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Effect of Silica Nanoparticles and BTCA on Physical Properties of Cotton Fabrics

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Silica nanoparticles particles were synthesized from rice hulls and characterized. The particles were found to be amorphous in nature, ranging in size from 50 to 100 nm. The concentration of silica nanoparticles, pH and curing time were taken as independent variables to design the experiment. Box-Behnken method has been used to derive the experimental plan and fifteen experiments were conducted. Regression equations have been formed with the dependent and independent variables and the results of all possible combinations have been derived. The combination of optimized concentration of BTCA and SHP were used as crosslinking agent and catalyst respectively and silica nano particles were used to enhance the physical properties of the cotton fabric. The effect of pH and curing time on physical properties were analysed by FTIR studies. By ranking method the best combination of process parameters were identified. From this study, it was observed that higher concentration of silica nanoparticles with BTCA improve the crease recovery angle and tensile strength. FTIR studies revealed that the increase of pH and curing time increases the ester carbonyl band intensity ratio.

Keywords: *silica nanoparticles, BTCA, box Behnken method, crease recovery*

1. Introduction

Finishing is a process step to add value to the textile product. It is classified into two categories such as physical finishes and chemical finishes. Functional finishes are one of the chemical finishes incorporated into the textile materials to enhance the functional and comfort properties[1]. Nanotechnology has the potential to create new bulk materials with new bulk properties in textile coating and finishing[2]. Nano particles can provide high durability for fabrics, because of its larger surface area-to-volume ratio and high surface energy. These can present better affinity for fabrics and leading to an increase in durability without affecting their breathability or hand feel[3-5].

Cotton is poor in crease resistance due to its free hydroxyl groups. To enhance the crease recovery property of cotton, the free hydroxyl groups are either removed or cross linked by applying formaldehyde and non formaldehyde based cross linking agents. The hazardous nature of the formaldehyde cross linking agents is replaced by non formaldehyde cross linking agents such as citric acid and BTCA (1,2,3,4-butanetetracarboxylic acid). The decrease of whiteness index of cotton fabric treated with citric acid tends to increase the usage of BTCA.

The mechanism of cross linking of cellulose with polycarboxylic acids is given in Figure 1. The mechanism of polycarboxylic acids is carried out in two steps by cellulose esterification. In the first step the formation of a cyclic anhydride intermediate by the dehydration of two carboxyl groups is carried out. In the second step the ester crosslinks are formed by the reaction between the anhydride with cellulose[6]. Polycarboxylic acid esterifies cellulose through the formation of a five-membered cyclic anhydride intermediate by the dehydration of two adjacent carboxyl groups[7]. The formation of cyclic anhydrides at lower temperatures is slow without catalyst, but the catalyst sodiumhypophosphite (SHP) accelerates the cyclic anhydride process[8,9]. High curing temperature and high BTCA concentration reduce the tensile strength and increase the crease recovery angle[10]. The whiteness index of the treated fabrics seems to decrease as the concentration of cross linkers increases[11-15]. The quantity of the cyclic anhydride intermediate formed in a cotton fabric increased as the pH of a finish bath was reduced from 4.5 to 1.5[16,17].

The metal oxide nanoparticles such as SiO_2, TiO_2, ZnO and Fe_2O_3 are used as UV blocking agents, antimicrobial agents, water repellents etc.[18-20].

The thermodynamic affinity of fibres form noncovalent interactions such as dipolar–dipolar and hydrogen bond, should be the key interactions for the adhesion of silica particles. The Si–O–cellulose covalent bonds in coated cellulose fabrics, make less contribution towards the adhesion of particles. Moreover, the cellulose in cotton (~98%) fabric plays a decisive role in the intensity of covalent bond between the cellulosic hydroxyl group and –Si–OH in silica particles[21,22]. The bond formation can improve the crease resistance property of the cotton fabrics. The crease recovery finishing process with DMEU, tetraethoxysilane (TEOS) and isoproponal at various volumes improved the crease resistance and tensile strength[23].

In most of the techniques to synthesis the nano silica, the base material is taken from the chemically available precursors such as tetraethoxysilane (TEOS) by sol-gel method[23]. But in the case of thermal degradation, the source material taken from natural resource such as rice hulls is the simplest and cost effective method for large scale production of silica nano particles[24]. In this research work, the synthesized silica nanoparticles are coated on the surface of the fabric with BTCA and SHP to analyse the crease recovery and other physical properties of the fabric and also the process parameters are optimized.

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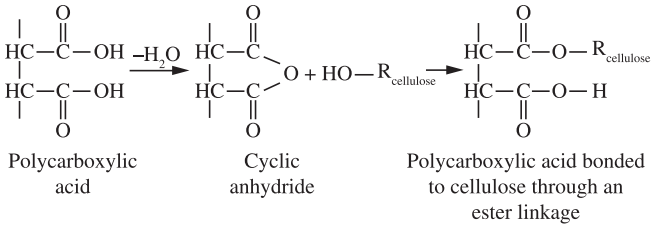

Figure 1. The mechanism of cross linking of cellulose and polycarboxylic acids.

Table 1. Experimental plan of process parameters with different levels for silica nanoparticles and BTCA coating process.

Variables	Process parameters	Levels		
		–1	0	+1
X_1	Concentration of silica nanoparticles (%)	1	1.5	2
X_2	pH	1.52	2.39	3.01
X_3	Curing time in seconds	60	120	180

2. Experimental

Amorphous silica nanoparticles were prepared by thermal degradation method from natural resource of rice hull. The rice hulls were rinsed, dried and aged at 80 °C for 1 hour and converted to micro particles. The particles were refluxed with 10% HCl for 30 minutes and dried at 80 °C. The residue was kept in a muffle furnace at 750 °C for 3 hours to convert nano particles. The synthesized silica nanoparticles were characterized by X-Ray Diffractometer (LabX XRD-6000) to find out the nature of the particles. Scanning Electron Microscope (HITACHI S-3400 SEM) and High Resolution Transmission Electron Microscope (FEI Quanta FEG 200) are used to find out the size of the silica nanoparticles.

Three levels and three variables were selected to design the experiment by Box-Behnken method and it is given in Table 1. Fifteen different combinations of experiments were derived and conducted, each combination has different process parameters such as concentration of silica nanoparticles, pH and curing time and it is given in Table 2. In each experiment, the pad solution contains different concentration of silica nanoparticles (1, 1.5 and 2%), 1,2,3,4-butanetetracarboxylic acid (BTCA) 6.5% and sodium hypophosphite (SHP) (both from Sigma-Aldrich) 4.5% with a fabric softener of silicone emulsion(1%, Finox HSS), acrylic binder (Texacryl binder ELT) and wetting agent (0.1%, Lissapol-N). The pH (1.52, 2.39 and 3.01) of the each experiment varied as per the experimental plan. The prepared solution has been coated on the surface of cotton fabric (134 ends.inch^{-1}, 64 picks.inch^{-1}, 120 g.m^{-2}, 40 × 40 seconds count and plain weave) to impart crease recovery property by pneumatic padding mangle with the material-to-liquor ratio of 1:5 and 100% wet pickup was maintained for all experiments. Then, it was squeezed and dried at 85 °C for 5 minutes and subsequently cured at 180 °C at different curing intervals of 60, 120 and 180 seconds. The cured fabrics were treated with 0.1 M NaOH solution at room temperature for two minutes to convert the free carboxyl groups to carboxylate, rinsed with cold water and dried at 80 °C for 5 minutes.

A quadratic polynome was used to analyze the relationship of each response with the three independent variables. The multiple polynomial regression equation was used to derive the regression co-efficients to analyse the fabric properties and is given in Equation 1.

$$Y = b_0 + \sum_{i=1}^{3} b_i x_i \sum_{\substack{i=1 \\ i<j}}^{3} b_{ij} x_i^2 + \sum_{i=1}^{3} b_{ij} x_i x_i \quad (1)$$

where b_0, b_i, b_{ii} and b_{ij} are the co-efficients of the regression equations, i, j are integers and Y is the response of the dependent variable.

The physical properties of treated and untreated fabrics were tested as per ASTM test methods such as crease recovery angle (D 1295-67), tensile strength (D 5035-95), tearing strength (D 1424-96), weight loss due to abrasion (D 4966-98), air permeability (D 737-96), flexural rigidity (D 1388-08) and whiteness index (E 313-98) and the results are given in Table 2.

2.1. Quality factor and ranking method

Quality factor has been derived for fabric properties which consist of the importance of each property given by this experiment. Higher priority has been given to crease recovery angle and surface coating processes generally reduce the strength behavior and permeability of the fabric. Hence it is considered next to crease recovery respectively. Equation 2 has been used to calculate the quality factor. The quality factor has been ranked and it is given in Table 4.

$$X = a\,0.3 + b\,0.2 + c\,0.15 + d\,0.1 + e\,0.1 + f\,0.05 + g\,0.1 \quad (2)$$

Where X = quality factor

- a = crease recovery angle in degrees
- b = whiteness index
- c = tensile strength in kg
- d = tearing strength in g
- e = flexural rigidity in g.cm^{-1}
- f = weight loss due to abrasion in g
- g = air permeability in cm^3.cm^{-2}/s

3. Results

3.1. Characterization of silica nanoparticles

The synthesized silica nanoparticles were characterized by X-ray Diffractometer (XRD) using CuK_α ($\lambda = 1.54$ Å) as a radiation source. The XRD patterns of the silica nanoparticles obtained from rice hulls is shown in Figure 2. The powder diffraction pattern indicates a broad peak at $2\theta = 22°$, which reveals the amorphous nature of the silica nanoparticles. Further, the XRD pattern confirms the absence of any ordered crystalline structure.

High Resolution Transmission Electron Microscope (HRTEM) was used to characterize the particle size and the image of nano particles are shown in Figure 3. It shows that the particles size is below 100 nm.

3.2. Analysis of physical properties

The physical properties of the coated and uncoated fabrics were analysed and are given in Table 1. Regression equations were derived for each property and are given in Table 3. The results of all possible combinations of process parameters have been derived and are given in Table 4.

3.3. FTIR analysis of coated fabric

The infra red spectroscopy data demonstrated that ester cross linkages in a finished cotton can be examined and evaluated on a semiquantitative basis by measuring the ester band intensity and carbonyl band intensity ratio (ester/carboxylate). The carbonyl band intensity ratio (ester/ carboxylate) is a function of the average number of ester groups formed for each BTCA molecule, so it represents the effectiveness of this bonded BTCA molecule as a crosslinking agent[17].

Table 2. Properties of untreated, silica nanoparticles and BTCA coated samples.

Sample No.	Process parameters									
	Nano Silica %	pH	Curing time in seconds	Crease recovery angle (w + f)	Whiteness index	Tensile strength in kg	Tear strength in g	Flexural rigidity ($mg.cm^{-1}$)	Weight loss (%) due to abrasion	Air permeability ($cm^3.cm^{-2}/s$)
U1	-	-	-	94	82.2	57.6	1312	1622	1.49	26.25
T1	1	1.52	120	180	71.5	63.4	1314	1822	2.93	26.20
T2	2	1.52	120	200	68.2	66.3	1386	1891	2.58	21.30
T3	1.5	1.52	180	196	69.1	62.5	1374	1838	2.64	24.30
T4	1.5	1.52	60	192	70.2	64.7	1365	1832	2.50	24.89
T5	2	2.39	180	221	67.2	71.2	1461	1916	2.05	21.90
T6	1	2.39	180	194	70.5	65.4	1340	1820	2.16	26.30
T7	2	2.39	60	218	68.5	69.3	1442	1902	1.95	21.78
T8	1	2.39	60	189	72.3	64.2	1335	1826	1.98	26.80
T9	1.5	3.01	60	212	72.5	68.5	1415	1843	1.64	24.30
T10	1.5	3.01	180	215	71.8	65.2	1424	1876	2.48	24.60
T11	2	3.01	120	225	70.5	73.5	1485	1920	1.84	21.90
T12	1	3.01	120	201	72.5	67.1	1398	1824	1.74	26.30
T13	1.5	2.39	120	205	69.8	67.8	1407	1856	1.82	24.90
T14	1.5	2.39	120	207	70.2	67.5	1405	1860	1.89	24.50
T15	1.5	2.39	120	205	70.1	67.4	1405	1859	1.85	24.80

U – untreated, T – treated.

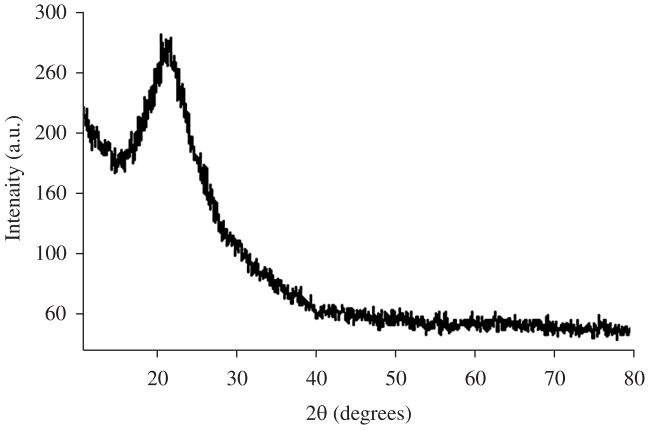

Figure 2. XRD pattern of silica nanoparticles.

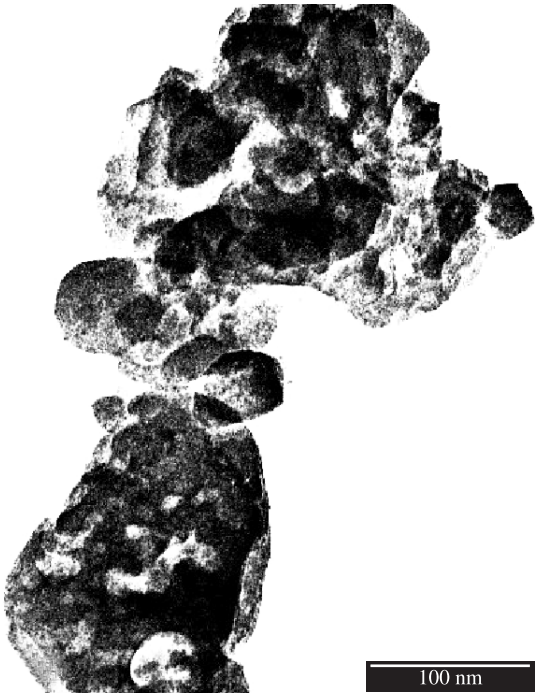

Figure 3. HR TEM image of silica nanoparticles.

Figure 4 shows the FTIR spectroscopy of silica nanoparticles and BTCA coated fabrics with different pH and curing time. From the figure it was observed that the carbonyl band at 1722 to 1735 cm^{-1} represents the carbonyl of ester while the band at 1577 to 1586 cm^{-1} and it represents the carbonyl of carboxylate[17]. The above two are the basic forms of the free carboxylic acids.

Table 5 shows that the values of ester carbonyl band intensity and carbonyl band intensity ratio. The values for the above are 0.516 to 0.545 and 0.619 to 0.636 respectively. By increasing the curing time from 60 to 180 seconds and the pH value of 1.52 to 3.01 with the BTCA concentration of 6.5%, the ester carbonyl band intensity and carbonyl band intensity ratio were increased. Carboxyl is the predominant species in the BTCA solution when the pH ranges from 1.52 to 2.39 which is demonstrated by the intense carboxyl carbony band at 1725 cm^{-1}[17].The conversion of carbonyl to carboxylate reaches maximum when the pH of the BTCA solution is 3.01[17]. When the carbonyl band intensity ratio increases, the cross linking increases. It is evident that the crease recovery angle has been increased proportionally. Choosing the optimum pH range will reduce the quantity of BTCA for desirable fabric performance, thus reducing the cost of non formaldehyde agents[17].

Table 3. Regression equations for silica nanoparticles and BTCA coated fabric properties.

S. No.	Property	Regression equation	R	R^2	F-ratio
1.	Crease recovery angle	$Y = 101.277\ X_1 + 67.022X_2 + 0.264\ X_3 - 18.367\ X_1^2 - 8.896X_2^2 - 4.110X_3^2 - 0.0801X_1X_2 - 0.045\ X_2X_3$	1.000	0.935	2572
2.	Whiteness index	$Y = 72.462 - 3.008X_1 - 1.526X_2 - 0.010X_3$	0.897	0.805	15.17
3.	Tensile strength	$Y = 45.899X1 + 21.343X_2 + 0.161X_3 - 6.658X_1^2 - 2.857X_2^2 - 0.001X_3^2 - 1.854X_1X_2 - 0.029X_1X_3 - 0.015X_2X_3$	0.999	0.722	1048
4.	Tear strength	$Y = 761.826X1 + 458.910X_2 + 3.031X_3 - 151.203X_1^2 - 60.028X_2^2 - 0.005X_3^2 - 56.395X_1X_2 - 0.505X_1X_3 - 0.385X_2X_3$	1.000	0.690	1135
5.	Weight loss	$Y = 3.839X1 - 1.048X_2 + 0.012X_3 - 0.832X_1^2 + 0.184X_2^2 - 0.241X_1X_2 - 0.006X_1X_3 + 0.001X_2X_3$	0.987	0.539	49
6.	Flexural rigidity	$Y = 1683.152 + 86.75X1 + 15.997X_2 + 0.074X_3$	0.966	0.933	50.75
7.	Air permeability	$Y = 15.388X1 + 11.210X_2 + 0.067X_3 - 5.032X_1^2 - 1.744X_2^2 - 1.32X_1X_2 - 0.012X_1X_3 - 0.008X_2X_3$	0.999	0.846	767

Table 4. Calculated silica nanoparticles and BTCA coated fabric properties using regression equations.

Sample No.	Nano silica %	PH	Curing time	Crease recovery angle (w + f)		Whiteness index		Tensile strength		Tear strength		Flexural rigidity $mg.cm^{-1}$	Weight loss in %	Air permeability in $cm^3.cm^{-2}/s$	Quality factor	Rank
				Degrees	Increase in %	WI	Retention %	kg	Increase in %	g	Increase in %					
1	2	3.01	180	226	140.43	69.24	84.23	70.74	22.81	1410	7.47	1918	1.95	24.05	427.6	5
2	2	3.01	60	223	137.23	70.44	85.69	92.35	60.33	1451	10.59	1909	1.59	21.78	433.1	1
3	2	3.01	120	222	136.17	69.84	84.96	85.14	47.81	1448	10.37	1914	1.77	22.91	431.9	2
4	2	1.52	180	204	117.02	66.97	81.47	67.77	17.66	1403	6.94	1894	2.72	25.2	417.1	14
5	2	1.52	60	200	112.77	68.17	82.93	86.7	50.52	1374	4.73	1885	2.54	21.5	414.8	15
6	2	1.52	120	202	114.89	67.57	82.20	80.83	40.33	1407	7.24	1890	2.63	23.35	418.4	13
7	2	2.39	180	218	131.91	68.29	83.08	71.04	23.33	1439	9.68	1908	2.17	25.47	427.1	6
8	2	2.39	60	218	131.91	69.49	84.54	91.54	58.92	1451	10.59	1899	1.89	22.6	430.4	4
9	2	2.39	120	218	131.91	68.89	83.81	84.89	47.38	1463	11.51	1904	2.03	24.03	431.1	3
10	1.5	3.01	180	215	128.72	70.74	86.06	64.75	12.41	1424	8.54	1875	2.39	28.23	421.2	10
11	1.5	3.01	60	214	127.66	71.94	87.52	84.69	47.03	1434	9.30	1866	1.67	25.24	423.9	8
12	1.5	3.01	120	214	127.66	71.34	86.79	78.32	35.97	1447	10.29	1870	2.03	26.73	424.7	7
13	1.5	1.52	180	196	108.51	68.47	83.30	60.4	4.86	1375	4.80	1851	2.98	28.39	407.1	16
14	1.5	1.52	60	187	98.94	69.67	84.76	77.65	34.81	1316	0.30	1842	2.44	23.97	400.0	23
15	1.5	1.52	120	192	104.26	69.07	84.03	72.63	26.09	1363	3.89	1846	2.71	26.18	406.0	18
16	1.5	2.39	120	210	123.40	70.4	85.64	64.48	11.94	1445	10.14	1860	2.22	27.44	420.1	11
17	1.5	2.39	60	207	120.21	71	86.37	83.3	44.62	1417	8.00	1856	1.89	25.65	418.8	12
18	1.5	2.39	180	212	125.53	69.8	84.91	77.49	34.53	1436	9.45	1865	2.54	29.24	422.3	9
19	1	3.01	180	200	112.77	72.25	87.90	59.5	3.30	1362	3.81	1831	2.42	29.89	405.8	19
20	1	3.01	60	195	107.45	73.45	89.36	73.69	27.93	1342	2.29	1822	1.33	26.18	403.3	20
21	1	3.01	120	197	109.57	72.85	88.63	68.16	18.33	1370	4.42	1827	1.88	28.03	406.5	17
22	1	1.52	180	179	90.43	69.97	85.12	58.52	1.60	1271	-3.13	1808	2.83	29.07	387.4	25
23	1	1.52	60	165	75.53	71.17	86.58	65.28	13.33	1282	-2.29	1799	1.92	23.93	384.1	27
24	1	1.52	120	172	82.98	70.57	85.85	61.09	6.06	1275	-2.82	1803	2.38	26.5	385.4	26
25	1	2.39	180	196	108.51	71.3	86.74	59.26	2.88	1357	3.43	1821	2.49	30.49	402.9	21
26	1	2.39	60	187	98.94	72.5	88.20	71.73	24.53	1308	-0.30	1813	1.48	26.18	396.2	24
27	1	2.39	120	192	104.26	71.9	87.47	66.76	15.90	1350	2.90	1817	1.98	28.34	401.6	22

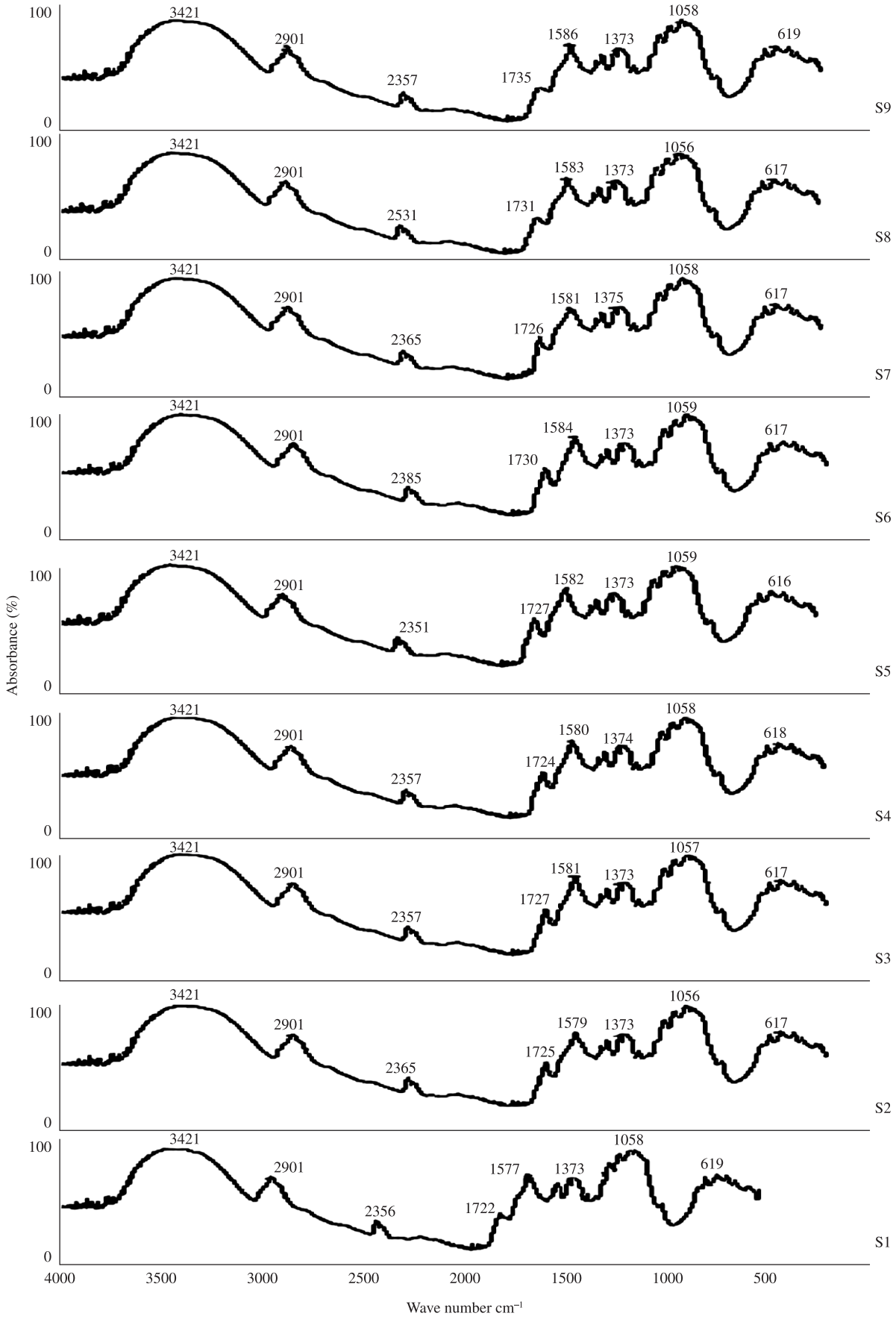

Figure 4. FTIR spectroscopy of silica nanoparticles and BTCA treated fabrics.

4. Discussion

4.1. Influence of process parameters on crease recovery angle

Nanometregrade metal oxides such as TiO_2, Ag, $MgCl_2$, SiO_2 and ZrO_2 are used as a co-catalyst along with non formaldehyde cross linking agents to improve the crease recovery properties of cotton fabric[25]. As the particle size of the TiO_2 is in nano-scale, the nano TiO_2 could fill the amorphous region of the CellOH, and hence, the presence of nano TiO_2 inside the fibre would probably restrict the molecular movement of cellulose[25].

Silica nanoparticles intrude more easily into the interior of cotton fibre and adhere more tightly to the fibre structure[21]. It was observed that, the increase of concentration of silica nanoparticles contributes more to increase the crease recovery angle of the fabric due to restriction of the molecular movement in the fibre structure than pH. During the curing process, the polycarboxylic acid reacts with the cellulose molecules of cotton fabric most probably through the formation of cyclic anhydrides as reactive intermediates, which, in turn, esterify cotton cellulose. Due to increase of ester carbonyl band intensity ratio while increasing the pH from 1.52 to 3.01, the crease recovery property has also been increased[17]. The increase in curing time increased the crease recovery angle due to the formation of more esterification of cellulose and the Figure 5 shows its contribution is minimum when compared to silica nanoparticles and pH.

4.2. Influence of process parameters on whiteness index

Citric acid is one of the nonformaldehyde cross linking agents, the cotton fabric yellowing caused by citric acid at elevated temperatures and the yellowness increases as curing temperature, curing time, and CA concentration increase. Using sodium hypophosphite as a catalyst causes less fabric yellowing than monosodium phosphate. Therefore, yellowing caused by citric acid and other hydroxyl multi functional carboxylic acids can probably be attributed to the formation of unsaturated polycarboxylic acids[26].

High acidity of chemicals is a source of causing fabric tendering and yellowing[27], hence the pH increases the acidity level reduces and the whiteness of the fabric has been increased and it is shown in Figure 6. The increase in curing time and silica nanoparticles concentration gradually reduces the whiteness index of the fabric. The reduction of whiteness index up to 19% is due to higher concentration of silica nanoparticles, higher curing time and lower pH.

4.3. Influence of process parameters on tensile strength

Cross linking between cellulose molecules causes stiffening of the cellulosic macromolecular network and fibre embrittlement, thus reducing the mechanical strength of the treated cotton[28]. The increase in pH improves the tensile strength of the fabric due to its decrease of acidic nature of the solution. The acidity of the cross linking treatment had a severe effect on the reduction of strength of the treated cotton fabrics and trapping of nano metal oxide particles

Table 5. Influence of curing time and pH on ester carbonyl intensity and carbonyl band intensity ratio of Silica nanoparticles and BTCA treated fabric.

Samples	S1	S2	S3	S4	S5	S6	S7	S8	S9
pH		1.52			2.39			3.01	
Curing time	60	120	180	60	120	180	60	120	180
Ester carbonyl band intensity	0.516	0.519	0.524	0.527	0.529	0.532	0.536	0.540	0.545
Carbonyl band intensity ratio (Ester/Carboxylate)	0.619	0.623	0.626	0.610	0.612	0.615	0.629	0.632	0.636

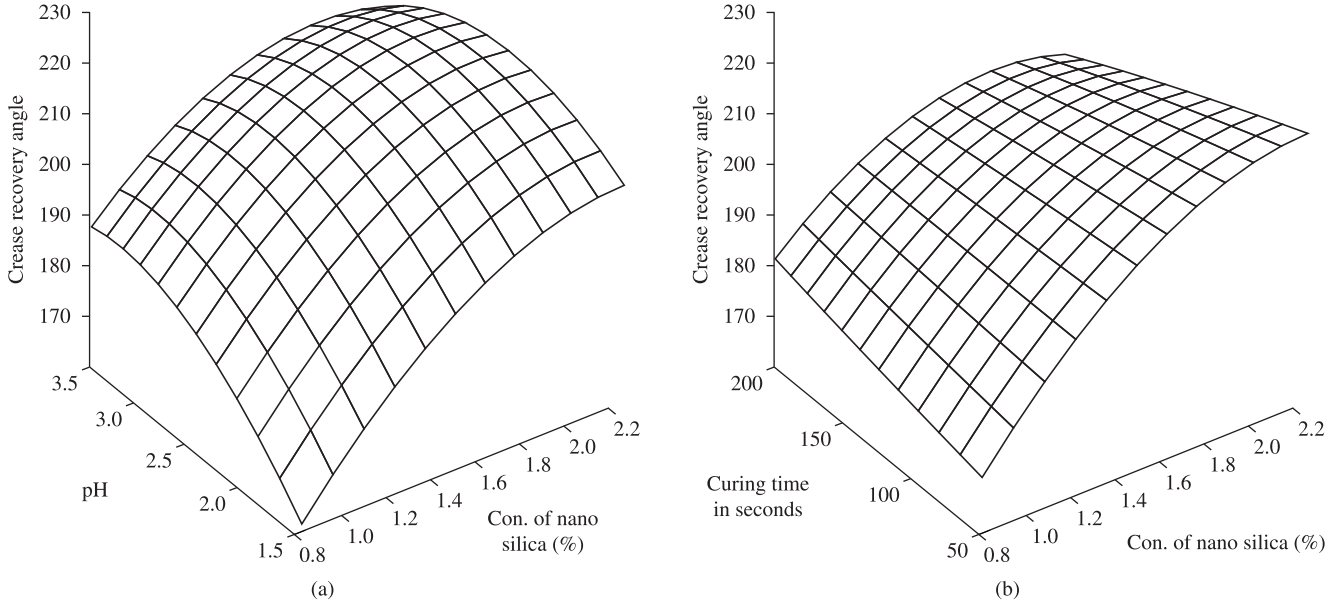

Figure 5. Influence of silica nanoparticles, pH and curing time on crease recovery angle.

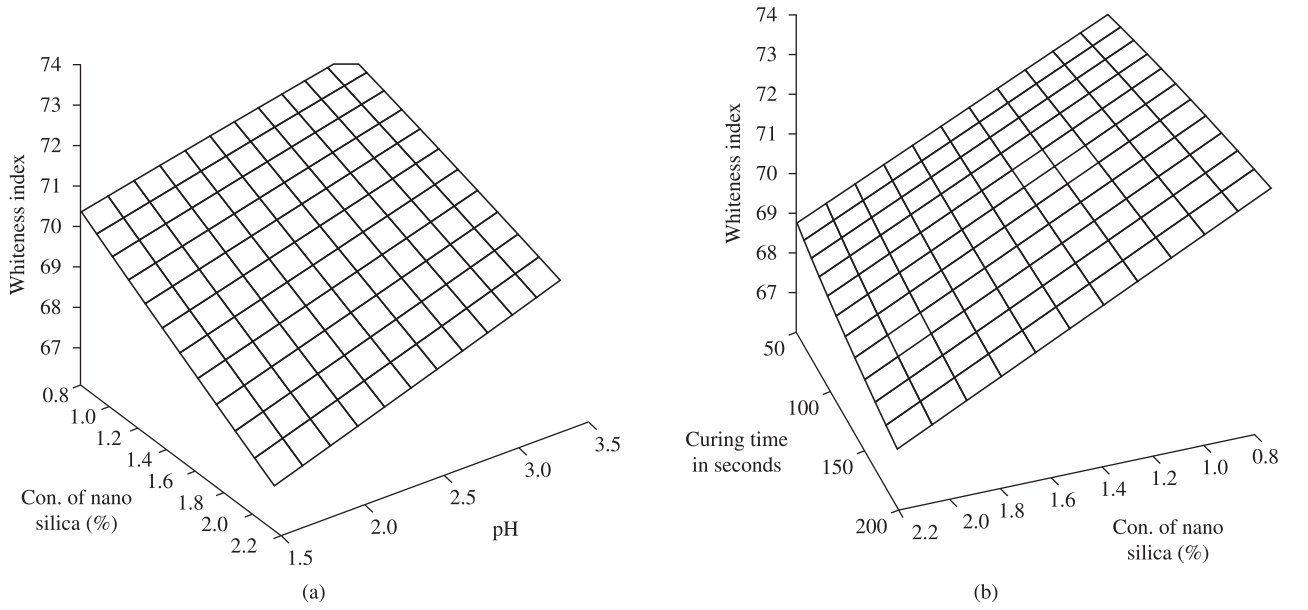

Figure 6. Influence of silica nanoparticles, pH and curing time on whiteness index.

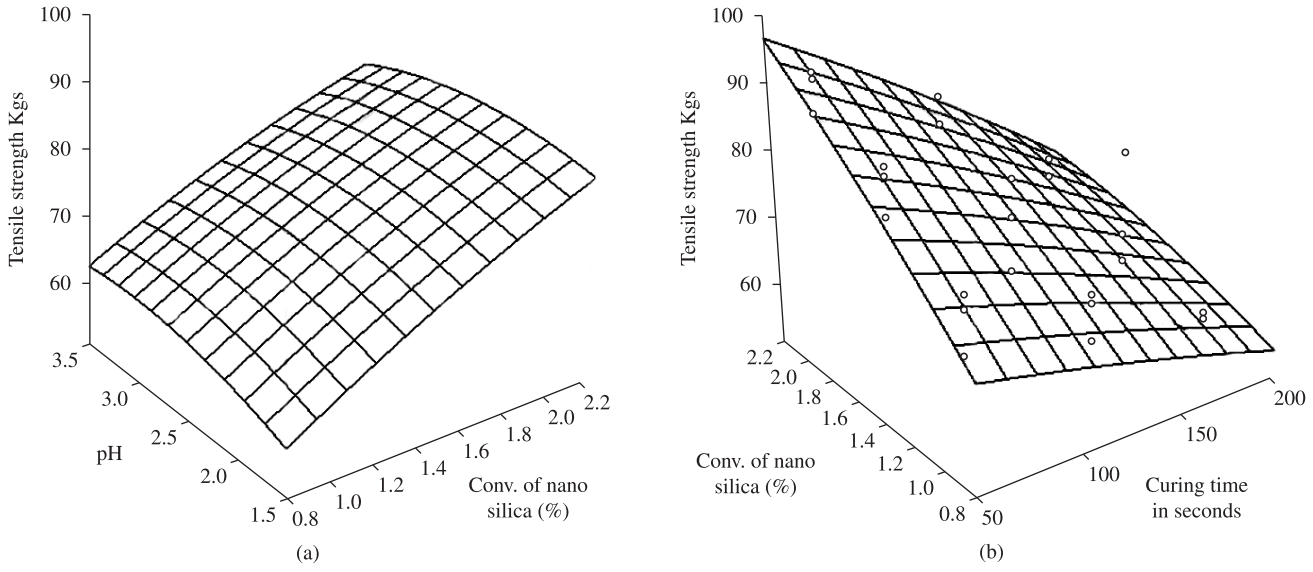

Figure 7. Influence of Silica nanoparticles, pH and curing time on tensile strength.

improves the strength of the fabric[27]. Hence, the higher concentration of nano metal oxides results increases the strength of the cotton fabric and it is shown in Figure 7 The increase of curing time reduced the tensile strength of fabric due to more esterification (acid dehydration at high temperatures and high curing time) and cross linking, when it was treated with BTCA/SHP. The same trend has been followed for tear strength.

The investigated process parameters showed insignificant effect on properties such as flexural rigidity, weight loss due to abrasion and air permeability

5. Conclusions

The silica nanoparticles were synthesized by thermal degradation method from natural resource such as rice hull and the particles are amorphous in nature with the size of 50 to 100 nm. Silica nanoparticles particles along with optimized concentration of BTCA of 6.5% as a cross linking agent and optimized concentration of SHP of 4.5% as a catalyst were applied on the cotton fabric as per the experimental plan. The BTCA and SHP accelerates the esterification of cellulose to form more number of cross links between the molecules. The effect of curing time and pH on ester carbonyl intensity ratio has been studied and the ester carbonyl intensity ratio significantly increases while increasing the pH value and curing time. The combination of crosslinking of cellulose and trapping of nano silica particles significantly increases the crease recovery angle of the fabric. The higher pH level and lower curing time significantly retains the whiteness index of the fabric, since the curing process contributes maximum to reduce the whiteness index of the fabric. The tensile strength of the fabric was significantly increased due to the intreraction of nano silica with cotton fibres and other process parameters. The weight loss of the fabric was increased in the range of 1.33 to 2.83%. The influence of the process parameters on flexural

rigidity and air permeability characteristics of the fabric was observed. There is no significant difference and specific trend. The concentration of silica nanoparticles of 2%, pH of 3.01 and curing time of 60 seconds observed as best combination to impart crease recovery finish. The Fourier Transform Infra Red spectrometer (FTIR) studies revealed that the formation of ester carbonyl band and carboxylate band intensity ratio. Both were increased by increasing the pH vale from 1.52 to 3.01 and the curing time from 60 to 180 seconds. The increased crease recovery angle of the fabric is due to the formation of more cross links, silica nanoparticles trapping and also noticed that the increase in ester carbonyl intensity from 0.516 to 0.547 and carboxylate band intensity ratio from 0.619 to 0.637.

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