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COMPARATIVE STUDY ON PHYSICO-CHEMICAL PROPERTIES OF ROSELLE (*HIBISCUS SABDARIFFA*L) AND RAMIE FIBRE

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ABSTRACT

Natural fibre seems to be the most imminent alternative for chemically treated synthetic fibres. Natural fibre based products are now getting more preference across the globe over synthetic fibre products which have less bio degradable characteristics. Roselle, one of the most important horticulture crop it could be utilize for extraction of fibre. Fibres extracted from Roselle and ramie degummed with 3% Na_2Co_3 solution at 95°C for 2 hours and Bleached with hydrogen peroxide at 90°C for 60 minutes in a close vessels and analysed for their physico-chemical and mechanical properties. The physical dimensions of fibres such as length, diameter, and wall thickness were measured. The tensile strength of these fibres varies from 30 g/tex to 40 g/tex and elongation were maximum after bleaching process. The untreated fibres have more tensile strength than the treated one. The chemical compositions like cellulose (64.50) were high after bleaching was as, hemicelluloses content decreased. The SEM and FTIR test were done for the mechanical analysis of the fibres. By comparing all the properties, it may be said that, the blending of the roselle-ramie can take place for the production of good quality textiles.

KEYWORDS: *Bleaching, Degumming, Fibre Fineness, Tensile Strength, SEM, FTIR*

Article History

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INTRODUCTION

Roselle is *Hibiscus sabdariffa* L., belongs to the Malvacea family and is found abundantly in tropical areas. It can grow annually and attain a height between 2 and 2.5 m. These plants are commonly used as an infusion and to produce bast fibres[2]. Roselle fibre is a type of bast fibre. The difference between roselle fibres and other fibres lies in their composition, *i.e.*, the ratio of cellulose; hemicellulose, lignin and orientation or the spiral angle of the cellulose microfibril[1]. The roselle plant may have been brought to the west from India by Muslims from Turkey. The plant was initially known by the name “Sabdariffa,” a Turkish word, which confirmed the origin of roselle. The scientific name for the bark roselle plant is fibrous and has a good potential of becoming an excellent source of fibre in manufacturing different valuable textiles as it is or after blending with other fibers[3]. The cross-section roselle plant stem, the most outer part of the bark provides protection from extreme temperature changes and excessive loss of moisture while hardening thestem. The fibres are hidden under the bark inside the phloem. The fibres keep the stem strong by supporting the conductive cells of the phloem. Roselle is cultivated primarily for the bast fibre obtained from the stems. Recently, roselle fibres have attracted many researchers to explore their potential as reinforcement materials due to their similar properties to other established natural fibres such as jute[4].

Ramie, one of the oldest textile fibres of plant origin ruled the textile world as king of natural fibres. Its popularity in the textile world is limited due to the difficulty in degumming and lack of knowledge of mechanical processing. Increasing ecological consciousness has accelerated interest in ramie originating from plants that are safe, biodegradable and recyclable. Ramie is highly adorned for its lustre, strength, excellent microbial resistance and valuable hygienic properties. Some of the demerits are encrusting gummy materials and its cohesiveness. If the apparent demerits can be masked, an excellent diverse range of product can be engineered by exploiting the intrinsic properties of ramie[5].

In the present study physical and chemical analysis of roselle and ramie fibre was done with different chemical agents and its effect on tensile strength, elongation and fineness properties of fibre was studied. Very few studies have been reported till now on the use of the roselle fibre and no study has been conducted on blending of these two fibres. Hence, this study is an effort towards to know the possible uses of these two fibres. Bleaching of fibres was also carried out to enhance the colour value of fibres. Physical and chemical properties of roselle and ramie fibres were also tested after each step of processing.

MATERIALS AND METHODS

Materials

Selection of Plant

The roselle plant utilized for the present study was AS73, CP 560 variety, collected from the farmer's field of Potya gaon, Jorhat, Assam. . It is an annual or perennial herb, growing to 2-3m (6-8ft) tall. The ramie fibre variety R1412 were selected for the study and collected from Ramie Research Station, Shorbhog, Assam. It is herbaceous perennial growing to 2-2.5m tall.

Methods

Extraction of Roselle Fibre

The roselle fibres were extracted by using a water retting process. Table.1 shows the yield of roselle fibre in different duration of days. In 26 day maximum yield were found (4.5%). The retted stem of roselle plant was washed in running water, and the fibres were removed manually. Next, fibre were cleaned, and then dried in sun light.

Table 1: Retting Parameter of Roselle Fibre

Bath Ratio (M:L, kg/lit)	Duration (Days)	Temperature (°C)	Yield of Fibre (%)	Observation
1:10	20	Room Temp.	1.8	Partial removal of gummy substances, fibre were stiff
1:10	23	-do-	2.5	soft fibre, low yield
1:10	26	-do-	4.5	soft fibre, more yield
1:10	29	-do-	3	fibre disintegrate

a). Roselle Plant **b).** Water Retting Process **c).** Retted Fibre

Figure 1: Extraction of Roselle Fibre.

Degumming and Bleaching Process

For degumming process, 1:10 bath ratio was maintained than extracted fibre treated with 3% solution of Na_2CO_3 at 95°C for 2 hours and rinsed with cold water, neutralized with 2% acetic acid, rinse again until neutral pH and dried at 50°C in a vacuum oven.

Bleaching were done by using alkaline hydrogen peroxide with material to liquor ratio 1:10 at 90°C, pH 10-11 for 60 minutes in a close vessels, washed properly and air dried.

a). Degummed and Bleached Roselle Fibre b). Degummed and Bleached Ramie Fibre

Figure 2: Degummed and Bleached Roselle and Ramie Fibre.

Physical Dimensions of Fibres

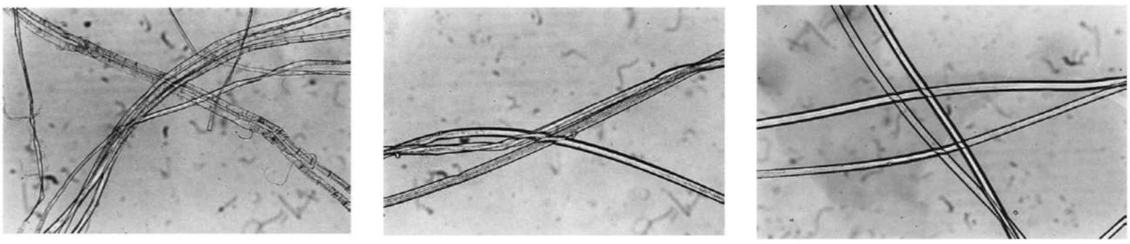
The physical properties like length, diameter, wall thickness and fibre fineness of the fibres were determined by using a Dokuval photomicroscope (JEOL, Japan). From this method diameter and wall thickness of the fibre was measured at a time. Themoisture regain of raw, degummed, and bleached fibres were measured by the oven dry method following ASTM-D2654-76 under standard conditions of 65% relative humidity at 27°C.The wicking height of the fibre was tested by calculating the time taken to absorb the water.

Analysis of Chemical Composition of Fibres

The chemical analysis were done with TAPPI standard methods (TAPPI1980)[7.] The ash, hemicellulose and cellulose (TAPPI Standard methods T429), lignin content (T-222), moisture content (T-412) were determined by the method suggested by Goswami and Saikia[8].

Microscopic Evaluation

Microscopic view of retted, degummed and bleached roselle fibre examined under microscope and photographs were taken with the help of Karl Zeiss optical microscope.



a). Retted Roselle Fibre **b).** Degummed Roselle Fibre **c).** Bleached Roselle Fibre

Figure 3: Longitudinal View of Roselle Fibre.

Physical Properties

The tensile strength and elongation were measured by stelometer by taking a bundle of fibres of 25cm long and measure of tenacity at ½ gauge lengths (Booth[9]) and the average values of 10 observations were recorded. For density ASTM[10] standard method was used.

Mechanical Analysis of Fibre

Scanning Electron Microscopy (SEM) Analysis

All the samples were tested and SEM images were obtained to analyse the morphological structure. The specimen containing the fibres were arranged on a pin stub and then positioned on a holder that can be inserted into the SEM. High acceleration voltage (15 kV) and 100 µA beam current were used to obtain high-resolution imaging. The longitudinal views of fibres were analysed.

Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

The macromolecular behaviour was examined using Bruker/ALPHA FTIR spectrometer equipped with an attenuated total reflection (ATR) attachment. The fibre samples were analysed following ASTM E168-06 and they were placed directly under the crystal and ATR attachment. The transmittance spectra were recorded at wave numbers from 500 to 4000cm^{-1}.

RESULTS AND DISCUSSION

Physical, chemical and mechanical properties of fibre from roselle and ramie of untreated, scoured and bleached fibre were shown in Table 2 and Table 3 and table 4 respectively.

Physical Dimensions of Fibres

Length, diameter, wall thickness, fibre fineness, wicking height and moisture regain were examined and presented in Table 2. The staple length of retted fibre was found to be highest in roselle and ramie fibres, which can suit to develop quality yarn since fibre length mainly influences spinability and yarn quality. The similar work related to the fibre length was reported by Kalita *et al.*[11].

In regarding to the diameter and wall thickness it's decreased after bleaching (11.90µm, 8.00µm) (2.10, 2.00) in roselle and ramie respectively due to the removal of gummy substances. Fibre diameter plays a vital role in determining the quality of yarn and fabrics. Maximum fineness found in retted roselle and ramie fibre (3.90g/tex and 3.50g/tex) and minimum in bleached fibre. After bleaching, there is an improvement in fibre fineness due to the removal of lignin and gummy substance from the fibre. The maximum wicking height and moisture regain recorded in bleached roselle and ramie fibres it may be due to the removal of pectin and waxes from the fibre surface. The moisture regains and wicking height of the fibre got improved in each stage of chemical processing. Good wicking height and moisture regain is required for further wet processing like dyeing and finishing, apart from that, fibres with good wicking height and moisture regain will provide better wear comfort.

Table 2: Physical Dimensions of Roselle Fibre

Properties	Retted Fibre		Degummed Fibre		Bleached Fibre	
	Roselle fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre
Length(mm)	122	120	120	118	119	117
Diameter(µm)	14.10	10.00	12.60	9.50	11.90	8.00
wall thickness(µm)	3.50	2.50	2.60	2.25	2.10	2.00
Fibre fineness (g/tex)	3.90	3.50	3.40	3.00	2.35	2.20
Wicking height (cm)	13.00	13.00	15.00	14.00	17.00	16.00
Moisture regain (%)	8.40	8.50	8.80	8.80	9.10	9.00

Chemical Composition of Fibres

The fibres obtained after retting, degumming, and bleaching were evaluated for their chemical composition, and data are presented in Table 3. The maximum cellulose (89.2%) and (64.50%) was recorded in bleached roselle and ramie fibres. The table showed a decreasing trend in case of hemicellulose content of fibre. The hemicelluloses content of processed fibre was reduced due to the different chemical treatments. The results have shown in conformity with the work of Razali *et al.*[12]. The percent of cellulose is different in processed fibre may be due to the use of chemicals during degumming and bleaching process, which may remove lignin percent from the fibre. Lignin, ash and wax content were found to be reduced after chemical processing. The moisture content of both the fibres increased due to the removal of wax and gummy substances. The increase in moisture content of the fibre can ultimately increase the wear comfort of the apparel. Both degummed and bleached fibres have shown competitively good moisture content, which is higher than cotton. Bleaching increased the brightness of the fibre due to the removal of natural colouring matter and partial oxidation of chromophoric groups present in lignin.

Table 3: Chemical Composition of Roselle and Ramie Fibre

Constituents	Retted Fibre		Degummed Fibre		Bleached Fibre	
	Roselle Fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre
Cellulose (%)	56.25	80.10	58.63	86.10	64.50	89.2
Hemicellulose (%)	11.50	8.80	9.70	3.00	8.15	2.50
Ash (%)	2.08	1.38	1.03	0.52	1.25	0.45
Lignin (%)	7.25	9.45	3.00	-	1.00	-
Wax (%)	0.50	0.70	0.30	0.50	0.20	0.3
Moisture (%)	7.40	6.87	8.21	7.30	8.76	8.68

Physical Properties of Roselle and Ramie Fibres

The physical properties of raw, degummed and bleached roselle and ramie fibres were analysed in Table 4. The maximum tensile strength was observed in retted roselle and ramie fibres. Tensile strength deteriorated in both the fibres from retted to bleached fibres; it may be due to the loss of hemicellulose content and lignin. In addition to this, the strength loss may also be contributed by the attack of alkaline hydrogen peroxide in the cellulose portion. Lignin and hemicellulose formed a continuous network of the fibre and directly influence the physical and mechanical properties of fibers[13]. On the other hand bleached roselle and ramie showed highest elongation. The elongation of the fibre affected the comfort and appearance of the fabric. The maximum density found in case of retted roselle fibre in compare to the ramie fibre it shows the roselle fibres are courser than ramie fibres. The increase in density may be due to the removal of hemicellulose and lignin during bleaching. The brightness was maximum in both the bleached fibres. Due to bleaching, the brightness index of the degummed roselle and ramie fibres were increased.

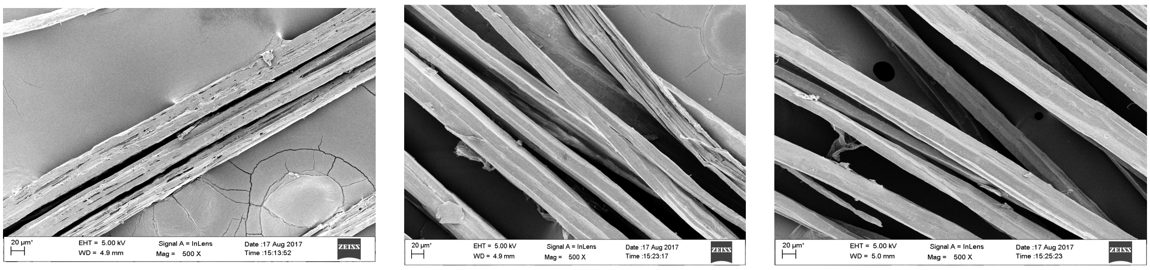
Table 4: Tensile Strength, Elongation and Density of Roselle and Ramie Fibres

Properties	Retted Fibre		Degummed Fibre		Bleached Fibre	
	Roselle Fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre	Roselle Fibre	Ramie Fibre
Tensile strength (g/tex)	30.00	40.00	25.00	35.00	23.00	30.00
Elongation (%)	1.59	1.60	1.65	1.70	1.95	2.00
Density (g/cc)	1.49	1.50	1.43	1.40	1.35	1.20
Brightness index (µm)	20.00	20.00	28.00	30.00	41.00	45.00

Mechanical Analysis of Fibre

SEM Analysis of Roselle Fibre

The surface appearance of raw, degummed, and bleached fibres was studied using FE SEM images as shown in Figure 4. The gum content of the raw fibres is clearly visible in the SEM image. The surface is found to have cracks and holes; this indicates the presence of gummy matter on fibre surface. The alkaline degumming almost removed the gummy matter Fig. 4(b) and bleaching process removed whole of the gum from the fibre surface Fig. 4(c). Degumming and bleaching resulted in individualization of fibre entity and smoothening of surface by removing the gum from the fibre.

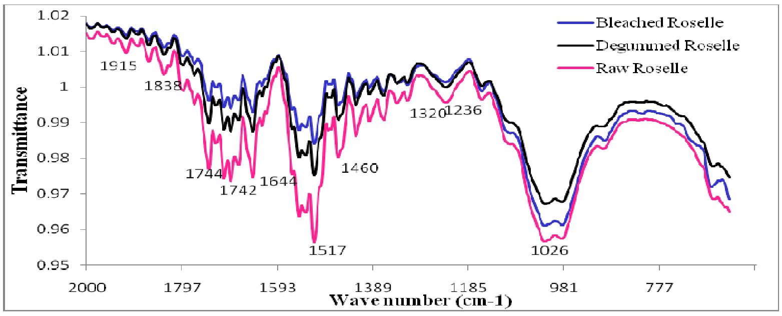


a). Retted Fibre **b).** Degummed Fibre **c).** Bleached Fibre

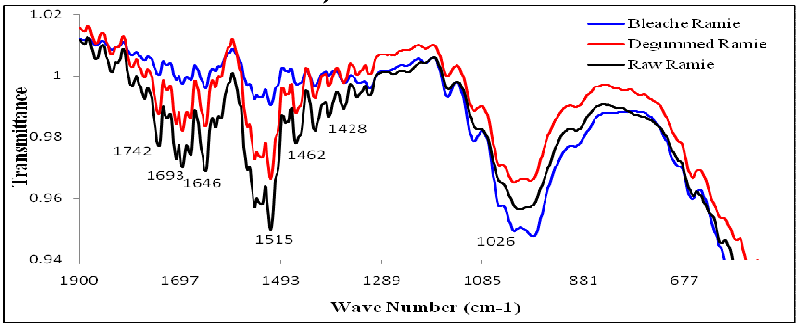
Figure 4: SEM Images of Roselle Fibre.

FTIR Analysis Fibres

The FTIR spectrum of raw, degummed and bleached roselle and ramie fibres is shown in Fig.5. The sharp peak indicates C=C aromatic symmetrical stretching of lignin in raw roselle and ramie fibres. The reduction in the intensity of these peaks indicates the removal of lignin during degumming. The carbonyl stretching vibrations of ester and carboxyl groups in hemicelluloses are attributed to the small band at 1744 and 1742 cm−1 Basu *et al.*[14]. The peak of both the spectrum shows deformation of lignin in raw fibre. The absence of this peak in degummed fibre indicates the removal of lignin[15]. In the figure is it shown that, the intensity of the peak is higher in the degummed fibre than raw fibre. It indicates that, the cellulose content of the fibre got increased during bleaching due to the removal of no cellulosic components like lignin, hemicelluloses and pectin's.



a). Roselle Fibre



b). Ramie Fibre

Figure 5: FTIR Analysis.

CONCLUSIONS

Roselle fibres are comparable with other established bast fibres in terms of their physical, chemical, tensile, and thermal properties. Fibre extracted from *H. sabdariffa* by water retting followed by alkaline degumming. Bleaching was performed to enhance the aesthetic value of the fibre. Chemical treatments enhanced the fibre fineness, density, crystallinity, and water absorbency of the roselle and ramie fibre. The removal of hemicellulose as well as lignin from the raw fibre during degumming and bleaching was confirmed by chemical component analysis, FTIR, and SEM analysis. The removal of gummy substance from the fibre surface by chemical treatments was confirmed by SEM analysis. By comparing all the properties, it may be said that, the blending of the roselle-ramie can take place for the production of good quality textiles.

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