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Enhancing Cotton Fabric Softness and Wrinkle Resistance through Optimized Formulation of Nano ZnO and Benzalkonium Chloride-Enriched Rinse-Cycle Softener

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ABSTRACT

This work attempts optimization of a multifunctional single rinse softener formulation for cotton fabric for domestic usage, catering to the growing customer demand for softeners with enhanced performance. Present section is part-I of the entire study. The primary objective is to determine the effects of optimized and developed formulation on softness and wrinkle resistance properties. We synthesized polyvinylpyrrolidone (PVP) stabilized nano-ZnO particles using the sol-gel method and characterized them using UV-VIS spectrophotometry, particle size analyser and X-ray diffraction. Atomic absorption spectrometry (AAS), and Fourier transform infrared spectroscopy (FTIR) techniques were utilized for fabric sample analysis. Objective measurements using the Kawabata evaluation system, subjective softness assessments and crease recovery angle were recorded. The results demonstrated a significant enhancement in fabric softness, crease recovery and low stress mechanical properties through the application of this single rinse softener formulation. Any improving or diminishing effects on softness and crease recovery angle due to the presence of nano-ZnO (nano zinc oxide) and BAC (Bezalkonium chloride) were insightful.

***Key words**: Nano-ZnO, Nano finishing, silicone softener, Wrinkle-free finish, Laundering, washing.*

Introduction

Cotton, as a natural fiber, holds paramount significance in the world of apparel due to its exceptional moisture absorption properties, making it a versatile material for various end-uses. Nevertheless, certain aspects of cotton's characteristics necessitate improvement, including its susceptibility to wrinkling, post-laundry roughness, and limited resistance to microbial activity. The issue of fabric harshness following laundry gave rise to the development of rinse cycle softeners during the 1960s. Softeners, by definition, are a category of chemicals designed to impart a softer texture to laundered textiles Zhang *et al.* (2010). In the case of cationic softeners, they achieve this by coating the fabric's surface with long alkyl chains featuring negatively charged tail ends. This process reduces inter-fiber friction and the charge on fibers, causing them to repel each other Igarashi *et al.* (2016). While enhancing softness remains a primary objective, researchers and developers are now striving to incorporate a spectrum of functionalities into these formulations, aligning with evolving consumer demands related to health and hygiene. One such contemporary demand is the need to protect wearers from harmful UV rays and pathogenic microorganisms that contribute to odors and infections reported Schages *et al.* (2020); Dewey *et al.* (2022). Microbial growth on textiles after washing with low-temperature detergents has been proven to be directly linked to malodorous fabric and potential health concerns infections by Munk *et al*. (2001). Antimicrobial substances can either eliminate or inhibit the growth of these pathogens. Various antimicrobial agents, such as quaternary ammonium compounds, triclosan, metal salts, polybiguanides, and natural biopolymers (Morais *et al.* 2016), along with UV absorbers like derivatives of o-hydroxybenzophenones and o-hydroxyphenyl triazines Mavriæ *et al.* (2018), can be incorporated to confer these properties upon cotton textiles. In the realm of technological and scientific advancements, nanotechnology has emerged as a promising frontier. Nanosized particles of metals and metal oxides, including silver (Ag), copper (Cu), titanium dioxide (TiO_2), zinc oxide (ZnO), and magnesium oxide (MgO), have demonstrated exceptional potential for enhancing textile finishes owing to their antibacterial, UV-absorbing, and photo-catalytic properties reported Vigneshwaran *et al.* (2007); Bhandari *et al.* (2022). Nano-ZnO, in particular, has garnered attention as an antimicrobial, UV-protective, and flame-retardant agent, owing to its chemical inertness, white color, and cost-effectiveness as stated by Samanta *et al.* (2017); Arputharaj *et al.* (2020). Prasad *et al.* (2016) and Arputharaj *et al.* (2017) indicated that various efforts have been made to develop durable finishing methods by employing curable polymer binders or in-situ generation of nanoparticles on cotton substrates. Nonetheless, these methods come with their own set of limitations, including stiffer fabric feel, reduced air permeability, complex treatment processes, and economic impracticality.

Present work is section one of the entire study which aims to create a multifunctional formulation capable of providing a soft texture, reduced proneness to wrinkling, antimicrobial activity, and UV protection to cotton textiles under typical home laundering conditions. We have optimized a rinse-cycle softener formulation, incorporating silicone and cationic softeners fortified with nano-ZnO and benzalkonium chloride (BAC). However, here are the results of its effects on performance variables of crease recovery angle, objective and subjective softness of the fabric.

Materials and Methods

The raw material for this study consisted of greige cotton fabric procured from the local market. The fabric possessed the following specifications: plain weave, 108 ends per inch (41 Ne), 103 picks per inch (42 Ne), and an areal density of 137 g/m2. Commercial amino-silicone and cationic softeners were generously provided by M/s Siligenz Technologies, Bangalore, India. Benzalkonium Chloride (95%) was sourced from M/s Sigma Aldrich Chemicals Private Limited. Analytical grade chemicals including zinc nitrate hexahydrate (99%), ammonium hydroxide (99.9%), methanol (99.8%), polyvinylpyrrolidone (PVP) (molecular weight 40,000 purity 99%), hydrogen peroxide (50% w/w), sodium hydroxide (98%), sodium chloride (99%), and sodium silicate (99.9%) were obtained from M/s Fischer Scientific India. Additionally, for comparative analysis, nano-ZnO with a particle size of 100 nm was purchased from M/s Sigma Aldrich Chemicals Private Limited.

Methods

To remove the sizing materials from the cotton fabric, an enzymatic desizing process was carried out in an autoclave. The fabric was immersed in water at a 1:10 material liquor ratio, to which 50 g/l amylase and 10 g/l sodium chloride were added. Desizing was performed at 70 °C for 1 hour as per the method described by Ul-Haq and Nasir (2011). Afterward, the temperature was raised to 100 °C to deactivate the enzymes. The desized fabric was then rinsed with water and air-dried at ambient temperature. Subsequently, a single-step scouring and bleaching process was conducted using a modified method outlined in the work by Morshed *et al*. (2014). This process involved the use of 8 g/l hydrogen peroxide, 3 g/l sodium hydroxide, and 1.5 g/l sodium silicate at 110°C for an hour, with a 1:20 Material-to-Liquor Ratio (MLR), within the same autoclave.

Synthesis of nano-ZnO particles

Nano-ZnO particles were synthesized using a modified sol-gel method inspired by prior research of Bandekar *et al.* (2014); Alwan *et al.* (2015); Al Abdullah *et al.* (2017). To form Nano-ZnO, 13 g of zinc nitrate hexahydrate was dissolved in 0.6 M methanol with the assistance of a magnetic stirrer. PVP (1% of the weight of the precursor) was added at 50 °C. The solution was heated to 80 °C, maintaining stirring for an hour. Subsequently, ammonium hydroxide was added drop wise at room temperature until the pH reached 11. The resulting sol was dried at 150 °C until gel formation occurred. Multiple methanol washes of the precipitate were performed to eliminate impurities. The precipitate was once again dried at 150 °C and calcinated at 500 °C for 3 hours.

Characterization of nano-ZnO particles

The particle size of the Nano-ZnO was determined using the PSS-NICOMP, USA particle size system. Prior to measurement, Nano-ZnO particles were dispersed in an aqueous medium and subjected to sonication to ensure uniform dispersion. The synthesized Nano-ZnO powder was characterized through powder X-ray diffraction using 'X'Pert PRO, PANalytical, Netherlands, equipped with Cu Ká (k = 1.54 Å). This analysis was conducted in the 2è range spanning from 20° to 80°. Additionally, UV-visible spectra of the synthesized ZnO in deionized water suspension were recorded using a Specord 205 double-beam spectrophotometer (Analytik Jena®) at wavelengths ranging from 240 nm to 400 nm, with sonicated samples ensuring uniform dispersion.

Optimization of softener concentration

The concentration of softeners that yields perceptible softness to human touch is a crucial factor for a product intended for domestic laundry. Therefore, sensory screening was employed to optimize the concentration of the selected softeners. The supplier recommended an optimal concentration range of 5-10 g/l for silicone and 60-70 g/l for the cationic softener. Based on these recommendations, both softeners were applied to the bleached cotton fabric at three concentrations: 5, 10, and 15 g/l for silicone, and 50, 60, and 70 g/l for the cationic softener. A panel of thirty textile experts was asked to select the sample with the most favorable hand feel compared to a control sample. The concentrations of 5 g/l for silicone and 60 g/l for the cationic softener were preferred by the majority of panelists, as indicated in Table 1. Consequently, the resulting combination of both softeners was used, resulting in a 65 g/l softener mix.

Table 1. Sensory evaluation preference to silicone and cationic softener concentrations

Softener concentrations (g/L)	No. of evaluators preferred as 'Best'
Silicone Softener 5	18
Silicone Softener 10	8
Silicone Softener 15	4
Total	30
Cationic Softener 50	7
Cationic Softener 60	20
Cationic Softener 70	3
Total	30

Preparation and application of rinse-cycle formulation

In prior works of Kriangkraipipat *et al.* (2020) and Fernandes *et al.* (2021), the concentrations of BAC required for microbial inhibition of textile materials through an exhaust bath were reported to range from 5 g/l to 20 g/l. For this study, BAC was utilized at a concentration of 8 g/l. In the case of the synthesized Nano-ZnO, a concentration of 10 g/l was applied, as previously established by Li *et al.* (2007). Based on the above findings and preliminary investigations, the optimized concentration of BAC, Nano-ZnO, and the softener mix was determined to be 8 g/l, 10 g/l, and 65 g/l, respectively. The entire formulation, containing 65 g/l of the softener mix, 10 g/l of Nano-ZnO, and 8 g/l of BAC, was prepared using distilled water. Combinations of these independent factors in the form of treatments were applied to the fabric samples as per treatment plan indicated in Table 2.

Treatment of fabric

For treatment, the bleached cotton fabric was first immersed in water at room temperature for 10 minutes. Excess water was removed by passing the samples through a nip of padding rollers. The wetted-out cotton fabric was then immersed in the treatment bath for 10 minutes at room temperature and squeezed through padding mangle. Following application, the samples were dried in a tumble dryer at 60 °C for 30 minutes.

Characterization of treated samples

FTIR spectra were recorded using Shimadzu IR-Prestige-21® in 4000-400 cm^{-1} range. For the quantitative determination of zinc content in the treated fabric, atomic absorption spectroscopy was em-

Table 2. Treatment plan with 3 active agents

Sample Name	Remarks	Softener (g/L)	BAC (g/L)	Nano-ZnO (g/L)
Pristine Control	No treatment	0	0	0
T1	Only Softener	65	0	0
T2	Only BAC	0	8	0
T3	Softener + BAC	65	8	0
T4	Only nano-ZnO	0	0	10
T5	Softener + nano-ZnO	65	0	10
T6	BAC + nano-ZnO	0	8	10
T7	Softener + BAC + nano-ZnO	65	8	10

ployed using GBC scientific equipment, Avanta PM®, Australia. For this, finely cut samples (approximately 0.5 g) were dissolved in a mixture of nitric and sulfuric acid under heating. The analysis was performed using this solution after cooling.

Analysis of physico-mechanical properties of the fabric

The control and treated fabrics were analyzed for response to low stress mechanical forces like tensile, bending, shearing, compression, surface friction and roughness, using the Kawabata Evaluation System (Kato Tech. Co. Ltd, Japan). The crease recovery angle of cotton specimen was evaluated according to AATCC Test Method 66-2003 using crease recovery tester (M/s Tex Lab Industries, India).

Subjective assessment of softness

To evaluate the softness imparted by formulation, samples treated with all 4 softener-containing treatments, i.e. T1, T3, T5 and T7 (Optimized formulation) along with control, were subjected to sensorial assessment. Conditions were same during the assessment by 30 panelists. Order of samples was kept random. Each sample was graded for its overall sensorial soft feel. A 4-point likert scale with polar attributes was used (1- poor, 2- average, 3- good, 4- excellent). Number of responses was multiplied by the given ranking to arrive at weighted scores for each sample. Another comparative assessment was done for softness preference to one out of the following three samples- control, developed formulation and commercial formulation.

Results and Discussion

Characterization of synthesized nano-ZnO particles

UV Absorption

Nanoparticles have unique optical properties which are sensitive to their size, shape, and agglomeration state. This makes UV-Vis absorption studies an important tool for identifying, characterizing and studying various properties of nano materials (Abbas, 2019). UV visible absorption spectrum of synthesized PVP stabilized nano-ZnO, commercial nano-ZnO, formulation with added PVP stabilized nano-ZnO and formulation without nano-ZnO was

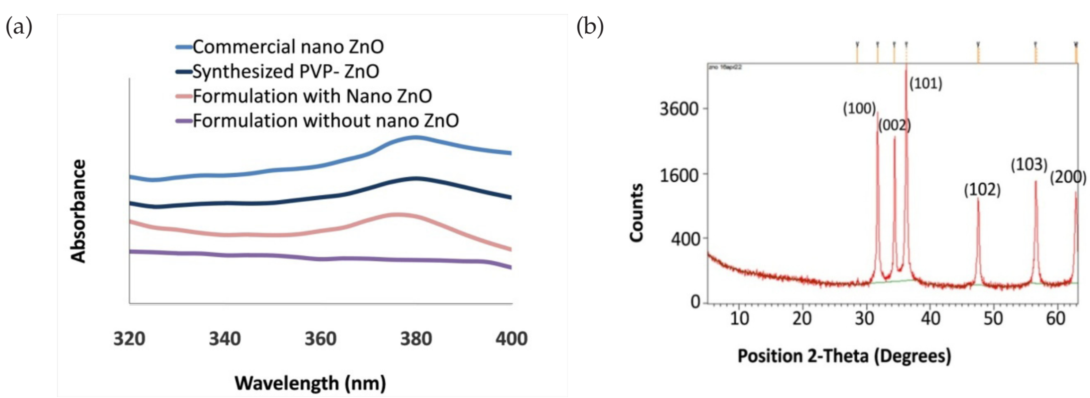


Fig. 1. Characterization of synthesized PVP-stabilized-nano-ZnO particles (a) UV-Vis spectrum, (b) XRD pattern

recorded and given in the Fig. 1 (a). It can be seen that all forms of nano-ZnO is having λ_{max} in the range of 375-380 nm. Presence of peak at 380 nm (Fig. 1a) in synthesized PVP-stabilized-nano-ZnO and nano-ZnO added formulation similar to commercial nano-ZnO confirms the formation of nano-ZnO.

Size Analysis

Particle size of the synthesized ZnO was determined using DLS particle size analyzer. Particle size analyzer gives hydrodynamic diameter of the particles. Average particle size of the prepared nano-ZnO was found to be 94.5 nm. X-Ray diffraction was used to determine the crystalline structure of synthesized nanoparticles. The peaks denoting diffraction from numerous crystal planes inform about the structure of nano-ZnO (Fig.1b). Synthesized nano-ZnO showed diffraction peaks with 2 values of 31.75p, 34.41p,36.24p, 47.65p, 56.57p and 62.82p corresponding to the crystal planes of (100), (002), (101), (102) (103), and (200) respectively (Elmorsi *et al.* 2017). Presence of these diffraction peaks is in good agreement with standard reference for ZnO (JCPDS card No. 36-1451) denoting a hexagonal wurtzite polycrystalline structure (Collins *et al.,* 2012). Broadening of peaks in diffractogram confirms the presence of nano crystal in the prepared ZnO. Using Scherer equation, the grain size of the particle was found to be 28 nm.

Characterization of Treated Fabrics

FTIR analysis

FTIR spectra of control and treated samples are shown in Fig. 2. Control fabric shows CH_2 and C–OH bending peaks at 2885 cm^{-1} and near 3300-3200 cm^{-1} respectively which are the typical characteristic peaks of cellulose of cotton as stated in the works by Chung *et al.* (2004). In comparison to control sample, samples T3 and T7 showed dual peaks at 2900 and 2850 cm^{-1} due to symmetrical and asymmetrical stretching of C-H groups' vibration of the alkyl groups present in the BAC. Bands corresponding to Zn–O stretching vibration was observed around 450 to 457 cm^{-1} as reported by Shamhari *et al.* (2018). Characteristics peak of silicone can be seen at 1095-1075,1055-1020, 1110-1080 cm^{-1} attributed to Si–O–Si stretching (Abidi *et al.,* 2007). These observations confirm the presence of ZnO, BAC and silicone on the respective treated fabric samples.

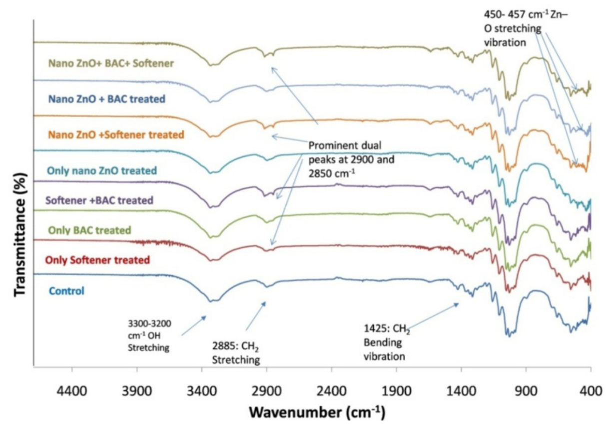


Fig.2. FTIR spectra of control and treated samples

Crease Recovery Angle

It was observed that the control fabric was having good crease recovery property as evident from its crease recovery angle, i.e. 245 (Table 3). Significant improvement in crease recovery angle up to 265 for T1 was found as compared to control, i.e. 245. Bao and Yun-Jun (2018) used modified amino polyether siloxanes and observed similar results. Possible explanation was suggested by Kumar *et al.* (2019) for improved CRA based on droplet size of softener emulsion. Sample with only nano-ZnO treatment, with corresponding 1.9% zinc content (Table 3), also showed an increment in CRA. In their study, similar positive effect of nano-ZnO on crease recovery was reported by Tania and Ali (2021) who can be due to surface smoothening effect of nano particles.

Table 3. Physical and Mechanical characteristics of Samples

Sample Name	Zinc content (% on weight of the fabric)	CRA (Degree)
Pristine Control	ND	245±2
T1	ND	265±3
T2	ND	248±3
T3	ND	262±2
T4	1.9	247±3
T5	1.8	263±2
T6	1.7	248±3
T7	1.7	262±2

Low stress mechanical characteristics

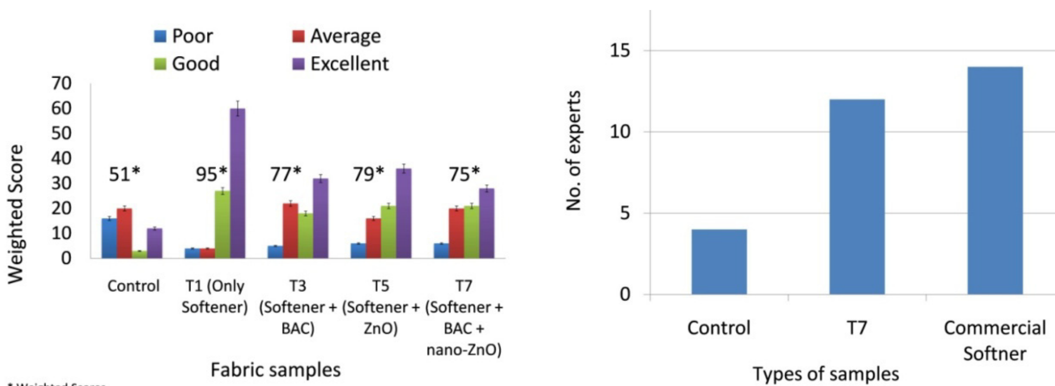
The Kawabata Evaluation of properties of fabrics, including tensile energy (WT), tensile resilience (RT), surface friction (MIU) and extensibility (EMT) is important because apparel usually undergoes small extension and relaxation during wear. Even miniscule variations in these values affect overall handle of a fabric.

The RT of the treated fabric was lower (29.48) than control fabric (29.65) indicating its reduced ability to absorb energy while elastic deformation. Similarly, reduction in bending rigidity from 0.0643 to 0.0620 gf cm^2/cm was observed (Table 4) indicating reduction in stiffness in T-7 formulation treated samples. Good increment in EMT value of the treated sample was observed (17.40) as compared to control fabric (11.06) which is attributed to the increase in the stretching property of the treated fabric. Reduction (22.79%) in shear rigidity (G) after treatment indicated that decrease in resistance to shearing motion provided better drapability to the fabric. Softener improves intrinsic low stress mechanical characteristics such as reduction in bending rigidity by reducing inter yarn and fiber friction (Tang *et al.,* 2017). Surface friction was also reduced in T-7 formulation treated samples (0.296) compared to control (0.243). Application of softener covers the fiber surface with homogeneous softener molecules and reduces surface friction reported Abolhasani *et al.* (2020) in their work. It is evident from the analysis that incorporation of nano-ZnO particles in the formulation did not hinder the softener induced reduction in surface roughness, possibly due to miniscule particle size (Ibrahim *et al.*, 2017).

Subjective assessment of softness

Enriching softener formulation with other functional agents without affecting its subjective softness performance efficacy is critical to product upgradation. Sample treated with only softener had highest score of 95 (Fig. 3a) as compared to control (51). This difference in scores confirmed that softener deposition was sensorially perceivable. Reduction in softness scores was observed with the addition of antimicrobial agents, *i.e.* nano ZnO and BAC.

To evaluate the softness provided by softening treatments in comparison to commercially available one, a comparative test was also performed. Out of 30 respondents, T7 treated and commercial softener



(a) (b)

Fig. 3. Subjective evaluation of softness (a) Subjective softness rating and weighted scores; (b) Preference given to developed formulation and commercially available softener formulation

Table 4. Low stress mechanical properties of control and formulation treated cotton fabrics

Property	Control	T-7treated	Percentage difference
Tensile resilience (RT) %	29.65	29.48	“0.57
Maximum elongation (EMT) %	11.06	17.40	57.19
Tensile energy (gf cm/cm^2)	8.28	11.83	43.11
Shear rigidity (G) g/cm	1.36	1.05	“22.79
Bending rigidity (B) gf cm^2/cm	0.0643	0.0620	“3.58
Surface friction (MIU)	0.296	0.243	“17.91
Primary hand values			
Koshi (Stiffness)	6.16	6.01	“2.44
Numeri (smoothness)	5.79	5.96	2.94
Fukurami (fullness and softness)	8.32	8.38	0.72
Total Hand Value	3.91	4.00	2.30

treated fabrics were preferred by 43.3% (13) and 46.6% (14) respondents respectively (Fig. 3b). It is evident from the result that the difference between the tactile feel of T7 and commercial formulation treated fabric did not differ significantly. Results of sensorial evaluation were in agreement with the results achieved from CRA and low stress mechanical characteristics. Similar correlation has been reported by Yildiz and Özdil (2014).

Conclusion

Herein, nano-ZnO was successfully synthesized using PVP as stabilizer using sol-gel method. A multifunctional rinse-cycle softener formulation incorporating nano ZnO and BAC for cotton fabric that can provide softness, wrinkle resistance, antimicrobial activity, and UV protection was optimized. Results of CRA, low stress mechanical characteristics and sensorial softness evaluation were reported in this paper which confirmed the improvement in softness both subjectively and objectively. As the presence of nano-ZnO and BAC did not significantly affect softening properties of the formulation, the developed rinse-cycle softener at the selected concentration was able to impart perceivable softness to cotton fabric along with better crease recovery. Moreover, this formulation has been developed using common chemicals and simple procedures which make it very easy for the adoption by the textile auxiliary manufacturers.

Conflict of Interest - None

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