

## Reclaiming peracetic acid bleach baths for sequential bleaching of lignocellulosic fibres

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This study presents an innovative and eco-friendly process using peracetic acid (PAA) for lignocellulosic fibres viz., banana, flax, jute and ramie at lower temperatures (70-76 °C) and pH conditions (7-8). Bleaching of these fibres with different proportions of cellulose, hemicellulose and lignin is carried out using optimised recipe for the individual fibres using statistical design. After bleaching, the whiteness index (WI) of all the fibres significantly improves with maximum retention of fibre strength (75 – 83 %). To make the process sustainable, the remnant PAA bleach bath is reclaimed twice for bleaching scoured fibres. Results show that the left-out liquor can be reused effectively twice to produce semi-bleached (WI: 64.5 - 69.1) to quarter-bleached (WI: 54.2 - 64.1) fibres, which may be dyed in medium to darker shades. The concentration of PAA in the bath is gradually depleted in the successive bleach baths, resulting in maximum utilisation of PAA. The discharged bleach liquor is assessed for total dissolved solids, turbidity, chemical oxygen demand, pH, electrical conductivity and residual PAA, keeping in mind the sustainability of the process.

**Keywords:** Bleaching, Flax, Lignocellulosic, Ramie, Reuse, Whiteness index

### 1 Introduction

Different agro-climatic zones exist in various parts of our country, where varieties of fibre-yielding plants are cultivated or grown naturally. India is blessed with many fibre-yielding plants that produce several distinct natural cellulosic and lignocellulosic fibres. A few popular lignocellulosic fibres include jute, flax, ramie, banana, sisal, pineapple, hemp and nettle, which are used for making textile and non-textile products for different industrial applications<sup>1-5</sup>. In the category of textile end uses, comparatively coarser yarns are produced from these fibres than cotton yarn. Such yarns are mainly used in making packaging materials, low-cost bags, and handicraft items, i.e., in the development of low-value products. The key potentials of these fibre products are biodegradability, environment friendliness, renewability, availability in plenty, and rationally low cost<sup>6</sup>. These fibres are preferred for making a number of value-added products over their traditional products portfolio after suitable chemical and mechanical intervention. In recent times, a lot of work has been carried out to produce finer yarns after suitable blending with finer fibre, improved extraction process for obtaining finer

fibres, chemical modification to make finer and softer fibre, enzyme treatment of fibres to produce ethnic home furnishing material, lifestyle products, fashion accessories, upholsteries and even outer garments<sup>7-12</sup>.

From a chemical composition point of view, ramie and flax fibres contain lower lignin percentages than banana and jute fibres. Such fine fibres are produced naturally during the bio-synthesis in the plant body. To produce value-added textile products, requisite chemical modification of coarser (jute & banana) as well as finer fibres in terms of degumming, scouring, bleaching, dyeing, printing, finishing, etc., are very important, as these chemical treatments have profound effects on fibre's colour, feel, look, fineness and rigidity<sup>8</sup>. As far as bleaching is concerned, hydrogen peroxide, an oxidative bleaching agent, is generally used for discolouration/de-pigmentation of cellulosic and lignocellulosic plant fibres and also protein fibres (silk & wool) to change their natural yellow colour to milky white colour. Degumming/scouring followed by bleaching could also enhance the liquid absorbency of the fibre due to the removal of natural and added oil, fat and wax from the fibre surface. Hydrogen peroxide bleaching is conventionally practised at high alkaline conditions and temperatures of 80–90 °C. Such a bleaching process, although it leads to the achievement of milky

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white and absorbent fibre, also deteriorates the fibre quality as far as strength is concerned. It is more important in the case of lignocellulosic fibres, as those fibres are more sensitive to alkaline solutions at elevated temperature, resulting in a significant loss in fibre's weight and strength due to partial removal of hemicelluloses and lignin macromolecules. Research has been carried out to bleach lignocellulosic fibres at much lower temperatures using hydrogen peroxide to maintain their original properties to the possible extent<sup>13-15</sup>. Research has been carried out to reduce the number of chemical processing steps in the textile process so that the material handling can be reduced, and the water, as well as thermal energy, are judiciously used to reduce the processing costs and improve the fibres' physical properties<sup>16-17</sup>. In this context, the reuse of bleach bath, simultaneous bleaching-dyeing, simultaneous alkali treatment-bleaching, and reuse of ambient temperature bleach bath are some of the essential attempts made in the case of lignocellulosic plant fibres<sup>13,18-20</sup>. PAA, a strong oxidising bleaching agent with high reactivity, may also be used to bleach lignocellulosic fibres swiftly at lower temperatures and mild alkaline conditions<sup>21</sup>. It is an environment-friendly bleaching agent that is fast-acting, non-foaming, and water-soluble<sup>22</sup>. The PAA bleaching of the textile substrate is a little costly compared to hydrogen peroxide ( $H_2O_2$ ) bleaching, and the improvement in whiteness indices is also a little lower compared to ( $H_2O_2$ ) bleaching. In the water bath, PAA remains in equilibrium with hydrogen peroxide and acetic acid, and the bleaching mainly occurs due to epoxidation of double bonds of impurities and coloured compounds to produce white fibres with retention of maximum fibre's inherent properties. Scanty and sporadic research has been reported on PAA bleaching of cotton, jute fibres and abaca pulp, followed by its effect on optical, physical and structural properties<sup>21,23-26</sup>. However, the detailed study on PAA bleaching of flax and ramie fibres has not been reported elsewhere to our knowledge.

An alternate source of bleaching agent having an effective output on whiteness indices with retention of maximum tenacity is very much needed for lingo-cellulosic fibres like banana, jute, flax and ramie, which are now being used as biodegradable textile materials. So, an effort was directed to study the effect of peracetic acid (PAA) as a bleaching agent on these fibres' physical, optical and structural

properties. Moreover, in order to make the process sustainable, bleach baths have been reused, and the progressive changes on different properties of fibres have also been studied. A comprehensive study of such types is scanty and needs to be explored. In this study, the bleaching efficacy of PAA on four important lignocellulosic fibres, viz., banana, flax, jute and ramie, have been studied using the optimised bleaching recipe obtained from Taguchi-experimental-design (TED), altering the PAA concentration, time, temperature and pH. The novelty of the process is to make the bleaching process more sustainable and economical, the bleach bath is reused two times without the addition of any bleaching chemicals. The process requires less chemicals and water, as the same water bath is used successively thrice. Water quality parameters, before and after the bleaching, are studied thoroughly. The research finding postulates that the fibre bleached in the reused baths may be used for dyeing in medium to dark shades to economise the process while reducing the water and chemical requirements. Important optical, physical, tensile, and chemical properties of the unbleached and bleached fibres were evaluated after each chemical treatment to study the effectiveness of reuse bleach bath.

## 2 Materials and Methods

### 2.1 Materials

The flax and ramie fibres were procured from the ICAR-Central Institute of Research on Jute and Allied Fibres, Barrackpore, West Bengal, India. Banana fibre was procured from the state of Maharashtra, India in reed form, and the raw jute fibre (*Corchorus olitorius* L.) of TD - 4 grade as per IS 271-2003 was procured from the Jute Corporation of India, West Bengal. These fibres were initially scoured for cleaning and then used for studies on PAA bleaching.

### 2.2 Chemicals

For the bleaching of banana, flax, jute and ramie fibres commercial grade PAA with concentration of 35 % (w/v) and specific gravity of 1.12-1.13 g/cc was procured from the Chemtex Speciality Limited, Kolkata, West Bengal, India. Tetrasodium-pyrophosphate (TSPP), sodium carbonate and acetic acid of analytical grade were purchased from the MERCK, Mumbai, India Ltd. The laboratory-grade ultravon JU (non-ionic surface-active agent) was procured from the Charminar Enterprise, Kolkata.

### 2.3 Mild Scouring and PAA Bleaching

All the fibres viz., banana, flax, jute and ramie were initially treated with 2 % sodium carbonate (on weight of fibre) and 2 g/L Ultravon JU, a non-ionic surface-active agent at a temperature of 50 °C for 30 min, keeping MLR ratio of 1:20. Ramie fibre, as it contains gummy material, prior to scouring it was degummed with 10 % NaOH. Bleaching of mild scoured fibres was performed in an enclosed vessel with a fibre-to-water ratio of 1:30 in the presence of tetrasodium pyrophosphate (TSPP): 3 g/L, non-ionic surface-active agent: 0.5 mL/L and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>): 1.8 g/L, by altering the three important bleaching parameters viz., time from 60 to 180 min, temperature from 60 to 80°C and PAA concentration from 10 to 30 g/L to optimize the bleaching recipe using Taguchi-Experimental-Design (TED). During the bleaching, pH was adjusted using sodium carbonate and TSPP. After bleaching, the fibres were washed thoroughly in clean water and finally dried in air. The original bleach bath was utilised two times for successive bleaching of scoured fibres for effective utilisation of different chemicals and water, wherein in the first reuse bath, nothing was added, and in the second reuse bath, the pH and MLR ratio were adjusted.

### 2.4 Fibre Properties Evaluation

#### 2.4.1 Optical Properties

The whiteness index (WI) and yellowness index (YI) of all the raw, scoured and bleached fibres were measured on the 'HUNTER' and 'ASTM D 1925' scales, respectively, using Spectrascan-5100 computerised colour matching system. Other colour parameters viz., colour strength (*K/S*), *L\** (lightness -darkness), *a\** (red–green colour) and *b\** (yellow–blue colour) were also measured in the same equipment in the presence of D65 illuminant and 10° angle of observer. A Canon 5D Mark III SLR camera (Japan) was used to capture the optical images of the fibres. The reported whiteness index, yellowness index, *K/S*, *L\**, *a\** and *b\** colour values are the average three replications and three readings on each sample at different locations.

#### 2.4.2 Bundle Strength

The bundle strength of unbleached and bleached banana, flax, jute and ramie fibres were measured after conditioning the sample in a standard atmosphere of 65±2 % relative humidity, 27±2 °C temperature for 48 h following the BIS recommendation (IS: 6359-1971). Bundle strength of

the fibre was measured using two unequal arms-based bundle strength testers, working on the principle of beam balance following BIS, 1986<sup>8</sup>. The reported values are the average bundle strength of 15 fibre replications.

#### 2.4.3 Scouring and Bleaching Weight Loss

The alteration in fibre sample weight owing to the scouring and bleaching of four different bast fibres was gravimetrically estimated by measuring the sample weight before (*M*<sub>1</sub>) and after (*M*<sub>2</sub>) various chemical treatments. Then, the weight loss percentage was calculated using the following expression: weight loss% = [(*M*<sub>1</sub>-*M*<sub>2</sub>)/*M*<sub>1</sub>] × 100. The reported weight loss percentage is the average of five replications.

#### 2.4.4 Fibre Fineness Measurement

Fibre fineness in terms of linear density, i.e., mass per unit length of the fibre after each chemical treatment (as procured, scouring, bleaching, bleaching in reuse bath), was measured by the standard gravimetric method as reported in literature<sup>27</sup>. In the measurement, 2 mm fibre samples were initially cut from different parts of the fibre, and then they were mixed. The weight of such 100 cut fibres was taken to calculate the 'tex' value, considering the weight of 1000 m fibre length in grams. The reported result is the average of three replicas.

### 2.5 Chemical Analysis Fibre and Bath

The Fourier transform infrared spectroscopy (FTIR) of the banana, flax, jute and ramie fibres was carried out in FTIR-Prestige 21 Bruker with HATR multiple reflection accessory using ZnSe prism over the wavelength of 500 to 4000 cm<sup>-1</sup>, Make: Bruker, Germany, Model – ALPHA in attenuated total reflection (ATR) mode. Cellulose, hemicellulose and lignin content in the raw and PAA bleached fibres were estimated following TAPPI method 17 for cellulose, TAPPI method 223 for pentosan (hemicellulose) and TAPPI method 222 for lignin. For the measurement of PAA concentration in the original and reuse bleach baths, a UV-visible spectrophotometer was used. It is estimated in the principle of rapid oxidation of excess potassium iodide by PAA in the presence of ammonium molybdate to change to yellow-coloured triiodide ions (I<sub>3</sub><sup>-</sup>) that could be quantitatively measured at a wavelength of 350 nm in UV-Vis spectrophotometer<sup>28</sup>. The concentration of the PAA in the different bleached baths was calculated using the below equation:

$$\text{PAA concentration (in ppm)} = \frac{A_{350} \cdot V_{\text{final}}}{K \cdot V_{\text{sample}}} \dots (1)$$

where  $K$  is the slope of calibration curve;  $A_{350}$ , absorbance of sample at 350 nm wavelength;  $V_{\text{final}}$ , final volume after the addition of all reagents in quartz cell (2.5 mL); and  $V_{\text{sample}}$ , volume of original bleaching liquid added into the 1 cm quartz cell (1.0 mL).

**2.6 Estimation of Bleach Bath Water Parameters**

Various water quality parameters obtained from the original, after bleaching, after 1<sup>st</sup> reuse and 2<sup>nd</sup> reuse bleach baths were considered for measurement of chemical-oxygen-demand (COD), total dissolved solids (TDS), turbidity, pH and electrical conductivity (EC). The EC and pH of the bleach water bath were evaluated using a digital pH meter (Model Gold 533) and Metler Toledo digital conductivity meter (Model FEP 30, Switzerland) following Method 4500-H pH, respectively. The COD was measured according to method 5210 (B). TDS of water were measured by a digital conductivity meter (Mettler ToledoAG, Switzerland), using the standard 2540 method.

**3 Results and Discussion**

**3.1 Optimisation of Bleaching Parameters**

All four lignocellulosic fibres—banana, flax, jute and ramie—are first scoured and then bleached using PAA under varying process conditions. The bleaching is conducted in a closed vessel using a non-ionic surfactant (0.5 ml/L), tetrasodium pyrophosphate (TSPP, 3 g/L), and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>, 1.8 g/L). Key parameters—bleaching time (60, 120, and 180 min), temperature (60 °C, 70 °C, and 80 °C), PAA concentration (10, 20, and 30 g/L), and bath pH (6.5, 7.0, and 8.0)—are systematically varied using the Taguchi Experimental Design (TED) method.

The TED approach, which considers both the mean and variance of responses, identifies optimal process conditions efficiently. Table 1 presents the optimised bleaching parameters for each fibre. Notably, all fibres bleach effectively around 75 °C and within a mild alkaline pH range (7–8). The required PAA concentration varies with fibre composition and is highest for jute and ramie, reflecting their greater lignin content. Ramie bleaches efficiently in just over an hour, whereas the other fibres require at least 2 hours to achieve a Whiteness Index (WI) >70, up from raw values of 33–53.

The bleaching process significantly reduces yellowness index (YI), from 32–47 in raw fibres to 20–33 in bleached fibres. Optical and physical

characterisation (Table 2) shows that banana, jute, and ramie retain superior bundle strength post-bleaching. Ramie emerges as the finest fibre (0.80 tex), while banana remains the coarsest (5.3 tex), reflecting their inherent structural differences.

**3.2 Bleaching Performance with Reused Bleach Baths**

Reuse of the bleaching bath is an important unit operation, as far as maximum utilisation of costly bleaching chemicals, or other auxiliaries and water utilisation are concerned. In this study, the original bleach bath is reused two times for successive bleaching of scoured fibres: (i) in the first reuse, without any additions, and (ii) in the second reuse, with adjustment of pH and MLR (material-to-liquor ratio) but without adding more PAA. This ensures maximum utilisation of PAA.

**3.2.1 Banana Fibre**

Figure 1 shows the WI and YI of raw, scoured, and bleached fibres in the original, as well as the 1<sup>st</sup> and 2<sup>nd</sup>

Table 1 — Optimised bleaching parameters for lignocellulosic fibres

Parameter	Banana	Flax	Jute	Ramie
Temperature, °C	73	75	75	76
Time, min	135	125	127	69
Concentration, mL/L	15.4	10	20	18
pH	7.5	7.8	8.0	7.0

Table 2 — Optical and physical properties of lignocellulosic fibres at optimum bleaching conditions

Property	Banana	Flax	Jute	Ramie
Whiteness index	73.8	74.8	74.1	76.3
Yellowness index	32.7	29.6	24.6	20.1
Fineness (tex)	5.3	3.0	2.9	0.80
Bundle strength (cN/tex)	21.8	13.1	18.3	19.3

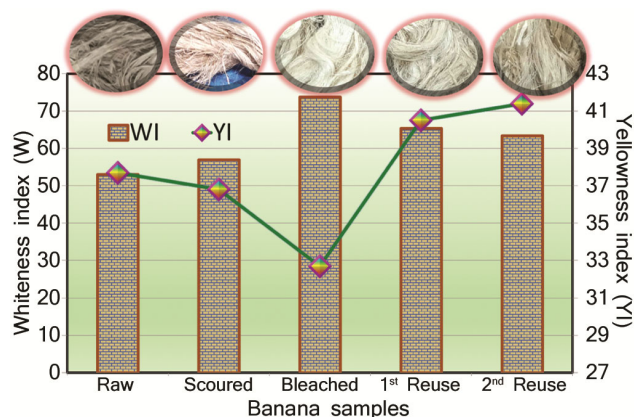


Fig. 1 — Effect of PAA on bleaching and reuse of bleach bath on whiteness and yellowness indices of banana fibre

reuse bleach bath samples. WI gradually increases from 53.1 to 73.8 in the raw to bleached fibres. The 1<sup>st</sup> and 2<sup>nd</sup> reuses achieve WI values of 65.4 and 63.4, respectively, corresponding to the gradual depletion of PAA to 66 % and 32 % (Table 3). In the final discharge effluent bath, only 1.1% PAA is present, showing the maximum utilisation of the bleaching agent. As the WI of the samples increases due to bleaching action, there is a concurrent decrease in fibre YI. Also, the brightness index (BI) of bleached fibres nearly doubles post-bleaching. Thus, the fibre looks much glossier. As expected, BI in the 1<sup>st</sup> and 2<sup>nd</sup> reuse bath samples are lower than the original bleached samples. As WI and reflectance improve, colour strength (*K/S*) decreases, confirming the bleaching effect. Colour strength values in the bleached fibre samples are lower than the raw and scoured samples. Other colour parameters like lightness-darkness (*L\**), red–green (*a\**) and yellow–blue (*b\**) are found to increase in the raw to bleached fibre samples (Table 3).

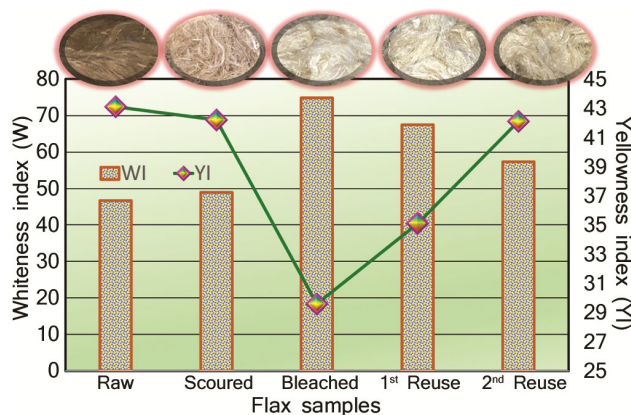


Fig. 2 — Effect of PAA on bleaching and reuse of bleach bath on WI and YI of flax fibre

### 3.2.2 Flax Fibre

Similar trends are observed in flax fibre (Fig. 2). WI notably increases from 46.7 (raw) to 74.4 upon PAA bleaching. Scouring of flax fibres in the presence of soap and mild alkali could improve the WI value marginally. When the scoured flax fibres are bleached in the reclaimed bleach baths, utilising its 1<sup>st</sup> and 2<sup>nd</sup> reuse, the achievable WI value is notably lower, i.e., 67.5 and 57.3, respectively, compared to the original bleach sample. The possible reason may be the reduction of PAA concentration from 100% to 44%, with 26 % remaining in the final effluent (Table 4). Unlike WI, YI follows the opposite trend and gradually drops from 43.1 to 29.6 in bleached samples but increases in reused baths due to lower bleaching efficiency. After the 2<sup>nd</sup> reuse, there was only 26 % PAA in the discharge bath, which is notably higher than the banana bath. BI follows the WI trend. The improvement in WI and BI, along with a reduction in YI, results in the decrease in colour strength (*K/S* value) from 3.79 (raw) to 0.60 (bleached). Images of samples in the figure visually corroborate these data.

### 3.2.3 Jute Fibre

Table 5 shows the spectroscopic colour parameters of the raw, scoured and bleached jute fibres. WI value steadily increases from 42.6 to 74.1. Unlike the other lignocellulosic fibres, there is a slight reduction in WI in jute fibres upon scouring. The 1<sup>st</sup> and 2<sup>nd</sup> reuse baths yield WI values of 68.4 and 54.2, with PAA depletion to 43 % and 27 %, respectively (Fig. 3). Only 1.5 % PAA is left in the effluent discharge. BI of jute fibres is significantly more than the other fibres, resulting in a shinier appearance of the fibre.

Table 3 — Colour parameters and PAA strength in the original and reused bleach baths for banana fibre

Sample	<i>L*</i>	<i>a*</i>	<i>b*</i>	BI	<i>K/S</i>	% PAA
Raw fibre	65.0	6.2	20.1	22.3	1.98	NA
Scoured fibre	68.2	5.1	18.4	26.6	1.82	NA
PAA bleached fibre	84.5	0.42	18.0	46.4	0.78	100
1 <sup>st</sup> reuse fibre	77.1	2.4	25.2	32.8	1.60	66
2 <sup>nd</sup> reuse fibre	75.9	2.5	26.2	31.8	1.67	32→1.1 in the effluent

Table 4 — Colour parameters and PAA strength in the original and reused bleach baths for flax fibre

Sample	<i>L*</i>	<i>a*</i>	<i>b*</i>	BI	<i>K/S</i>	% PAA
Raw fibre	59.4	1.5	22.3	16.9	3.79	NA
Scoured fibre	61.7	3.7	22.3	18.6	3.09	NA
PAA bleached fibre	84.2	-0.4	19.2	48.0	0.60	100
1 <sup>st</sup> reuse fibre	78.6	-0.4	22.0	37.6	1.09	80
2 <sup>nd</sup> reuse fibre	70.2	2.7	24.7	25.3	2.02	44→26 in the effluent

Table 5 — Colour parameters and PAA strength in the original and reused bleach baths for jute fibre

Sample	L*	a*	b*	BI	K/S	% PAA
Raw fibre	53.1	4.8	14.2	15.0	3.20	NA
Scoured fibre	51.8	7.8	18.0	12.6	4.10	NA
PAA bleached fibre	86.4	-0.6	17.3	53.3	0.44	100
1 <sup>st</sup> reuse fibre	82.3	0.2	18.5	45.8	0.64	43
2 <sup>nd</sup> reuse fibre	65.8	4.2	19.4	23.8	1.90	27 → 1.5 in the effluent

Table 6 — Colour parameters and PAA strength in the original and reused bleach baths for ramie fibre

Sample	L*	a*	b*	BI	K/S	% PAA
Raw fibre (degummed)	45.1	7.3	19.8	8.1	7.1	NA
Scoured fibre	66.0	4.8	14.6	26.2	1.34	NA
PAA bleached fibre	82.8	1.5	12.1	50.7	0.34	100
1 <sup>st</sup> reuse fibre	78.0	2.6	13.1	41.7	0.57	60
2 <sup>nd</sup> reuse fibre	73.3	3.3	14.0	35.8	0.75	34 → 6 in the effluent

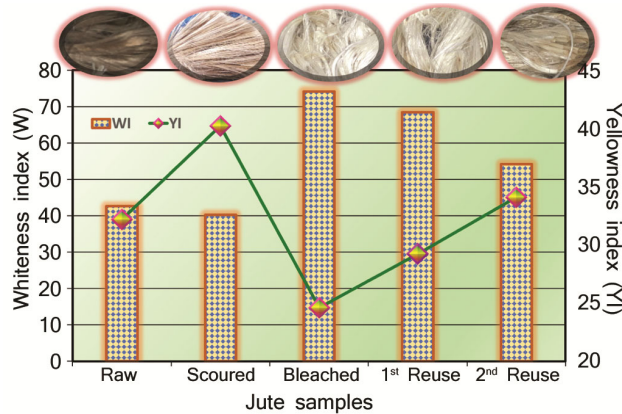


Fig. 3 — Effect of PAA on bleaching and reuse of bleach bath on whiteness and yellowness indices of jute fibre

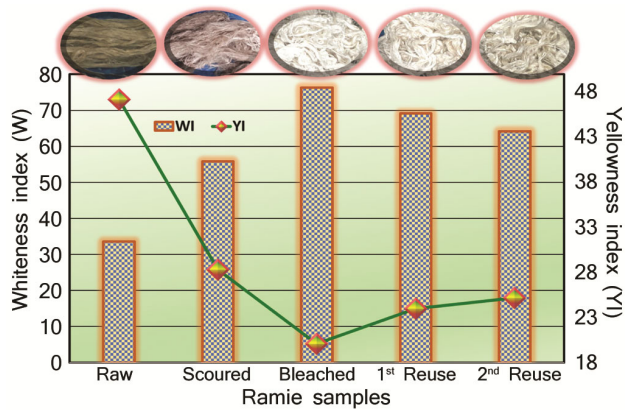


Fig. 4 — Effect of PAA on bleaching, and reuse of bleach bath on whiteness and yellowness indices of ramie fibre

The spectroscopic colour measurement in terms of wavelength vs. reflectance percentage of light was also measured. It is seen that due to the depigmentation of colour compound or removal of lignin macromolecules during the PAA bleaching process, the light reflectance percentage increases

from the white and glossy bleached jute fibre surface. When the scoured jute is bleached in the 1<sup>st</sup> reuse and 2<sup>nd</sup> reuse baths, light reflectance percentages are lower due to lower and higher values of WI and YI, respectively. As a result, K/S values decrease initially, then rise again in the second reuse due to reduced WI (Table 5).

It was observed that fibres from the original bleach bath with a WI value of 74.1 may be dyed both in light as well as dark shades. Bleached fibres obtained from the 1<sup>st</sup> reuse and 2<sup>nd</sup> reuse baths having lower whiteness indices of 68.4 and 54.2 may be dyed in medium to dark colour. This may ultimately reduce the cost of dyeing, as the samples need not to bleach to their fullest extent (WI of >70). At the same time, as maximum PAA could be utilised, it may also reduce the effluent discharge load.

### 3.2.4 Ramie Fibre

Table 6 and Figure 4 show the spectroscopic colour parameters of different ramie fibres under various treatments. Ramie, with a fineness of 0.80 tex), is the finest fibre among those studied and exhibits the highest achievable WI. It may be due to better penetration and bleaching action of PAA with lignin and/or natural pigments facilitated by the larger surface area of finer fibres. It is also supported by the highest cellulose and lowest lignin content among all the natural fibres, further supporting its high WI and lowest YI (20.1). This leads to the highest BI (50.7). Even after bleaching with the 1<sup>st</sup> and 2<sup>nd</sup> reuse bleach baths, ramie fibres exhibit higher WI of 69.1 and 64.1. The fibres obtained from the reuse bleached baths show much lower colour strength (K/S) of 0.57 and 0.75, corresponding to PAA concentrations of 60 % and 34 %, respectively (Table 6). The YI after bleaching in

Table 7 — Physical and mechanical properties of banana, flax, jute and ramie fibres after various treatments

Sample	Bundle strength, g/tex	Fineness, tex	Weight loss, %
Banana			
Raw fibre	27.3	6.2	-
Scoured fibre	25.3	5.7	15.3
PAA bleached fibre	21.8	5.3	12.8
1 <sup>st</sup> reuse fibre	23.3	5.5	11.6
2 <sup>nd</sup> reuse fibre	22.8	5.5	11.8
Flax			
Raw fibre	16.2	3.9	-
Scoured fibre	15.7	3.4	12.1
PAA bleached fibre	13.1	3.0	10.7
1 <sup>st</sup> reuse fibre	13.5	3.1	10.6
2 <sup>nd</sup> reuse fibre	14.1	3.2	9.3
Jute			
Raw fibre	24.6	3.4	-
Scoured fibre	22.9	3.2	6.2
PAA bleached fibre	18.3	2.9	4.1
1 <sup>st</sup> reuse fibre	19.8	2.9	3.9
2 <sup>nd</sup> reuse fibre	18.6	3.0	3.2
Ramie			
Raw fibre (degummed)	23.2	1.0	-
Scoured fibre	20.9	0.9	6.9
PAA bleached fibre	19.3	0.8	5.2
1 <sup>st</sup> reuse fibre	19.6	0.9	5.7
2 <sup>nd</sup> reuse fibre	19.8	0.9	5.3

the reuse baths (24 and 25.2) also remains much lower than that of banana, flax and jute fibres.

The results reveal that the original PAA bleach bath can be judiciously reused once without adding bleaching agents/ aids. However, during the 2<sup>nd</sup> reuse, water and pH must be adjusted to achieve satisfactory whiteness and brightness. The PAA concentration declines progressively with each reuse, resulting in a lower chemical load in the effluent, thereby supporting a sustainable bleaching strategy.

### 3.3 Physical and Mechanical Properties of Fibres

Table 7 shows the bundle strength, fineness, and weight loss of banana, flax, jute, and ramie fibres subjected to different chemical treatments. The maximum reduction in bundle strength due to PAA bleaching action on these lignocellulose fibres is approximately 16.8 - 25.6 %. Retention of fibre strength is greater when the fibres are bleached in the reuse bath, possibly due to a lower degree of bleaching action in the mild alkaline bath. Fibre fineness in terms of 'tex' is also

found to improve, i.e., fibres become finer after either individual (scouring) or sequential chemical treatments (scouring followed by bleaching). Among the lignocellulosic fibres, ramie is the finest, and banana is the coarser fibre; flax and jute fibres are found to be in between. As far as weight reduction percentage is concerned, due to either only scouring or scouring followed by the bleached process, it is much lower in the case of jute and ramie fibres. Higher weight loss in the banana and flax fibres may be due to the removal of dust, dirt, fine stick, partly lignin, pectin and hemicellulose. Reduction in weight is less when the scoured fibres were bleached in 1<sup>st</sup> and 2<sup>nd</sup> re-use baths, indicating their relatively lower content of easily extractable non-cellulosic material.

### 3.4 FTIR Analysis

ATR-FTIR spectroscopy is performed to examine the chemical changes in all four fibres following scouring and bleaching processes. The effect of low temperature and mild alkali in the PAA bleaching bath on chemical properties is studied. Figure 5 shows the FTIR spectra of these fibres at raw and after the scouring and bleaching process. A sharp peak is visible in the wavenumber range of 3000-3600 cm<sup>-1</sup> for the -OH group in the cellulose and hemicellulose macromolecules, as well as due to the presence of trapped moisture in fibres. The intensity of the peak is reduced with scouring and bleaching treatments, in the case of banana, flax and jute fibres, indicating the removal of hemicellulose and lignin. In contrast, it increases in ramie fibres after treatment, where hydrocarbon gummy material is removed concurrently with exposure to the hydrophilic cellulose macromolecules. The peak at 1420-1430 cm<sup>-1</sup> for -CH<sub>2</sub> bending and 895 cm<sup>-1</sup> for -C-O stretching is due to the crystalline portion versus the amorphous portion in the lignocellulosic fibres. No major change is visible at a wavenumber of 1028 cm<sup>-1</sup> for the -C-C, -C-OH, -C-H ring and side group vibrations. However, it is found to increase in ramie fibres and decrease in jute fibres. These peaks correspond to dislocation regions associated with in-plane CH bending at wavenumbers of 1370 cm<sup>-1</sup> to 1318 cm<sup>-1</sup>, showing some decrease, possibly due to the removal of hemicelluloses. Absorption bands at 1740 cm<sup>-1</sup> and 1236 cm<sup>-1</sup> are related to hemicellulose-induced C=O and C-O stretching of the acetyl group. The absorption in the wavenumbers of 1103 cm<sup>-1</sup> and 1028 cm<sup>-1</sup> appeared by cellulose's C-O-C pyranose ring skeletal vibration and asymmetric stretching of C-O-C in cellulose and hemicelluloses.

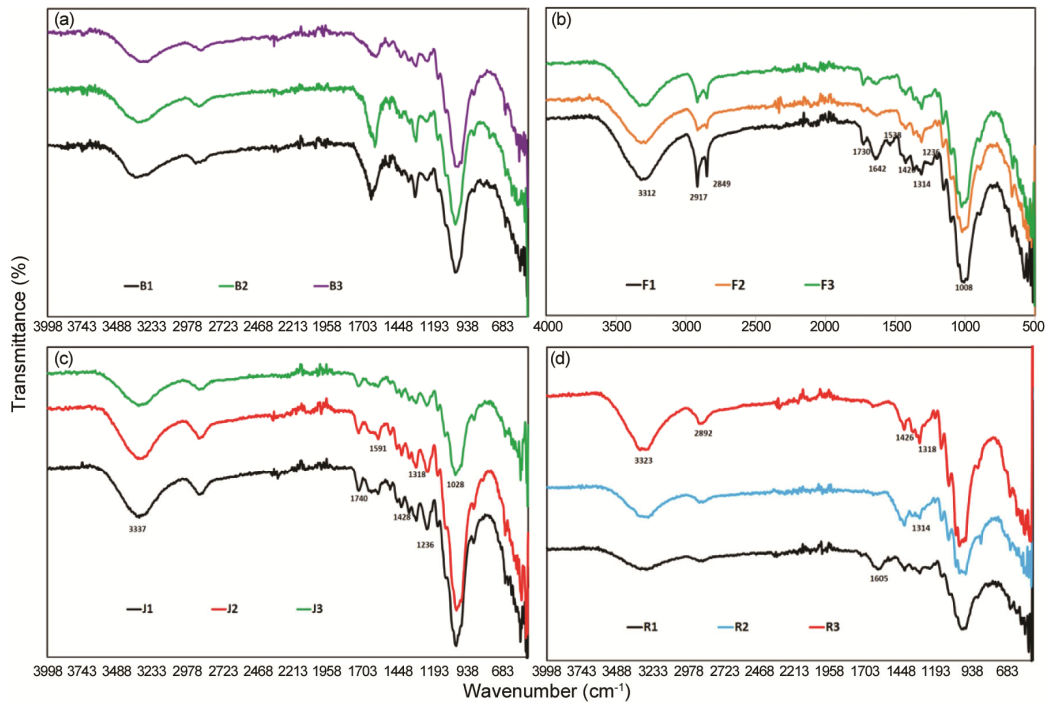


Fig. 5 — FTIR spectra of fibres (a) banana, (b) flax, (c) jute, and (d) ramie, showing changes at different stages - raw (1), scoured (2), and bleached (3). Fibre codes: B - Banana, F - Flax, J - Jute, R - Ramie.

Table 8 — Effluent characteristics of original and reused bleach baths

Sample details	pH	EC (mS/cm)	TDS (PPT)	Turbidity (NTU)	COD (mg O <sub>2</sub> /L)
Banana					
PAA bath (fresh)	7.5	6.20	3.05	2.20	2560
After original bath	7.8	6.20	3.14	131.8	2266
After 1 <sup>st</sup> reuse	7.7	7.41	3.59	257.5	1980
After 2 <sup>nd</sup> reuse ( <i>in the effluent</i> )	7.8	7.52	3.76	328.8	1080
Flax					
PAA bath (fresh)	7.8	7.91	3.05	2.20	2560
After original bath	8.1	8.62	3.41	113.6	2080
After 1 <sup>st</sup> reuse	8.0	8.27	3.67	164.6	1840
After 2 <sup>nd</sup> reuse ( <i>in the effluent</i> )	8.2	8.19	3.92	157.2	640
Jute					
PAA bath (fresh)	8.0	8.09	4.29	2.20	2560
After original bath	8.0	11.1	5.28	46.3	2400
After 1 <sup>st</sup> reuse	7.8	10.92	5.62	48.9	2080
After 2 <sup>nd</sup> reuse ( <i>in the effluent</i> )	7.9	11.12	5.83	50.0	1200
Ramie					
PAA bath (fresh)	7	5.79	2.89	2.20	2560
After original bath	7.6	5.90	3.18	14.6	2240
After 1 <sup>st</sup> reuse	7.7	6.82	3.40	17.8	1920
After 2 <sup>nd</sup> reuse ( <i>in the effluent</i> )	7.7	7.43	3.76	19.1	1120

**3.5 Effluent Parameters of Original and Reused Bleach Baths**

Table 8 shows that the pH of the bath could be maintained properly by adding sodium carbonate and TSPP to the original and reused bleach baths. Bleaching process is accompanied by loss in weight

due to removal of hemicellulose, pectin, lignin, etc., resulting in an increase in TDS and turbidity in the original, 1<sup>st</sup> and 2<sup>nd</sup> bleach baths for all the fibres. The increase in turbidity is much higher than in TDS, resulting in visibly darker bleach baths, as shown in Figure 6 for the jute fibres.

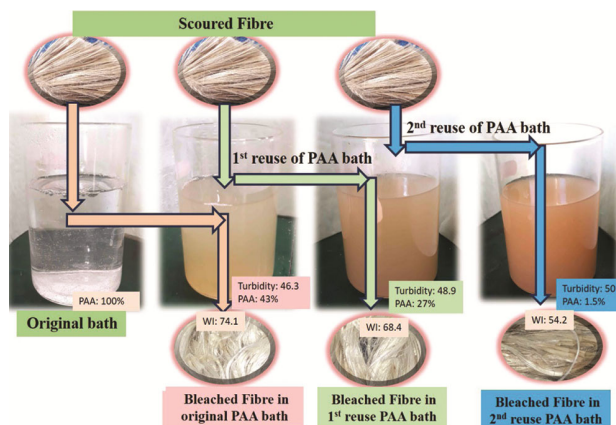


Fig. 6 — Bleaching of jute fibres in original and reused bleach baths along with residual PAA and turbidity

A gradual decrease in PAA concentration, along with an increase in turbidity, hinders the bleaching action, resulting in less improvement in WI. It also results in a decrease in chemical oxygen demand (COD). The measured electrical conductivity (EC) is related to TDS and turbidity. Due to the increase of these parameters, the EC also increases, confirming the ionic nature of dissolved species. In the 1<sup>st</sup> reuse bath, WI values of 65.4 (banana), 67.5 (flax), 68.4 (jute) and 69.1 (ramie) are quite good and may be considered for successive dyeing for medium to light shades. This may also reduce the cost of production, as in the 1<sup>st</sup> reuse bath, no addition of chemicals/ auxiliaries is required. Likewise, when the bleach bath is used 2<sup>nd</sup> time, achievable WI are 63.4 (banana), 57.3 (flax), 54.2 (jute) and 64.1 (ramie), which are suitable for successive dyeing for dark shades. The results indicate the feasibility of bleach bath reuse for economic and environmental benefits, including reduced chemical use, water consumption, and thermal energy.

#### 4 Conclusion

The study demonstrates the effective scouring and bleaching of lignocellulosic fibres—banana, flax, jute, and ramie—using PAA, highlighting improvements in fibre fineness, whiteness, and tenacity retention. The original PAA bleach bath can be reused twice with minimal adjustments, achieving good whiteness indices (64.5–69.1 in the 1<sup>st</sup> reuse; 54.2–64.1 in the 2<sup>nd</sup> reuse) along with satisfactory brightness values. The 1<sup>st</sup> reuse of the bleach bath requires no additional chemicals, water, or pH adjustment, while the 2<sup>nd</sup> reuse needs only minor compensation in terms of water and pH.

Fibres bleached in the original bath are suitable for light to dark shade dyeing, while those from the 1<sup>st</sup> and 2<sup>nd</sup> reuse baths are appropriate for medium to dark shades. Analysis of residual bleach baths at different stages indicates a progressive decrease in PAA along with an increase in TDS and turbidity. The reuse of PAA bath could enhance the whiteness and fineness of bleached fibres with acceptable retention of tensile strength. The process reduces the bleaching cost for maximum utilisation of bleaching agents while producing minimum effluents.

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