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Surface Modification of Cotton, viscose, and polyester/cotton blend fabrics using nano carboxyethylchitosan Before Dyeing with commercial acid dyes and Antimicrobial Activity Evaluation

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Chitosan(CS) and acrylic acid were combined in a Michael addition process to successfully produce N-carboxyethylchitosan (CECS). Then carboxyethyl chitosan undergo ionic gelation method to prepare carboxyethylchitosan nanoparticles (CECSNPs). The prepared Chemical material replacement is appropriate for modern chemical processes and ecofriendly. Cotton, viscose, and polyester/cotton blend fabrics were all treated with nano carboxyethylchitosan at varying concentrations. 0.5, 1, 1.5 wt%, then dyed with commercial acid dyes. We also tested the dyed fabrics' antimicrobial resistance to two model gram-negative and gram-positive bacteria. Further, The K/S, light, rubbing, prespiration, and washing fastness values were estimated. This paper aims to treatment Cotton, viscose, and polyester/cotton blend fabrics with different concentration of nano carboxyethylchitosan and apply two commercial acid dyes on it. These dyes are used in dyeing these treated fabrics without any additives and all treated fabrics have good K/S, light fastness, and are also excellent for washing, rubbing and prespiration fastness. Also, they have highly antimicrobial activity.Use of nanocarboxyethylchitosan to treatment of many fabrics before dyeing will bring a number of benefits to society including higher fastness properties without any additives thus they save a lot of money, materials and have ecofriendly to environment

Keywords: cellulosic fabrics, nano carboxyethylchitosan, Dye-ability, Fastness properties, Antimicrobial.

1. Introduction

Chitosan is an incredibly interesting natural polymer. It is made up of β-1,4-N-glucosamine and β-1,4-N-acetyl glucosamine units, with the proportion of N-glucosamine units exceeding 50%. Chitosan contains some residual acetyl groups as a result of incomplete deacetylation of chitin. Chitosan has positively charged water-soluble macromolecules in slightly acidic solutions due to protonation of amine groups, indicating that it is a cationic polysaccharide. [1, 2].

Innovative biological characteristics of chitosan include biocompatibility, biodegradability, and non-toxicity. Additionally, it has strong immunological, antimicrobial, antioxidant, and hemostatic properties. [3-6]. These characteristics support the use of chitosan in biomedical and pharmaceutical applications as a material to promote wound healing. However, due to its rigid crystalline structure and the presence of strong intermolecular hydrogen bonding, chitosan-related applications are restricted by its insolubility in water at pH levels higher than 6 [7-9].

Ionic substituents that are water-soluble are found in acid or anionic dyes. These dyes are entirely ionised in the acidic conditions used in the dyeing process [10]. Acid dyes adhere to wool both electrostatically and because of their affinity for the material. Wool and silk fabrics are dyed using mono and diazo-acid dyes made from coumarin derivatives. [11]. Eight novel monoazo acid dyes with heterocyclic bases were created using different substituted imidazol-4-one as the diazo component in combination with different amino-naphthol sulphonic acids. The finished dyes were used to dye wool fabric. [12].

Many dyes migrate in hot, humid conditions because the bonds between dye anions and amino groups in the fibre are easily broken and reformed. Although the ease of migration is beneficial and has good levelling properties, it results in poor wet treatment fastness. [13].

Acid dyes are categorised based on their chemical makeup, affinity, and dyeing capabilities. Numerous acid dyes, such as anthraquinone-based acid dyes, acid nitro dyes, and triphenylmethane acid dyes, have been reported in the literature. [14]. The light fastness of triphenylmethane acid dyes is poor. [15]. The class of

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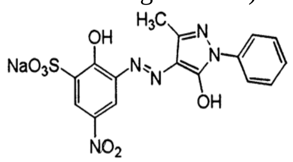
azo dyes is significant due to their broad colour spectrum, brightness, simplicity, and ease of manufacturing and dyeing performance. [16]. They are utilised to colour food, cosmetics, leather, paper, and textiles.[17]. Additionally, they are utilised in high-tech products like fuel cells, dye-sensitized solar cells, metallochromic indicators, thermal transfer printing, nonlinear optical systems, lasers, and photodynamic therapy. [18, 19]. To enhance cotton's dyeability and antimicrobial activity, synthetic acid and basic dyes are used, followed by a layer of nano-sized zinc oxide or titanium isopropoxide, or both.[20]. Cosmetics, medications, food, and chitosan are examples of biotechnological applications.[21]. Succinyl- and carboxymethyl-chitosan are examples of products that have been developed to be water-soluble in an effort to expand their applications in a number of industries, including but not limited to plant protection and human health. These chitosan derivatives had greater water solubility over a wider pH range, which contributed to their higher anti-inflammatory and anticancer properties. [22-24].

A significant water-soluble chitosan is carboxyethyl chitosan. Numerous studies have demonstrated that the production of carboxyethyl chitosan from the hydrolysis of chitosan and dehydrogenation reactions with halo propionic acids can be achieved with sufficient sodium hydroxide.[25]. The aim of this work is to improve the dyeability, fastness and antimicrobial properties of many fabrics towards two commercial acid dyes through treatment with nano-carboxyethyl chitosan

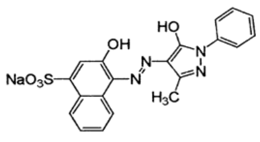
2. Experimental

2.1. Material

Viscose fabric was purchased from Abou ElOla Company for spinning and weaving in Egypt, while cotton fabric (130 g/m^2; Misr El- Mahalla Co., Egypt) was mill-scourged and bleached. The fabric weighs 110 g/m^2, has a warp count of 375/10 cm, a weft count of 320/10 cm, and is a 65/35 blend of polyester and cotton. CS Chitosan (Aldrich, viscosity 1860cps, deacetylation degree 85.0 %). Reagent grade acrylic acid LR 99 percent (AA) was bought from Molychem in Mumbai, India. are employed without further purification Every other substance, including reagents, was of analytical grade and was used right away. Acid Orange 142 (Molecular Formula: $C_{16}H_{12}N_5NaO_7S$, Molecular Weight: 441.35) and Acid Violet 90 (Molecular Formula: $C_{40}H_{27}CrN_8O10S_2$.2Na, Molecular Weight: 944.8) are the two acid dyes.



Acid Orange 142(E1)



Acid Violet 90(E2)

2.2. Methods

2.2.1. Preparation of N-Carboxyethyl chitosan (CECS)

N-carboxyethyl chitosan prepared using the same procedure as before [26]. 2.5 g chitosan powder was added to 120 ml of 5% acrylic acid in a three-necked flask, stirred, and the system became transparent. NCECS can be obtained after a 12-hour reaction at 50 ^{0}C. To purify NCECS, acetone was added to the system in sufficient quantities, and the milk-white flocculent was precipitated. To obtain the final NCECS, the precipitate was washed three times with acetone and vacuum dried at 40 ^{0}C.

2.2.2. Preparation of N-carboxyethyl chitosan nanoparticles (CECS nanoparticles)

The previously described ionic gelation method was used to create CECS nanoparticles. CMCS aqueous solution (1 mg/mL, pH 7.4) and CS acetic acid solution (1 mg/mL, pH 6.0) were prepared separately. For 30 minutes, CMCS solutions were prepared with magnetic stirring. This solution was dropped into the obtained CMCS mixture and stirred continuously for 1 hour, until nanoparticles formed. CMCS-NP were collected by ultracentrifugation at 12,000 rpm for 30 minutes at 4°C and washed twice with sterile ultrapure water. The formed nanoparticles, after centrifugation, were then resuspended in distilled water using water bath sonication for storage and characterization [27].

2.2.3. Dyeing procedures

The amount of acid dyes E1 and E2 applied on treated cotton, viscose, and polyester/cotton by nano carboxyethylchitosan 0.5, 1, and 1.5 percent at different temperatures (40, 60, 80 °C) in an Ahiba dyeing machine without salt or alkaline medium, the dying bath at liquor ratio 40.1 The temperature was then raised at a rate of 2 °C min-1 to the fixation temperature, and the cotton, viscose, and blend fabrics were air dried.

2.3 Measurements and Testing

2.3.1. Dye Exhaustion:

Using a calibration curve previously obtained using known dye concentrations (g/l) on a Shimadzu UV-2401PC UV/Vis spectrophotometer, the uptake of acid dyes by cotton, viscose, and polyester/cotton fabrics pretreated with nano carboxyethylchitosan was measured at the maximum value. Using Eq. 1, the dyebath exhaustion percentage (E percent) was calculated.

$$\%E \left[1 - \frac{C2}{C1}\right] x\ 100 \quad (1)$$

Where $C1$ and $C2$ are the dye concentrations in the dyebath before and after dyeing, respectively.

2.3.2 Color measurements:

The colour parameters of untreated dyed cotton, viscose, and polyester/cotton fabrics as well as fabrics treated with nano carboxyethylchitosan were measured using an Ultra Scan PRO spectrophotometer (Hunter Lab) outfitted with a D65 illuminant and a 10° standard observer. The corresponding K/S values were computed using reflectance data collected at the height of the dyeing process.

2.3.3 Fastness Testing:

Standard ISO procedures were followed to test samples of dyed cotton, viscose, and polyester/cotton fabrics pretreated with nano carboxyethylchitosan after washing with 2 g/L nonionic detergent at 80°C for 15 minutes. The ISO Gray Scale was used to visually evaluate the wash fastness (ISO 105-C02 (1989), crock fastness (ISO 105-X12 (1987), and perspiration fastness (ISO 105-E04 (1989)) for both colour change (AATCC Evaluation Procedure (EP) 1-similar to ISO 105-A02) and colour staining (AATCC EP 2-similar to ISO 105-A03). Using ISO 105-B02, the light fastness (carbon arc) was assessed.

2.4 Evaluation of antimicrobial activity in vitro:

2.4.1. Materials:

Gram-positive pathogens Escherichia coli and Staphylococcus aureus (ATCC 6538) (E. coli, ATCC 11229). Aspergillus Niger, ATCC 13497 and other fungi were found to have antifungal activity (Candida, ATCC 10231). The most prevalent microbes causing wound infections led to the selection of these bacterial and fungal strains as test cells. Fresh inoculants were prepared in nutrient broth for 24 hours at 37°C in preparation for antimicrobial testing.

2.5.2. Test method

Using the disc diffusion method, the antimicrobial and antifungal effects of treated and coloured samples were evaluated on an agar plate.[28, 29]. Following solidification, cotton fabrics with a 1 cm diameter were cut, placed in 10 ml of nutrient agar, and then inoculated with 10 l of microbe culture. The diameter of the inhibition zone was measured and noted after 24 h of incubation at 37 ^{0}C.

3. Results and Discussion

3.1. Preparation of CECS and CECS-NPs

Chitosan (5 gm) and acrylic acid (10.97 mL) were combined using the Michael reaction with water serving as the solvent over a two-day period at 60 ^{0}C. The Michael reaction is viewed as a typical 1,4 nucleophilic addition because it produces the carbonyl group by adding the hydrogen to the -carbon and the nucleophile to the -carbon. As a result, carboxyethyl chitosan is produced.

The nitrogen content of chitosan has been used to confirm the conversion of chitosan into carboxyethyl chitosan by lowering the percentage of nitrogen from 6.9 percent for native chitosan to 3.4 percent for carboxyethyl chitosan. The nitrogen concentration changes as NH_2 and CH_2OH are converted into $NHCH_2COOH$ and CH_2OCH_2COOH, respectively.

Structures of chitosan and carboxyethyl chitosan can be determined using infrared spectroscopy (FT-IR). The FT-IR spectra of native and carboxymethyl chitosan are displayed in Figure 1. Chitosan's spectra revealed bands for NH and OH stretching at 3423 cm^{-1}, NH bending (amide I and II) at 1647 cm^{-1} and amide III at 1401 cm^{-1} and 1375 cm^{-1}.[30, 31]. The spectra also show CO bridging absorption bands at 1151 cm^{-1} 1074 cm^{-1} and CO stretching peaks at 1074 cm^{-1}and 1025 cm^{-1}. Strong absorption is observed at 1553 cm^{-1} and 1404 cm^{-1} for both asymmetric and symmetric stretching of the carboxyethyl chitosan COOH groups, respectively.. [25, 32]. Chitosan's transformation into carboxyethyl chitosan is confirmed by the shifting of amino acid amino groups caused by interference between prepared carboxylic groups and amino groups [33-36].

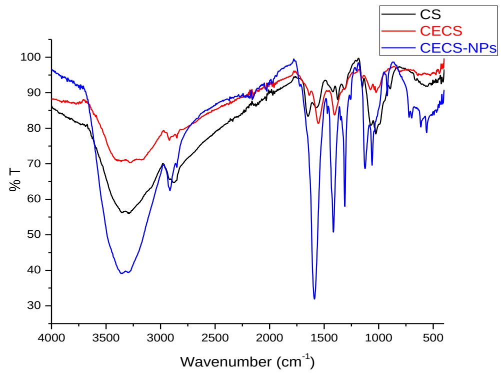


Figure 1: FTIR of carboxyethyl chitosan (CECS), chitosan (CS), and chitosan nanoparticles (CECS NPs)

3.1. effect of temperature

E1 and E2 were applied at 0.5 %, 1 %, and 1.5 %, respectively, to cotton, viscose, and polyester/cotton fabrics that had been treated with nano carboxyethylchitosan. The temperature of the dyeing was varied at a liquor ratio of 1:40. (40, 60, and 80 ^{0}C). The traditional method was used to dye the treated fabric without the use of salt or alkalinity. The temperature differences of two dyes on cotton, viscose, and polyester/cotton fabrics treated with nano carboxyethylchitosan are shown in Figure 2 and 3. All data indicated that the 1 percent treatment on fabrics resulted in the greatest exhaustion compared to the other concentrations. Figure 2 displays the higher exhaustion values for E1 at 60 0C, whereas Figure 2 also demonstrates that E2 has a higher concentration of 1% at 80 ^{0}C due to planarity and having a higher molecular weight than orange dye, which increased the exhaustion.

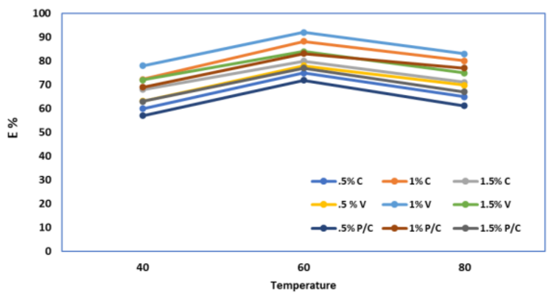


Figure 2: Exhaustion of Cotton(C), Viscose(V) and Polyester/ Cotton (P/C) treated by nano

carboxyethylchitosan at 0.5%, 1%, and 1.5%, dyed with acid orange 120 (**E1**) at different temperature

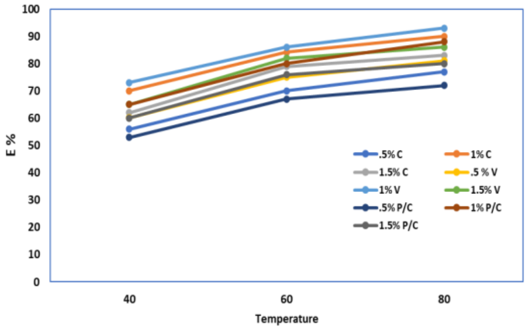


Figure 3: Exhaustion of cotton (C), viscose (V), and polyester/cotton (P/C) treated with nano carboxyethylchitosan at 0.5, 1%, and 1.5 percent and dyed with acid violet 90 (E2) at various temperatures

3.2 *Colorimetric and Fastness properties:*

The colour parameters were assessed using the Cielab system and the modified CIE L* C * ho (D65/10o) system. For the dyed samples, the digital Cielab system determined the following colour parameters: L* stands for lightness, a* for positive or negative redness, b* for positive or negative yellowness, h for colour hue, X for coordinate x, Y for coordinate y, and Z for coordinate z. C* stands for chromaticity. The colour parameter for acids 120 orange and 90 violet at 60 C is shown in Tables I and II, respectively.

The following colour parameters for the dyed samples were obtained by the digital Cielab system: L* – lightness, a* – redness if positive coordinate, or greenness if negative coordinate, b* – yellowness if positive coordinate, or blueness if negative coordinate.

Table I: Colorimetric data of the dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at concentrations of 0.5, 1 and 1.5 using acid orange 142 at 60 °C

ID	L*	a*	b*	dE*	C*	h◦
.5%C	71.79	21.88	25.40	36.40	33.52	49.26
1%C	64.00	27.06	22.22	41.34	35.01	39.39
1.5%C	68.10	24.59	22.81	38.01	33.54	42.85
.5% V	65.90	29.83	29.59	46.57	42.02	44.77
1.5% V	61.34	27.92	25.43	45.09	37.77	42.33
1.5% V	67.55	25.53	23.34	39.20	34.59	42.43
.5% B	70.12	24.60	24.47	38.16	34.70	44.85
1% B	67.59	21.31	16.67	32.71	27.06	38.02
1.5% B	67.53	23.55	22.51	37.43	32.57	43.70

Figures 4, 5, and 6 show the K/S, absorbance, and transmission of dyed cotton, viscose, and polyester/cotton treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % for acid orange and Figures 7, 8 and 9 for acid violet at 60 ^{0}C. All figures show that the absorbance and K/S of 1 % treated with nano carboxyethylchitosan for all fabrics have higher exhaustion and absorbance than other concentrations due to fabric aggregation. Furthermore, for all dyes, viscose fabrics have higher K/S and absorbance than other fabrics.

Table II: Colorimetric data of the dyed cotton **(C)** viscose **(V)and** polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5%, 1%, and 1.5% using acid violet 90 at 60 °C

ID	L*	a*	b*	dE*	C*	h◦
.5%C	64.33	23.97	-5.44	30.92	24.57	347.22
1%C	60.50	25.58	-12.11	34.69	28.30	334.66
1.5%C	63.36	25.15	-8.95	32.25	26.70	340.41
.5% V	52.07	31.44	-5.38	44.85	31.89	350.29
1.5% V	52.23	29.88	-8.18	43.56	30.98	344.69
1.5% V	60.39	26.71	-8.65	35.42	28.07	342.04
.5% B	62.15	24.98	-6.08	33.08	25.71	346.33
1% B	60.07	22.59	-8.15	32.86	24.02	340.16
1.5% B	61.12	26.60	-8.46	34.84	27.91	342.36

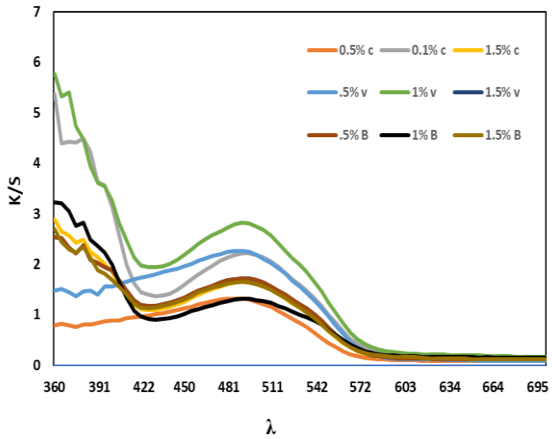


Figure 4: K/S of dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid orange 120 at 60 ^{0}C.

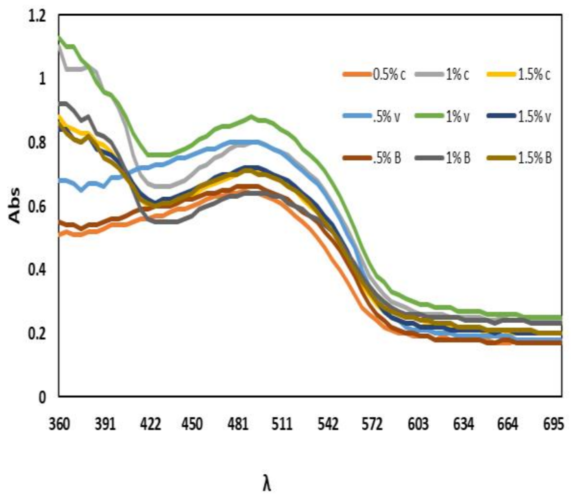


Figure 5: Absorbance of dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid orange 120 at 60 ^{0}C.

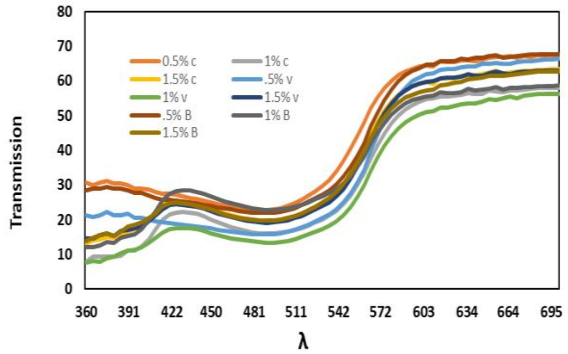


Figure 6: Transmission of dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid orange 120 at 60 ^{0}C.

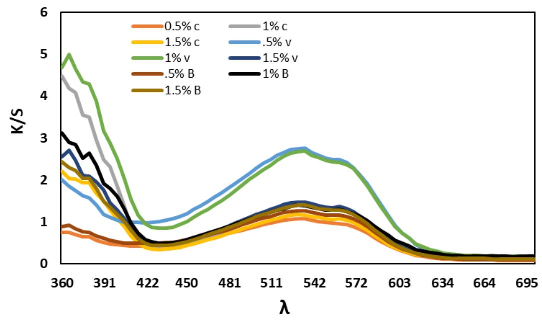


Figure 7: K/S of dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid violet 90 at 60 ^{0}C.

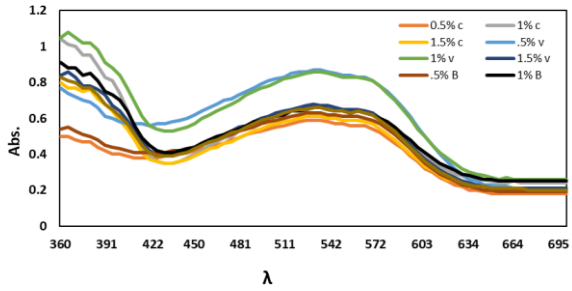


Figure 8: Absorbance of dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid violet 90 at 60 degrees Celsius.

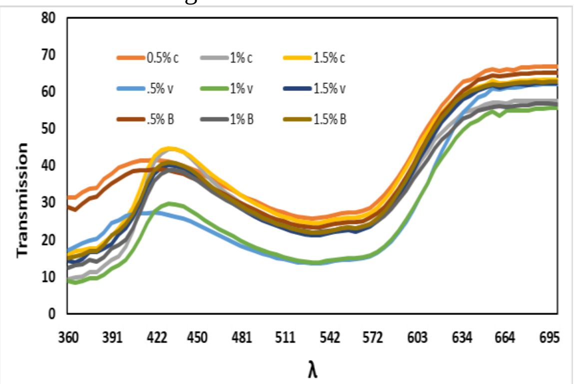


Figure 9: Transmission of the dyed cotton **(C)** viscose **(V)** and polyester/cotton fabrics (B) treated with nano carboxyethylchitosan at 0.5%, 1%, and 1.5% using acid violet 90 at 60 °C

Table III shows that the light fastness, rubbing, washing, and perspiration resistance of all dyed treated samples was excellent.

Table III: The fastness properties of treated dyed cotton (C), viscose (V), and polyester/cotton fabrics (B) with nano carboxyethylchitosan at 0.5, 1, and 1.5 % using acid orange 120 and acid violet 90 at 60 ^{0}C were investigated.

Dyes	**Treated Cotton Fabrics**	**Fastness to Wash**			**Fastness to Perspiration**						**Fastness to Rubbing**		**Light**
					Acidic			**Alkaline**					
		SC	**SW**	**Alt**	**SC**	**SW**	**Alt**	**SC**	**SW**	**Alt**	**Dry**	**Wet**	
Acid orange 120	**0.5%C**	4	4	4	4	4	4	4	4	4	4	4	5
	1%C	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1.5%C	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	.5%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1.5%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	.5%B	4	4	4	4	4	4	4	4	4	4	4	4
	1%B	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
	1.5%B	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
Acid violet 90	**0.5%C**	4	4	4	4	4	4	4	4	4	4	4	5
	1%C	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1.5%C	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	.5%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	1.5%V	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	.5%B	4	4	4	4	4	4	4	4	4	4	4	4
	1%B	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
	1.5%B	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4

Antimicrobial Activity of CECS nanoparticles treated and dyes fabrics

Ammonium ion is created when chitosan dissolves and has an antimicrobial effect. The mechanism of chitosan and its derivatives' antimicrobial action may be explained by the amino groups of chitosan binding to the surface of bacteria to inhibit growth..[1]. Low concentrations could cause bacterial cell death by binding to the negative charges on bacteria and leaking its contents through the cell membrane. [1, 22]. When CS concentrations were high, it coated bacteria's outer surfaces, preventing bacterial growth and causing bacterial cell death. [1, 22].

The chemical structure of carboxyethyl chitosan contains both carboxyl and amine groups, which can be changed into carboxylate anion and ammonium cation, respectively

In order to enhance the antimicrobial effect of carboxyethylchitosan, the negative charges on the carboxylate anions substrate and the positive charges on the ammonium cations may interact with bacterial cell membranes very effectively.[37].

In addition, S. aureus was more resistant to the antimicrobial effects of CSCE than E. coli. This is so because the cell wall layer structures of S. aureus and E. coli differ. S. aureus, a Gram-positive bacteria, contains peptidoglycan with numerous holes in the outer layers, in contrast to Gram-negative *E. coli*, which has lipopolysaccharide and phospholipid protein that created a closed bilayer cell wall and had stronger antimicrobial properties..[30, 31].

As was already mentioned, chitosan nanoparticles have a similar inhibition zone but show greater antimicrobial activity against both Gram-positive and Gram-negative bacteria. This is due to the fact that chitosan nanoparticles are polycations with a high surface charge density that interact with bacteria and are tightly absorbed onto the surface of bacterial membranes to disturb the membranes of both Gram-positive bacteria and Gram-negative bacteria, ultimately leading to the death of bacterial cells in a similar ratio. [1, 38].

Figures 10 and 11 illustrate the antimicrobial qualities of cotton, viscose, and cotton/polyester blended fabrics treated with carboxyethyl chitosan nanoparticles and coloured with two common acid dyes. The antimicrobial activity was measured by the percentage of bacterial growth that was reduced, as mentioned in the experimental section.

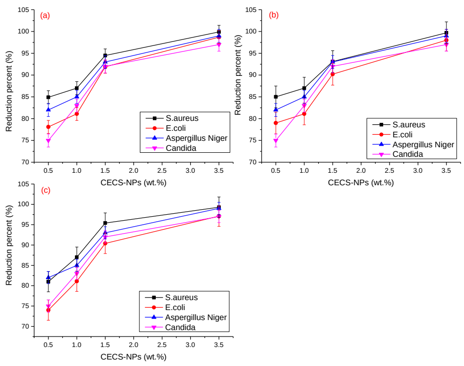


Figure 10: Antimicrobial activity of cellulose-based fabrics dyed with acid orange and treated with nano carboxyethyl chitosan (0.5-3.5 wt%) in the following order: cotton, viscose, and cotton polyester

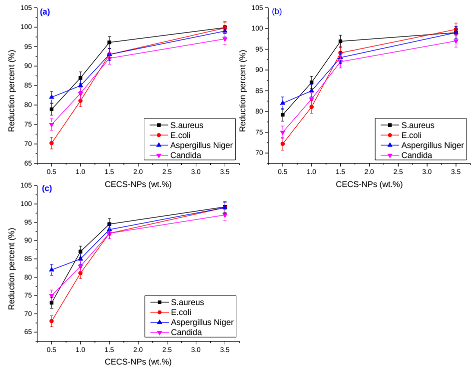


Figure 11: Antimicrobial activity of cellulose-based fabrics dyed with acid violet 90 and treated with nano carboxyethyl chitosan (0.5-3.5 wt%) and treated with cotton, viscose, and cotton polyester.

An increase in reduction percent demonstrates how effectively microbial growth is restricted by higher concentrations of carboxyethyl chitosan. It can be assumed that the colours added after the carboxyethyl chitosan act as potent antimicrobials. The increase in acid orange 120 dye is significantly greater than that of acid violet 90 dye. So, it can be used as antibacterial materials due to its bactericidal effect.

4. Conclusion

The Michael reaction between chitosan and acrylic acid results in nano carboxyethylchitosan. Fabrics made of cotton, viscose, and polyester/cotton were given different treatments (0.5 -1.5 %) to enhance their antimicrobial properties and ability to take acid orange 120 and acid violet 90 dyes. Furthermore, the combination of low acid dye fixation and high salt concentrations results in highly coloured effluent. These problems can be resolved by significantly enhancing fabric dye in the absence of salt and treating the fabric with various Carboxyethylchitosan concentrations. When the Nano Carboxyethylchitosan concentration reached 1 wt %, the antimicrobial activity increased and then stabilized at roughly constant levels. Nano Carboxyethylchitosan has antimicrobial activity in treated fabrics because it contains amino and carboxyl groups in this moiety. Additionally, Gram-positive bacteria are more resistant to antibiotics than Gram-negative bacteria.

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