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Enhancing Printability of Natural Fabrics via Pre-treatment with Polycationic Polymers

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Abstract

In this study natural fabrics such as cotton (C), wool (W) and cotton/wool (CW) fabrics were treated using polycations (PC) such as polyethyleneimine (PEI) and chitosan (CH) to enhance the printing results for natural and synthetic dyes. The effect of polycation concentration, printing paste pH, time and temperature of fixation on printing performance were studied. SEM, methylene blue test, FTIR, reflectance spectroscopy, water contact angle measurements, and antibacterial test were used to investigate the cationic group content, chemical surface modification, whiteness index, surface tension, and antibacterial properties of the modified cotton samples. Bactericidal inhibition tests revealed that the modified fabric with chitosan/TPP bilayers may enhance the degree of inhibition on E. coli and S. aureus bacteria. It was a simple and cost-effective approach.

Keyword: Natural fabrics – polycation – chitosan – polyethyleneimine – pre-treatment – printing

1. Introduction

Traditional printing methods include hand printing, discharge printing, [1] flocking printing, [2] block printing, and engraved copper printing. [3] Mechanical Printing Methods include Screen printing, Transfer printing, Digital inkjet printing. [3]

Printing paste is the primary component of printing that enables the creation of specific patterns. Printing paste is made up of pigments, thickeners, binders, and auxiliaries. As a result, each of the printing paste parts must be treated with care. All of the aforementioned ingredients are not utilised continuously in any pigment printing paste; instead, relevant components are sleeted in the creation of printing paste based on the kind of pigment used and the method of printing used. [4]

Finishing is well known for the fact that the surface characteristics of the fibres play an important role in the aesthetic and functional properties of the fabrics, which improves textile properties in general and aids in achieving desired properties, giving the textile its final commercial value and character. [5]

Polymers have been widely used in recent years instead of simple chemicals to improve multiple functional properties at the same time, such as increasing absorbency, particularly for natural dyes, antimicrobial properties, primarily for natural fibres, and ultraviolet protection (UV) for most natural and synthetic fibres by any finishing or coating method. [6-8]

Polyelectrolyte adsorption has been studied extensively for cellulosic and keratin fibres for roughly 40 years; polyelectrolytes include chitosan and polyethyleneimine. Using polyethyleneimine (PEI) for fibre pre-treatment strongly favours the adsorption of colouring dyes in fibre and may be particularly important for the textile industry since it dramatically boosts antibacterial activity. [9] Adsorption activity is most likely owing to pure electro-sorption behaviour, with the first increase linked with the polyelectrolyte binding with increasing salt concentrations. The polymer may permeate certain big pores in the fibre wall since the fibres are exceedingly porous. This, in turn, increases polyelectrolyte adsorption. [10]

Chitosan is an ideal choice for the eco-friendly textile industry since it has a high Nitrogen Atom content and is also a cheap cost plentiful natural resource that requires minimal processing when seeking an eco-friendly procedure that can be carried out without dangerous textile chemicals. [11, 12]

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Anahita et al. concentrated on creating an antibacterial cotton fabric that is suited for sanitary and medicinal applications utilizing TPP as the anionic polyelectrolyte and chitosan as the cationic polyelectrolyte. [8] Hassabo et al. processed reactive dye effluents before releasing them into the environment. [13] Hassabo et al. improved the dyeability of wool and cotton fibres by depositing a functionalized polyethyleneimine dye on them. [14] Ibrahim et al. attempted to identify the best finishing formulations to add antibacterial functionality to cellulosic/wool blends by employing citric acid (CA) as a crosslinker, sodium hypophosphite (SHP) as a catalyst, carefully selected bio-active additives, and the pad-dry microwave fixing procedure. [15] Mohamed et al. increased the dyeability and antibacterial activity of cotton by carboxymethylation and then padding it in nano zinc oxide or titanium isopropoxide or both, and then dyeing cotton using synthetic acid and basic dye. [16]

Furthermore, Yavaş et al.,[17] obtained coloured patterns on wool fabric by printing chitosan prior to dying. The un-treated and printed areas of wool fabrics dyed with acid and reactive dyes, which are anionic in nature, showed a significant colour difference, demonstrating that various colour patterns could be obtained by printing with chitosan. Abdelslam et al., [18] compared the effect of chitosan and Chitosan Nanoparticles on the printing of natural fabric.

The main objective of this work was to improve the printing results of natural fabrics printed by natural (tea leaves extract) and synthetic dyes (reactive and acid) using different polycation compounds (chitosan and polyethyleneimine), as well as to supply them with additional functional features such as antibacterial and UV-protection capabilities. Aside from conserving the necessary quantity of dyestuff and chemicals, which saves money and generates less environmental impact.

2. Experimental

2.1. Materials

In this study cotton (C, 100 %; 225 g/m^2) supplied by El-Nasr Spinning and Weaving Co., wool (W, 100 %; 235 g/m^2) company – mill-scoured and bleached cotton/wool 70/30 (CW, 70/30; 195 g/m^2) were obtained from the Golden-Tex and used for the application. Polycation (PC) materials (Polyethyleneimine (PEI, water-free Mw=25000 g/mol, Aldrich Co., see **Figure 1** for chemical structure) – Chitosan (Ch) (low molecular weight, deacetylation 82.9% Dalton, Vanson Inc. Co. USA), sodium carbonate, non-ionic surfactant, sodium carbonate (Na_2CO_3), methylene chloride (CH_2Cl_2), acetic acid (CH_3COOH), sodium thiosulfate ($Na_2S_2O_3$), hydrochloric acid (HCl), sodium chloride (NaCl), sodium hydroxide (NaOH), distilled water, non-ionic detergent (Hostapal NG). Reactive dye (Synozol red k-3BS, CI Reactive dye) and acid dye (Acid Bordeaux RLS, CI Acid Red 27) were kindly supplied from Newtrac company and their chemical structure is illustrated in **Figure 1**. Dried tea leaves were purchased from the Egyptian local market.

2.2. Methods

2.2.1. Preparation of tea leaves extract

Our prior work was used to extract tea leaves. [19] In brief, 40 g dried tea leaves were boiled for 24 hours in 1 L water in the Soxhlet system. After that, the filtrate containing extracted dyes was collected to use in fabric printing. Natural dye concentration was: 0.5 % (extraction in double amount of water), 1 % (normal extraction as it is), 2% (extraction in half amount of water) and 3% (extraction in one third amount of water)). The water extract was concentrated using a rotary evaporator.

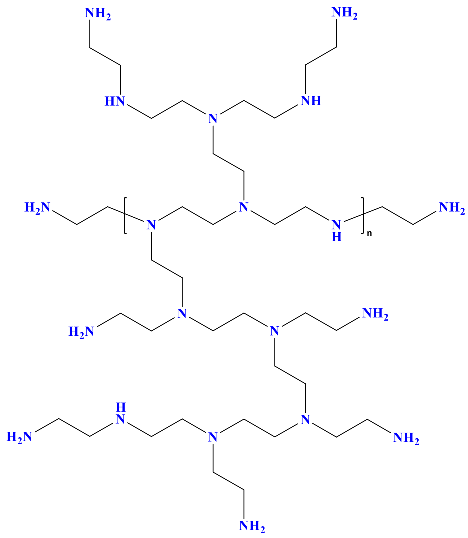
2.2.2. Fabric scouring

Before being treated with PEI or chitosan polymers, the cotton and wool textiles were washed to remove waxy components and impurities from the outer surface of the fibres. Cotton and cotton/wool fabrics were scoured in a 2 g/L (Na_2CO_3) solution for 15 minutes, while wool fibres were scoured in non-ionic detergent 2 g/L for 15 minutes, both at 40°C and then dried at room temperature.

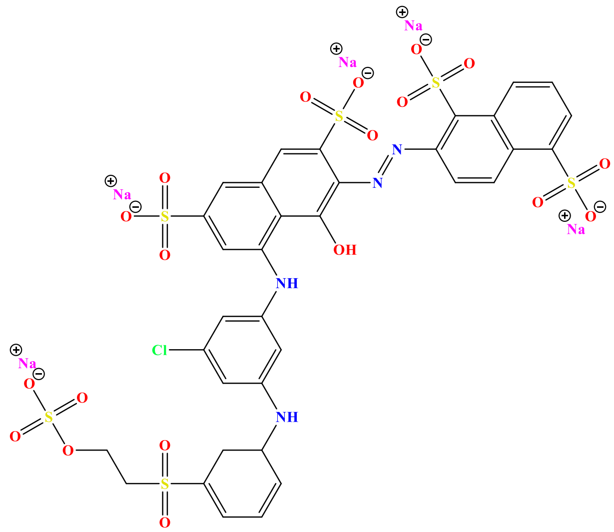
2.2.3. Fabric treatment with polycation compounds

Chitosan and polyethyleneimine solutions in various concentrations (0.5, 1, 2, or 4% w/v) were made by dissolving chitosan powder in a 1% acetic acid solution and dissolving PEI in distilled water under continuously stirring.

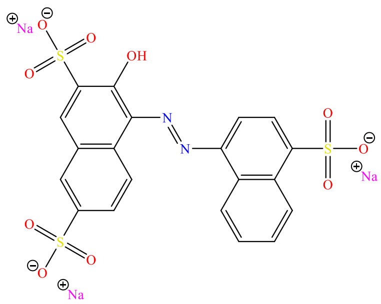
Washed fabrics were treated with citric acid; 10 g/L) and sodium hypophosphite (SHP; 5 g/L). Fabrics were then submerged in the polycation solutions for about 20 minutes at 40°C. After that, the samples were padded to a 100% wet pick-up and dried for 5 minutes at 100°C. To remove unreacted chemicals, the treated textiles were washed in a washing solution containing 2 g/L non-ionic detergents.



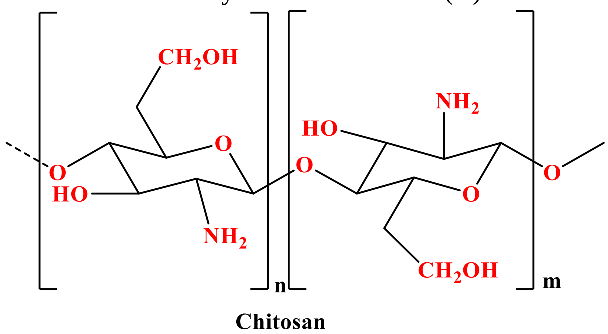
Poly(ethyleneImine) PEI



Synozol RED K 3BS (A)



Acid Red 27



Chitosan

Figure 1: Chemical structure of PEI, and Synozol RED K 3BS and Acid Red 27

2.2.4. Preparation of printing paste

The following recipe was used to prepare the printing paste for different fabrics using reactive or acid dye.

Printing paste consistent	Cotton	Wool	Cotton/Wool
Reactive dye	0.5, 1, 2, 3 g	0.5, 1, 2, 3 g	0.5, 1, 2, 3 g
Thickener	70 g	70 g	70 g
Urea	15 g	15 g	15 g
Sodium carbonate	till pH=9	till pH=5.5	till pH=6
Glycerin	2 g	2 g	2 g
Water	Y g	Y g	Y g
Total	100 g	100 g	100 g

After preparing the paste, it was stored for 2–3 hours before applying it to the materials (to allow paste to relax after mixing and cool to its internal temperature). The samples were then printed using the created paste using a manual silkscreen.

2.2.5. Printing Procedure

The fabric samples were printed using synthetic dyes (reactive or acid dyes) and natural dye (tea leave extract). Effect of polycation type and concentration (PEI, chitosan; (0.5, 1, 2, or 4% w/v)) and dye concentration (0.5, 1, 2 %) were investigated as effective printing factors on the colour performance. Finally, the printed fabrics were dried at 100°C for 5 minutes and cured at 140°C for 5 minutes in an air oven. [20, 21]after that, the printed fabrics were then rinsed with water for about 15 min. at 50°C in the presence of 2 g/L non-ionic detergent, then dried at 85°C for 3 minutes. Flat-screen printing was used during this research.

2.3. Measurements

2.3.1. Determination of the amine content

The amine content of prepared materials and/or textiles is determined using the modified method for carboxyl content. [22] in this modified method: 0.25 g of the sample and 40 ml of HCl (0.1 M, 30 g NaCl + 10 g HCl in one litre) were added to a 250 ml stoppered conical flask. Filter and titrate the filtrate with phenolphthalein as an indicator against NaOH (0.1 M) after leaving the conical flasks overnight with occasional shaking. The following equation was used to calculate the proportion of amino acids:

$$\text{amine contents (\%)} = \frac{(V_B - V_S) \times M_{NaOH}}{W_t} \times 100$$

which V_B is the Volume of NaOH consumed by blank experiment, V_s is the Volume of NaOH consumed by sample, M_{NaOH} = Molarity of NaOH, and W is the sample weight.

2.3.2. Colour Measurements

A computer-based automated philtre spectrometer (Data Colour Model 3890, Marl Co., Germany) was used to determine the colour strength K/S of untreated and treated printed fabrics. The Kubelka-Munk equation was used to determine the K/S values as follows: [23-30]

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-R_o)^2}{2R_o}$$

Where: R and R_o are representing the colour reflection of printed sample and uncoloured, respectively.

2.3.3. Colourfastness properties

The AATCC test method 61-2013 was used to measure colourfastness to washing using a Launder-Ometer. [31] The AATCC test method 8-2016 was used to measure colourfastness to rubbing (dry and wet). [32] The AATCC test method 15-2013 was used to measure colourfastness to perspiration (acid and alkaline). [33] The AATCC test method 15-2013 was used to determine the colourfastness to light. [34] The colour shift grayscale reference was used to assess washing, rubbing, and perspiration fastness properties, while the colour change blue scale reference was utilized to evaluate light fastness property for coloured materials.

2.3.4. Dye Fixation Measurement

To evaluate dye fixing, the printed fabric was washed at 50°C for 30 minutes. The colour strength values of the printed fabric were determined after and before washing. The dye fixation (F %) was calculated using the equation below

$$F\ (\%) = \frac{(K/S)_a}{(K/S)_b} \times 100$$

where $(K/S)_a$ and $(K/S)_b$ are the colour strength of printed fabric after and before washing respectively.

2.3.5. Physical and Mechanical properties

The AATCC Test Method 66 – 2014 was used to determine the dry crease recovery angle (CRA). [35] Fabric roughness was determined using a SE 1700 Surface Roughness Measuring Instrument according to ASTM Test Method D 7127 – 13. [36] ASTM Test Method D5035-2011 was used to determine tensile strength and elongation at a break. [37] The ASTM D737 standard technique was used to measure the air permeability using the TEXTEST FX-3300 at a pressure gradient of 100 Pa. [38]

2.3.6. Antimicrobial Measurement

Gram-negative bacteria (Escherichia coli; ATCC 25922), gram-positive bacteria (Staphylococcus aureus; ATCC 6538), and fungi (Candida albicans; ATCC 10231) were used in this investigation. **[39]**

The samples were applied to these tested microorganisms using the shake flask method to calculate antimicrobial activity throughout reduction (%) of the growth of these selected microbial strains was detected by optical density (OD) at 600 nm, and the antimicrobial activity was measured throughout the relative [OD (%)] reduction of these microbial strains compared to the control of these microbial strains. The following equation was used to express all of the results:

$$Relative\ (OD\ Reduction\ (\%)) = \frac{A - B}{A} \times 100$$

where A and B are the (OD) of the control flask and tested flask respectively.

3. Results and Discussion

3.1. Characterization of the treated fabric

When the polycations concentration is increased during the pre-treatment procedure, pre-treated fabrics have a greater amine content than the untreated ones. Furthermore, pre-treated textiles with PEI had a higher amine content than pre-treated fabrics with chitosan at all investigated concentrations (see **Table 1**). In addition, increasing the polycation concentration by more than 1% results in a slight increase in the amine content values of treated fabrics, leading to the conclusion that one percent polycation compound is more appropriate for this study.

The FTIR spectrum of the treated textiles is shown in **Figure 2**. The band at 1650 cm^{-1} is due to the amino groups in the polycations chain. When fabrics were treated with each polycation compound, a shoulder band appeared at 1722 cm^{-1}, suggesting the production of new amide groups as a result of the interaction between the amine groups of polycations and the carboxyl groups of each crosslinker or wool fibres.

The NH_2 group was found in the 3000-3200 cm^{-1} a broad band and a sharp band 1590 cm^{-1} in the FTIR spectra of treated fabrics with each polycation, as shown in **Figure 2**. A stretching band has been observed at 1245 cm^{-1}, which is linked to C-O from phenol or alcohol. **[40]** In the FTIR spectra, the ester or amide group displays long wavelengths ranging from 1643 to 1657 cm^{-1}. The amplitude of waves at 1643 and 1657 cm^{-1}, which was the area for the C=O group, was enhanced owing to pre-treated textiles with citric acid.

Tensile strength, elongation at break, roughness, and crease recovery angle of cotton, wool, and cotton/wool textiles in the warp and weft directions were measured before and after they were treated with 1% polycations (PEI, and chitosan), and the results are presented in **Table 2**. Both chitosan and PEI have a favourable effect on textile fabrics, as seen by an increase in amine percent.

Table 2 shows that raising the amine content in treated textile materials causes changes in physicomechanical properties, which were of special interest. While the crease recovery angle was raised, fabric roughness, tensile strength, and elongation at the break are decreased. This indicates that the polycations under investigation were firmly embedded in the textile fabrics' microstructure, generating a thin coating layer on the fabric's surface that was responsible for the observed changes. [41-44]

Table 1: Amine content for treated fabrics using different polycation compounds

Fabric	Polycation type	Polycation (%)	NH_2 (mg/ g fabric)	NH_2 %
Cotton	PEI	0	0.32	0.17
		0.5	298.72	20.52
		1	351.88	41.04
		2	369.60	47.88
		4	372.25	52.58
	Chitosan	0	0.32	0.17
		0.5	254.42	12.90
		1	263.28	16.32
		2	289.86	26.57
		4	298.10	28.68
Wool	PEI	0	18.57	1.42
		0.5	362.20	24.88
		1	426.66	49.76
		2	448.14	58.05
		4	451.35	53.75
	Chitosan	0	18.57	1.42
		0.5	308.48	15.64
		1	319.23	19.79
		2	351.46	32.22
		4	361.45	34.77
Cotton / Wool	PEI	0	14.94	1.30
		0.5	330.46	22.70
		1	389.27	45.40
		2	408.87	52.96
		4	411.80	58.17
	Chitosan	0	14.94	1.30
		0.5	281.45	14.27
		1	291.25	18.05
		2	320.66	29.40
		4	329.77	31.72

Treatment conditions: Polycations (X %); temperature: 50°C, Time: 15 min, drying for 5 min at 80°C

Table 3 and **Figure 3** demonstrate the amine content and colour strength (K/S) values for printed pre-treated textiles with varied concentrations of PEI and chitosan (0.5, 1, 2, and 4%), and different dyes (acid, reactive, and natural dye (tea leaves extract)).

The amine content of the printed fabric rose when the concentration of polycation compounds in the pre-treatment step was raised, which boosted the colour strength of the printed fabrics with all examined dyes (synthetic or natural) on all fabric types compared to the untreated one. Furthermore, pre-treated printed fabrics with PEI had a greater amine content and higher K/S values than pre-treated printed fabric with chitosan at all concentrations.

The key finding is that increasing the polycation concentration in the pre-treatment process by more than 1% leads to a slight increase in K/S values of printed textiles, leading to the conclusion that pre-treatment of fabrics with 1% polycations compound is more suitable for this study.

3.2. Optimisation of the printability parameters

3.2.1. Effect of polycation concentration

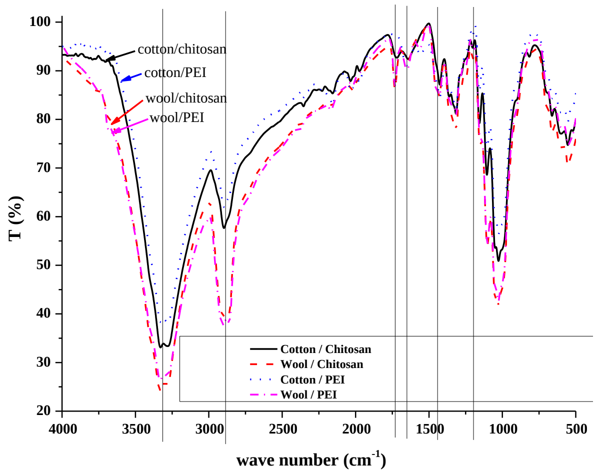


Figure 2: FT-IR spectra for treated fabrics with different composites

Table 2: Physical and mechanical properties for treated fabrics using different polycation compounds

Fabric	Polycation (1 %)	Physical and Mechanical properties			
		R (µm)	Tensile strength	Elongation at a break (%)	CRA (W+F°)
Cotton	without	20.9	154.0	36.1	221.6
	PEI	18.6	113.1	25.8	206.3
	Chitosan	19.8	115.1	28.6	188.0
Wool	without	21.4	158.0	37.0	227.4
	PEI	19.1	116.0	26.4	211.7
	Chitosan	20.4	118.1	29.3	192.9
Cotton / Wool	without	22.0	162.1	38.0	233.3
	PEI	19.5	119.0	27.1	217.1
	Chitosan	20.9	121.2	30.1	197.8

Pre-Treatment conditions: citric acid (10 g/L); sodium hypophosphite (5 g/L); drying for 5 min at 80°C
Treatment conditions: Polycations (1 %); temperature: 50°C, Time: 15 min, drying for 5 min at 80°C

R: Roughness, CRA (W+F): average Crease Recovery Angle in warp and weft directions

Table 3: Amine content and colour strength (K/S) values for printed treated fabrics using different polycation compounds

Fabric	Polycation type	Polycation (%)	NH_2 (mg/1 g fabric)	NH_2 %	Colour Performance					
					Reactive dye (λ = 540)		Acid dye (λ = 510)		Natural dye (Tea leaves, λ= 270)	
					K/S	Dye fixation (%)	K/S	Dye fixation (%)	K/S	Dye fixation (%)
Cotton	PEI	0	0.32	0.17	4.98	75.49	3.12	46.32	6.20	45.04
		0.5	298.72	20.52	10.70	88.42	6.18	69.98	11.97	69.96
		1	351.88	41.04	10.91	83.66	6.43	83.10	15.15	84.26
		2	369.60	47.88	10.91	86.50	6.41	83.13	15.72	87.95
		4	372.25	52.58	10.21	83.70	6.40	80.36	15.66	81.19
	Chitosan	0	0.32	0.17	4.98	75.49	3.12	46.32	6.20	45.04
		0.5	254.42	12.90	8.97	87.66	7.42	45.10	10.48	60.67
		1	263.28	16.32	9.21	97.12	7.45	47.14	10.92	63.45
		2	289.86	26.57	9.51	94.58	7.25	47.79	11.16	67.74
		4	298.10	28.68	8.39	95.13	8.67	51.90	10.95	75.04
Wool	PEI	0	18.57	1.42	3.40	94.08	4.31	50.55	5.94	59.83
		0.5	362.20	24.88	9.78	94.35	8.26	58.00	12.55	75.05
		1	426.66	49.76	10.25	88.02	8.43	59.79	14.53	81.38
		2	448.14	58.05	10.57	89.34	8.40	59.84	15.41	82.35
		4	451.35	63.75	10.40	91.20	8.56	59.25	15.40	80.18
	Chitosan	0	18.57	1.42	3.40	94.08	4.31	50.55	5.94	59.83
		0.5	308.48	15.64	6.85	80.99	8.41	56.12	9.98	75.79
		1	319.23	19.79	6.91	76.35	8.44	60.82	10.62	82.71
		2	351.46	32.22	6.90	75.44	8.45	61.24	10.54	84.40
		4	361.45	34.77	6.92	80.15	8.46	64.66	10.54	82.25
Cotton / Wool	PEI	0	14.94	1.30	4.79	74.27	2.63	44.11	6.70	63.92
		0.5	330.46	22.70	13.29	84.94	8.18	79.41	12.42	83.87
		1	389.27	45.40	13.29	86.66	7.73	76.27	14.23	91.11
		2	408.87	52.96	9.04	80.92	3.44	47.25	13.53	87.87
		4	411.80	58.17	9.77	84.06	3.54	47.84	12.84	84.64
	Chitosan	0	14.94	1.30	4.79	74.27	2.63	44.11	6.70	63.92
		0.5	281.45	14.27	8.94	91.47	7.76	45.50	13.11	66.32
		1	291.25	18.05	9.27	94.42	7.65	43.72	14.00	71.29
		2	320.66	29.40	9.46	94.57	7.39	39.52	14.15	78.50
		4	329.77	31.72	9.52	94.68	7.34	39.60	14.72	82.10

Pre-Treatment conditions: citric acid (10 g/L); sodium hypophosphite (5 g/L); drying for 5 min at 80°C
Treatment conditions: Polycations (X %); temperature: 50°C, Time: 15 min, drying for 5 min at 80°C
Printing conditions: dye concentration (5 g/L, **Natural dye concentration was**: normal extraction as it is); Polycations (X %); drying for 5 min at 110°C, curing for 3 min at 140°C

Dye fixation (%) = $\frac{K/S_{after\ washing}}{K/S_{before\ washing}} \times 100$

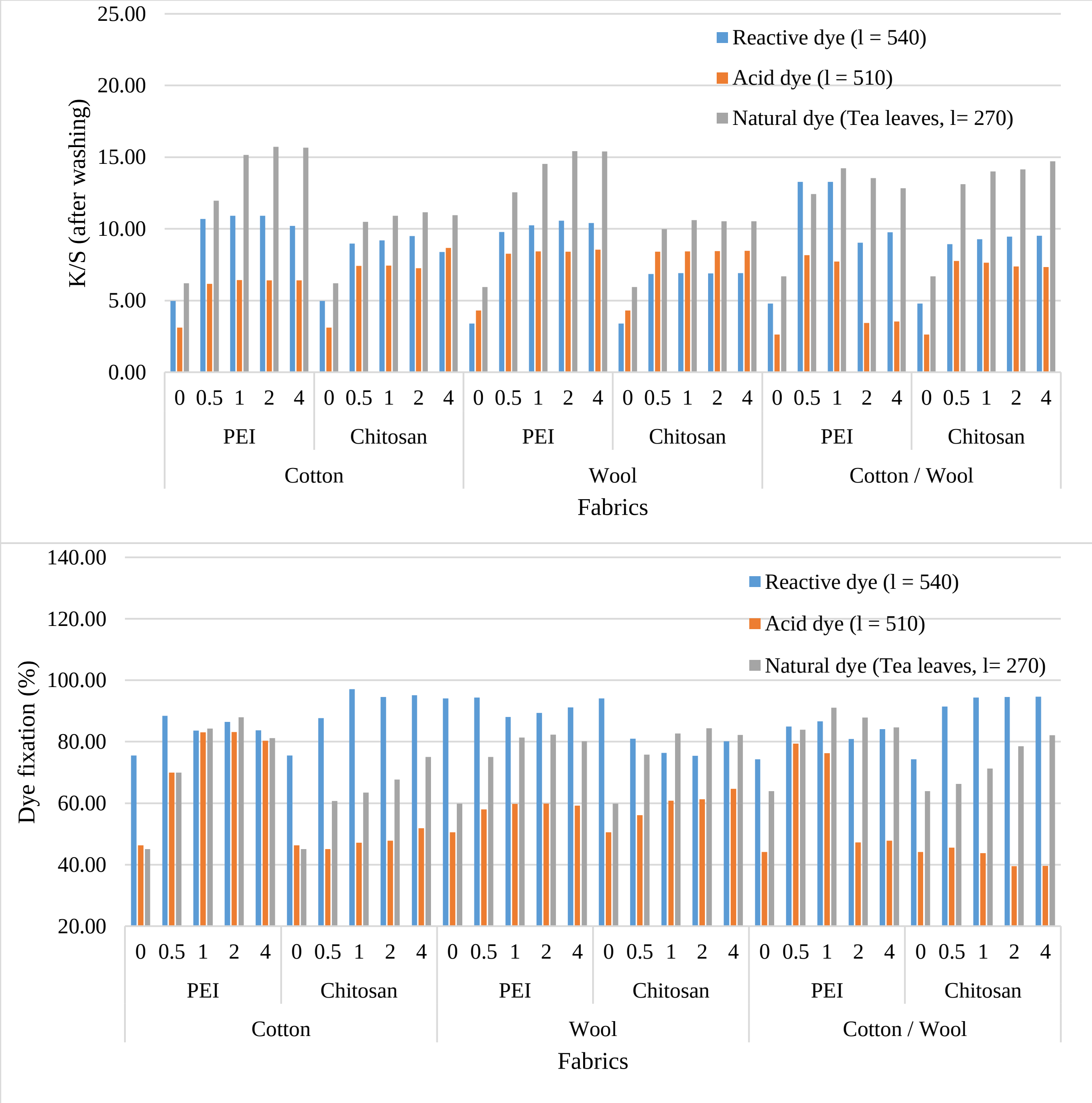


Figure 3: Colour strength (K/S) values and dye fixation (%) for printed treated fabrics with different concentrations of polycation compounds

When it comes to dye affinity, it's apparent that textiles made of cotton or wool, or their blends, give high colour strength and dye fixation when treated with polycation compounds.

Table 3, treatment with polycation compounds improved the dye-fixing of the utilised dyes (reactive, acid, and/or natural dye) on all substrates. PEI-treated textiles had a higher increase than chitosan-treated textiles. When both polycations were used, chemical interactions between (i) amino groups from polycation compounds, (ii) hydroxyl groups on the cotton surface or carboxyl groups

The dye fixation of printed untreated and treated textile fabrics was calculated using the equation presented in the experimental section. As demonstrated in

from the wool surface or crosslinker, and (iii) dye molecules dramatically increased dye-fixing.

3.2.2 Effect of dye concentration

The colour strength (K/S) values for printed treated fabrics (cotton, wool and cotton/wool) with polycations (1%) at various dye concentrations (0.5, 1, 2 and 3 %) compared to untreated fabrics printed with 3 % dye concentration are shown in **Error! Reference source not found.** and **Figure 4**. As the dye concentrations rise from 0.5 to 2 %, the colour strength of printed fabrics improves.

During this investigation, untreated fabrics were printed using suitable printing paste containing 3 % dye concentration to compare the effect of dye concentration on the colour performance of treated fabrics.

From the results, it is clear that increasing the dye concentration led to increasing the colour strength and dye fixation for each treated fabric with each polycation. In addition, printed fabrics with each polycation using 0.5 % dye concentration provide colour strength closely with untreated printed fabric using 3 % dye concentration, which provides the role of pretreatment with polycation compounds. These results confirm that during this study pretreatment the fabric with a low amount of each polycation decrease the dye used as well as the cost with similar or high colour strength values.

Table **5** shows the fastness properties of printed treated fabrics (cotton, wool, and cotton wool) with a polycation (PEI and chitosan; 1%) using three different dye natures (reactive, acid, and nature (tea leaves extract)) with 0.5 % as concentration

Treated textiles showed superior washing, perspiration, lighting, and rubbing fastness properties than untreated materials. Because the polycation treated textiles absorb more dye molecules (resulting in the presence of amino group), chitosan and polyethyleneimine treatment significantly enhanced the washing and lightfastness qualities of coloured fabrics using all investigated dyes as compared to untreated materials. The chemical interactions created between polycations-treated materials and dye molecules are responsible for this enhancement.

Furthermore, the dye molecules are bonded to the textile surface due to an increase of hydrogen and chemical bonding between dye molecules, polycations, the fabric surface, and the crosslinker. The dye molecules are absorbed better and more uniformly as a result of the homogeneous coated film on the fabric surface.

1.1.1. Mechanical and physical properties

Tensile strength, elongation at a break, bending length, crease recovery angle, and surface roughness of printed textiles have all been investigated, and the results are presented in **Error! Reference source not found.**.Tensile strength, elongation at a break, crease recovery angle in the warp and weft directions, and surface roughness of cotton, wool, and cotton/wool textiles were measured before and after treatment with 1% polycation polymers, with the findings shown in **Error! Reference source not found.**. The presence of amino groups in both

As shown in **Error! Reference source not found.** and **Figure 4**, raising the dye concentrations (0.5, 1, and 2 %) increased the dye-fixing of the used dyes (reactive, acid, and/or natural dye) on all substrates, resulting in minor changes in colour fastness and dye fixation. PEI-treated textiles increased more than chitosan-treated textiles because PEI contains more amine, which allows for more interactions between dye molecules and the fabric surface.

Another finding is that reactive dye has a higher dye-fixing percentage than the other dyes studied (acid or natural). Furthermore, raising the dye concentrations improves the colour strength of textile materials with natural dye.

3.3. Characterization of the printed textile fabrics

3.3.1. Fastness properties

polycation polymers has been proven to improve the performance of textile fibres.

As shown in **Error! Reference source not found.**, the presence of a amino group in treated textile materials causes changes in physicomechanical parameters. When the crease recovery angle was raised, the fabric roughness, tensile strength, and elongation at the break all decreased. This shows that the polycation polymers under investigation were firmly embedded in the textile fabrics' microstructure, resulting in a thin coating layer on the fabric's surface that produced the observed changes. [41-44]

The pre-crosslinking treatment of cotton, wool, and/or cotton/wool textiles with citric acid catalysed by sodium hypophosphite (SHP) had a substantial impact on the performance of the previously examined attributes. During this pre-treatment, covalent crosslinking connections between neighboring cellulosic or proteinic chains would form, giving the cotton structure rigidity. Meanwhile, the citric acid and catalyst would degrade the cotton structure chemically. Stiffness and chemical degradation were reasonable causes of the decline in tensile strength. The creation of an extensive network with a high degree of crosslinking via covalent chemical bonding most likely produced the massive increase in the crease healing angle.

1.1.2. Antimicrobial activity

Error! Reference source not found. shows the antimicrobial effects of cotton, wool, and cotton/wool fabrics treated with one percent polycations and printed with three different dye natures (reactive, acid, and natural (tea extract) using three different microorganisms (i) gram-positive (Staphylococcus aureus), (ii) gram-negative (Escherichia coli), and (iii) fungal (Candida Albicans).

The results show that two types of bacteria (gram-positive and negative bacteria), as well as a fungus (Candida Albicans), had a lesser inhibitory influence on untreated printed textile materials than treated printed textile fabrics. This action is caused by the presence of amino groups from each polycation compound.

Because the cell walls of both the tested bacterial strains differ in composition, the treated fabrics are more efficient against gram-negative bacteria than gram-positive bacteria.

The polycations in use also block ergosterol, a critical component of the fungal cell membrane. **[29, 45-50]**

Table 4: Colour strength (K/S) values and dye fixation (%) for printed treated fabrics using different dyes concentration (%)

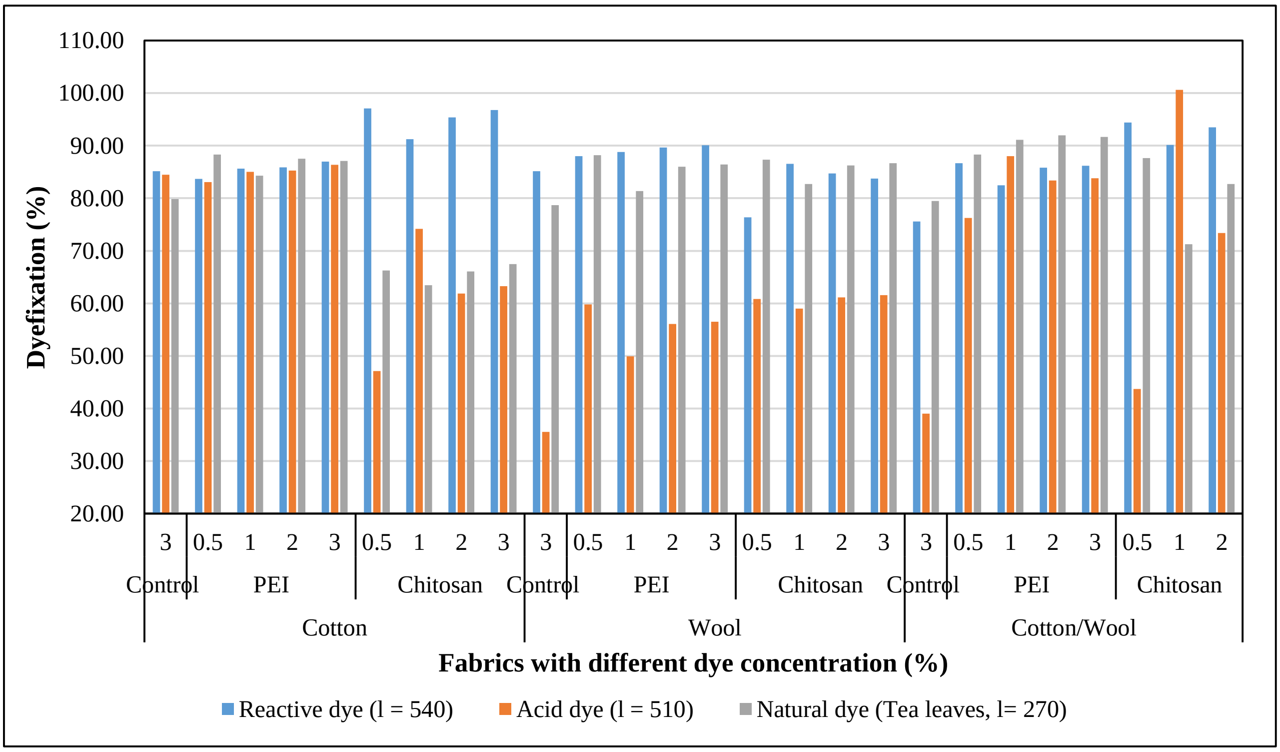
Fabric	Polycation type	Dye Concentration (%)	Colour Performance					
			Reactive dye (λ = 540)		Acid dye (λ = 510)		Natural dye (Tea leaves, λ= 270)	
			K/S	Dye fixation (%)	K/S	Dye fixation (%)	K/S	Dye fixation (%)
Cotton	Control	3	10.97	85.15	6.53	84.45	14.39	79.87
	PEI	0.5	10.91	83.66	6.43	83.10	12.11	88.32
		1	12.17	85.63	7.71	85.01	15.15	84.26
		2	12.77	85.87	8.30	85.28	16.86	87.52
		3	13.70	86.98	9.23	86.37	17.23	87.12
	Chitosan	0.5	9.21	97.12	7.45	47.14	8.11	66.25
		1	13.12	91.22	9.55	74.16	10.92	63.45
		2	14.39	95.40	9.73	61.88	10.75	66.08
		3	14.47	96.81	11.14	63.29	12.16	67.49
Wool	Control	3	8.85	85.12	7.01	35.55	10.71	78.69
	PEI	0.5	10.25	88.02	8.43	59.79	8.19	88.17
		1	11.78	88.80	9.95	49.90	14.53	81.38
		2	12.25	89.64	10.42	56.08	14.59	86.00
		3	12.66	90.05	10.83	56.49	14.67	86.41
	Chitosan	0.5	6.91	76.35	8.44	60.82	7.25	87.32
		1	11.83	86.58	11.12	59.02	10.62	82.71
		2	11.60	84.69	11.01	61.15	11.17	86.25
		3	11.35	83.77	11.42	61.56	11.91	86.66
Cotton/Wool	Control	3	12.71	75.58	5.51	39.05	13.61	79.47
	PEI	0.5	13.29	86.66	7.73	76.27	9.84	88.32
		1	14.35	82.47	7.97	88.01	14.23	91.11
		2	15.05	85.79	9.08	83.37	14.26	91.94
		3	15.46	86.20	9.49	83.78	15.01	91.69
	Chitosan	0.5	9.27	94.42	7.65	43.72	9.36	87.66
		1	14.61	90.15	10.09	100.64	14.00	71.29
		2	13.17	93.51	10.10	73.41	14.91	82.70
		3	13.58	93.92	10.51	73.82	14.99	81.78

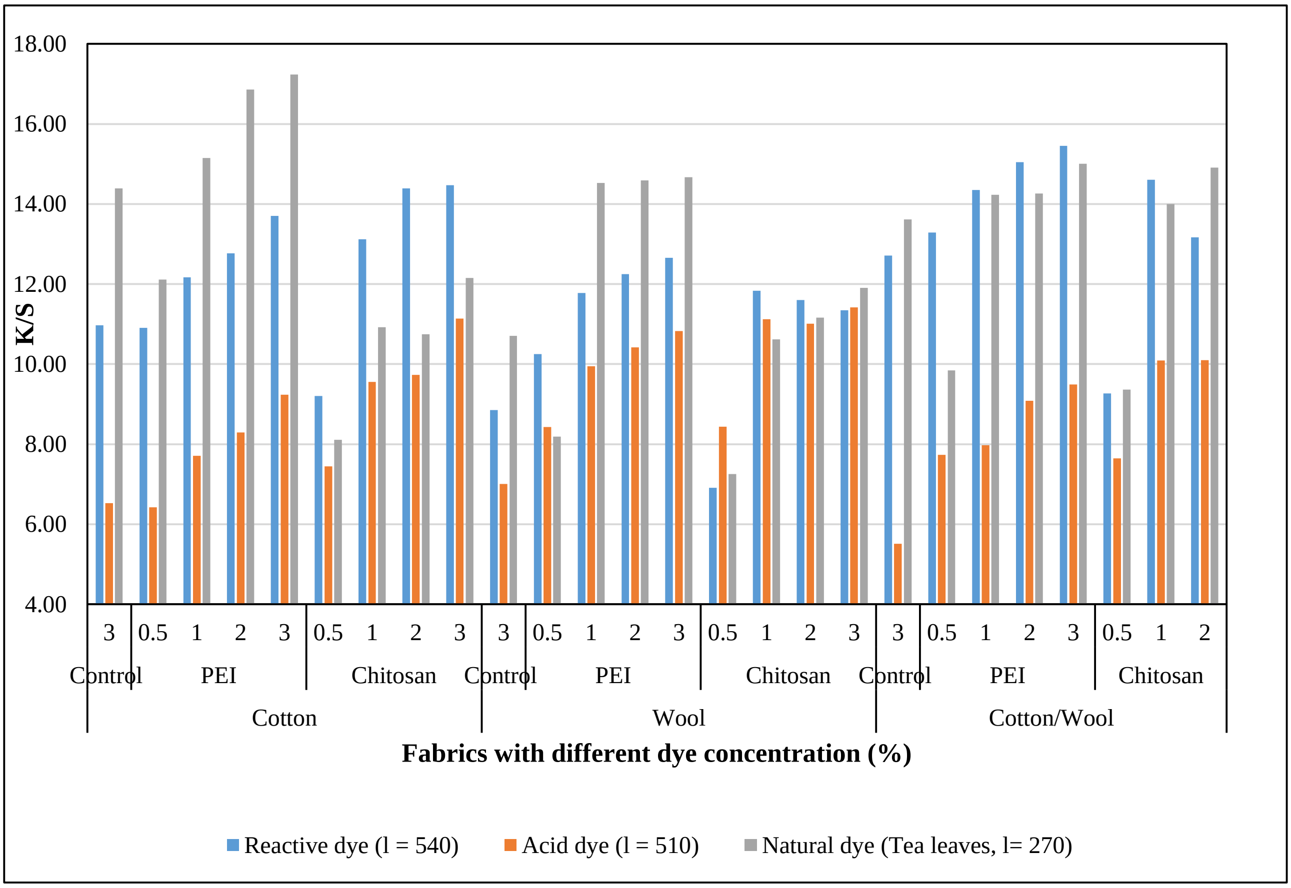
Pre-Treatment conditions: citric acid (10 g/L); sodium hypophosphite (5 g/L); drying for 5 min at 80°C
Treatment conditions: Polycations (1 %); temperature: 50°C, Time: 15 min, drying for 5 min at 80°C

Printing conditions: dye concentration (X (%), **Natural dye concentration was**: 0.5 % (extraction in double amount of water), 1 % (normal extraction as it is), 2% (extraction in half amount of water) and 3% (extraction in thirdth amount of water)); Polycations (1 %); drying for 5 min at 110°C, curing for 3 min at 140°C

Dye fixation (%) = $\frac{K/S_{after\ washing}}{K/S_{before\ washing}} \times 100$

Figure 4: Colour strength (K/S) values and dye fixation (%) printed treated fabrics using different dyes concentrations (%)




18.00
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K/S
3 0.5 1 2 3 0.5 1 2 3 3 0.5 1 2 3 0.5 1 2 3 3 0.5 1 2 3 0.5 1 2
Control PEI Chitosan Control PEI Chitosan Control PEI Chitosan
Cotton Wool Cotton/Wool
Fabrics with different dye concentration (%)
Reactive dye (l = 540)
Acid dye (l = 510)
Natural dye (Tea leaves, l= 270)

Table 5: Fastness properties for the printed treated fabrics with polycations using different dyes nature

Fabric	dye	Polycation (1 %)	Fastness properties											
			Washing		Rubbing				Perspiration				Light
					Dry		Wet		Acidic		Alkaline		
			Alt	St	Alt	St	Alt	St	Alt	St	Alt	St	
Cotton	reactive dye	without	3	2-3	2-3	2-3	2-3	2-3	3	2-3	3	3	4
		PEI	4	4	4-5	4-5	4-5	4-5	3	4-5	4-5	4-5	5
		Chitosan	3-4	3	3-4	3	3	3	3	3	3	3	5
	acid dye	without	2-3	2-3	2-3	2-3	2-3	2-3	2	2	2	2	4
		PEI	4	4	4	4	4	4	4	4	4	4	5
		Chitosan	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	5
	natural dye	without	2	2	2	2	2	2	2	2	2	2	4
		PEI	4	4	3	4	4	3	3	4	4	3	4
		Chitosan	3-4	3-4	3	3-4	3-4	3	3-4	3-4	3	3	4
Wool	reactive dye	without	3-4	3	3	3-4	3-4	3	3-4	3-4	3	3	4
		PEI	4	4	4	4	4	4	4	4	4	4	5
		Chitosan	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	3-4	5
	acid dye	without	3	3	3	3	3	3	3	3	3	3	4
		PEI	4	4	3-4	3-4	3-4	3-4	3-4	4	4	4	5
		Chitosan	3	3	4	4-5	3	3-4	3	3-4	3	3	5
	natural dye	without	3	3	3	3	3	2-3	3	2-3	3	2-3	4
		PEI	4	4	4	4	3-4	4	3-4	3-4	3-4	3-4	5
		Chitosan	4	3-4	3-4	4	3-4	3-4	3-4	3-4	3	3	5
Cotton / Wool	reactive dye	without	3	3	3	3	3	3	3	3	3	3	4
		PEI	4	4	3-4	3-4	3-4	3-4	3-4	4	4	4	5
		Chitosan	3	3	4	4-5	3	3-4	3	3-4	3	3	5
	acid dye	without	3	3	3	3	3	2-3	3	2-3	3	2-3	4
		PEI	4	4	4	4	3-4	4	3-4	3-4	3-4	3-4	5
		Chitosan	4	3-4	3-4	4	3-4	3-4	3-4	3-4	3	3	5
	natural dye	without	3	3	3	3	3	2-3	3	2-3	3	2-3	4
		PEI	4	4	4	4	3-4	4	3-4	3-4	3-4	3-4	5
		Chitosan	4	3-4	3-4	4	3-4	3-4	3-4	3-4	3	3	5

Pre-treatment condition: 10 g/L citric acid and 5 g/L sodium hypophosphite

Treatment condition: polymer conc. 1 %, temperature: 50°C, Time: 15 min, drying at 110°C for 5 min.

Printing condition: Dye concentration 0.5 g/L (for reactive and acid dyes) and 100 % (natural dye extract), drying at 110°C for 5 min. and curing for 140°C for 3 min

Table 6: physical and mechanical properties for untreated and treated printed fabrics

Fabric	Polycation (1 %)	NH_2 (mg/1 g fabric)	NH_2 %	Physical and Mechanical properties			
				R (µm)	Tensile strength	Elongation at a break (%)	CRA (W+F°)
Cotton	without	15.32	1.17	19.7	133.5	30.9	214.0
	PEI	351.88	41.04	19.2	137.4	32.2	220.4
	Chitosan	263.28	16.32	20.6	136.6	32.8	217.3
Wool	without	18.57	1.42	20.3	137.0	31.7	219.6
	PEI	426.66	49.76	19.7	140.3	32.8	222.3
	Chitosan	319.23	19.79	21.2	140.1	33.7	223.9
Cotton / Wool	without	16.94	1.30	20.8	140.5	32.6	225.2
	PEI	389.27	45.40	20.2	142.1	32.6	227.5
	Chitosan	291.25	18.05	20.9	141.2	32.6	227.8

Fabric	Dye	Polycation (1 %)	Microbial Reduction %					
			E. coli (ATCC 25922)		S. Aureus (ATCC 29213)		C. Albicans (ATCC 10231)	
			before washing	after 10 washing cycles	before washing	after 10 washing cycles	before washing	after 10 washing cycles
Cotton	Reactive dye	without	23.3	19.4	24.2	18.6	22.7	19.4
		PEI	81.6	80.3	84.5	77.6	79.2	70.6
		Chitosan	72.2	70.5	74.8	68.1	70.1	62.5
	Acid dye	without	40.7	37.6	42.2	36.2	39.6	33.8
		PEI	89.6	88.5	92.7	85.6	86.7	80.6
		Chitosan	78.3	75.2	79.5	72.7	75.1	70.9
	Natural dye	without	58.1	55.8	60.3	53.8	56.5	48.3
		PEI	97.6	96.8	100.8	93.6	94.3	90.6
		Chitosan	84.5	80.0	84.2	77.4	80.1	79.3
Wool	Reactive dye	without	38.7	35.4	40.0	34.1	37.4	34.3
		PEI	87.3	86.0	90.1	83.1	84.3	80.4
		Chitosan	78.3	76.7	81.0	74.2	75.8	70.0
	Acid dye	without	52.4	49.7	54.2	48.0	50.8	46.5
		PEI	89.5	86.9	91.0	85.4	86.5	82.5
		Chitosan	81.5	78.3	82.6	77.4	78.9	72.8
	Natural dye	without	66.2	64.1	68.5	61.9	64.1	58.8
		PEI	91.8	87.9	92.0	87.7	88.7	84.6
		Chitosan	84.7	79.9	84.1	80.6	82.0	75.7
Cotton /	Reactive	without	36.0	32.5	37.2	31.4	34.7	34.3

Wool	dye	PEI	83.0	81.4	85.6	78.8	80.0	79.0
		Chitosan	77.0	75.1	79.3	72.6	74.1	73.3
	Acid dye	without	57.7	55.0	59.5	53.2	55.6	54.9
		PEI	88.4	85.6	89.7	84.2	85.1	84.1
		Chitosan	83.8	80.5	84.6	79.5	80.7	79.8
	Natural dye	without	79.3	77.6	81.7	75.0	76.4	75.5
		PEI	93.7	89.8	93.8	89.6	90.3	89.2
		Chitosan	90.6	85.8	89.9	86.4	87.3	86.2

Table 7: CFU reduction (%) of microbial strain cells for untreated and treated printed fabrics before and after washing

Textiles treated with PEI have a stronger antibacterial impact than those treated with chitosan. This is owing to the presence of a significant number of amino groups, which play a key role in disrupting microbial cell membranes while also having a strong antibacterial impact. **[19, 41]**

Natural dye extract (tea leaves extract) contains metals, phenolic acids, and flavonoids that have successfully interacted with the bacterium's cells. The cover's polycation chemicals spread and stabilise the extract dyes' components (metals, phenolic acids, and flavonoids) well on the fabric surface, decreasing their potential to interact with bacterial cells. **[51-54]**

Furthermore, treated and printed textile materials have superior antibacterial characteristics before and after washing than untreated textile fabrics. After washing, the treated materials' resilience gives an excellent antibacterial action against all microbial tested.

2. Conclusion

Textile materials (cotton, wool, and cotton/wool fabrics) were studied for polycation modification. Both treatments with both polycation compounds enhanced the colour performance of printed textile fabrics with distinct dye natures (reactive, acid, and natural (tea leaves extract) dyes). The dye concentration from 0.5 to 2 % has been demonstrated. Polycation treatment increased colour strength, fastness properties and dye-fixing of investigated textile materials (cotton, wool, and cotton/wool fabrics). The FTIR spectra showed that polycations adhered well to the surface of cotton and wool fibres. This easy and eco-friendly process might be a feasible alternative to the classic printing method.

3. Conflict of interest

The authors declare that there is no conflict of interest

4. Acknowledgements

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