International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 3 Issue 4, April - 2014

Croslinking of Chitosan with Cotton using Polycarboxylic Acids

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***Abstract*--Chitosan can be crosslinked with cellulose using PCAs and therefore better bonding has resulted between chitosan and cotton fabric. Because of the better bonding of chitosan and the crosslinking of cellulose by PCAs, an optimum blend of functional properties viz., antimicrobial protection, low flammability characteristics, improved crease recovery with greater strength retention, excellent soil release properties and reduced stiffness can be achieved in cotton. Antimicrobial property exhibited by the fabric treated with all combination samples is good with zone of inhibition ranging from 21 to 25mm for E.coli and 24 to 27mm for S. aureus. All the chitosan and PCAs combination samples show class I flammability which means that these samples are suitable for applications that require lower flammability. 5% MA and chitosan sample show a flame propagation time of 16.84s, which is the highest among all the samples and is comparable to that of THPC treated control fabric. Combination of PCAs with chitosan has imparted excellent soil release characteristics in the finished fabric with a maximum soil release grade of 5. This enhances the easy-care nature of the fabric. CRA of the finished sample has increased up to 261^{o} which is very much closer to the value of 265^{o} for the resin treated control sample. Strength loss values are very low compared to the control samples. 18.7% of tensile strength loss has been obtained for 5% MA and chitosan combination. In case of tearing strength, almost all samples exhibited strength loss values in the range of 13% - 15%. On comparison with resin treated sample, all the samples treated with combination of PCAs and chitosan have better strength retention both in tensile and tearing strength. Though all combinations of PCAs and chitosan have been found to be successful in imparting the functional properties, MA combinations with chitosan have an edge over combinations of chitosan with other two PCAs.**

Keywords - Polycarboxylic acids, Chitosan, antimicrobial property, flammability, crease recovery angle, soil release property, bacteriostasis

I. INTRODUCTION

Crosslinking Of Chitosan With Cotton Fabric Using Polycarboxylic Acids Can Impart Polyfunctional Finishes In Cotton Fabric. Polyfunctional Finish Is The Latest Attractive Terminology In Textile Wet Processing Industry As It Offers The Advantage Of Achieving More Than One Functional Property In One Single Application Process. The Process Also Claims The Benefits Of Saving In Time, Money, Labor, Reduced Lock In Period and increased production. Specific chemical finishing treatments are required for 100% cotton fabric material to impart the functional properties viz., improved crease recovery with reduced tensile strength loss and tearing strength loss, greater whiteness retention percentage, enhanced soil release characteristics and reduced flexural rigidity. Antimicrobial property and thermal resistance are among a few other functional properties which make the fabric suitable for specific end uses. Any single chemical finishing treatment that may improve a few of the above properties, to make the fabric easy-care and protective, will be highly advantageous and preferable.

Chitosan and polycarboxylic acids applied in different combination are capable of imparting these functional properties on cotton fabric in single application. Chitosan is the deacetylated compound of chitin, which is one of the most abundant polysaccharides found in nature, derived from marine shells and mollusks . Chitin is made up of a linear chain of acetyl glucosamine `group. The process of deacetylation involves in the removal of acetyl groups from the molecules of chitin leaving behind chitosan, with a high degree of chemically reactive amino group [Figure1]. Polycarboxylic acids(PCAs) are nowadays considered as a successful alternative crosslinking agent for carcinogenic resins, which can impart polyfunctional finishes. Combination of PCAs and chitosan is expected to impart many functional finishes by optimizing the advantages of both chemicals.

Chitosan is found to be the new range of chemicals used in imparting antimicrobial property [1-4] on cotton textiles. The antimicrobial activity of chitosan is influenced by a number of factors that include the type of chitosan, the degree of deacetylation, molecular weight and other physicochemical properties. The antimicrobial activity of chitosan is also sensitive to pH, with higher activity at lower pH values. Shin [5] investigated the effect of the molecular weight of chitosan on antimicrobial activity. Chitosan of

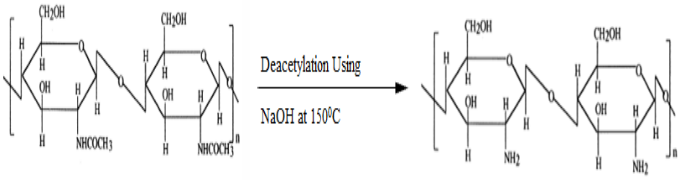

Fig.1 Chitin deacetylated to Chitosan

higher molecular weight were found to be effective in inhibiting the bacterial growth even at low concentration. A fibre reactive chitosan derivative, *O*-acryl amido methyl-*N*-[(2-hydroxy-3-trimethyl ammonium)propyl] chitosan chloride (NMA-HTCC), was applied to cotton fabrics [6], by a cold pad-batch method in the presence of an alkaline catalyst to evaluate its use as a durable antimicrobial textile finish. Cotton fabrics treated with NMA-HTCC at a concentration of 1% on weight of fabric showed 100% of bacterial reduction. Carboxymethyl chitosan (CMCTS) was applied to cationised cotton [7] with different concentrations. The results showed that the antibacterial property and physical properties were improved by increasing the concentration of CMCTS.

Huang [8] studied the effect of using low-molecular-weight chitosan (LWCS) for anti-creasing treatments of cotton fabric. The LWCS was mixed with dimethylol dihydroxyl ethylene urea (DMDHEU) and applied on cotton fabric. The addition of LWCS increased the tensile strength retention and creasing resistance of the treated fabrics. Tahlawy [9] used chitosan phosphate to produce an eco friendly flame retardant cotton textiles. It was found that phosphate derivatives reduce the heat emitted by increasing the CO/CO_2 ratio, which reduces the rate of propagation and the afterglow. In another study [10], it was shown that chitosan could increase the efficiency of sodium stannate/phosphate flame retardants on cotton. Recently, several works [11-14] were carried out by incorporating nano particles in the chitosan formulation and then applying on cotton fabric. Silver, silver oxide and copper oxide nano particles were extensively used along with chitosan. Samples treated with the nanocomposites showed excellent and durable antibacterial activity.

Tahlawy [15] treated cotton fabrics with chitosan and two crosslinking agents 1,2,3,4 butane tetra carboxylic acid (BTCA) and Arcofix NEC (low formaldehyde resin). Fabrics treated with BTCA and chitosan were found to possess better antibacterial activity than the fabrics treated with Arcofix NEC. Montazer and Afjeh [16] worked on simultaneous crosslinking and antimicrobial finishing of cotton fabric using chitosan and *N*-(2-hydroxy) propyl-3-trimethyl ammonium chitosan chloride (HTCC) with different crosslinking agents including citric acid (CA), BTCA and glutaraldehyde. This treatment improved the laundering durability of antimicrobial treatment. Kim et al [17] used *N*-(2-hydroxy)propyl-3-trimethylammonium chitosan chloride (HTCC) as an antimicrobial agent for cotton fabrics. Crosslinkers such as DMDHEU, BTCA, and CA were used with HTCC to improve the laundering durability of HTCC treatment. The polycarboxylic acid treatment was superior to the DMDHEU treatment in terms of prolonged antimicrobial activity of the treated cotton after successive 20 laundry cycles.

In the studies carried out so far by different researchers using the combination of PCAs and chitoson, PCAs had been used only for making stronger bonds between chitoson and cellulose by way of formation of covalent bonds. The main objective of these studies was to improve the durability of antimicrobial activity.

In our work, three PCAs maleic acid itaconic acid and citric acid were used along with chitoson with objectives of imparting more functionalities like soil release property, resistance to flammability, crease recovery behavior with good strength retention in addition to antimicrobial activity, in cotton fabric to make it suitable for the production of regular apparels, sanitary wears, kitchen and laboratory aprons, hand gloves etc.. The crosslinking characteristic of PCAs and antimicrobial property of chitosan have been effectively utilized to achieve maximum functionalities in cotton. 100% cotton fabric samples were treated with combination of chitosan & single PCAs and combination chitosan & mixed PCAs to study which PCA in single and in combination of PCAs contribute much in imparting these functional properties in an efficient manner. All treated samples were tested for the functional properties as per international standards.

II. MATERIALS AND METHODS

A. Fabric

100% cotton woven undyed fabric with the constructional parameters of warp count 40Ne, weft count 40Ne, 106 ends per inch, 96 picks per inch and an areal density of 101 g/m^2 has been selected for the study. A sample size of 50 cm x 50 cm was used for each application.

B. Chemicals

Chitosan (90% deacetylated), maleic acid (M.A., Two carboxylic acid groups), itaconic acid (I.A., Two carboxylic acid groups) , citric acid (C.A., Three carboxylic acid groups), acetic acid for dissolving chitosan and sodium hypophosphite(catalyst for PCAs) were purchased in analytical reagent grade.

C. Samples

Cotton fabric samples were treated with combination of 1% chitosan and 4% of each acid, 1% of chitosan and 5% of each acid, 1% of chitosan and binary combinations of acids with total acid concentration of 4% and 5%. The samples were padded in solution containing necessary quantities of PCAs, catalyst and chitosan, squeezed in LABTEX laboratory padder with a pressure of $3kg/cm^2$.These samples were dried at 80^oC for 10 minutes in hot air oven and curing was carried out at 170^oC for 3minutes in high temperature curing oven. The samples were washed and dried.

Two control samples were prepared using commercially successful chemicals one for comparing crease recovery finish and another for flame retardency. First sample was prepared by padding in a solution containing 100 g/l DMDHEU resin (this concentration was arrived based on the laboratory trials), 20 g/l magnesium chloride 6 H_2O, 25 g/l polyethylene emulsion, 30 g/l acrylic emulsion and 3 g/l non-ionic wetting agent. Padded fabric sample was squeezed with a pressure of 3 kg/cm^2, dried at 80^oC for 10 minutes followed by curing at 160^oC for 5 minutes. Then the sample was washed and dried.

Another sample of fabric was prepared with commercial flame retardant chemical Tetrakis Hydroxy Methyl Phosphonium Chloride (THPC), by padding the sample in a solution containing 16.0% THPC (this concentration was arrived based on the laboratory trials), 9.5 % methylol melamine 3.0% triethanol amine and 9.0% urea. Padded fabric sample was squeezed with a pressure of 3 kg/cm^2, dried at 80^oC for 10 minutes followed by curing at 140^oC for 5 minutes. Then the sample was washed and dried.

D. Testing Methods

All the samples were conditioned for 8 h at 65%$\pm$2% RH and $20^o\pm2^oC$ in a conditioning chamber. Functional properties viz., antimicrobial activity (AATCC 147) , flammability (45^o Flame test ASTM D 1230- 94) , soil release properties (AATCC 130 :2000), crease recovery angle (AATCC 66-2008), tensile strength (ASTMD 5035 – 06 – 2008) and tearing strength(ASTMD 1424 – 09) were tested.

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III. RESULTS AND DISCUSSION

A. Antimicrobial Activity

Antibacterial activity in terms of zone of bacteriostasis(mm) was tested for all samples treated with chitosan and PCAs using the test method AATCC 147. Escherichia coli ATCC 11 229 and staphylococcus aureus ATCC 6538 were the bacterial strains used. The zone of bacteriostasis values are 0 mm for parent sample against both the strains.

Combination of chitosan with PCAs is found to impart antimicrobial property. Zone of bacteriastasis for samples treated with chitosan and PCAs is in the range of 21mm to 25mm for bacterial strain escherichia coli and it is in the range of 24mm to 27mm for the strain staphylococcus aureus. These values are obtained for the combination of single PCAs and chitosan. As there is no significant difference between these values and the inhibition values of 100% chitosan treated samples[18], it can be inferred that the imparting antimicrobial activity in cotton is mainly by the chitosan. The presence of PCAs influences this property in a negligible manner. The zone of inhibition values for itaconic acid & chitosan and maleic acid & chitosan are found to be slightly better than the citric acid & chitosan as could be referred from figure 2.

The mechanism of antimicrobial activity [4] exhibited by chitosan can be explained in the following manner. Chitosan is a polycation. The amine groups present in chitosan bind with negatively charged residues of the cell surface of bacteria and such interaction causes extensive changes in the cell surface and cell permeability, leading to leakage of intracellular substances, thus inhibit the growth of bacteria. Thus chitosan acts like a biocide.

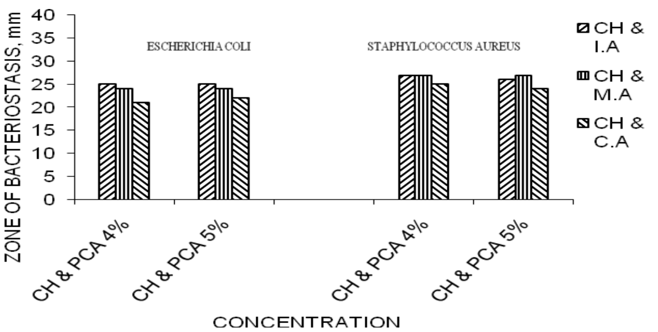

Fig .2 Effect of concentration of chitosan and PCAs on Anti Bacterial activity.

B. Flammability test

Samples were tested for flammability using test method ASTM D1230-94. Samples were held in a holder at an angle of 45^{o} and ignited at the bottom. Flammability was measured in terms of rate of propagation of flame over a fixed distance marked on the fabric. Flame propagation time is 6s for untreated fabric, and 18s for THPC treated control fabric.

Based on the flame propagation time, degree of flammability of the samples can be categorised into three classes viz., *Class I* refers to normal flammability, *class II* intermediate flammability and *class III* rapid flammability . Degree of flammability is class II for parent sample and class I for all the chitosan and PCAs treated samples. Chitosan and PCAs treatment has reduced the degree of flammability from class II to class I and therefore the treated fabric can be used for applications that require lower flammability. The flammability values in terms of flame propagation time are recorded slightly lower for samples treated with itaconic acid combination and citric acid combination than maleic acid combination. Maleic acid and chitosan treated samples produced a maximum protection with a flame propagation time of 16.84s which is almost nearer to that of the control sample. Maleic acid/sodium hypophosphite system [18] has been found to possess good flame resistant characteristics by retaining more phosphorous in cellulose. Therefore in combination with chitosan also, the same trend was observed [Figure 3]. When single PCA is combined with chitosan, samples treated with chitosan and 4% M.A. & 5% M.A. have been found to exhibit better thermal resistance on comparing with other PCAs combinations. Interestingly when mixed PCAs are combined with samples with M.A. combinations show greater flame propagation time.

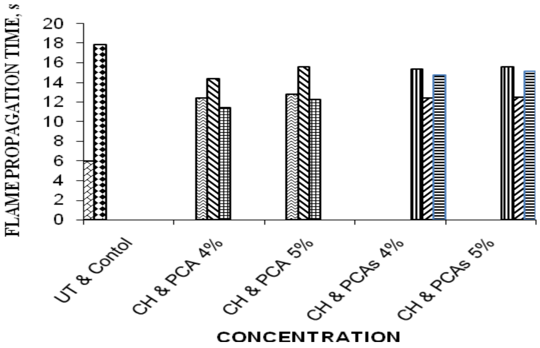

Fig.3. Effect of concentration of chitosan and PCAs (single and mixed) on Thermal Resistance.

C. Crease recovery property

Crease recovery behaviour has been measured in terms of total crease recovery angle, CRA which is the sum of crease recovery angle measured in warp way and weft way. CRA is only 215^{o} for untreated fabric and for all chitosan and PCAs treated fabrics the values range from 248^{o} to 261^{o}. Increase in CRA is mainly due to the crosslinking of cellulosic chains. PCAs form ester cross links with cellulose through esterification reaction and therefore able to impart crease recovery behavior. Addition of chitosan has not affected this crosslinking. As chitosan does not involve in crosslinking of cellulosic chains, improvement in CRA of combination treated samples is mainly due to the presence of PCAs. A few works have already established that though chitosan does not involve in any cross linking with cellulose, when PCAs are applied along with chitosan, they help to crosslink chitosan with cellulose through covalent bonds [15,16].

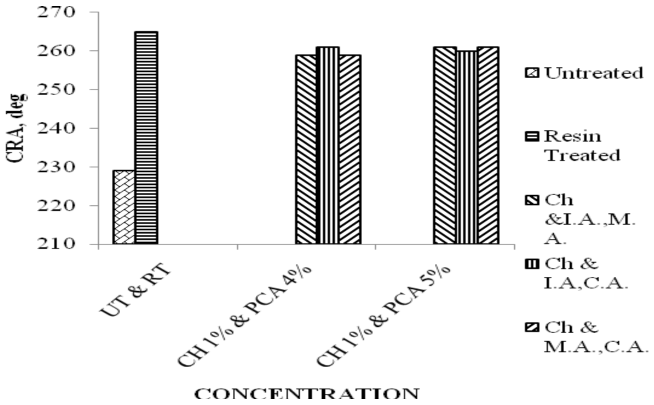

Fig.4. Effect of concentration of chitosan and mixed PCAs on Crease Recovery Behaviour .

The crease recovery angle for chitosan and 4% single acid combination ranges from 248^o – 254^o, whereas 5% concentration of acids when combined with chitosan increases the crease recovery values upto 258^o (table 1). Similar trend is observed for samples treated with combinations of mixed PCAs and chitosan with maximum CRA recorded as 261^o (figure 4). When mixed PCAs are combined with chitosan, difference in CRA values among different combinations is very meager.Combinations with C.A. has shown the best CRA values in 4% as well as 5% combinations. Though citric acid is found to have difficulties in crosslinings when applied in single, it has been established that citric acid exhibits synergic effect in crosslinking cellulose when combined with other pieces. Combinations of C.A. with other PCAs increase the functionality of PCAs in terms of increased Carboxylic acid groups. From the results of the previous part of the study, as 100 % chitosan treated samples show only a meager increase in CRA values to the range of 220^o - 230^o, it may be inferred that chitosan cannot improve the crease recovery property cotton fabric significantly, but addition of PCAs help to improve this behaviour and makes the fabric more functional.

D. Strength properties

Since there are no crosslink formation between chitosan and cellulose, there is no much loss in strength because of chitosan treatment. But when combined with PCAs, significant strength loss to the tune of 11%-18% was observed in samples treated with different combinations of chitosan and PCAs, the reason being that the formation of intermolecular and intramolecular crosslinks reduces the possibility of equalizing the stress distribution causing reduction in the capacity to withstand load. But on comparing with the strength loss of resin crosslinked fabric, the PCAs crosslinks produced samples with greater strength retention properties. Further, PCAs crosslinking makes cellulose softer than resin treated one and allows more stress to be borne.

Chitosan and 5% maleic acid show maximum strength loss and chitosan & citric acid combination show minimum strength loss both in tensile and tearing strength properties(Table 1). Strength properties of the chitosan and mixed PCAs are given in Table 2. Maleic acid combination samples show higher strength loss both in cases when it is combined alone or its mixture with other PCAs are combined with chitosan as shown in Figure 5 & Figure 6. Though maleic acid has only two carboxyl groups, its structure is linear and when polymerized can form stronger crosslinks. Further it can copolymerize easily with other PCAs enhancing its crosslinking capacity by way of increasing the number of carboxyl groups [20]. Though the samples with maleic acid show maximum strength loss compared to other PCAs combination, the values are lower than that of resin treated fabrics and further its performance in terms of functional properties like CRA, soil release characteristics, low flammability values is better and therefore reduction in strength retention could be tolerated.

E. Soil release property

Soil release property was tested for all samples as per AATCC 130: 2000 using Soil Release - Oil Stain Release Method. Washing procedure - III was adopted with corn oil as staining agent and AATCC- WOB as detergent. Soil release grade 3 was obtained for untreated fabric, it has increased to maximum grade of 5 for samples treated with combinations of PCAs and chitosan.

Surface property alteration of cellulose towards hydrophilic / hydrophobic nature, affects the soiling characteristics of the fabrics. All chitosan and PCAs treated samples have shown the same level of soil release property irrespective of the type of PCAs added. PCAs treatment introduces carboxyl groups [19] in cellulose and chitosan has amine group in its structure which may probably be the reason for increasing hydrophilic nature, thus improving its soil release characteristic. Since the soil release grade of all treated fabric samples has increased from grade 3 to grade 5, an increase of 2 units in grade is significant to infer that the fabric has acquired very good soil release nature because of the PCAs and chitosan treatment.

Table 1: CRA and strength properties of combination of chitosan and single PCAs treated samples

Description of the samples	Total CRA (W+F) deg	Tensile Strength				Tearing Strength			
		Warp Way kg	Loss %	Weft way kg	Loss %	Warp Way gm	Loss %	Weft Way gm	Loss %
Untreated	215	34.02	*	27.22	*	1120	*	989	*
Resin	265	20.41	40.0	17.69	35.0	768	31.5	656	33.7
Ch & I.A.	250	29.03	14.7	23.13	15.0	958	14.5	872	11.9
Ch & I.A.-	253	29.94	12.0	22.68	16.7	960	14.3	860	13.1
Ch &	254	28.58	16.0	22.68	16.7	962	14.1	856	13.5
Ch &	258	29.03	14.7	22.23	18.4	959	14.4	860	13.1
Ch &	248	29.49	13.4	24.04	11.7	960	14.3	880	11.1
Ch &	249	29.49	13.4	24.04	11.7	963	13.9	865	12.6

*-reference value based on which strength loss % is calculated

Table 2 Strength properties of combination of chitosan and mixed PCAs treated samples

Combinations & Concentration	Tensile Strength, kg		Tearing Strength, g	
	Warp-way	Weft-way	Warp-way	Weft-way
CHT & IA 2% MA 2%	28.58	23.59	976	864
CHT & IA 2.5 % MA 2.5%	29.03	23.13	944	848
CHT & IA 2% CA 2%	29.94	24.04	960	864
CHT & IA 2.5% CA 2.5%	29.49	23.59	976	848
CHT & MA 2% CA 2%	29.49	23.59	976	880
CHT & MA 2.5% CA 2.5%	28.58	23.59	944	864

IV. CONCLUSION

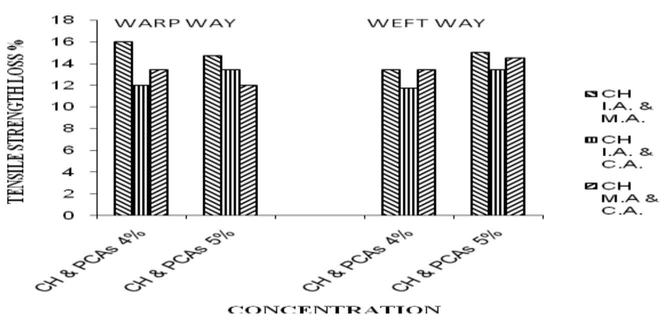

Fig.5. Effect of concentration of chitosan and mixed PCAs on Tensile Strength loss %.

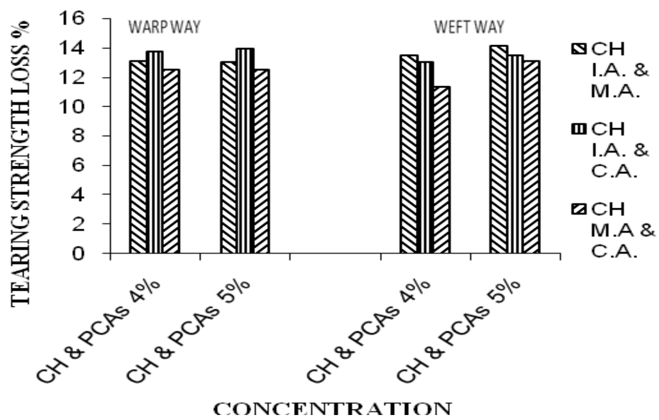

Fig.6. Effect of concentration of chitosan and mixed PCAs on Tearing Strength loss %.

following conclusions are made based on the results obtained. All combinations of chitosan and PCAs treated samples offer great level of inhibition for both bacterial strains viz, *escherichia coli* and *staphylococcuss aureus* with a zone of bacteriostasis ranging from 21mm-25mm and 24mm-27mm respectively. The values are not much influenced by the type of PCA added. Similarly, the flame resistance is found to be good with class I flammability rating for all chitosan and PCAs combinations, especially flame propagation time for maleic acid combination with chitosan is found to be better than other PCAs combinations with chitosan.

When PCAs are combined with chitosan, crease recovery properties are also imparted in the fabric in addition to the antimicrobial property. CRA for samples treated with combination of PCAs and chitosan is almost comparable to that of resin treated control samples, while the strength loss is only about 10% - 15%. Binary combinations comprising of CA and chitosan treated samples show a maximum CRA of 261^o because of the synergic effect exhibited by CA with other PCAs. Soil release characteristics are rated as grade 5 for all samples against grade 3 for untreated fabrics. Addition of PCAs with chitosan has helped to improve the hydrophilic nature of cellulose, resulting in improved soil release property. In single PCAs combination, 1% chitosan and 5%maleic acid and in binary combination of acids, 5% of maleic acid & itaconic acid(total concentration is 5%) and 1% chitosan are the optimum concentrations for achieving the multifunctional properties in an efficient manner.

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