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Synthesis, Characterization and Application of Nano Cellulose for Enhanced Performance of Textiles

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Abstract

In the present investigation cellulose nano whisker is separated from industrial waste viscose rayon fibre, characterized by SEM images and FTIR spectroscopy. The size and size distribution of these nano crystals have also been examined using particle size analyzer; the average size of the particles is found to be 348 nm. The findings support the size and shape of the synthesized nano cellulose particles. These nanoparticles have been applied to polyester fabric by padding technique and manifested the improved physical and thermal properties. The dyeing behaviour of the treated fabrics with direct dye has also been studied and the build-up of dyes, measured as colour strength in terms of *K/S* values, reported. The higher *K/S* values are obtained when the cellulose nano is anchored in the fibre matrix, i.e. when the fibre is pre-treated and dyed with direct dyes. Improved colour strength with good resistance towards soaping is obtained after treatment of fabrics with nano cellulose.

Keywords: Absorbency; Cellulose nano; Direct dye; Physical property; Thermal property

Introduction

Textile materials made from natural fibers have played an important role in the life of human beings from time immemorial and still are widely used in the modern textiles industry for their unique properties as high quality textile materials. Due to the variation in staple length, the natural fibers with short staple length can't be used to spin yarns. Consequently, natural fibers such as wool, silk, cotton or hemp are wasted during processing and final usages. A new way of reusing these fibers has large marketing potential because of their excellent intrinsic properties. Meanwhile, not only the textile industry, but many other industries like the bio-medical industries need such bio-compatible materials [1-3].

Fine/super fine powder prepared from protein or cellulose fiber is generally known as nano-whiskers, which can impart various functional properties not only to the textiles but also contribute significantly in the field of electronics and medicines. Some potential applications of nano cellulose in the field of paper and paperboard applications as dry strength agent, surface strength agent or nanocoatings/nanobarriers, bio-nanocomposites, food applications, cosmetics/skin creams, medical/pharmaceutical applications, hygiene/absorbent products, emulsion/dispersion applications and oil recovery applications. Many researchers have reported new method of synthesis nano-whiskers and their application in bio-technological and bio-medical fields [4-7].

In this paper, nano scale cellulose polymers were prepared from viscose rayon yarns by a novel technique. The prepared cellulose nano whiskers were characterized for their size, shape and chemical composition. Nano scale cellulose was applied to polyester textiles by padding technique. Changed in physico-chemical characteristics and thermal behaviour of the new polyester fiber incorporated with nano cellulose were analyzed by SEM, FTIR, Image analyzer and computer colour matching system, the thermal behaviour of polyester cellulose nano composite were analyzed using DSC.

Materials and Experimental Methods

Material

Pure polyester woven fabric with specification as mentioned in Table 1 was used. The fabrics was cleaned with 2% sodium carbonate and 5% nonionic detergent at 70°C temperature for 15 minute then again washed and neutralized before used.

Experimental methods

Nano-cellulose was prepared by treating waste viscose rayon fibers with freshly prepared solution of sodium zincate.

Preparation of sodium zincate solution: Sodium zincate was prepared by adding 180 gms of NaOH to 200 ml of water then 80 gms of ZnO was gradually added with constant stirring. The solution was kept for 24 hours in a container. Finally, the solution was filtered using Whatman No.1 filter paper to get sodium zincate solution.

Preparation of nano cellulose: In this study, suspensions of nanocrystals were prepared from waste viscose rayon fibers the scheme for the preparation of nano cellulose is illustrated in the following Figure 1.

The waste viscose rayon fibers were ground to smaller than 20 mesh powder. Ground viscose rayon fiber powder was mixed with sodium zincate in a ratio of 1:9 (g/ml). A reaction temperature of 50°C was maintained for the diffusion of sodium zincate into the amorphous region of the fibers resulting in a subsequent cleavage of the glycosidic bonds. After 1 hour the particles were neutralized by glacial acetic acid solution. The suspension was washed and further filtered by Whatman No.1 filter paper. The colloidal suspension was evaporated aconverted in powder form. The powder was washed with distilled water and dried.

Characterization of nano cellulose particles: The particle size and size distribution of the cellulose nano were analyzed on the particle size analyzer (Malvern Instrument, MAL501131, DTS version 5.03,

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ample	Material Specification						
	Count/Denier		Ends/inch	Pick/inch	Type weave	Wt.gm/ sq.m.	Thickness (mm)
	Warp	Weft					
100% polyester	128d	146d	90	72	Plain	109.7	0.21

Table 1: Specifications of polyester fabric.

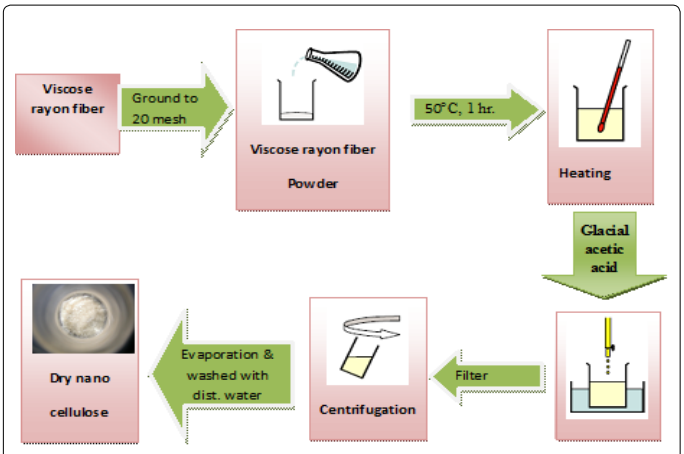

Figure 1: Scheme for preparation of cellulose nanocrystals.

U.K.). The morphology of cellulose nano nanoparticles was examined on scanning electron microscope (SEM) (Model JSM5610LV, version 1.0, Jeol, Japan). Chemical composition of prepared nano powder was analyzed by FTIR Spectroscopy Nicolet is10 FT-IR Spectrometer (Thermo Scientific).

Application of nano cellulose by *Pad–dry–cure method:* Polyester fabric samples were padded with varying concentrations of nano cellulose suspension viz., 1 gpl, 5 gpl, and 10 gpl. For 1 gpl solution, 0.1 gm nanoparticle was added in 100 ml liquor with 5 gm lissapol L surfactant. The mixture was then stirred using magnetic stirrer at 250 rpm for 30 minutes at 50°C temperature. Likewise all concentration solution was prepared. Polyester fabric samples (size: 40cm × 30cm) were immerged in padding liquor at room temperature for 10 minutes and then passed through a two bowl laboratory padding mangle, which was running at a speed of 15 rpm with a pressure of 1.75 Kg/cm^2 using 2-dip-2-nip padding sequence at 70% expression for polyester fabric. The padded substrates were dried at 80°C. The dried samples were cured in a preheated curing oven at 180°C temperature for 60 seconds.

Testing and analysis

Fabric characterization: The morphology of cellulose nanoparticles deposited on polyester fabric was observed by SEM. The samples were also observed on microscope at 100 X magnification. The images at selected places of the specimen were captured by digital camera attached to the microscope. These images were transferred to image analyzer in computer. Image analyses of these samples were carried out using Image-Pro® Plus, Version 4.1 Software of Media Cybernetics, USA. The presence of cellulose in the polymer structure was detected by FTIR Spectroscopy. The thermal characterizations of polyester fabric and nano cellulose were analyzed using Differential Scanning Calorimetric measurements (DSC) (Model 6000 from PerkinElmer, Singapore) in the temperature range 50°C to 300°C. Nano cellulose and polyester-nano cellulose composites were scanned on an X-ray diffractometer (X'Pert-Pro®, PAN Analytical, Singapore). Cu Kα radiation at 45 Kv and 40 mA was utilized and scanned for 2θ between 5°C and 40°C.

Physical testing: Before physical testing the samples were dried and conditioned at 65± 2% RH and 27 ± 2°C temperature.

Determination of tensile properties: 2 cm x 8 cm fabric samples were tested at 100 mm/min traversing speed for the determination of breaking load, breaking elongation, stress and strain. The test was performed as per B.S. 2576:1959

Determination of crease recovery angle: The test specimen was folded and compressed under controlled condition of defined force to create a folded angle, the specimen was suspended in an instrument for a controlled recovery and the recovery angle was measured. The test was performed as per AATCC test method 66-2003.

Determination of bending length: The stiffness in terms of bending length of nano treated and untreated samples were measured as per AATCC Test Method 115-2005 using Prolific stiffness tester (India).

Determination of absorbency by wicking test: Wicking behavior of the treated and untreated samples were evaluated as per T-PACC standard method.

Evaluation of water permeability: These test methods provide procedure for determining the hydraulic conductivity (water permeability) of textiles materials in terms of permittivity under standard testing conditions in uncompressed state. The test was conducted using ASTM D 4491 (Constant Head Method) water permeability test method.

Evaluation of air permeability: The air permeability of treated and untreated polyester fabric samples were measured on Metefem air permeability tester as per ASTM D 737 test method. The result of the test measured reported in m^3/h/m^2 to three significant digits.

Dyeing of nano cellulose treated and untreated fabric samples

Mild scoured polyester fabric samples (size: 40cm × 30cm) were immerged in the padding liquor containing 3 gram and 5 gram direct dye, 2 gram sodium carbonate and 5 gram glauber's salt in 100 ml liquor. Polyester sample were entered in above liquor at room temperature for 10 minutes and then passed through a two bowl laboratory padding mangle, which was running at a speed of 15 rpm with a pressure of 1.75 Kg/cm^2 using 2-dip-2-nip padding sequence at 70% expression for polyester fabric. The padded substrates were dried at 80°C. The dried samples were cured in a preheated curing oven at 180°C temperature for 60 seconds.

Evaluation of dyed samples

Measurement of colour strength value (K/S Value): The dyed samples were assessed for *K/S* values using computer colour matching system (illuminant D65/100 observer, Spectra scan 5100 RT, Spectrophotometer, Premier Colourscan Instrument, India).

Fastness tests: The light fastness of the dyed samples was tested on Fad-o-meter (FDA-R, Atlas, U.S.A.) after partially exposing the samples to the xenon arc lamp for 16 h and graded for the colour change with the ratings. The wash fastness of the samples was performed as per ISO-2 tests using launder-o-meter (Digi.wash, Paramount Scientific Instruments., India). Samples were also evaluated for the rating in terms of colour change

Results and Discussion

This section of the paper discussed the results of preparation of nano cellulose particles and their application to polyester fabrics by pad-dry-cure techniques. The prepared nanoparticles were characterized

using particle size analyzer, the morphology of the particles was observed using SEM. The nano cellulose polymers were using FTIR. The functional properties of nano treated fabrics were tested as per the standard methods of testing.

The rod-like particles that were produced as a result of treatment were dried and again washed with distilled water and dried. Figure 2 represent organization and separation of nano cellulose from the fiber.

Characterization of prepared nano cellulose

The dried powder of nano cellulose prepared is in the insect photographs shown in Figure 3. The analysis of the sample of cellulose powder dispersed in water by particle size analyzer showed a narrow and sharp peak at around 348 nm diameter.

Figure 4 shows the scanning electron micrographs of prepared nano cellulose particles deposited on carbon coated aluminum sheet. It can be seen from the figure that the shape of prepared nano cellulose particles was rod-like. The breaking of the cellulose chain, which contains high order crystalline regions, connected with low order amorphous regions, which appear like individual rods. These rod-like particles are commonly called as whiskers. FTIR spectrum of the nano cellulose powder is shown in Figure 5 and X-ray diffraction pattern of nano cellulose are shown in Figure 6.

The absorption in the region of 3600-3100 cm^{-1} was due to the stretching of -OH group and at 3000 to 2800cm^{-1} to the CH stretching. The band observed at 1642 cm^{-1} across from the H-O-H bending of the absorbed water. The symmetric C-H bending occurred at 1400 cm^{-1}; the FTIR absorption band at 1430 cm^{-1}, assigned to a symmetric CH_2 bending vibration, decreases. This band is also known as the

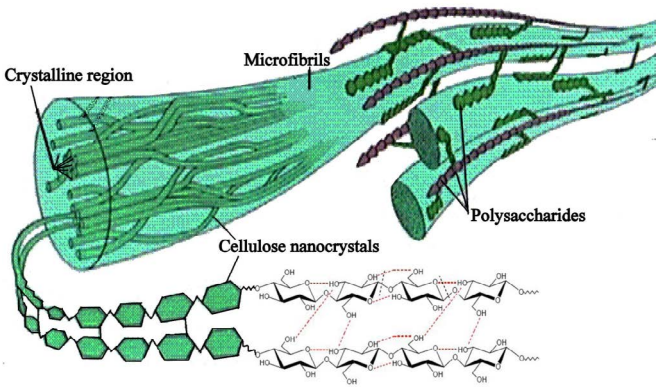

Figure 2: Chemical structure and organization of cellulose in fiber.

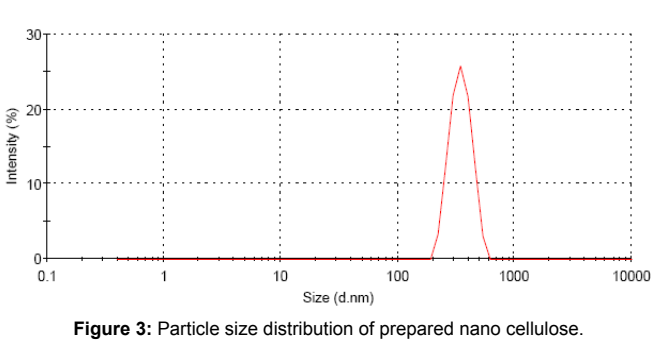

Figure 3: Particle size distribution of prepared nano cellulose.

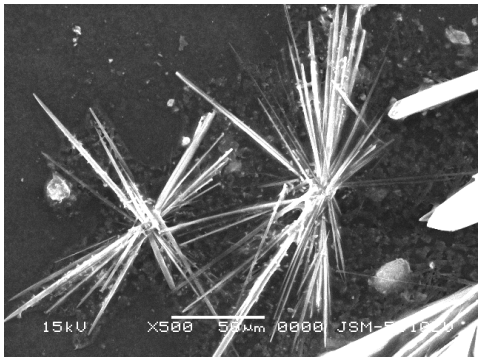

Figure 4: Scanning electron microphotographs of nano cellulose deposited on carbon coated aluminum sheet.

"crystallinity band", indicating that a decrease in its intensity reflects reduction in the degree of crystallinity of the samples. The main characteristic peaks were detected at absorption band 898 cm^{-1}, assigned to C–O–C stretching at β-(1→4)-glycosidic linkages, is designed as an "amorphous" absorption band. The IR spectra confirmed the presence of amorphous microcrystalline celluloses structure.

Characterization of polyester/nano cellulose composite

The dispersion of nanoparticles on the surface of the fiber and their penetration in the polymer matrix were examined by image analyzer (100 X). Cross sectional and longitudinal view of the sample were prepared in laboratory as per AATCC Test method 20-2005. (AATCC Technical manual, vol. 81, 2006, pp-40.). The prepared cross sectional and longitudinal sections were further stained with direct dye and examined under image analyzer. The images captured by image analyzer are shown in Figure 7.

Cellulose particles applied on 100% polyester fabric, the treated fabric was then stained with a direct dye (Congo Red BDC) to highlight the cellulose particles. Deposition of cellulose particles is seen on the surface of polyester fiber, from the longitudinal view of the polyester fiber presented in Figure 7a. Dispersion of these particles in the polymer matrix is also observed from the Figure 7b which represents the cross sectional view of polyester fiber.

Figure 8 shows the SEM images of polyester fiber surfaces after the nano-cellulose treatment. The Micro photographs captured at different magnifications i.e 500 X, 1000 X, 1500 X and 2000 X show that the fiber surface is covered with nano-cellulose particles after treatment.

The FTIR spectra of the polyester fabric before and after nano cellulose treatment are illustrated in IR spectra Figure 9a and 9b respectively. The peaks in the IR spectra of the polyester loaded with nano cellulose and untreated fabric appeared in the range of 600-4000 cm^{-1}. The waves were assigned as follows: 1715 cm^{-1} (C=O), 1409 cm^{-1} (aromatic ring), 1331 $cm^{-1}$and 1021 cm^{-1} (carboxylic ester or anhydride), and 1021 cm^{-1} (O=C–O–C or secondary alcohol), 967 cm^{-1} (C=C), 869 cm^{-1} (five substituted H in benzene). The peak at 1409 cm^{-1} corresponded to the aromatic ring. It was the characteristic absorption peak of PET. The peak at 1715 cm^{-1} was assigned to the ester group.

No significant changes in the spectra were observed after nano cellulose treatment for polyester portion appears over the course. But the spectra also confirmed the presence of cellulose from the absorption peaks in the region of 3600-3100 cm^{-1} due to the stretching of –OH group; at 3000 to 2800 cm^{-1} to the CH stretching; 1642 cm^{-1} across from

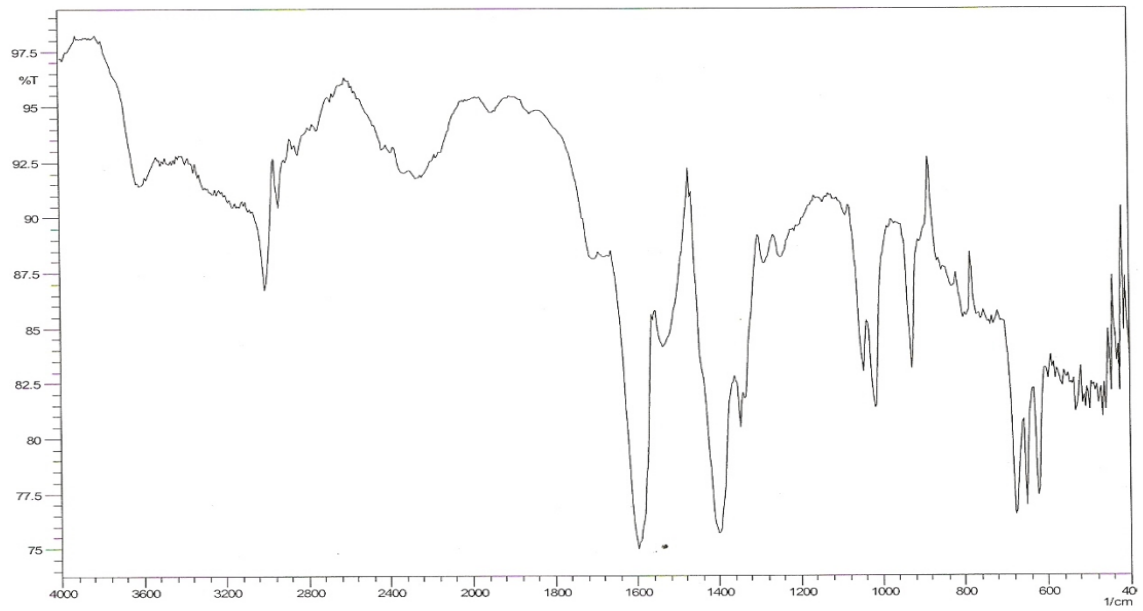

Figure 5: IR Characterization absorption peak of prepared nano cellulose powder.

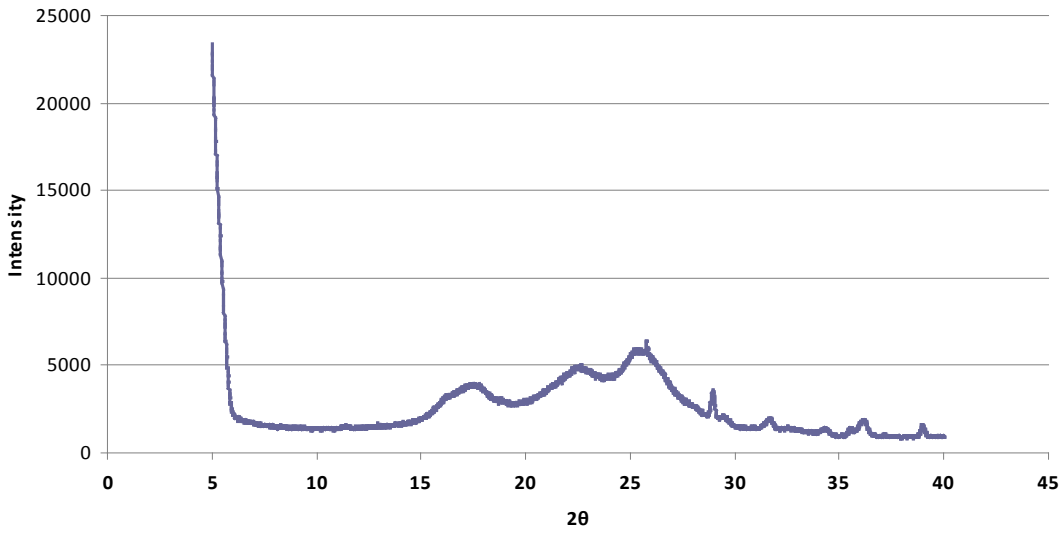

Figure 6: X-ray diffraction pattern of nano cellulose.

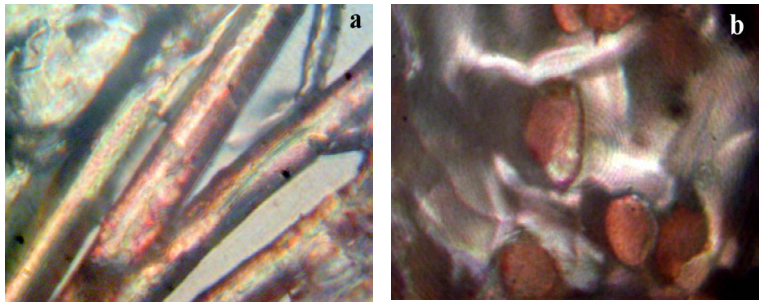

Figure 7: Images (100X) of polyester substrate treated with nano-cellulose and stained with congored direct dye (a) longitudinal and (b) cross sectional view.

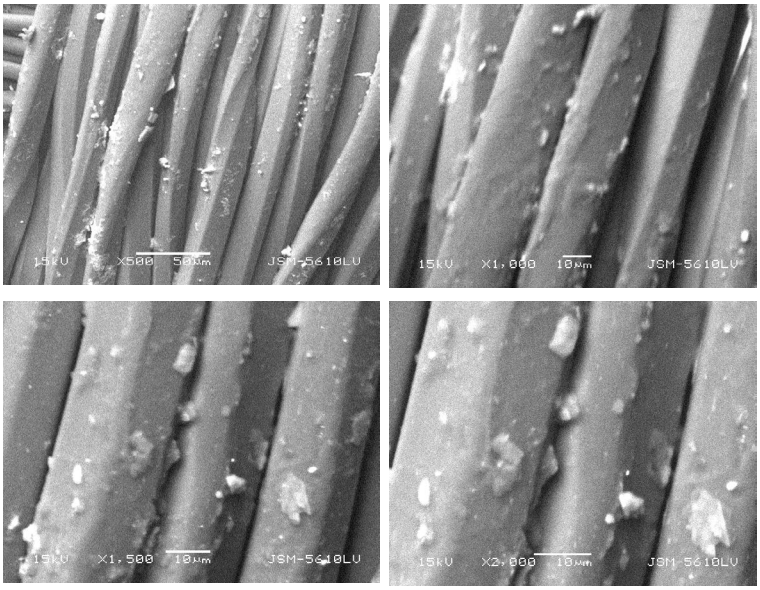

Figure 8: Polyester fiber surface morphology after treatment with the nano-cellulose treatment.

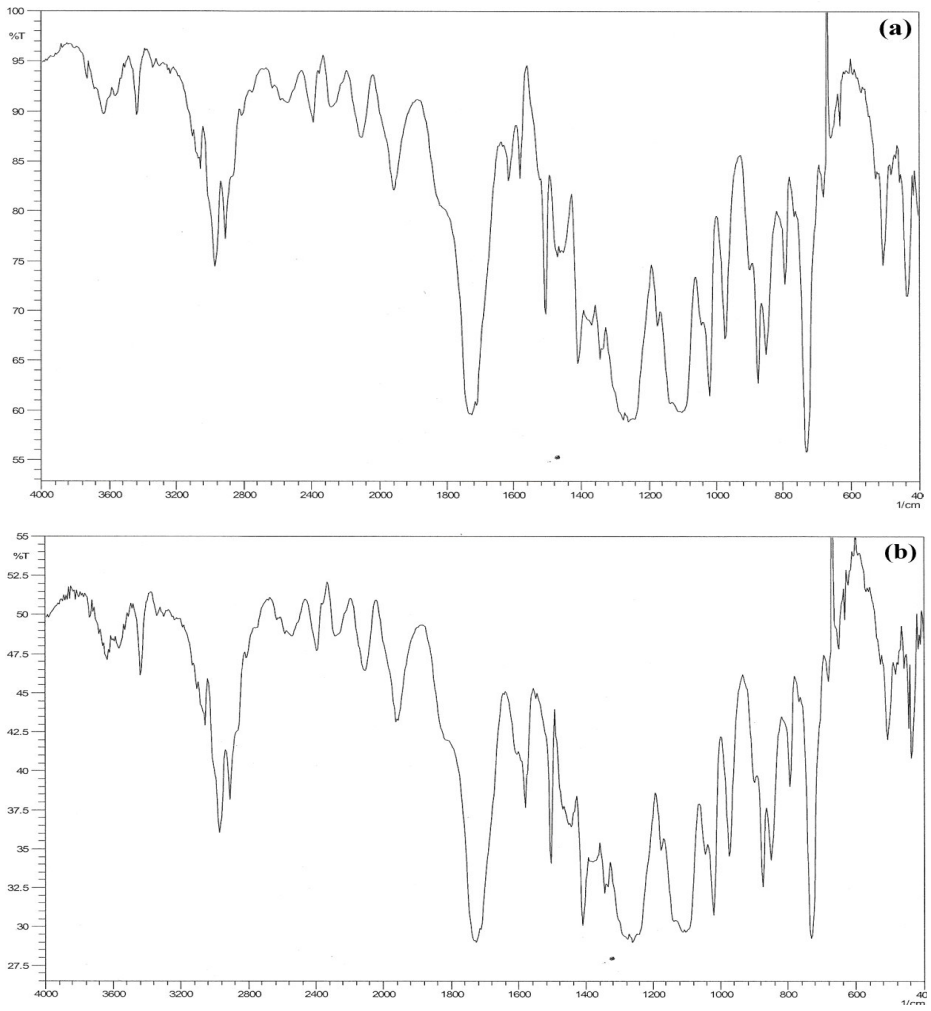

Figure 9: FTIR spectra of (a) pure polyester fabric and (b) polyester fabric treated with nano-cellulose.

the H-O-H bending of the absorbed water; symmetric C-H bending occurred at 1430 cm^{-1}; at absorption band 898 cm^{-1}, assigned to C–O–C stretching.

Thermal analysis

The thermal reactivity of synthesized nano powder (C), polyester fabric treated (B) and untreated (A) were then studied. The onset temperatures for samples were determined using Differential Scanning Calorimetric measurements (DSC). The sample A and B showed the onset temperature at 250.32°C and 250.73°C respectively. The slight increase in onset temperature can be attributed due to the incorporation of cellulose nano into the polyester fiber matrix; however, the effect is not very significant. For the nano cellulose powder the onset temperature at 54.29°C. The reactivity of the untreated PET was then compared with the PET treated with cellulose nano powder, which revealed little difference in reactivity as recorded by DSC. The various onset temperatures for samples are shown in Table 2 and DSC curves registered in the 50-300°C temperature range are shown in the Figure 10.

To study the effect of nano cellulose treatment on thermal degradation of polyester fabric was analyzed. The endothermic peaks characteristic of nano cellulose, occurred at 57.59°C on the DSC curves is presented in Figure 11. A shift of the maximum temperature of the nano treated sample to higher values may be observed with the decrease of the crystallinity degree. Thus, in the case of untreated polyester fabric A, the maximum temperature of the peak appears at 251.72°C, followed by the PET sample treated with nano cellulose B at 253.18°C noticed for the 50-300°C region, which corresponds to the incorporation of nano cellulose in polyester fiber matrix. This behavior is explained by the fact that the thermal degradation reaction starts in the amorphous domain of the cellulosic materials by statistical degradation of cellulose. In the present case, the amorphous content increases from pure polyester to polyester/nano cellulose material.

Effect of nano cellulose on physical properties of polyester fabric

Treated and untreated polyester fabrics were evaluated for the change in physical properties in terms of breaking load and crease recovery angle.

Effect on tensile strength: The treated and untreated polyester

Compound	Onset temperature in °C	Endothermic peak °C	ΔH, J/g	Area of the endothermic peak, mJ
A- Pure PET	250.32	251.72	67.77	67.77
B- PET/Nano-cellulose	250.73	253.18	67.28	45.75
C- Nano - cellulose powder	54.29	57.59	121.27	179.48

Table 2: DSC results.

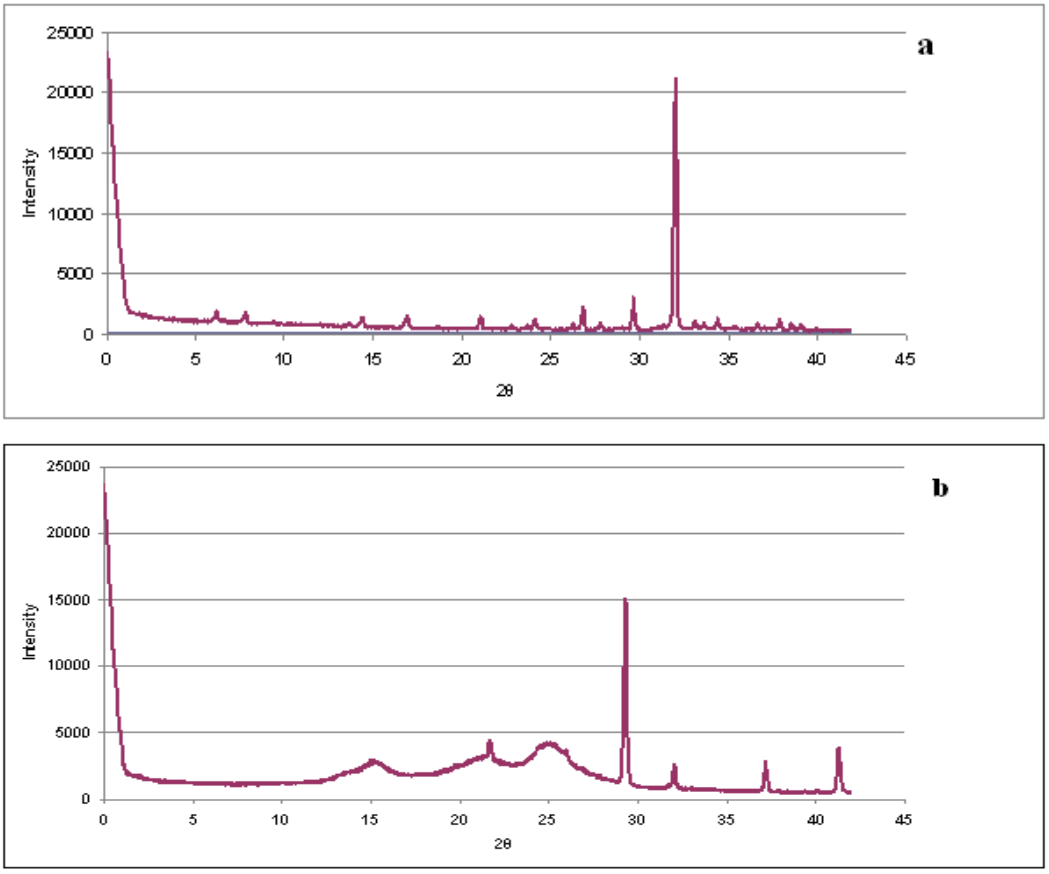

Figure 10: X-ray diffraction pattern of a) pure polyester and b) polyester treated with nano cellulose.

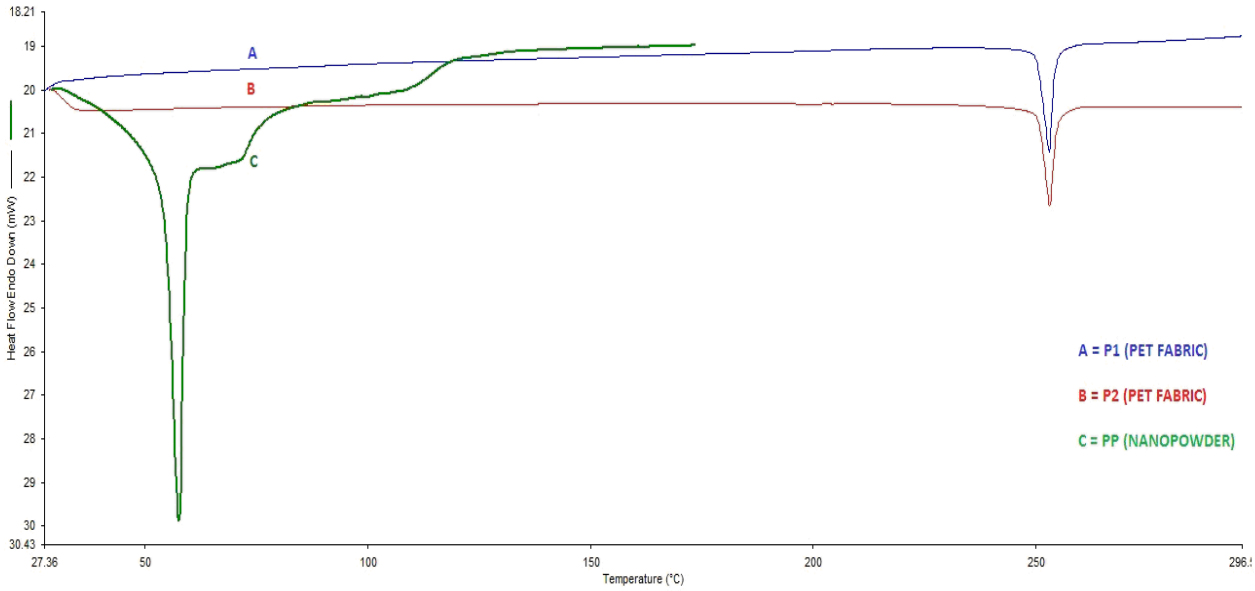

Figure 11: DSC curves registered in the 50-300 °C temperature range for untreated polyester (A), polyester treated with nano-cellulose (B) and nano-cellulose powder (C).

samples were tested to evaluate change in tensile strength; the results are shown in Table 3. The results show that the application of nano cellulose particles to polyester fiber causes an improvement in the load bearing capacity of the fiber. It may be due to the more amount of nano cellulose diffused in the polymer matrix.

Effect on crease recovery: The result presented in Table 4 shows minor improvement in crease recovery angle of the treated samples. The nano cellulose particles because of their small size can enter in between the polymer molecules and perhaps act as filler or cross linking agent. The crease recover angle of fabric was improved with increase in the concentration of nanoparticle. The improvement in physical properties is due to the mechanical interlocking caused by the mechanical anchoring of the nano cellulose in the intermolecular pores.

Effect of nano cellulose on water absorbency of polyester fabric

The absorbency of polyester fabric treated with nano cellulose was measured by drop test and wicking behavior test. Tables 5 and 6 show results of absorbency of the fabric using drop test and wicking height.

It is found from the results shown in Table 4 that the water absorbency of polyester fabric treated with 1 gpl concentration of nano cellulose take less time for the absorption of water indicates improvement in absorbency. But as the concentration of nano cellulose was increased to 5 gpl and 10 gpl, the water droplet took more time to absorb water drop indicating reduction in absorbency of nano treated material.

The improvement in hydrophilicity of polyester treatment due to low dosage of nano cellulose treatment is because of the inherent hydrophilicity of cellulose. But interestingly as the concentration of nano cellulose was increased it started hindering the penetration of the water molecules as they start acting as nano whiskers and do not allow the water drop to be accommodated within the interpolymeric spaces.

Effect of nano cellulose on water permeability of polyester fabric

Table 7 shows reduction in water permeability through the polyester fabric sample treated with nano cellulose compared to untreated sample, it may be attributed due to the resistance offered by the nanoparticle present in the polymer matrix towards the flow of water through the fabric. It can also be seen that as the concentration of nanoparticle in fabric was increased, the permeability of water was reduced.

Effect of nano cellulose on air permeability of polyester fabric

The treated polyester fabric were tested for air permeability and compared with air permeability of untreated polyester sample. The results presented in Table 8 show that the air permeability of polyester fabric treated with nano cellulose was reduced compared with the untreated sample. It may be due to the resistance of the nanoparticles present in the polymer matrix towards the flow of air through the fabric sample.

Effect of nano cellulose on dyeing of treated fabric with direct dye

The dyeing of polyester fabric treated with 5 gpl nano cellulose using pad-dry-cure method and subsequently dyed with direct dye (Congo red BDC) using pad-dry-cure method. The results in terms of colour strength and colour coordinate values are reported in Table 9. From the table it can be seen that the polyester fabric dyed without nano cellulose treatment is stained only. It can be observe from the table that as the concentration of dye in the dye bath increases the *K/S* value of the sample also increases in case of treated polyester fabric subsequently dyed with direct dye. The treated and dyed samples were further soaped with 5% (v/v) non ionic detergent at 70°C temperature for 15 minutes. The results shows that the loss in *K/S* values of these samples still remain higher than the samples dyed without the nano treatment. The dyeing of nano cellulose treated polyester fabric with

direct dye may be attributed due to the presence of cellulose particles in the polyester structure.

Conclusions

- FTIR spectral analysis results confirm the formation of nano cellulose from the waste viscose rayon fiber by treatment with sodium zincate.
- The z-average size of the separated cellulose nano whiskers is found to be 348 nm. The SEM images indicate that cellulose crystals produced are of rod-like shape.
- The treatment with cellulose nano whiskers improves the breaking load and crease recovery angle with almost no effect on rigidity of the material.
- Cellulose nano treatment to polyester also alters the thermal property, It is found that incorporation of cellulose nano slightly increase the onset temperature and The endothermic peak of nano cellulose treated polyester, occurred at 253.18°C.
- Cellulose nano treatment improves absorbency and reduces water and air permeability of polyester fabric.
- Nano cellulose treatment enhances the colour strength of polyester fabric dyed with direct dyes and also improves the fastness towards soaping.

Sample	Tensile Strength (kgf)	
	Bre.load(kgf)	Extention(mm)
Control Polyester	105.6	63.07
Polyester treated with 1 g/L nano cellulose	106.7	63.03
Polyester treated with 5 g/L nano cellulose	108.3	64.77
Polyester treated with 10 g/L nano cellulose	110.5	66.05

Table 3: Effect of nano cellulose treatment on tensile strength of sample.

Sample	Crease recovery angle (°)
Control Polyester	148
Polyester treated with 1 g/L nano cellulose	157
Polyester treated with 5 g/L nano cellulose	158
Polyester treated with 10 g/L nano cellulose	162

Table 4: Crease recovery angle of nano cellulose treated polyester fabric.

Polyester fabric treated with nano cellulose (grams/liter)	Time (sec)
Untreated sample	20.10
1	12.0
5	76.0
10	110

Table 5: Effect on absorbency of polyester fabric due to the nano cellulose treatment.

Polyester fabric treated with nano cellulose (grams/liter)	Wicking height(mm)		
	1min	5 min	10 min
Untreated sample	20	40	55
1	22	44	57
5	15	29	41
10	11	22	31

Table 6: Effect of nano cellulose treatment on wicking height (mm) of polyester fabric.

Polyester fabric treated with nano cellulose (grams/liter)	Ψ-Water Permeability (S^{-1})
Untreated sample	0.3253
1	0.3223
5	0.3087
10	0.2965

Table 7: Effect of nano cellulose on water permeability of polyester fabric.

Polyester fabric treated with nano cellulose (grams/liter)	Air permeability ($m^3/m^2/h$)
Untreated sample	223.14
1	215.33
5	211.47
10	195.21

Table 8: Effect of nano cellulose treatment on air permeability of polyester fabric.

Concentration of dye (gpl)	sample	*K/S*	Sample	*L**	*a**	*b**
50	Treated with 5 gpl nano cellulose	23.84		66.81	29.66	7.51
	Soaped	14.44		66.51	27.97	6.84
	Without nano cellulose treatment	6.05		66.06	20.15	6.80
30	Treated with 5 gpl nano cellulose	21.71		66.83	29.48	7.65
	Soaped	5.15		65.94	23.78	5.74
	Without nano cellulose treatment	2.29		66.34	14.05	7.77

Table 9: Colour strength and co-ordinate values of polyester fabric pretreated and dyed with direct dye (Congo red BDC).

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