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An Innovative and Sustainable Approach for Quantifying Cotton and Ramie Fibre Blend Ratios

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Abstract

Cotton, a widely used cellulose fibre, is often blended with other fibres such as Ramie, Jute and Hemp to enhance textile performance. However, a reliable method for accurately determining the blend ratio of these fibres is lacking. This study addresses this gap by utilizing the moisture regain properties of these fibres to quantify blend ratios. Various blends in different ratio of Cotton and Ramie were created, and their composition was confirmed using FTIR spectral analysis and Xray-diffraction. The Moisture regain values of these blends were then measured under varying humidity and temperature conditions using standardized testing methods. A calibration curve was established to correlate blend percentages with their respective moisture regain values, enabling precise determination of unknown blend ratios. This research offers a dependable method for quantifying fibre blend ratios of Cotton and Ramie, contributing to the development of standardized techniques for assessing and characterizing fibre blends, and facilitating improved control and utilization in textile applications.

Keywords: Calibration curve, Cotton, blend, Ramie, moisture regains

INTRODUCTION

The increasing global awareness and societal concern about the environmental consequences of the textile industry have underscored the urgent requirement to adopt eco-friendly and sustainable practices across the industry's supply chain. In response to escalating population growth and the higher demand for textile fibres, it has become imperative to explore sustainable raw materials and processes to address these challenges (Felgueiras et al., 2021)**.** Cotton is the most widely used natural fibre, representing about 90% of all natural fibres and being extensively utilized in apparel, home furnishings, and industrial applications (Yu, 2015). However, other fibres like flax, ramie,

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jute, kenaf, and sisal are also used to meet the increasing demand. Blending cotton with fibres such as Ramie, jute, hemp, and banana not only enhances the quality of the final product but also helps conserve cotton for other uses. Environmentally, it is important to gradually replace cotton with pulp-based fibres. Although cotton covers only 2.4% of arable land, it dominates the global insecticide market. Among alternative textile materials, ramie is considered a sustainable substitute for cotton due to its lower water requirements. In agriculture, ramie acts as a natural pesticide, effectively controlling pests and diseases. Its high productivity in terms of fibre, biomass, and nutritional quality has made it increasingly valuable as an industrial crop. Ramie's unique properties, such as resistance to microbial attacks, high strength, durability, absorbency, and excellent lustre, make it ideal for a range of high-value products, including clothing fabrics (Rehman et al., 2019).

The chemical composition of Ramie fiber consists of cellulose (67-99%), hemicellulose (13.1-16.7%), pectin (1.9-2.1%), lignin (0.5-1%), and fat & wax (0.3%) (RAMIE FIBRE, n.d.). Various studies have explored the structural properties and applications of Cellulosic fiber, including its surface morphology, physical characteristics, and potential uses (Wang et al., 2009) (Garside & Wyeth, 2003). However, there is currently no method available for quantifying fibers in blends of Ramie and Cotton. While several techniques, such as chemical, microscopic, and gravimetric methods, exist for identifying and quantifying blends like cotton/polyester, cotton/wool, and cotton/viscose (Greaves, 1990) (AATCC 20A Test Method for Fiber Analysis: Quantitative – AATCC Test Methods, n.d.), quantifying blends involving unconventional fibers like Ramie, Jute, Banana, etc. is challenging due to the fact that both Cotton and these unconventional fibers are cellulose-based. The lack of a standardized method for quantifying Ramie/Cotton blends highlights the need for developing a reliable procedure to accommodate the growing use of these blend types. In this study, the moisture regain property of Ramie and Cotton fibers was employed to determine the blend percentage.

MATERIALS AND METHODS

For this study cotton and Ramie fibres and Yarn were sourced from NITRA. Yarn and Fabric of unknown blend ratio of Cotton and Ramie was purchased from market.

Five blend ratios of Cotton and Ramie (80%Cotton:20 %Ramie, 60%Cotton%: 40% Ramie, 50% Cotton: 50% Ramie, 40% Cotton:60%Ramie, 20% Cotton:80% Ramie) were prepared at the

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NITRA pilot plant. Along these blend 100% Cotton and 100% Ramie fibre were also taken for study. Two known blends of Cotton and Ramie fibres were also produced: one as a yarn by twisting yarn of both the fibres, and the other as a fabric using the union method, with Cotton Yarn as the warp and Ramie Yarn as the weft.

Identification of Fibres

Identification of fibres was performed following AATCC 20 guidelines, utilizing several methods including the burning test, solubility test, microscopic analysis, FTIR, and X-ray diffraction.

Burning Test: To evaluate the burning behaviour of Cotton and Ramie fibres, small samples of each fibre were held with tweezers and exposed to a small flame from a spirit lamp. Observations were made regarding characteristics such as melting, shrinkage, and continuous burning. Additionally, the type of odour produced during burning was noted.

Solubility Test: The solubility of Cotton and Ramie fibres was tested by placing 10 mg of each fibre into separate test tubes, followed by the addition of 1 ml of 70% sulfuric acid to each tube.

Microscopic Analysis: The longitudinal and cross-sectional structures of the fibres and their blends were examined using a Zeiss high-resolution projection microscope.

Fourier Transform Infrared (FTIR) Study: The presence of Cotton and Ramie fibres was confirmed using Fourier Transform Infrared Spectroscopy with the ATR technique on a Perkin Elmer UATR TWO instrument. FTIR spectra were recorded over a range of 450 cm^{-1} to 4000 cm^{-1}.

X-Ray Diffraction Test: X-ray diffraction was used to determine the degree of crystallinity of the fibres. This method helped characterize Cotton and Ramie fibres and their blends, as each fibre type exhibits a distinct crystallinity index due to differences in polymer arrangement.

Moisture Regain

The moisture regain of the fibres and their blends were determined following the guidelines specified in IS 199 test methods. First, an accurately weighed specimen of the fibre/blend sample was placed in a clean and dry tared weighing beaker. This beaker, along with the test specimen, was then placed in a drying oven and heated at 105 ±3°C until a constant mass was achieved. The oven dry mass of the specimen was determined through this process. Next, the fibres/blends samples were individually exposed to three different relative humidity (RH) and temperature conditions. These conditions included 65 ±% RH and 27 ±2°C, 80 ±2% RH and 20 ±2°C, and 50

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±2% RH and 30 ±2°C, which were maintained in a conditioning chamber for 24 hours. Three readings were taken for each sample, and the average was calculated.

Based on the obtained data, a calibration curve was plotted, correlating the blend percentage of fibres with their corresponding moisture regain at different RH and temperature conditions. The same process was repeated for known and unknown blends samples of cotton and Ramie at standard conditions.

Statistical Analysis

The experimental data obtained from the study were analysed using the statistical software SPSS (version 20). The null hypothesis (H0) proposed that there is no relationship between the percentage of fibre blend and moisture regain. Conversely, the alternative hypothesis suggested the existence of a potential relationship between the fibre blend percentage and moisture regain. The null hypothesis (H0) would be rejected if the p-value obtained from the statistical analysis is lower than the predetermined significance level, typically set at 0.05.

RESULT AND DISCUSSION

Identification of fibres in the known and unknown blend:

Burning test: The burning test conducted in the study revealed that fibre and their known and unknown blends did not exhibit melting or shrinking behaviour, resembling the burning characteristics of paper. This experimental test provided evidence that the fibres and blends consisted of cellulosic fibres.

Solubility test: The solubility test demonstrated that all fibres and their blends were successfully dissolved when exposed to 70% sulphuric acid. This additional test confirmed that the fibres were indeed of cellulosic nature.

Microscopic test: To further examine the fibres, both the known and unknown blends were subjected to microscopic evaluation. Longitudinal and cross-sectional structures of the fibres were observed under the microscope. The fibres were extracted from both the known and unknown blends. Figure.1 displays the structures of the fibres, with Fig. 1(a) and Fig. 1(b) showed the longitudinal and cross-sectional views of cotton fibres, respectively.

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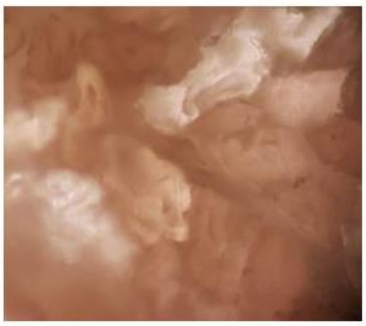
The longitudinal view of ramie fibre is irregular, displaying cross markings, as depicted in Figure 1(c). This view highlighted the unique irregularities and patterns typical of ramie fibres. In contrast, Figure 1(d) presented the cross-sectional view, revealing elongated, curved, and flattened fibre cells. These characteristics are distinctive to ramie fibres and contribute to their structural properties. (Kalia & Sheoran, 2011). Longitudinal and cross-sectional view of fibre extracted from cotton/Ramie blend showed the characteristics of both Cotton and Ramie fibreFigure1(e) and 1(f). From this analysis, it is clear that the fibres are Cotton and Ramie.

(a) Longitudinal view of cotton Fibre

(b) Cross-sectional view of cotton fibre

(C) Longitudinal view of Ramie Fibre



(d) Cross-sectional view of Ramie fibre

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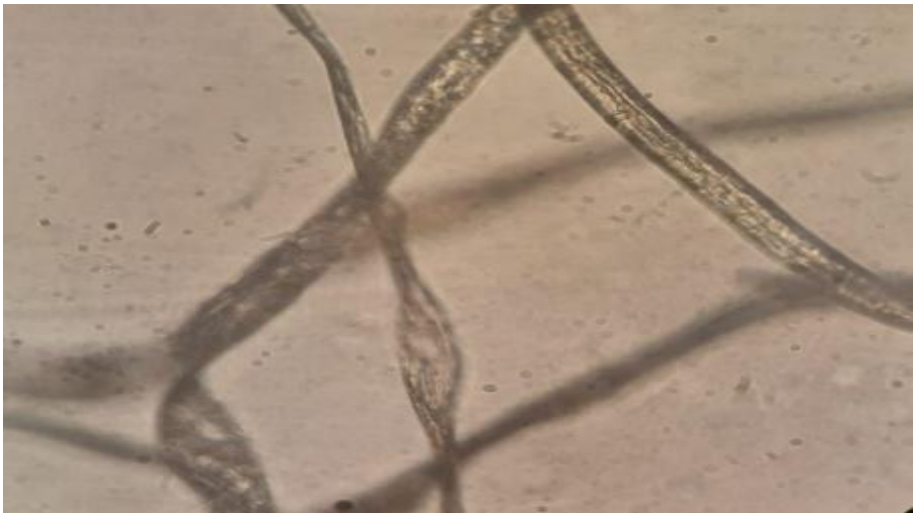

(e)Longitudinal view of Cotton Ramie blend fibre

f) Cross-sectional view of Cotton Ramie blend fibre

Fig.1 Longitudinal and cross-sectional view of Cotton (a and b), Ramie (c and d) and fibres from their blend (e and f)

Fourier transforms infrared (FTIR) study:

To further support microscopic analysis and FTIR study was carried out of cotton and Ramie fibre and their blends. The result of FTIR study of blends was compared with FTIR of individual Cotton and Ramie fibres. FTIR spectra of Cotton, Ramie and Blends are shown in Fig-2. Typical Functional group and their corresponding wavenumbers are given in Table-1.

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The involvement of certain fibre molecule functional groups is an important characteristic of any lignocellulosic fibre, such as Ramie fibre, and can be investigated using Fourier Transform Infrared (FTIR) spectroscopy. The primary components of any lignocellulosic fibre, cellulose, hemicelluloses, and lignin, are represented by the FTIR spectra of alkanes, esters, aromatic ketones, and alcohols with various oxygen-containing functional groups present in it (Simonassi et al., 2017)

A broad peak around 3330 cm^{-1}(Table-1) indicates the presence of the –OH functional group in cellulose, common to all fibers analyzed (da Silva et al., 2016). Peaks near 2850 cm^{-1} and 2901 cm^{-1} correspond to aliphatic –CH stretching in cellulose and hemicellulose (Cecci et al., 2020), while a peak at 1646 cm^{-1} is linked to –OH bending in absorbed water (Cecci et al., 2020). A peak between 1508 and 1587 cm^{-1}, found in ramie, suggests lignin presence, which is absent in pure cotton due to its lack of lignin (Jose et al., 2016). Additional peaks at 1430 cm^{-1}, 1370 cm^{-1}, and 1280 cm^{-1} are associated with cellulose and hemicellulose structures (Jose et al., 2016). The peak at 898 cm^{-1}, indicative of β-glycosidic linkages, provides further structural details, while peaks at 894 cm^{-1} and 661 cm^{-1} correspond to COC, CCO, CCH groups, and C-OH bending, respectively (Simonassi et al., 2017).

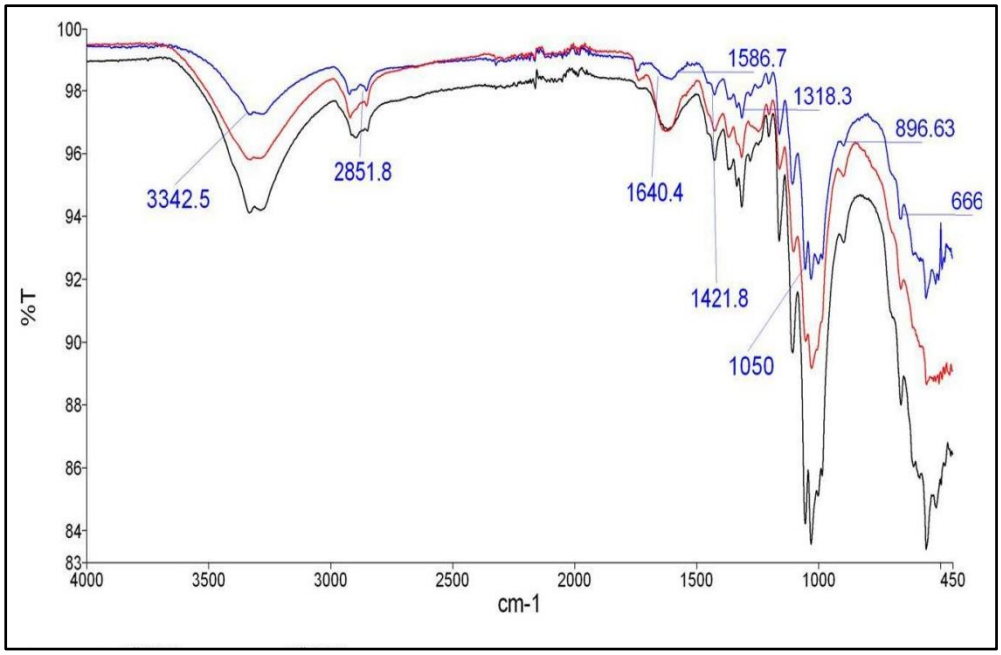

Fig. 2 FTIR spectra of ● Cotton ● Ramie ● Cotton Ramie Blend

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Wavenumber(cm-1) of cotton as per Fig.2	**Wave number (cm-1) of Ramie as per Fig.2**	**Wave number (cm-1) of cotton and Ramie blend as per Fig.2**	**Peak Characteristics**	**References**
3330	3352	3342	OH stretching	(da Silva et al., 2016)
2857	2860	2851	CH_2 and CH_3 stretching	(Cecci et al., 2020)
1635	1735	1640	H-O-H bending of absorbed water	(Cecci et al., 2020)
-	1545	1586	Benzene ring Stretching (Lignin)	(Jose et al., 2016)
1427	1436	1421	CH2 bending in lignin	(Jose et al., 2016)
1370	1374	1318	CH3 bending (deformation stretch)	(Jose et al., 2016)
1274	1205	1239	C-O stretching of acetyl(lignin)	(Jose et al., 2016)
1028	1024	1050	C-O stretching in cellulose, hemicellulose and lignin	(Simonassi et al., 2017)
890	897	898	B-Glucosidic linkage	(Simonassi et al., 2017)

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663	659	666	out of –plane OH bending	(Simonassi et al., 2017)

Table:1 IR Absorbtion frequencies of Cotton, Ramie and their blend

Characterisation of fibres by x-ray diffraction method:

The crystallinity index (CI) is a parameter used to quantify the relative amount of crystalline material in cellulose. Fibre strength is not solely dependent on cellulose macromolecular weight but also heavily relies on fibre crystallinity. Higher crystallinity greatly contributes to fibre strength. (Pervez et al., 2017).

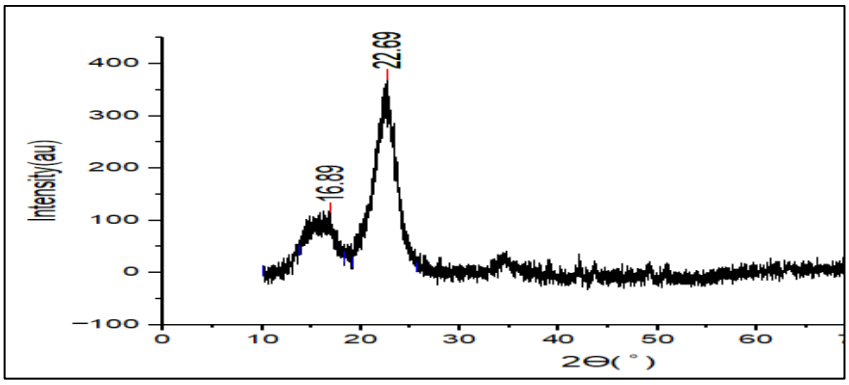
The crystallinity index (Ic) of the fibre was calculated using the following formula coming from the empirical method described by (Segal et al., 1959)

$$Ic = \frac{I22^{o} - I18^{o}}{I22^{o}} \times 100$$

The counter reading of peak intensity at 22º (at 2 theta scale) represents crystalline material and the peak intensity at 18º (at 2 theta scale) corresponds to amorphous material.

The Crystallinity Index of cotton was calculated as 85.79% using the Segal equation, which is similar to the 84.7% and 85% values for untreated cotton reported by (Nam et al., 2016)

The Crystallinity Index of ramie was calculated as 80.08%, similar to the 79.75% value reported by (Akbar et al., 2020) using X-ray diffraction.



a) XRD graph for Cotton fibre

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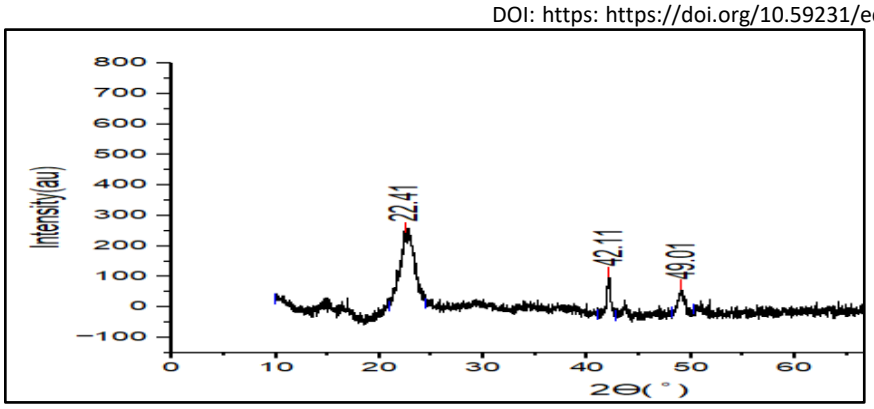
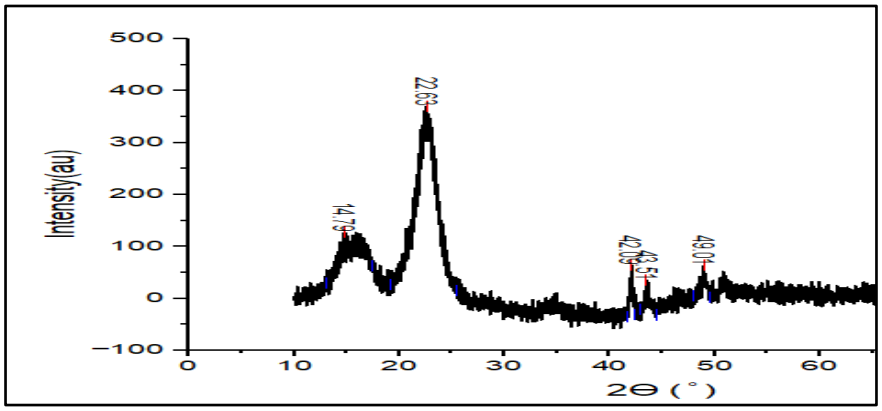


b) XRD graph for Ramie fibre



c) XRD graph of fibre from Cotton and Ramie Blend

Fig. 3 X-Ray Diffraction graphs of Cotton(a), Ramie(b) and Cotton and Ramie Blend(c)

Table2: Crystallinity Index of Cotton, Ramie and their Blend

S.No.	Fibre	I_{22} (at 2 theta scale)	I_{18} (at 2 theta scale)	Crystallinity %
1	Cotton	241	85	85.79%
2.	Ramie	387	111	80.08%
3.	Cotton/Ramie	366	130	81.41%

Crystallinity Index of Cotton and Ramie blend showed a decrease in crystallinity index compared to pure cotton. Specifically, the crystallinity index of blends was lower than that of 100% cotton and closely approximated the crystallinity index of the Ramie fibres blended with cotton. However, it is important to note that no references were provided to validate these observations.

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3.2 Moisture Regain of Fibre Blend

The moisture regains of cotton, Ramie and their prepared blend at fibre stage was measured according to the IS199 standard after being exposed to different conditions for 24 hours. These conditions include 65±2% RH and 27±2°C temperature, 80±2% RH and 20±2°C temperature, and 50±2% RH and 30±°C temperature. Moisture Regain value are tabulated in table:3 The effect of relative humidity and temperature on moisture regain was shown in Figure 3.

Table:3 Moisture Regain of Cotton/Ramie Blend at Different RH* and Temperature

S. No	Blend Percentage	Moisture Regain at 65%RH* and 27%T**	Moisture Regain at 80%RH* and 20%T**	Moisture Regain at 50%RH* and 30%T**
1	100%Cotton	8.03503	11.8827	5.3274
2	80%Cotton20%Ramie	8.9436	12.5616	6.0342
3	60%Cotton 40%Ramie	9.6453	13.2687	6.7379
4	50%Cotton50%Ramie	10.1981	13.6184	7.0903
5	40%Cotton60%Ramie	10.6598	13.9703	7.4426
6	20%Cotton80%Ramie	11.6542	14.6723	8.1467
7	100%Ramie	12.5573	15.3746	8.8513
RH*-Relative Humidity, T**- Temperature				

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From the fig.4, it is evident that as the percentage of Ramie fibre in the blend increases, the moisture regain percentage of the blend also increases. Additionally, with an increase in humidity percentage, the moisture regain of the fibres also increases.

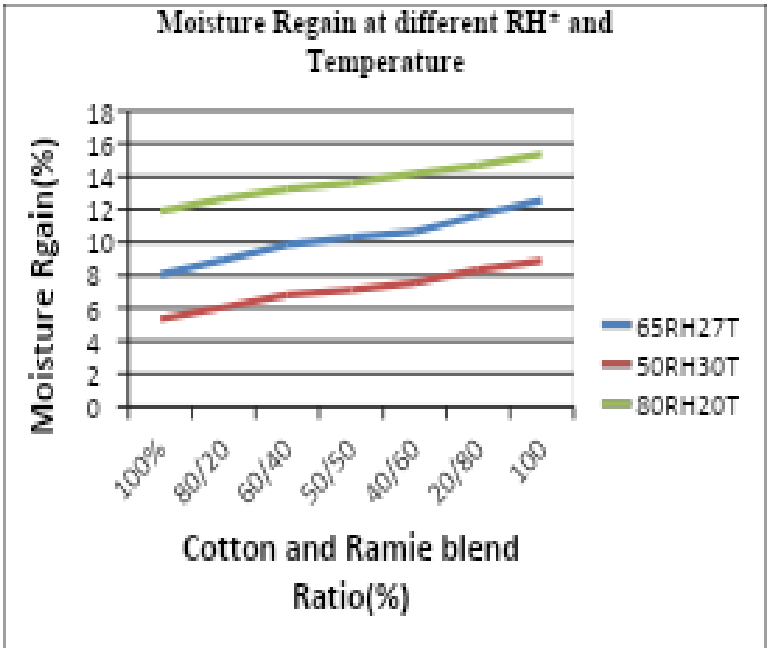

Fig. 4 Comparison of Moisture regain (Cotton/Ramie blends) at different RH and Temperature

*RH-Relative Humidity

Since the exposure conditions of 65±2% RH and 27±2°C are standard for testing textile fibres according to IS:6359, these conditions were selected for further experiments. A calibration curve (Fig. 4) was plotted to depict the relationship between various fibre blends and moisture regain. This calibration curve serves as a reference for determining the blend ratio of unknown blends.

3.3 Statistical Analysis

The analysis of the relationship between blend percentage and moisture regains in the blend of cotton and Ramie using the ANOVA test at standard conditions (65±2% RH and 27±2%℃ temperature) resulted in the rejection of the null hypothesis (Ho). This rejection was based on the p-value being lower than the predetermined significance level of 0.05.

Furthermore, the regression coefficient (R2) value of 0.991 was obtained for the blend of cotton and Ramie. This high R2 value indicates a strong relationship between the blend percentage and moisture regain in the blend.

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Table:4 Univariate Annova between Blend Percentage (Cotton/Ramie) vs. Moisture regain

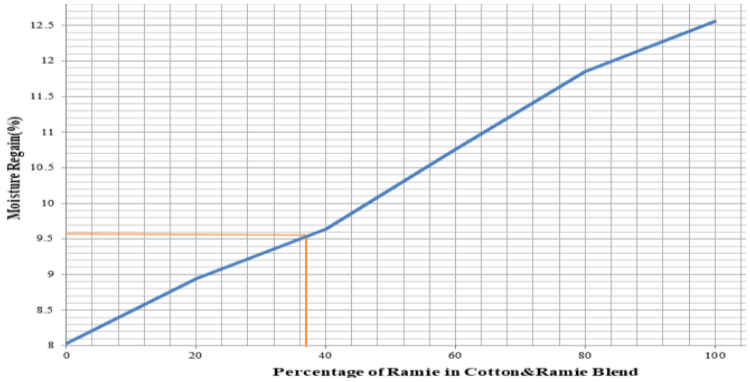
Dependent Variable: Moisture Regain

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	43.735a	6	7.289	265.692	.000
Intercept	2210.240	1	2210.240	8.056E4	.000
Blend percentage	43.735	6	7.289	265.692	.000
Error	.384	14	.027		
Total	2254.359	21			
Corrected Total	44.119	20			

a. R Squared = .991 (Adjusted R Squared = .988)

3.4 Moisture Regain of Known and Unknown Blend

In Table 5, the moisture regains values of the prepared blended yarn and fabric with known ratio -60% Cotton40% Ramie and 70% Cotton and 30% Ramie respectively and unknown blends procured from market sample-1(Yarn) and sample 2(Fabric) were determined using the standard method and compared to the calibration curve in Figure 4. The intersection point on the calibration curve represents the blend ratio. To validate this new approach to determine blend composition, two known blends were also prepared (yarn and Fabric), and their results are provided in Table 5.



a) Known Cotton 60% Ramie40% blend (Yarn)

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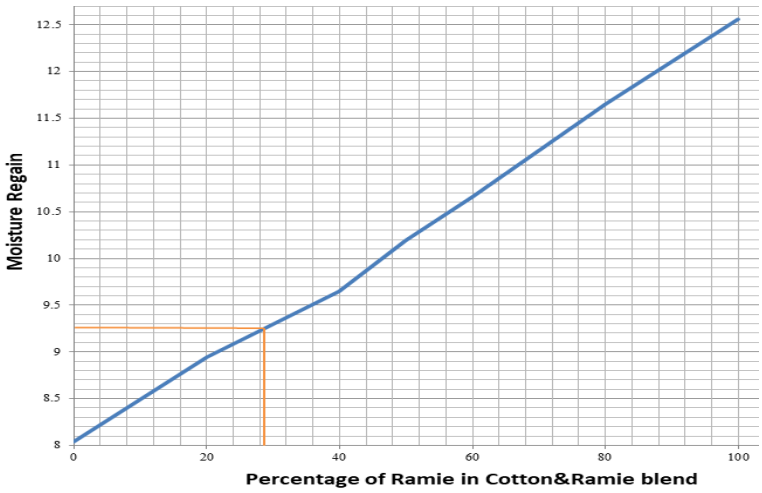

b) Known Cotton 70% Ramie30% blend (Fabric)

Table- 5: Moisture regain of known and Unknown blends

Cotton /Ramie blend Ratio	**Moisture regain (%) as per standard method**	**Cotton/Ramie Blend Ratio as per new approach**
	65%±2%RH 27°C±2°Temperature	
60%Cotton40% Ramie (Yarn)	9.5767	63%Cotton37% Ramie
70%Cotton30%Ramie (Fabric)	9.2718	71% Cotton29% Ramie
Unknown Sample-1(Yarn)70%Cotton30%Ramie (as per source)	8.9308	80%Cotton20%Jute
Unknown Sample -2(Fabric)45% Cotton 55% Ramie (as per source)	10.25433	49%Cotton51% Ramie

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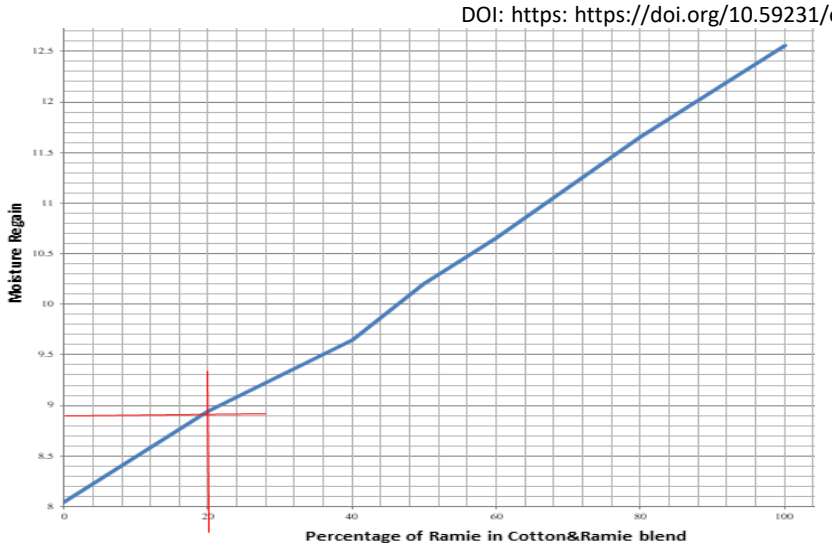
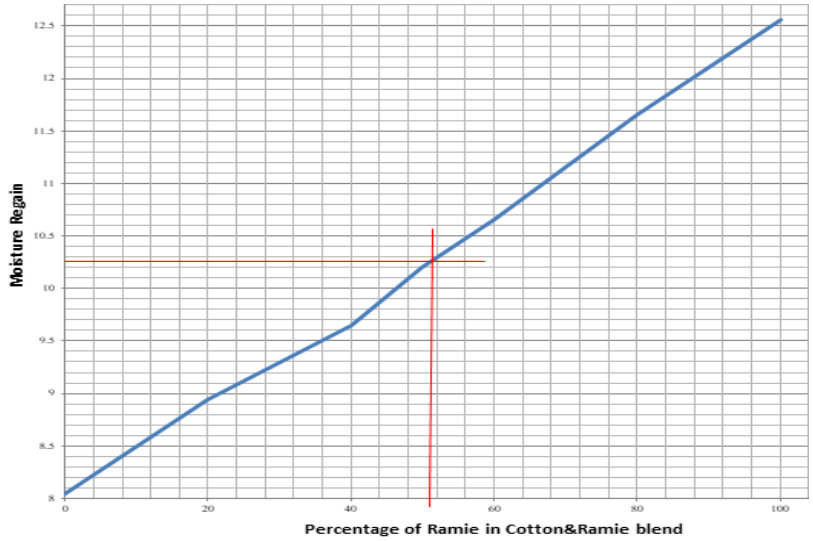


S-1 Unknown blend of Cotton and Ramie (Yarn)



S-2 Unknown blend of Cotton and Ramie (Fabric)

Fig 5. Blend percentage of Cotton and Ramie in known and Unknown blends

CONCLUSION

1. Based on the qualitative studies conducted, including burning, solubility, and dyeing tests, it was determined that both fibres in the blends are of cellulosic nature. The burning test demonstrated that the fibres burned similarly to cellulose, without melting or shrinking. The

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solubility test further supported this finding, as the fibres dissolved in sulphuric acid, a characteristic of cellulose-based fibres.

2. Microscopic analysis of the fibres longitudinal and cross-sectional views revealed the presence of both cotton and Ramie fibres in the blend, confirming the visual identification.

3.The Fourier transform infra-red (FTIR) spectra of the cotton and jute fibres exhibited similarities, with the only notable difference in the Ramie fibre spectra being the presence of a peak at 1508cm-1, indicating the presence of lignin. This peak was also observed in the cotton-Ramie blended fabric, further confirming the inclusion of Ramie in the cotton-Ramie blend.

4. X-Ray diffraction showed that cotton has the highest crystallinity among the studied fibre and blend.

5. Through the univariate ANOVA analysis of blend percentage versus moisture regain, the null hypothesis (Ho) was rejected, as the p-value was lower than the predetermined significance level of 0.05. The high rejection coefficient (R2) value of 0.990 indicated a strong relationship between the blend percentage and moisture regain.

6. The analysis of the known and unknown blends using a novel blend analysis approach revealed that the values of the known blends closely matched the actual blend percentages of cotton and Ramie fibres. For the unknown blends, the new approach determined that one contained approximately 80% cotton and 20% ramie, while the other had about 49% cotton and 51% ramie, in comparison, the labels on the unknown yarn and fabric indicated 70% cotton and 30% ramie, and 45% cotton and 55% ramie, respectively. This discrepancy suggests that the manufacturer did not account for fibre wastage when labelling the blend ratios, an issue that was negligible in the known blends.

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