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# Effect of enzymatic process on characteristics of cottonized industrial hemp fibre

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This paper reports an investigation on the bacteria-based enzyme's cottonization of industrial hemp fibre. The industrial hemp fibres are enzyme processed to eliminate massive non-cellulosic portions from the fibre to enhance their fineness and softness. Box- Behnken response surface methodology is applied to optimize the effect of different concentrations, temperature and time by using enzymes on chemical and physical properties, like weight loss, average strength, length, chemical composition, and surface modification to simulate cotton feel touch. The results show that the pectin, lignin, hemicellulose, and other impurities are removed under the enzyme and alkali refining processes. The effect of the enzyme concentration, treatment time, and treatment temperature is found significant on weight loss, fibre diameter, crystallinity, cellulose, hemicelluloses content, and tenacity. The effluent load of the enzyme process by measuring COD is also found less than that of the alkali processing for industrial hemp fibre. Industrial hemp fibre can be a perfect model of the sustainability of fibre from the plant's inception into the entire life cycle, leading to the biodegradable product reducing less effluent load in an environment.

Keywords: Cottonization, Delta-9 tetrahydrocannabinol, Enzymatic process, Industrial hemp fibre

# **1** Introduction

The hemp plant has been cultivated from the ancient period for many applications comprising textiles. In the twentieth century, the hemp plant was prohibited in many countries, including India, due to a high amount of tetrahydrocannabinol compound, a psychoactive substance. Over the past few decades of increasing environmental awareness, there has been a growing profound interest in developing industrial hemp fibres for changing the technological aspects in the engineering pattern, textile production, and sustainable design in the global market<sup>1</sup>.

Industrial hemp is a genetically modified plant obtained from the *Cannabis sativa* L. containing <0.3% THC (delta-9-tetrahydrocannabinol), which does not have a psychoactive effect and is cultivated in a large array of the environment<sup>2</sup>. The industrial hemp plant is a versatile crop serving, mainly food, shelter, medicine, and textile applications. In the depletion of natural resources and pollution of the environment, the sustainability of natural fibre is gaining essential pinnacle requirements of the textile and fashion market. Industrial hemp is surging in attractive attributes in comfort and aesthetic prerequisite of fashion consumer. Industrial hemp can be grown strategically three crops in a year as compared to cotton. One crop required 3402 litre of water in evaluation to 9958 L/kg of cotton fibres. Industrial hemp emits less carbon dioxide with less energy and carbon sequestration than cotton and polyester fibres<sup>3</sup>. Furthermore, the industrial hemp plant offers three times more fibres than cotton in the same cultivated area. Industrial hemp consumes less energy to come in a position to offer fibre than cotton and polyester with the sustainable route of fibre manufacturing<sup>4</sup>.

The utilization of natural fibres accosts ecological issues, like bio-degradability, a renewable resource, low energy consumption, low cost & recyclability; and absorb carbon dioxide in the plant's growth period. The surface modification of natural fibre is further enhanced between polymer and fibre to improve thermo- composites, which leads to sustainable production and consumption<sup>5</sup>. Industrial hemp fibre is composed of cellulose, hemicellulose, lignin, pectins, and other natural contaminations. These natural contaminations cause fibre stiffness and obstruct spinning. Decorticated industrial hemp fibres are enzyme processed for achieving contaminations-free worthy cellulose fibres<sup>6</sup>.

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Fibres in the bast region of the industrial hemp plant are tiny bundles of primary and secondary These agglomerated fibres. fibres are of approximately the diameter and length of cotton fibres and remain adhered at the middle lamella by pectinacious material in the form of a bundle. Cottonization refers to the disaggregation of these bundles of fibres into fine cotton-like fibres by removing pectic substances. Researchers have investigated various methods to remove these contaminations by physical, chemical, and biological methods'. Due to the water resistance properties of the hemp fibre, it is difficult to ferment. Hence, the chemical method is the prime method used for most of the processing of hemp fibre. Simultaneously chemical process consumes a significant amount of water and energy, leading to environmental pollution and less sustainability. Bacterial-originated enzymes have a prominent role in dissolving pectin and hemicellulose from cell walls of the complex structure of industrial hemp fibre to construct apposite fibre for the subsequent textile applications<sup>8</sup>. Applying enzymes to facilitate hemp degumming effectively reduces the above problems, improves hemp quality, and reduces environmental pollution. Decorticated industrial hemp fibres are enzyme processed for achieving contaminations-free worthy cellulose fibres<sup>9</sup>.

For spinning, industrial hemp extraction needs intriguing research to make it close to other cellulosic fibres like linen. The coarseness and stiffness of industrial hemp fibre restrict its applications in the apparel sector<sup>10</sup>. It is also a need of the hour to identify and optimize eco-friendly processing with an economical, sustainable, and low-temperature method for extracting good fibre from the decorticated raw fibre stage without damaging fibre quality. Few researchers have worked on the enzymatic action on industrial hemp fibre for separating the fibre from the cell wall to enhance the fineness with lower stiffness, which is essential for converting it into spun yarn<sup>11,12</sup>. This study is planned to investigate a systematic approach for the wet extraction of Industrial hemp fibres using enzymes. The optimization of materialto-liquor ratio, treatment time, temperature, and enzymatic concentration was done using Box-Behnken response surface methodology. The response was recorded in the form of fibre diameter, fibre crystallinity, tenacity, weight loss, and contents of hemicelluloses and lignin in industrial hemp fibre

after enzyme cottonization. The impact of green processing of industrial hemp is investigated in terms of ecological values.

## 2 Materials and Methods

The industrial hybrid hemp seed Carmagnola CS was cultivated on an experimental basis in February 2019 in the licensed farm Udhamsingh Nagar at Uttrakhand, India. The Industrial hemp plant was harvested in May 2019 and 50 kg fibre was extracted for experimental purposes. The Bioprel 3000 enzyme was procured from Nova Enzyme. AR grade sulphuric acid, acetone, ethyl alcohol, potassium dichromate, ferrous ammonium sulphate, ferrin indicator, mercuric sulphate, silver sulphate and sodium hydroxide were used to determine cellulose and chemical oxygen demand.

The fibre treatment was performed with an HTHP dyeing machine (R B & Engg Pvt Ltd). Trash analyzer, Statex digital fibrograph, Stelometer, Instron tensile tester Testing (Instron) and Proto X-ray diffraction were used to estimate the trash content, bundle fibre strength, fibre tensile behavior, and crystalline content of industrial fibre, respectively after conditioning at  $65\%\pm2\%$  RH and  $20\pm2$  °C for 24 h.

The cottonization of decorticated industrial hemp fibre was performed by opting process sequence as given below:



The decorticated<sup>\*</sup> industrial hemp fibre was first hot rinsed for 20 min and then retted at three prescribed concentrations of the enzyme, viz. 5, 10, and 15 gpL, by keeping the temperature at 60°C, 70°C, and 80°C for three different durations (60, 90, and 120 min.) Total 15 experiments were designed as per Box-Behnken design, controlling three levels for three process parameters, keeping constant materialto-liquor ratio 1:10 (Table 1).

Quadratic polynomial equations were formulated for each industrial hemp fibre property in terms of independent variables, namely concentration of

Table 1 — Design plan of the experiments					
Parameters	Level/coded value				
	-1	0	1		
Enzyme ( $x_1$ ), gpL	5	10	15		
Temperature $(x_2)$ , <sup>O</sup> C	60	70	80		
Time $(x_3)$ , min	60	90	120		

Bioprel 3000 enzyme  $(x_1)$  temperature  $(x_2)$  and time  $(x_3)$ . The generalized polynomial regression equation is as follows:

$$Y = \mathbf{b}_0 + \sum \mathbf{b}_i x_i + \sum \mathbf{b}_{ij} x_i^2 + \sum \mathbf{b}_{ij} x_i x_j \qquad \dots (1)$$

In Eq. (1), the regression coefficients are represented in terms of  $b_0$ ,  $b_i$ ,  $b_{ij}$ ; and  $b_{ij}$ , i, j, the integers with i>j; and Y, the response or dependent variable (fibre property). The design of experiment software was used for statistical analysis. The weight loss percentage was calculated using the following equation:

$$\frac{\text{Weight loss (\%)} =}{\frac{\text{Fibre weight before process} - \text{Fibre weight after process}}{\text{Fibre weight before process}} \times 100$$
... (2)

The fibre diameter was measured by a projection microscope with the assistance of a fibre diameter analyzer at the magnification level of  $\times 10$ . The tenacity (gf) was estimated by keeping the gauge length 1/8 inch, as per ASTM D1445 / D1445M. The crystalline fibre content was calculated by Proto X-ray Diffraction machine in powder form by scanning in the range of 10-60° with a step of 0.008 and X-ray wavelength of 0.15406 nm. To determine the cellulose content, 1.5 g industrial hemp fibre was treated with 0.5% ethylenediaminetetraacetic acid (EDTA) at 105°C for 30 min, followed by cooling (weight  $a_0$ ), filtration and hot and cold rinsing. Industrial hemp fibre was treated with 75 mL of 0.5 mol/L HCl at 105°C for 30 min. The fibre sample was hot, and cold washed, dried at  $105^{\circ}$ C and weighed (weight  $a_1$ ). Acetone (100 mL) was used to isolate the residue fibre sample two times. The residual fibre was further treated with 22.5 mL H<sub>2</sub>SO<sub>4</sub> (72 w/v) in 150 mL distilled water and stored for 15 h at room temperature (27°C).

The solution was filtered, rinsed with hot water four times, rinsed with cold water, and finally weighed as lignin weight (weight  $a_2$ ). The cellulose content is determined using the following equations:

Cellulose (%) = 
$$\frac{a_1 - a_2}{a_0} \times 100$$
 ... (3)

The required amount of oxygen to completely oxidize the organic matter in a boiled-off liquor sample is called chemical oxygen demand (COD). The organic materials in boiled -off liquor are oxidized to  $CO_2$  and  $H_2O$  with strong oxidizing agents like potassium dichromate under acidic conditions<sup>13</sup>. The protocol to calculate COD during hemp processing is defined below:

- Boiled-off liquor sample (50mL) was taken in a 500 mL refluxing flask.
- Mercuric sulphate (1g), silver sulphate (1g) and several glass beads were added.
- Sulphuric acid (5mL) was added very slowly to dissolve mercury sulfate. Potassium dichromate (25 mL of 0.0417 M) was then added. Flask was attached to the condenser and turned on cooling water.
- The remaining amount of 70 mL sulphuric acid was added through the open end of the condenser. Condensor was covered with a beaker to prevent foreign materials and refluxed for 2 h.
- The flask was removed and cooled. Mixture was further diluted about twice its amount of distilled water.
- Potassium dichromate was titrated with ferrous ammonium sulphate by using 0.15 mL feroin indicator. The color was changed to reddishbrown at the endpoint.

Similarly, blank was run with distilled water by using the same amount of chemicals. COD chemical oxygen index was calculated by the following equations:

$$\operatorname{COD}\left(\frac{mg}{l}\right) = \frac{(A_2 - A_3) \times N \text{ of } FAS \times 8 \times 1000}{A_1} \qquad \dots (4)$$

where  $A_1$  is the volume of a boiled-off water (mL);  $A_2$ , the blank titer value in mL;  $A_3$ , the titre value with the water sample in mL; and N of FAS, the normality of ferrous ammonium sulphate solution.

#### **3** Results and Discussion

To identify the impact of the enzymatic process on the properties of industrial hemp fibre, weight loss %, fibre diameter, uniformity index, tenacity, crystallinity, cellulose %, and hemicellulose % are evaluated. Experimental results of 15 samples as per

			Та	ble 2 — Ex	xperimenta	al results of ind	ustrial hemp	fibres		
Sample No.	Enzyme gpL	Temp. °C	Time min	Weight loss, %	Fibre diam. u	Uniformity Index. %	Tenacity g/tex	Crystallinity %	Cellulose %	Hemi- cellulose, %
1	5	60	90	12.61	26.92	73.40	16.23	75.57	68.16	14.84
2	15	60	90	18.70	22.34	76.90	19.27	74.65	71.52	11.48
3	5	80	90	13.90	26.54	74.80	17.57	73.74	69.78	13.22
4	15	80	90	19.37	22.40	77.80	20.98	75.41	72.95	10.05
5	5	70	60	11.47	26.06	74.30	18.66	74.75	67.91	15.09
6	15	70	60	16.72	22.47	76.90	19.75	73.48	71.23	11.77
7	5	70	120	15.48	26.44	73.80	16.98	74.45	69.52	13.48
8	15	70	120	21.30	21.55	77.90	20.69	73.58	73.23	9.77
9	10	60	60	11.73	25.04	75.40	17.65	75.11	70.52	12.48
10	10	80	60	13.18	25.37	76.10	18.8	74.51	70.66	12.34
11	10	60	120	13.93	25.421	75.8	17.85	73.47	70.86	12.14
12	10	80	120	15.77	26.451	76.4	19.1	73.87	71.29	11.71
13	10	70	90	14.19	26.104	74.9	17.75	74.52	69.92	13.08
14	10	70	90	13.85	26.56	75.10	17.72	74.58	69.62	13.38
15	10	70	90	14.12	26.46	75.30	17.96	74.67	69.85	13.15
					Table 3 -	— ANOVA res	ults			
Parameters		Length	Weigł	nt loss Fibr	re diam	Crystalline	Cellulose	Uniformity	Tenacity	Hemi-
		%	9/	0	(μ)	%	%	Index, %	g/tex	cellulose, %
Enzyme, gp	ьL	NS	S	5	S	S	S	S	S	S
Temperatur	re, °C	NS	S	5	S	S	S	S	S	S
Time, min		NS	S	5	S	S	S	NS	S	S
A G×temp.		NS	Ν	S	NS	NS	NS	NS	NS	NS
A G×time		NS	S	5	NS	S	NS	NS	S	S
Temp×time	:	NS	Ν	S	NS	S	NS	NS	NS	NS
A G – Enzy	me gpL, Te	empTemp	erature,	NS–Non-si	gnificant,	S-Significant.				

box Behnken design are shown in Table 2. For statistical significance, ANOVA analysis is done and shown in Table 3.

# 3.1 Effect on Length and Uniformity Index %

Industrial hemp consists of different fibre lengths in terms of primary and secondary fibre. Process such as retting and extraction of industrial hemp leads to change in fibre length drastically, which compelled to estimate the uniformity index with the help of upper mean-length and mean length. The decortication process also adds length variations to hemp fibre which can be evident from Table 2. It is also observed that the enzyme treatment time does not introduce any remarkable change in the uniformity index of the processed fibre. However, the concentration of enzyme and treatment temperature significantly influence the uniformity index of processed hemp fibre, as shown in Table 3. The effect of enzyme concentration, processing time, and temperature on uniformity index can be modeled by following equations, with the assistance of response surface methodology:

Uniformity index =  $75.10 + 1.65X_1 + 0.45X_2 + 0.15X_3 + 0.21X_1^2 + 0.41X_2^2 + 0.41X_3^2 - 0.13X_1X_2 + 0.38X_1X_3 - 0.025X_2X_3$ ... (5)

#### 3.2 Effect on Surface Structure

Scanning electron microscope has been used to characterized the treated and untreated fibres surface structure of industrial hemp fibre. The SEM images of treated and untreated industrial hemp fibre are shown in Fig. 1. The surfaces of untreated fibre possess coated bundles with smooth layers and agglomerates. The enzymatic and other mechanical treatments registered remarkable alterations in surface morphology. It is observed that the untreated fibre surface is rough and stiff due to the presence of waxes, pectin, and lignin, as shown in Fig. 1. The untreated industrial hemp fibre bundle offers a light brown color with considerable variability in the diameter and length.

It is also observed that the enzymatic treatment increases the surface unevenness of hemp fibre. In untreated fibre, the hemicellulose and lignin are unevenly present. After removing hemicellulose and



Fig. 1 — SEM images of original and treated industrial hemp fibres (sample size  $20\mu$ , EHT 20KV, WD 8.mm, magnification ×824 and Zeiss machine)



Fig. 2 — Effect of enzyme, treatment time and temperature on weight loss %, diameter and tenacity of industrial hemp fibre (a) 60min, (b) 90min and (c) 120min

lignin through the enzymatic treatments, the unevenness of hemp fibre increases, as shown in Table 2. It is also observed that the treated fibre bundles are converted into the separated small units of fibre cluster, which ultimately leads to the higher available specific surface area. The enzymatic treatment drives extensive heterogeneous fibrillation within each batch, as shown in Fig. 1. The enzymatic treatments also show enough flaking due to degradation of the non-fibrous surface layers at the fibre surface by removing the massive non-cellulosic substance.

#### 3.3 Effect on Weight loss

The influence of enzyme concentration, temperature, and time on fibre mass loss is assessed by monitoring the weight loss after treatment. It is observed from Table 3 that there is a significant impact of the enzyme concentration, processing temperature, and processing time on weight loss. The minimum and maximum weight loss range from 10% to 21%. Figure 2 shows that enzyme concentration has a maximum impact on weight loss, followed by the temperature for all three treatments at 60, 90, and 120 min.

It is evident from Fig. 2 that as the processing time and concentration of enzyme increase, the weight loss also increases due to removal of non-cellulosic component. An enzyme reacts with hemicelluloses, pectin, and lignin in the fibre matrix and helps to remove these non-cellulosic components. It is also recorded that weight loss increases with the increase of processing time and temperature due to the higher reduction of residual pectin and other impurities in the fibre structure.

The effect of enzyme concentration, processing time, and temperature on weight loss can be modeled using the following equations with the assistance of response surface methodology:

Weight loss (%) =  $14.03 + 2.83X_1 + 0.66X_2 + 1.67X_3 + 2.35X_1^2 - 0.24X_2^2 - 0.14X_3^2 - 0.16X_1X_2 + 0.14X_1X_3 + 0.099X_2X_3$  ... (6)

# 3.4 Effect on Fibre Diameter

Fibre diameter has a profound role in the spinning of industrial hemp fibre which further influences the yarn and fabric properties. The diameter of 200 industrial hemp fibres is estimated by an optical microscope, and averaged values are reported. The untreated hemp fibre shows a broad range of diameters with very thick fibre bundles (150 microns to finer 25 microns). Fine cottonized fibres are collected for diameter estimation to avoid the bundling effect. The enzyme concentration significantly influences the fibre diameter, followed by processing temperature and processing time, as observed from Fig. 2.

It is observed from Fig. 2 that fibre diameter decreases with an increase in the enzyme concentration. It may be due to eroding of the surface and removal of non-cellulosic components. The treatment time of the process further reduces the diameter, which indicates that the temperature has a marginal impact on the fibre diameter. It is also observed that a high processing span leads to a lower fibre diameter which may be attributed due to the more available reaction time of enzymes and fibres. The polynomial quadratic regression equation for the average diameter is given by the following equations:

Diameter  $(\mu) = 26.39 - 2.15X_1 + 0.13X_2 + 0.12X_3 - 1.64X_1^2 - 0.20X_2^2 - 0.62X_3^2 + 0.11X_1X_2 - 0.33X_1X_3 + 0.18X_2X_3$ ... (7)

### 3.5 Effect on Tenacity

The fibre properties influence the mechanical properties of a staple fibre yarn. Tenacity is the measure of the mechanical behavior of fibre. The average tenacity of the treated fibre is given in Table 2. It is observed from Table 2 and ANOVA results (Table 3) that the tenacity of fibre is significantly influenced by the enzyme concentration and treatment temperature, while no significant impact of treatment time is found. Among all studied samples, the highest strength of enzymatic processed industrial hemp is 20.98 g/tex. Figure 2 is a graphical representation of the effect of the concentration level of the enzyme, treatment temperature, and treatment time on the tenacity of industrial hemp fibre.

The fibre tenacity increases by increasing the enzyme concentration during fibre treatment due to the high cellulose content after removing hemicellulose and other non-cellulosic impurities, as shown in Fig. 2. The applied treatment removes certain content present in the form of hemicellulose and wax material, which covers the hemp fibre's external surface, increasing the length/diameter ratio values. An increase in cellulosic content in the fibre content leads to an improvement in the mechanical



Fig. 3 — Effect of enzyme, treatment time and temperature on crystalline %, cellulose % and hemicellulose % of industrial hemp fibre (a) 60min, (b) 90min and (c) 120min

properties of the fibre. Higher processing temperature leads to higher tenacity as more impurities have been removed from the fibre. The polynomial quadratic regression equation for the average tenacity is:

Tenacity  $(g/tex) = 17.80 + 1.41X_1 + 0.68X_2 - 0.030X_3 + 0.69X_1^2 + 0.021X_2^2 + 0.53X_3^2 + 0.092X_1X_2 + 0.66X_1X_3 + 0.025X_2X_3$ ... (8)

# 3.6 Effect on Fibre Crystallinity

The enzymatic process removes massive noncellulosic components (hemicelluloses, lignins, and pectins) from the fibre bundles surface and increases cellulose content. The crystallinity result significantly changes due to enzyme concentration and processing time (Table 2). There is no significant change in the crystallinity observed due to temperature (Table 3). Figure 3 depicts the effect of enzyme concentration, treatment temperature, and treatment time on the crystallinity of processed industrial hemp fibre.

It is observed from Fig. 3 that the enzyme concentration has a significant impact on crystalline %. As the enzyme concentration increases, it reduces the amorphous content that appears due to hemicelluloses and wax. The treatment temperature also significantly affects the crystallinity %. Enzyme treatment leads to hydrolysis of the non-cellulose fraction, majorly representing amorphous areas and higher crystallinity values. The effect of enzyme

concentration, processing time, and temperature on crystalline content is modeled by following equation with the assistance of response surface methodology.

Crystallinity (%) = 
$$74.59 - 0.17X_1 - 0.16X_2 - 0.31X_3 + 0.040X_1^2 + 0.21X_2^2 + 0.56X_3^2 + 0.65X_1X_2 + 0.10X_1X_3 + 0.25X_2X_3$$
... (9)

#### 3.7 Effect on Hemicellulose and Lignin Contents

The high cellulose content, a chief component in hemp materials, facilitates the spinning process. The other components are hemicellulose and binding component of the elementary hemp fibres. From Table 3, it is observed that there is a significant impact on cellulose content of the enzyme concentration, time and temperature. The wet chemistry analysis of studied samples revealed that the highest cellulose content in the composition could be achieved by treating the sample with 15 gpL enzymes at 80 °C for 120 min. Table 2 shows that the experiment number 8 has the highest cellulose level, while experiment number 1 has the lowest value of cellulose among all the studied samples. Non-cellulosic compositions, such as hemicellulose and lignin, also decrease in the processed fibre due to the enzymatic treatments. Lignin upto 4.8% and hemicellulose upto 9.77% remain in the processed fibre. Figure 3 shows the effect of enzyme, time and temperature on cellulose % of industrial hemp fibre.

It is observed from Fig. 3 that as the concentration of the enzyme increases, cellulose content in the processed fibre increases. As discussed, a higher enzyme concentration reduces the noncellulosic content from the fibre and ultimately enhances cellulose's composition. Similarly, processing time and temperature also lead to high cellulose in the processed hemp fibre. The polynomial quadratic regression equation for the average cellulose % is given below:

Cellulose (%) =  $69.78 + 1.69X_1 + 0.45X_2 + 0.57X_3 + 0.23X_1^2 + 0.59X_2^2 + 0.46X_3^2 - 0.047X_1X_2 + 0.098X_1X_3 + 0.0273X_2X_3$  ... (10)

Hemicellulose (%) =  $13.22 - 1.70X_1 - 0.45X_2 - 0.57X_3 - 0.23X_1^2 - 0.59X_2^2 - 0.46X_3^2 + 0.047X_1X_2 - 0.098X_1X_3 - 0.073X_2X_3$  ... (11)

## 3.8 Effect on Ecological Value

Impact on the ecological value of liquor waste after the cottonizing process is comparatively studied with the traditional caustic soda process in chemical

Table 4 — Optimized value of industrial hemp fibre variables							
Parameter	Optimum values	Enzyme gpL	Temperature °C	Time min			
Weight loss, %	21.24	15	80	119.99			
Fibre diameter, µ	27.07	6.99	70.74	87.85			
Crystallinity, %	75.58	6.33	60.86	84.47			
Uniformity index,	77.98	14.09	78.77	118.21			
%							
Tenacity, g/tex	21.15	14.98	79.8	105.77			
Cellulose, %	73.48	14.95	77.85	118.61			
Hemi-cellulose, %	14.89	5.0	66.09	75.49			

oxygen demand. The Industrial hemp fibre is processed with the optimized recipe of alkali using caustic soda (10 gpL) soda ash (10 gpL) and lissapol D (1gpL) at 80°C for 2 h, keeping the M.L ratio at 1.10, A similar condition of time and temperature is given to enzyme, keeping strength 10 gpL. The quality of the residual degumming bath of waste liquor is also evaluated by measuring the chemical oxygen demand of both processes. The COD values of the wastewater obtained after the caustic soda and enzyme treatments of the industrial hemp fibre are 9768 mg/L and 3500 mg/L respectively.

It is observed that the COD is much lesser for the enzyme approach of treatment. Replacement of the caustic soda process with the enzymatic process provides less effluent load and seems more sustainable.

#### **3.9 Optimized Parameter**

untreated industrial hemp fibre The is unbendable/stiffened in nature. Fibre spinnability is considered as a prime requirement for yarn spinning, and the stiffened nature of untreated hemp fibre is not appropriate for yarn spinning directly. Therefore, the fibres are to be processed/treated in an optimized manner to extract the spinnable fibre. The fibre weight is essential for optimizing the process/treatment, as it removes impurities and other parameters directly, like diameter, strength, crystallinity, and cellulose content. The optimized values for variables of enzymatic treatment for cottonization of Indian industrial hemp fibre are given in Table 4.

## 4 Conclusion

The decorticated industrial hemp fibre is inappropriate for converting to yarn, fabric, and composites and can be processed by enzyme treatment. The enzyme retting process can successfully replace the traditional Indian water retting process of hemp. Based on the above study, following inferences are drawn:

**4.1** The processing of industrial hemp by the enzyme can be done in the harvesting field at atmospheric pressure

**4.2** The maximum weight loss of industrial hemp fibre is 21.24% after significantly eliminating hemicellulose and other impurities from the fibre. The minimum weight loss is found 12.60%.

**4.3** The optimal tenacity of fibre is achieved value 21.15 g/tex and elongation at 11%.

4.4 The crystallinity of fibres is increased after the enzymatic treatment. Higher temperature and time lead to higher crystallinity.

**4.5** The enzymatically retted and treated industrial hemp fibre shows the highest softness due to a significant reduction in fibre diameter.

**4.6** The COD chemical oxygen demand value in case of enzyme treatment is 3500 mg/L as compared to that is caustic soda treatment (9768 mg/L), which is much lesser for the sustainable enzymatic treatment.

4.7 The ideal fibre properties for cottonization can be achieved by using optimum value of enzyme, time, and temperature, keeping M:L ratio 1.10.

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## References

- Brodeur G, Yau E & Badal K, Collier J, Ramachandran KB & Ramakrishnan Subramanian, *Enzyme Res*, (2011). DOI: 10.4061/2011/787532.
- 2 Amaducci S & GusoviusH, Industrial Applications of Natural Fibres (John Wiley & Sons. Ltd, UK), 2010.
- 3 Cherrett N, Barrett J, Clemett A, Chadwick M & Chadwick M J, *Ecological Footprint and Water Analysis of Cotton, Hemp and Polyester* (Stockholm Environment Institute, Stockholm), 2005.
- 4 Gupta M K, Gond R K & Bharti A, *Indian J Fibre Text Res*, 43(2018) 313.
- 5 Pakarinen A, Zhang J, Brock T & Viikari L, *Biores Tech*, 107(2012)275.
- 6 George M, Mussone P G, Abboud Z & David B C, *Appl Surf Sci*, 314(2014)1019.
- 7 Garcia-Jaldon C, Dupeyre D & Vignon M, *Bio Bioenergy*, 14(1998) 251.
- 8 Sorieul M, Dickson A, Hill S J & Pearson H, *Materials*, 9(2016) 618.
- 9 Placet V, Day A & Beaugrand J, *J Mater Sci* 52(2017) 5759.
- 10 Moussa M, Hage R, Sonnier R, Chrusciel L, Ziegler I & Brosse N, Indu Crops Prod , 151(2020) 112.
- 11 Fang G, Chen H G, Chen A Q, Mao K W & Wang Q, *BioRes*, 12(2017) 1566.
- 12 Nykter M, Kymäläinen H R, Thomsen A B, Lilholt H, Koponen H, Sjöberg A M & Thygesen A, *Bio Bioenergy*, 32 (2008) 392.
- 13 Khanna D R & Bhutiani R, *Water and Waste Water Analysis* (Daya Publishing House) (2015) 39 – 41.