

## Efficacy of solvent, alkali and pectinase on removal of non-cellulosics from cotton fibres

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The physical properties of different varieties of cotton fibres of various origins have been studied after extraction using solvents and alkali scouring with reference to enzyme scouring process. Enzyme scoured samples show comparable results with that of solvent extracted and alkali scoured samples in terms of fibre fineness, weight loss, moisture regain, strength and elongation. The scouring processes improve the properties of the fibres in the order: solvent extraction < enzyme scouring < ammonium oxalate extraction < alkali scouring.

**Keywords:** Cotton, Elongation, Moisture content, Solubility parameter, Strength, Weight loss

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### 1 Introduction

Cotton fibre contains many natural impurities on its primary and secondary walls along with the impurities added during the yarn preparation. Besides removal of oils, fats and waxes present in the fibres, removal of pectin shows great advantages in terms of significant improvement in the absorbency. About 85% of carboxyl groups present in the cotton pectin are methylated, available in the form of insoluble calcium, magnesium and iron salts of polygalacturonic acid, which contribute to the hydrophobic characteristics of raw cotton.<sup>1</sup> In the past, extractions using different solvents, like hexane, methanol, ethanol, 1,1,1-trichloroethylene, and water, have been carried out as the means to remove the natural impurities.<sup>2-4</sup> Extraction of pectin present in cotton fibres has been tried by many researchers and the procedures are well documented.<sup>5-7</sup> Though these processes are the widely accepted methods to remove the natural impurities, the results vary widely among these methods. Several factors influence the successful use of enzymes in scouring cotton fabrics that includes the nature of substrates, type of

enzymes, enzyme activity, presence of surfactants and mechanical agitations employed during the process.<sup>8,9</sup> Effect of enzymatic treatment on different fibres, and the effect of acid pectinases, alkaline pectinase and combined enzyme systems on handle values and other properties have been studied extensively and significant differences have been pointed out irrespective of the varieties of cotton fibres or their mixtures.<sup>10-14</sup> The present work was undertaken to study the effects of enzyme treatment on various cotton varieties and to assess its relative performance with alkali scouring and extractions with the solvents of different solubility parameters.

### 2 Materials and Methods

#### 2.1 Materials

Different varieties of raw cotton such as Bunny, MECH and MCU 5, obtained from M/s Ramakrishna Mills, Sathyamangalam, were used in this study.

#### 2.2 Methods

##### 2.2.1 Physical Properties of Fibres

Physical properties of the fibres before and after extractions, like fineness, strength, elongation, moisture and trash contents, were measured using

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ASTM D 5867-05 with Uster high volume instrument 1000, which is capable of measuring these properties using very less amounts of sample.

### 2.2.2 Solvent Extraction

Carbon tetrachloride and ethanol were selected as the solvents for extracting various impurities present in the raw cotton, whose Hidebrand's solubility parameter values are 18.0 MPa and 26.2 MPa respectively.<sup>15</sup> Extractions were carried out using Soxhlet extraction apparatus, as specified in the ASTM D2257 – 04 at the rate of six extractions per hour for a total duration of 2 h. Firstly the samples were extracted with carbon tetrachloride followed by the extraction with ethanol. Extraction of pectin from raw cotton was also carried out separately using 50 mM solution of ammonium oxalate at 75°C (ref. 5).

### 2.2.3 Scouring

Alkali scouring was carried out using 1:20 liquor ratio with 4% (owm) sodium hydroxide and 1% wetting agent at boil for 1 h (ref. 16). Enzyme scouring was carried out using pectinase, supplied by M/s Rossari Biotech (India), in a rotating type Laundromat considering the optimum process parameters as suggested by the supplier (8.0 pH, 55°C temperature, 3 g/L concentration and 1 h treatment time).

### 2.2.4 Weight Loss

Weight loss after every extraction was calculated as the ratio of difference in weights before and after the treatment to the original weight. Before weighing, every sample was allowed to reach equilibrium under standard conditions with a relative humidity of 65% ± 2% and temperature of 25 ± 2°C.

### 2.2.5 Moisture Content

In order to find out the equilibrium moisture content values, the fibre samples were conditioned at 65% RH and 25° ± 2°C, then transferred to an air tight container and taken to the instrument for the testing. Moisture content of the fibre was tested in Mesdan semi automatic moisture tester using ASTM 2654-89a standard testing method. The ratio of difference in initial weight and final weight to the initial weight is expressed as the moisture content of the specimen.

### 2.2.6 FTIR Spectrum

To assess the extraction of the impurities from solvents and scouring processes, FTIR spectra of

samples after every extraction were taken using Shimadzu FTIR 8400S spectrophotometer. Pellets of the specimens were prepared using potassium bromide and twenty scans were taken for every specimen to reduce the influence of the noises. The spectra were obtained in the wavenumber range of 400 – 3500 cm<sup>-1</sup>.

## 3 Results and Discussion

Table 1 shows the results of untreated cotton fibres measured in the Uster high volume instrument. It is known that the extraction efficiency of a solvent molecule, to a larger extent, depends on the solubility parameter of the solvent with which the solvent molecules selectively interact with a particular polymeric molecule without dissolving other molecules present in the system. Alkali scouring of cotton fibres can result in higher extraction of natural impurities due to non-specific nature in the reactions compared to the solvent extractions and pectinase scouring, which are mainly substrate specific in their reaction and expected to result in lower weight losses. Any hindrance that prevents the access to the substrate in the case of pectinase scouring will not only result in partial removal of pectineous matters but also other hydrophobic impurities attached to it.

### 3.1 Weight Loss

The cumulative weight loss values of solvent and ammonium oxalate extractions are found to be lower than that of the alkali scoured cotton fibres but higher than that of the enzyme scoured samples (Fig. 1). Between the carbon tetrachloride and ethanol extractions, higher weight loss is observed in the case of carbon tetrachloride, perhaps due to the presence of more hydrophobic impurities in the raw cotton fibres compared to hydrophilic ones. High level of correlation is observed between the weight loss values of carbon tetrachloride – ethanol extraction and the enzyme scouring, compared to ammonium oxalate treatment and alkali scouring with a coefficient of 0.971.

Table 1 — Characteristics of raw materials

Fibre	Fineness µg / inch	Length mm	Strength gf/tex	Elongation %	Moisture content, %
Bunny	3.14	29.03	32.70	7.80	6.00
MCU 5	3.63	30.23	38.30	7.20	6.00
MECH	3.53	29.36	35.80	8.00	6.20

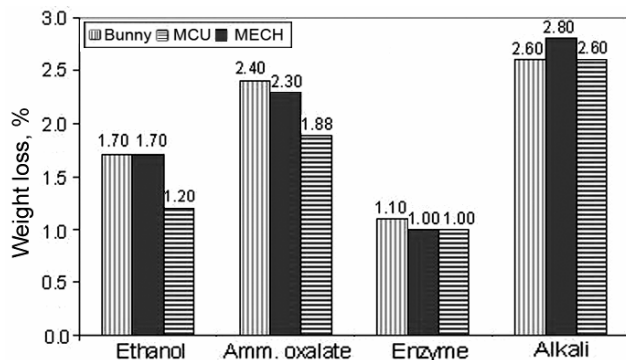


Fig. 1—Effect of solvent extraction and scouring on weight loss

### 3.2 Fibre Fineness

Fineness of the fibres ( $\mu\text{g}/\text{inch}$ ), to a larger extent, depends on the maturity of the fibres and, to some extent, is also influenced by the amount of the moisture present in the material.<sup>17</sup> Removal of hydrophobic impurities from the surface of the fibres is likely to increase the moisture regain of the fibres, which otherwise could reduce the surface bound moisture to the fibres.<sup>18,19</sup> The effect of the solvent extraction on resultant fineness of the fibres varies largely, that reflects the dissolution power of the solvents on selected hydrophobic impurities. In the case of alkali scoured samples, a significant increase in the fineness values is observed compared to the untreated cotton fibres and enzyme scoured samples, but the values are found to be lower than that of the ammonium oxalate extracted samples (Fig. 2). Lower values observed in the case of enzyme treated samples could be due to partial removal of waxy substances from the surface of the fibres as reported in the literatures.<sup>20</sup> In the case of fineness, the highest correlation is observed between enzyme treated samples and carbon tetrachloride-ethanol extracted samples with a correlation coefficient of 0.984, and similar correlation of enzyme treated samples with ammonium oxalate treated samples is also observed.

### 3.3 Fibre Strength and Elongation

Tensile strength of the fibres mainly depends on the ability of the polymeric molecules to distribute and withstand the load between the ordered and the disordered regions. Cotton fibres treated with solvents, alkali and enzymes show a reduction in the strength as compared to untreated cotton fibres (Fig. 3). Removal of impurities causes the structure of the fibres to become open and reduction in fibre strength. The impurities that are present tenaciously among the molecular chains in the

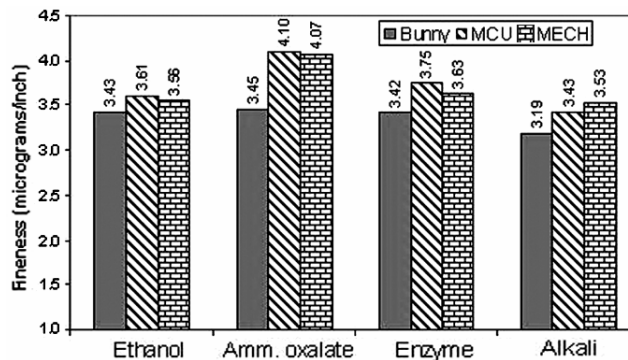


Fig. 2—Effect of solvent extraction and scouring on fineness

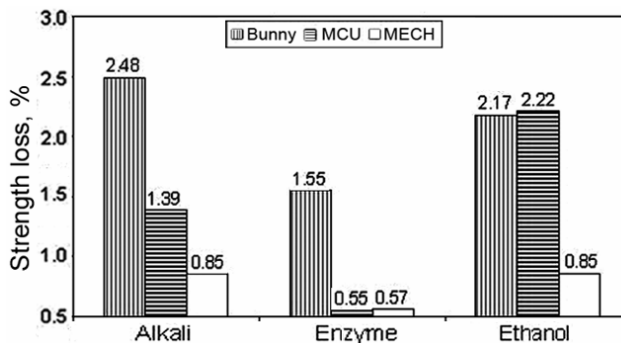


Fig. 3—Effect of solvent extraction and scouring on fibre strength

primary and secondary walls, otherwise involved in binding of molecular chains, could have contributed to the strength in the control samples. However in the case of yarns, contradictory results have been reported in the literature on account of different failure mechanisms.<sup>1</sup> The lowest reduction in strength is observed in the case of enzyme scoured samples and the highest reduction in the case of ammonium oxalate treated samples ( $>10\%$ ). Correlation of reduction in strength between carbon tetrachloride – ethanol extracted samples and enzyme treated samples shows the coefficient value of 0.932. Interestingly, higher correlation is also observed between alkali scoured samples and enzyme treated sample with the coefficient of 0.998.

As far as the elongation is concerned, the values are found to be less as compared to the untreated fibres, regardless to the nature of the reagents used in the extraction. However, much less decrease in the elongation values is observed in the case of enzyme scoured samples while higher reduction is observed in the case of ammonium oxalate extraction (Fig. 4), which appear to complement the results obtained in the tensile strength. The elongation values of the

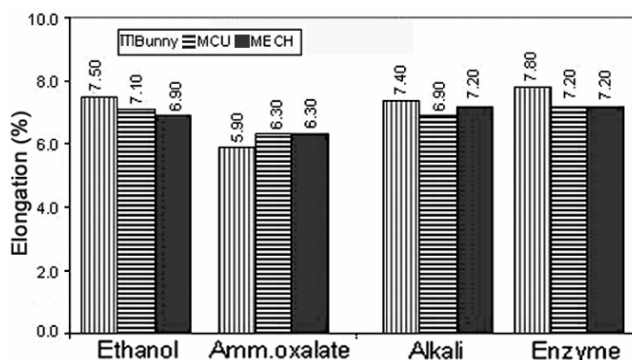


Fig. 4—Effect of solvent extraction and scouring on elongation

enzyme treated samples and alkali treated samples exhibited better correlation coefficient (0.989), while carbon tetrachloride-ethanol extracted samples results in the coefficient of 0.945.

### 3.4 Moisture Content

Moisture regain or moisture content of the cotton fibres is mainly influenced by the nature of the polymeric substance, accessible region in the fibres and amount of hydrophobic impurities present in the fibres.<sup>18,19</sup> Alkali scoured cotton fibres and solvent extracted fibres show higher regain values compared to that of enzyme treated samples. Extraction of hydrophobic impurities in the samples possibly could have resulted in higher moisture content values at equilibrium, however the difference in the extraction of hydrophobic impurities in the treatments appears to result in the variations in moisture content of the samples tested after the treatments (Fig. 5). In this case too, higher correlation coefficient of 0.945 is observed between enzymes treated samples and carbon tetrachloride – ethanol extracted samples.

### 3.5 FTIR Analysis

It is very difficult to obtain liquid spectra of extracted solvents from the fibres due to the highly volatile nature of the solvents and in many situations FTIR spectra of fibre samples have been used by many researchers.<sup>2</sup> FTIR spectra of untreated cotton fibres match with their reference spectra stated in the literature.<sup>21</sup>

In the untreated cotton fibres, the strong peaks are observed at the wavenumbers 1031, 1058, 1113 – 1163, 1629 – 1640, 2862 – 2920 and 3301 – 3380  $\text{cm}^{-1}$ , medium peaks at 1282, 1317-1340, 1372 and 1431  $\text{cm}^{-1}$  and weak peaks at 438 – 459, 518 – 532, 559, 617, 670, 703, 896, 1610 and 2331 – 2360  $\text{cm}^{-1}$  in the spectra. Significant differences are observed among the sample spectra obtained after various

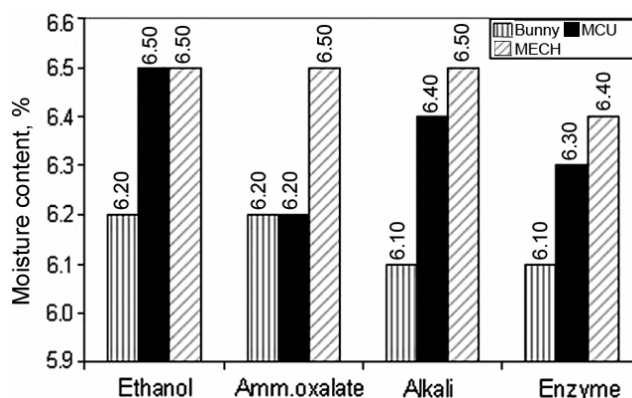


Fig. 5—Effect of solvent extraction and scouring on moisture content

extractions and scouring operations. Both enzyme and alkali scoured specimens show similarities in the removal of impurities as compared to the solvent extracted specimens as shown by the elimination of the characteristic peaks.

The peaks observed at 700  $\text{cm}^{-1}$  and 1610  $\text{cm}^{-1}$  are eliminated in the carbon tetrachloride extraction, probably associated with C – H bend, alkynes and C-C stretch in ring structures. The weak peaks at 2526 – 2560  $\text{cm}^{-1}$  and 1600 – 1700  $\text{cm}^{-1}$  regions are eliminated in the ethanol extraction process, probably associated with aldehydes and C-H stretch, carbonyl groups of esters, aldehydes, unsaturated esters, unsaturated aldehydes and the hydrogen bond interactions of very low molecular weight soluble impurities. In the enzyme scouring process, the characteristic groups like amides and carboxylic acids are eliminated as shown by the peaks in the range of 3301 – 3404  $\text{cm}^{-1}$  in the untreated cotton fibres and additionally, the peaks observed at 2831  $\text{cm}^{-1}$  and 1530  $\text{cm}^{-1}$  are eliminated in the alkali boil method, probably associated with aldehydes and protein substances.

## 4 Conclusions

Though the mechanism of removal of impurities differs among the various agents used in the experiments, some similarities in the results are observed in all the treated samples in various properties consistently. Ethanol, in spite of higher solubility value, could not yield higher extractable impurities compared to carbon tetrachloride whose solubility value is relatively less, possibly due to the high proportion of hydrophobic impurities in the

fibres. Similarly, enzymatic scouring results in lower weight loss and moisture absorption, and increase in fineness compared to alkali scoured and solvents extracted fibres, possibly due to partial removal of the natural impurities. Ammonium oxalate shows capability to remove the impurities to a higher extent, comparable to that of alkali scouring but the tenacity and elongation values obtained after the treatment are found to be much lower as compared to other treatments. Nevertheless, lower strength reduction in the case of alkali scoured sample as compared to the solvent extracted samples needs further investigations. However, the fibres extracted using carbon tetrachloride followed by ethanol show good correlation with enzyme treated samples in the case of fineness, moisture content, strength and elongation expressed by higher correlations though in certain cases similar relationships are observed with alkali scoured samples.

*Industrial Importance:* Though enzyme scouring has been widely experimented and adopted in the industrial practice, it is very much evident from this study that the results obtained in such process are comparable to those obtained in the conventional alkali scouring treatments.

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