fig. 2 : Fibre separation Process

Characterizations of HS stem fibre

Fourier Transform Infrared spectroscopy analysis

Fourier transform infrared spectroscopy was used to identify the functional groups present in extracted fibre. The fibre samples were dried, ground into fine particles and 2.5mg were mixed with diamond, compressed into pellets, and then analysed with Perkin Elmer Spectrum two FT-IR Spectrometer by using attenuated total reflection (ATR) contact sampling method. The test was conducted with a total of 32 scans for wave number ranging from 4000 to 400 cm^{-1} in room temperature. In FTIR spectroscopy, infrared radiations are passed through the fibre sample. The OH stretching bonds present within the functional groups absorb the radiation and vibrate with different frequencies (strong/medium/weak), depending on the strength of the bonds. Each molecule has a unique combination of bonds that produce peaks at specific frequencies. The obtained peaks indicate the molecule present in the fibre sample (Nagaraja Ganesh and Rekha, 2019).

X-Ray Diffraction (XRD) Analysis

X-ray diffractograms of the powdered samples of each were recorded in X'pert PRO-PAN analytical system using 40kV, 30mA and CuKα radiation. A step size of 0.05^0 and step scan of 10.16 sec was used for the entire reading range (10^0-80^0). Crystallinity was evaluated from the percentage crystallinity index (I_c), using Segal equation (Segal *et al*., 1959).

$$I_c = \left[1 - \frac{I_{am}}{I_{002}}\right] \times 100\% \quad (1)$$

Where I_{002} is the maximum peak intensity representing the crystalline region, and I_{am} is the minimum peak intensity representing the amorphous region

The crystallite size of the fibres was determined by Scherer's formula,

$$CS = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

Where, K = 0.89 is Scherer's constant, β is the peak's full width at half- maximum, λ is the wavelength of the radiation.

The analysis were done by using multipurpose X-ray defractometer equipped with a D/ taX ultra 250 1D detector and integrated with the SmartLab SE software.

Morphological study

The surface morphology of extracted fibres was studied by using scanning electron microscopic study. In SEM analysis the specimen was mounted on stubs using carbon tape and was over-coated with gold using JFC 1600. This iron-sputtering device performs rapid and efficient gold coating on the microscopic specimen, allowing surface visualization. The SEM measurements were performed at 15kV accelerating voltage. Different magnifications were used as indicated on the images.

Phytochemical analysis

Extraction of plant material: The collected samples of the stem were washed thoroughly under running water, air dried and powdered to enhance the contact of solvent and the plant material.

Hot water extract: The air dried finely powdered plant samples (1.0g each) were soaked in 20ml of water and boiled. The extract is filtered through Whatman No.1 filter paper. This procedure is repeated for one more time. The supernatant was collected, covered, and labelled then used for screening of various phytochemicals.

Alcohol extracts: The air dried finely powdered plant samples (1.0g each) were soaked in 100ml of ethanol for 24hours at room temperature. The supernatant was collected, covered, and labelled then used for screening of various phytochemicals (Sadasivam and Manickam., 1991).

Qualitative phytochemical analysis: The qualitative phytochemical analysis of test samples was determined by Harborne, J. B (2005) method. Molisch's test was used to determine the carbohydrates. Ferric chloride test used to determine tannin and form test was performed for the determination of saponin. Flavanoids was determined by using sulphuric acid test. Mayer's method was used to determine alkaloids and sodium hydroxide test was used to identify anthocyanin and betacyanin. Keller-killiani test was used for the determination of glycosides. Protein and sterols were determined by using Ninhydrin and Salkowski tests respectively.

Results and Discussion

Fourier Transform Infrared spectroscopy analysis

The interaction of active functional group with natural fibre is determined using Fourier Transform Infrared Spectroscopy (FT-IR). In the FT-IR spectrum bands studied under transmittance in the range from 400 to 4000cm^{-1} on fibre showed the presence of functional groups in respective peaks. The spectrum shows the absorption bands of lignocelluloses fibre compounds such as cellulose, hemicelluloses and lignin, composed of alkenes, aromatic groups and different oxygen containing functional groups. The FT-IR spectrum of HS stems fibre shows a broad peak at 3334cm^{-1} indicate (O-H) stretching vibration of fibre cellulose structure shown in figure 3. The C=O stretching vibration of carboxylic acid in lignin or ester group in hemicelluloses shows a peak at 1733cm^{-1} in HS stem fibre as in *Sansevieria cylindrica* (Sreenivasan *et al*., 2011). The peak at 1240cm^{-1} is associated with C-O stretching of lignin as seen in Okra and Artichok (Igor *et al*., 2010; Fiore *et al*., 2011). The bending vibration of CH-CO group of aromatic rings of hemicelluloses and lignin shows at 1031cm^{-1} and where the βglycosidic linkage of lignin shows at a peak 556 cm^{-1} in HS stem fibre. Here the absorbance at 409cm^{-1} in HS stem fibre shows C-OH bending similar to *Sida rhombifolia* (Gopinath *et al*., 2016).

X-Ray Diffraction (XRD) Analysis

The cellulose crystallographic structure of the extracted HS fibre was studied from the X-ray diffractogram. The 2θ location and its intensity are related to the crystalline content in extracted fibre. The XRD spectrum of investigated HS stem fibre is demonstrated in Figure 4. The angle 2θ contains a peak at 22.88° can be attributed to (110) cellulose crystallographic plane. It reveals a monoclinic structure. The angle 22.88° shows a broad reflection. The crystalline index (CI) refers to the amount of crystalline cellulose with respect to the global amount of amorphous materials. Segal equation was used to find the crystalline index. The crystalline index of HS stem fibre was found to be 58.24% which is comparatively greater than that of fibre such as ferula fibre 48% (Yoldas *et al*., 2013), Grewia tilifolia 41.7% (Jayaramudu *et al*., 2010), Acacia leucophloea 51% (Stuart, B., 2005). The crystalline size (CS) of the HS stem fibre using Scherer's formula in equation (2) was calculated as 14.7nm. The CS in HS stem fibre offers

chemical reactivity and water absorption capacity properties seems higher than other natural fibre such as flax fibre.

Morphological study

The scanning microscopic images of *Hibiscus surattensis* stem fibre (HS stem fibre) is illustrated in Figure 5. The morphological study of HS stems fibre shows that the surface of fibre has serrations. The surface of HS stem fibre bare long stretches which extend throughout its length with many minor cracks can be seen as in figure 5b and 5c with a magnification 100 and 20µm respectively (Ganesh *et al*., 2018). It is apparent that the surface of this fibre is rough and the structure can aid in good interfacial adhesion with polymer matrices. Some white coloured precipitation can be seen in the surface may be the cellulosic substance or wax present in them. The magnification 10 µm shows some separations or dislocations are illustrated in figure 5d. These are referred as cell wall folds, slip panels, zones of micro compression etc. (Qi *et al*., 2019). A study on ramie fibre shows that the dislocations are made by nodes surrounding the cell wall, and some authors describes them depending on the type of deformation (Nyholm *et al*., 2001). The diameter of HS stem fibre is 18.40µm is shown in figure 5f.

Phytochemical analysis

The preliminary qualitative phytochemical screening of water and alcohol extracts is demonstrated in the Table 1. The table revealed that the stem extract had the highest composition of alkaloids, carbohydrate, tannin, saponin, and flavanoids. Also shows the presence of phenol in alcohol extract. It has reported tannin shows antimicrobial activity through a variety of mechanism in *Sida acuta* (Ezeabara and Egenti, 2018). Were athocyanin/ betacyanin, glycosides are absent.

Conclusion

The cellulose fibre extracted from HS stem was tested to find their compatibility to reinforce polymer composites from their physical, chemical, morphological and surface properties. The test conducted on HS stem fibre revealed the properties of cellulose, considerably good than other cellulosic fibre. The surface of HS stem fibre shows a rough nature provides good interfacial adhesion with polymer matrices. The crystallinity index (CI) of HS stem fibre was found to be 58.24%, which indicate the presence of high crystalline cellulose.

The degree of crystallinity represents the packing order of the crystallites in the fibre. Higher the degree of crystallinity, higher is the mechanical strength of the fibre and its composites. In this study the degree of crystallinity of HS stem fibre was calculated as 58.24% a value that proves the crystallite packing is good and structural arrangement is strong enough for making durable biocomposites.

The *Hibiscus surattensis* parts are loaded with bioactive compounds that have powerful health benefits. The alkaloid occurred highest in the stem which is believed to be the major active component. The therapeutic value of the plant depends on their chemical component which produces specific pharmacological activities on human and animal body. Hence the fibre of the plant contains those phytochemicals could be the rich source. From the study, it can be concluded that the fibres obtained from the stem of *Hibiscus surattensis* can be effectively used as promising reinforcements for bio-composites.

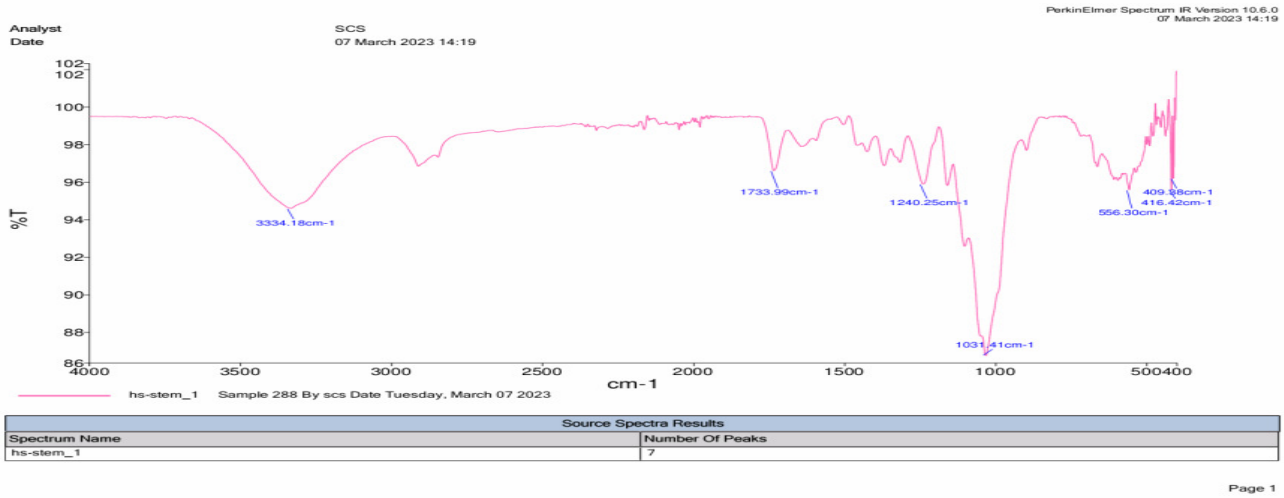


Fig. 3 : FTIR spectrum of HS-stem fibre in the frequency of 400-4000cm^{-1}

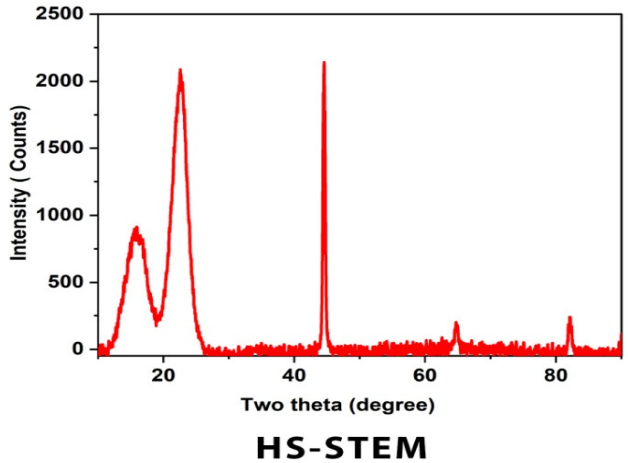
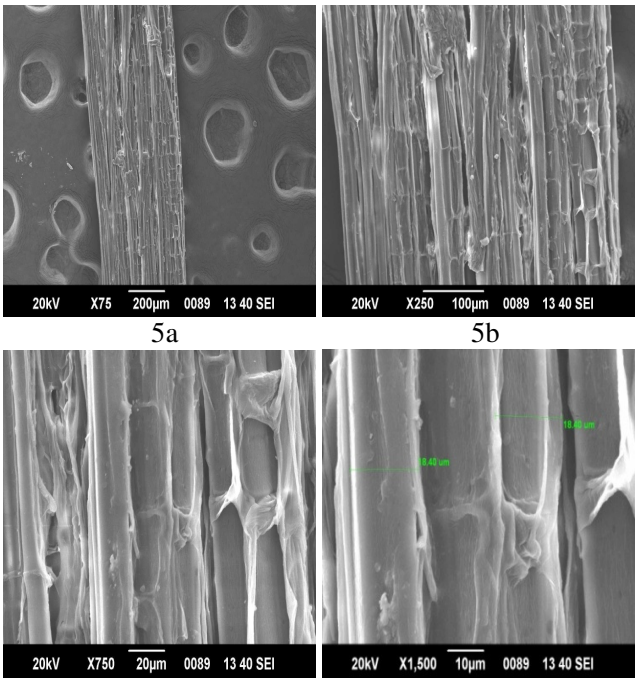


Fig. 4 : X-ray Diffractograms of HS-stem fibre



5a 5b

5c 5d

Fig. 5 : Scanning electron microscopic study of HS stem fibre

Table 1 : Phytochemical analysis

Metabolites	Sample	
	Water extraction	Alcohol extraction
Carbohydrates	+	+
Tannins	+	+
Saponins	+	-
Flavanoids	+	+
Alkaloids	+	+
Anthocyanin & Betacyanin	-	+
Glycocides	-	-
Proteins & Aminoacids	-	-
Steroids & Phytosteroids	-	-
Phenols	-	+

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