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Functionalizaton of Linen Fabric using Poly (N-vinyl-2-pyrrolidone)

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TO IMPROVE the resiliency as well as to functionalize linen fabric to resist *S. aureus* and *E. coli* bacteria and block UV-B radiation, PVP was incorporated into easy care finishing formulations to chelate the TiO_2 nano-particles and Cupper sulphate as a catalyst. Factors affecting the crosslinking process were studied. The obtained results indicate that:

- the easy care finishing of linen fabric using DMDHEU, 50-75 g/l, as a crosslinker enhances the nitrogen content, resiliency and yellowness along with decreasing the tensile strength as well as the wettability of the treated fabrics,
- incorporation of 4% PVP in the easy care finishing formulation of linen fabrics in presence of 50 g/l DMDHEU, enhances the nitrogen content, tensile strength and yellowness along with a decrease in the resiliency as well as the wettability of the treated fabrics,
- esterification of linen fabrics with CA or BTCA in presence of 4% PVP is accompanied by an enhancement in the nitrogen content as well as tensile strength along with a decrease in the resiliency, wettability and yellowness of the treated fabrics,
- incorporation of $CuSO_4$ as a catalyst or TiO_2 nano particles in the DMDHEU finishing baths in the presence of PVP enhances the extents of the antibacterial as well as the UV blocking properties of the treated fabrics,
- the functionalized linen fabric samples are durable up to 10 washing cycles with little drops in their functionalities.

Flax is used to produce woven and knitted fabrics with exceptional health, hygienic and aesthetic qualities. The principal physical characteristics of flax which are related to its application as textiles are rapid absorption and de-sorption of moisture, good conducting of heat leading to the cool handling and un-collection for the electrostatic charges, that is to say linen is beneficial for the human health as it provides comfort [1,2].

Besides, the high crystallinity of the cellulosic component of the flax fiber results in high creasability of linen fabrics, low extensibility of flax yarns, high tenacity of fibers and yarns, relatively poor abrasion resistance of linen fabrics, poor resistance to flexing and poor resistance to microorganisms after bleaching. Recently, certain nano-scale metals (Ag and Cu) and nano-metal oxides (TiO_2, or ZrO) are used on various textiles coatings to impart antibacterial properties and/or UV-protection [(3)].

On the other hand, Poly N-vinyl-2-pyrrolidone (PVP) is a synthetic, nontoxic, water-soluble polymer commonly used in a wide range of applications including several pharmaceutical applications. PVP polymers are film formers, protective colloids and suspending agents, dye-receptive agents, binders, stabilizers, detoxicants, and complexing agents [(4)].

The present study aims to improve the performance properties as well as to functionalize linen fabric to resist *S. aureus* and *E. coli* bacteria and block the harmful UV radiation via incorporation of PVP as complexing agent for the TiO_2 nano-particles and Cupper sulphate as a catalyst during the easy care finishing of these fabrics.

Experimental

Materials

Fabrics

The fabric used throughout this work was 100% linen fabric. Its specifications are shown in Table 1.

TABLE 1. Specifications of the experimental fabrics.

Property	**Specification**
Fabric Structure	Plain 1/1
Weight/area (g/m^2)	367
Thickness (mm)	0.6
Yellowness Index	29.45

Chemicals

Poly (N-vinyl-2-pyrrolidone) (PVP) of molecular weights 10000 and 40000 Dalton (Sigma – Aldrich), titanium tetraisopropoxide (Merck) was used. GL-300®, aqueous solution of dimethyloldihydroxyethylene urea (DMDHEU), Textchem Co, Egypt was used. Egyptol®, non-ionic wetting agent, supplied by the Egyptian Company for Starch and Yeast and Detergents, Egypt, was used. Sodium hydroxide, sodium silicate, sodium sulphate, acetic acid, sodium carbonate, boric acid, hydrochloric acid, sulphuric acid, ammonium chloride, copper sulphate, potassium sulphate, sodium hypophosphite monohydrate (SHP), citric acid (CA), 1,2,3,4-butanetetracarboxylic acid (BTCA),and hydrogen peroxide (35%) were of laboratory grade chemicals.

Methods

TiO_2 sol–gel preparation

The sol was prepared by hydrolysis and condensation of 5% titanium tetraisopropoxide in aqueous acidic medium containing 5% glacial acetic acid and 1% nitric acid. The mixtures were heated at 60 ^{O}C under vigorous stirring for 16 hr [5].

Fabric treatments

Scouring and bleaching of linen fabrics

The grey linen fabric was mill scoured by introducing the fabric into an aqueous bath containing 40 g/l sodium hydroxide and 5g/l Egyptol® with adjusting the material-to-liquor ratio at 1:20 then the temperature was raised to 95°C for 30 min. The fabric was then washed several times with boiling water and finally with cold water. After scouring, the fabric was bleached using the following recipe: H_2O_2, 10 g/l; sodium silicate, 4 g/l; NaOH, 2 g/l; Egyptol®, 2 g/l; organic stabilizer, 2 g/l; $MgSO_4$, 2 g/l, at 95 ^{O}C for 45 min. The fabric was washed several times with boiling water then with cold water and finally squeezed and dried at ambient conditions.

Easy care finishing of linen fabrics

The finishing treatment was performed by padding linen fabric strips (30X30 cm) twice, at wet pick-up 85%, in finish formulations containing different concentrations of PVP alone, PVP/DMDHEU (using ammonium chloride, magnesium chloride or copper sulphate as catalysts), PVP/CA, or PVP/BTCA (using SHP as a catalyst) to enhance linen fabrics performance properties. Another PVP/DMDHEU finishing bathes containing ammonium chloride as a catalyst as well as titanium dioxide nano-particles of specific concentrations was employed to enhance the antibacterial as well as UV protection properties of linen fabrics. The padded fabrics were dried at 85 ^{o}C for 5 min followed by curing in Wenner Mathis AGCH-8155 oven at specific temperature and intervals of time. The finished fabrics were then washed under occasional stirring (at 50 ^{o}C for 15 min), thoroughly rinsed and finally dried for testing.

Post treating of PVP crosslinked fabrics with iodine

The PVP containing crosslinked linen fabrics were post treated with 5% iodine solution, in absolute ethanol, at 40 °C for 5 hr and then thoroughly rinsed with n-heptane to remove the excess iodine followed by drying in air at room temperature.

Testing and analysis

The nitrogen content was determined according to Kjeldhal method [6]. The dry wrinkle recovery angle was determined according to ASTM method D-1296-98[7]. The tensile strength of the finished fabric sample was tested in the warp direction according to ASTM procedure D-2256-98 [8]. The wettability test was carried out according to AATCC Test Method 39-1980 [9]. The yellowness index was evaluated by using Color-Eye® 3100 spectrophotometer supplied by SDL Inter, England, according to the Standard Test Method ASTM E313 [10].

UPF values were calculated according to the Australian/New Zealand standard (AS/NZS 4399-1996) with a UV-Shimadzu 3101 PC spectrophotometer. According to the Australian classification scheme, fabrics can be rated as providing good protection, very good protection, and excellent protection if their UPF values are 15–24, 25–39, and greater than 40, respectively [11].

The antimicrobial activity of control and finished linen fabrics was performed according to the disc diffusion method, AATCC Test Method 147-1988 [12]. Nutrient agar was inoculated with 0.1 ml of an appropriate dilution of the tested culture. Linen fabric samples (1 cm diameter) were placed on the surface of the inoculated plates. The plates were incubated at the 37 ^{O}C for 24 hr and the diameter of inhibition zone (IZ), in mm, including the disc diameter was measured for each treated sample. The antibacterial activities of the untreated blank as well as finished fabrics were tested against the following bacteria:
(1) Gram-positive bacteria: *Staphylococcus Aureus (SA)*.
(2) Gram-negative bacteria: *Escherichia Coli (EC)*.

Durability to wash was assessed by subjecting the fabric to 1, 10 and 25 laundering cycles. Each laundering cycle consists of washing (10 min at 50 ^{O}C using 2 g/L nonionic surfactant followed by rinsing and air drying at ambient conditions.

Results and Discussion

To enhance the performance and functional properties of linen fabric, TiO_2 nano-particles were incorporated along with PVP, as a complexing agent for these nano-particles, in the easy care finishing formulations of these fabrics. Factors affecting fixation of PVP as well as such nano-particles onto linen fabric matrices were studied and the results obtained accompanied with appropriate discussion follow.

Factors affecting performance and functional properties of linen fabric via PVP

PVP concentration

For a given set of PVP fixation conditions, Table 2 demonstrates that increasing the PVP concentration up to 6% brings about an enhancement in the nitrogen content, tensile strength and yellowness indices along with a reduction in both the resiliency and wettability of the treated fabrics. This may be a direct consequence of the further fixation of PVP onto the fabric structure [13].

PVP molecular weight

Table 3 shows the effect of PVP molecular weight on some performance properties of treated linen fabric. The data in Table 3 signify that increasing the molecular weight of PVP, up to 40,000 Dalton, reduces the nitrogen content, resiliency and yellowness, along with improvement of the tensile strength and wettability of the finished fabrics. This is a direct consequence of increasing the viscosity of the PVP finishing bath that may lead to coat the fabric surface with PVP film which is swept away by washing [13,14].

TABLE 2. Effect of PVP concentration on some performance properties of treated linen fabric.

PVP (%)	% N	WRA (w +f)°	TS (Kg)	YI	W (S)
Untreated	0.0601	86	83.2	17.02	1.17
2	0.3141	142	71.1	17.97	1.39
4	0.3852	128	72.9	18.64	1.73
6	0.3939	117	73.6	18.78	1.95

[DMDHEU], 50 g/l; [NH_4cl], 5 g/l; PVP molecular weight, 10000 Dalton; wet pick up, 100 %; drying, 85 ^oC/5 min; curing, 160 ^oC/3 min.

TABLE 3. Effect of PVP molecular weight on some performance properties of treated linen fabrics.

PVP MW (Dalton)	% N	WRA (w +f)°	TS (Kg)	YI	W (S)
Untreated	0.0601	86	83.2	17.02	1.17
10000	0.3852	128	72.9	18.64	1.73
40000	0.3412	124	74.2	18.04	1.61

[DMDHEU], 50 g/l; [NH_4cl], 5 g/l; [PVP], 4%; wet pick up, 100 %; drying, 85 ^oC/5 min; curing, 160 ^oC/3 min.

DMDHEU concentration

Table 4 shows the performance properties of the linen fabric treated with finishing bathes containing different concentrations of DMDHEU, 50-75 g/l, in absence or presence of 4% PVP. It is clear that increasing DMDHEU concentration from 50 to 75 g/l, in absence of PVP, is accompanied by increasing the nitrogen content, resiliency and yellowness of the finished fabrics along with a reduction in tensile strength and wettability of the treated fabrics. This could be associated with increasing the extent of crosslinking and the subsequent increasing in the molecular degradation of cellulose structure [15]. Furthermore, incorporation the PVP in the finishing bath enhances the nitrogen content, tensile strength and yellowness along with decreasing the resiliency as well as the wettability of the treated fabrics. This can be associated with the accompanied increase in the finishing bath viscosity, which hinders the diffusion of DMDHEU inside the fabric structure as well as deposition of PVP onto the cellulose structure[15].

TABLE 4. Performance properties of linen fabric treated with different concentrations of DMDHEU in presence of PVP.

DMDHEU (g/l)	PVP (%)	% N	WRA (w +f)°	TS (Kg)	W (S)	YI
Untreated	**-**	0.0601	86	83.2	17.02	1.17
50	**-**	0.3059	149	70.5	1.65	18.32
50	**4**	0.3852	128	72.9	1.73	18.44
75	**-**	0.4786	171	62.7	1.80	18.94
75	**4**	0.5897	157	66.2	1.89	19.36

PVP molecular weight, 10000 Dalton; [NH_4cl], 0.1(based on DMDHEU concentration); wet pick up, 100 %; drying, 85 ^{o}C/5 min; curing, 160 ^{o}C/3 min.

Easy care finishing with polycarboxylic acids

Table 5 shows the performance properties of linen fabric crosslinked with CA or BTCA alone or in presence of PVP. Table 5 signifies that: i) estrification of linen fabrics with CA or BTCA in absence of PVP enhances the resiliency, wettability and yellowness along with a decrease in tensile strength of the treated samples, which can be attributed to esterification of the hydroxyl groups of linen cellulose with the carboxyl groups of the CA or BTCA via an anhydride intermediate mechanism as well as the formation of unsaturated acids bound to the surface of the finished fabrics [(13,16,17)] and ii) estrification of linen fabric with CA or BTCA in presence of PVP is accompanied with an enhancement in the nitrogen content as well as tensile strength along with a decrease in the resiliency, wettability and yellowness of the treated fabrics. This could be related to the decrease in the extent of ester crosslinking of the cellulosic hydroxyl groups via increasing the viscosity of the finishing bath thereby hindering the diffusion and penetration of CA or BTCA within the fabric structure and hence the altering in the values of the aforementioned properties [(16)] and iii) in absence or presence of PVP, BTCA improves the nitrogen content (only in presence of PVP), resiliency, wettability and yellowness of the treated fabrics compared with the fabrics treated with CA. The opposite holds true for tensile strength reflecting the differences between these crosslinkers in their reactivity, activation energy, functionality, structure, thermal stability level, extent of esterification, and number and length of crosslinks [(18)].

TABLE 5. Performance properties of linen fabric crosslinked with different types of polycarboxylic acids in the presence of PVP.

Treatment bath	% N	WRA (w +f)°	TS (Kg)	W (S)	YI
CA	-	149	45.7	1.14	20.23
CA + PVP	0.1322	138	48.6	1.21	19.71
BTCA	-	164	41.9	1.05	19.41
BTCA + PVP	0.1401	151	45.5	1.11	18.75

[PVP], 4%; PVP molecular weight, 10000; [poly carboxylic acid], 8%; CA/SHP molar ratio, 1; wet pick up, 100 %; drying, 85 ^{o}C/5 min; curing, 180 ^{o}C/90 sec. Control, sample treated only with 4% PVP.

Multi-functionalization of linen fabric using catalysts salts or nano particles

To multi-functionalize linen fabric, magnesium chloride or copper sulphate was utilized as catalysts in finishing baths containing DMDHEU or DMDHEU/PVP. Moreover, the sol of TiO_2 nano particles was introduced as additive in finishing baths containing DMDHEU or DMDHEU/PVP in presence of ammonium chloride as catalyst. The antimicrobial as well as the UV blocking properties of the treated fabrics are illustrated in Table 6. For a given set of finishing conditions, it is clear that:

i) among the aforementioned used catalysts, $CuSO_4$ imparts antimicrobial properties to the finished sample as well as UV-protection properties and these properties are enhanced remarkably in presence of PVP, most probably due to the chelation of the Cu^{2+} cations by PVP,

ii) the extent of the improvement in these properties follows the descending order: $Cu^{2+} > Mg^{2+}$,

iii) incorporation of TiO_2 nano particles in the DMDHEU finishing bath, in absence of PVP, enhances the antibacterial and UV blocking properties of the treated fabrics. Incorporation of PVP in the finishing bath enhances the extents of the aforementioned properties which can be explained by the ability of PVP to bind these nano particles via coordination bonds,

iv) the differences in the magnitudes of the aforementioned functional properties of treated fabrics are governed by the differences between the aforementioned cations or nano particles in the molecular weight, particle size, location, fixation and extent of distribution, and ability to bind to specific sites in the DNA[19-23] in the bacterial cells thereby inactivating and killing bacteria as well as in UV-blocking and absorbing capacity [24-27] and

v) the inactivation efficiency of *E. coli* (G -Ve bacteria) was lower than that of *S. aureus* (G +Ve bacteria), regardless of the finishing regime.

TABLE 6. Effect of catalyst or nano particle type on the functional properties of the treated linen fabric.

Treatment bath	Salt or nano particle type	UPF	IZ (mm)	
			SA	***EC***
Untreated	**-**	14	-	-
DMDHEU	**$MgCl_2$**	16	-	-
	$CuSO_4$	31	11	8
	Nano TiO_2	27	19	16
DMDHEU + PVP	**$MgCl_2$**	17	-	-
	$CuSO_4$	45	14	11
	Nano TiO_2	54	22	20

[PVP], 4%; PVP molecular weight, 10000; [DMDHEU], 50 g/l; [NH_4Cl, $MgCl_2$ or $CuSO_4$], 5 g/l; [nano TiO_2], 0.17 %; wet pick up, 100 %; drying, 85 ^oC/5 min; curing, 160 ^oC/3 min.

Durability to wash

Table 7 shows the effect of repeated washing cycles on the UPF and antibacterial activities of copper cations as well as TiO_2 nano particles loaded finished linen fabric samples. It is obvious that the antibacterial and UV protection properties of the aforementioned finished samples did not change after one washing cycle. Moreover, the repeated laundering, *i.e.* 10 cycles, results in a slight reduction in the imparted antibacterial and UV protection properties of the treated samples reflecting the strong interaction and fixation of the aforementioned metal cations or nano particles onto the finish/fabrics matrices.

TABLE 7. Effect of repeated washing cycles on the functional properties of finished linen fabric.

Treatment bath	Salt or nano particle type	UPF		IZ (mm)			
				SA	*SA*	*EC*	*EC*
		1 Cycle	10 Cycles	1 Cycle	10 Cycles	1 Cycle	10 Cycles
DMDHEU	$CuSO_4$	31	22	11	6	8	1
	Nano TiO_2	26	19	19	12	16	8
DMDHEU + PVP	$CuSO_4$	45	38	14	8	11	4
	Nano TiO_2	54	49	22	17	20	13

[PVP], 4%; PVP molecular weight, 10000; [DMDHEU], 50 g/l; [NH_4Cl, $MgCl_2$ or $CuSO_4$], 5 g/l; [nano TiO_2], 0.17 %; wet pick up, 100 %; drying, 85 ^{o}C/5 min; curing, 160 ^{o}C/3 min.

Characterization of finished linen fabric

TEM and EDX analysis

Figure 1(a) shows the TEM image of TiO_2-NPs where these particles are less than 10 nm in size and are relatively homogenous. Figures 1(b) and (c) show the SEM images of untreated and TiO_2-NPs loaded linen fabrics, respectively. Figures 1(d) and (e) show the EDX of Cu^{+2} and TiO_2-NPs loaded linen fabrics, respectively, confirming the presence of Cu^{+2} content of 0.93 and Ti- content of 1.19% (w/w) onto the treated fabrics.

Conclusions

To enhance the resiliency as well as impart functional properties to linen fabric, PVP, as a chelating agent, was incorporated in easy care finishing formulations in presence of TiO_2 nano-particles and Cupper sulphate as a catalyst. The proper conditions affecting the crosslinking process were studied. The obtained results indicate that:

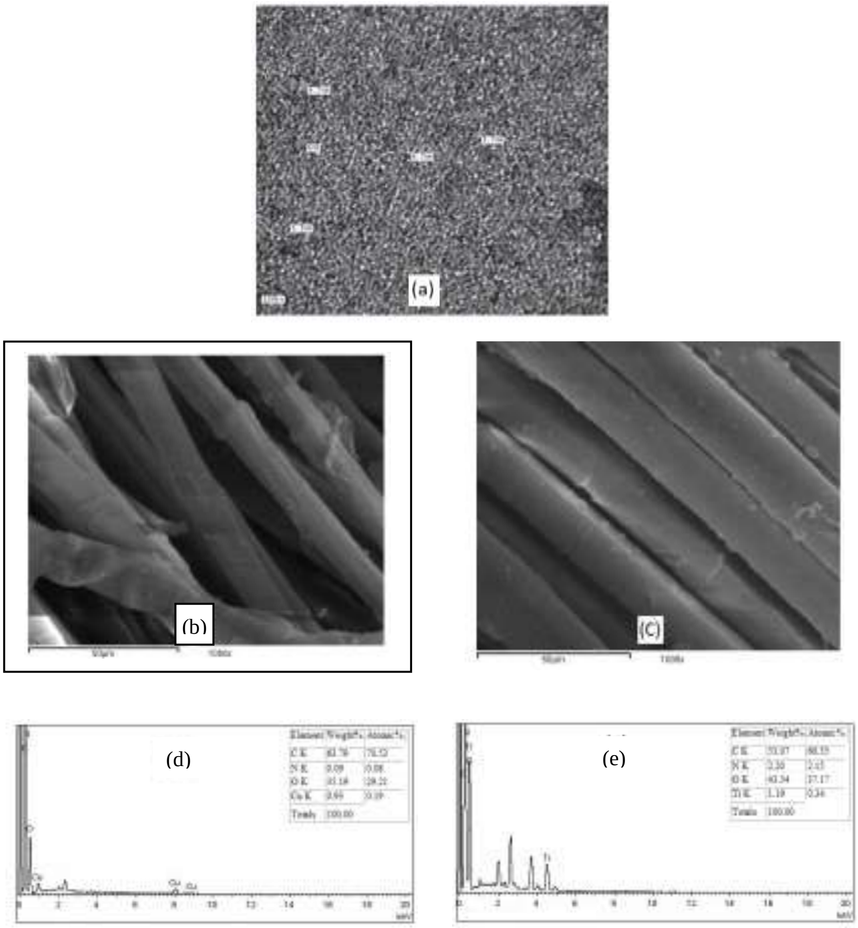

Fig. 1. (a). TEM image of TiO_2-NPs, (b) SEM of untreated linen fabric, (c) SEM of TiO_2- loaded linen fabric, (d) EDX image of Cu^{+2} loaded linen fabric and (e) EDX image of TiO_2-NPs loaded linen fabric.

ii) incorporation of 4% PVP in the above aforementioned easy care finishing formulations, enhances the nitrogen content, tensile strength and yellowness along with decreasing the resiliency as well as the wettability of the treated fabrics,

iii) estrification of linen fabric with CA or BTCA in presence of 4% PVP is accompanied by an enhancement in the nitrogen content as well as tensile strength along with a decrease in the resiliency, wettability and yellowness of the treated fabrics,

i) finishing linen fabric in presence of DMDHEU, 50-75 g/l, as a crosslinker, enhances the nitrogen content, resiliency and yellowness along with decreasing the tensile strength as well as the wettability of the treated fabrics,

iv) incorporation of 4% PVP in easy care finishing formulations using DMDHEU as a crosslinler and $CuSO_4$ as a catalyst or TiO_2 nano particles as a bio-additive and NH_4Cl as a catalyst, enhances the extents of the antibacterial as well as the UV blocking properties of the treated fabrics,

v) the aforementioned finished samples are durable up to 10 washing cycles with little drops in their extents.

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توظيف أقمشه الكتان باستخدام عديد فينيل البيروليدون

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لتعزيز خواص أقمشه الكتان ضد الكرمشه و اكسابها خواص مضاده للبكتريا و الأشعه فوق البنفسجيه تم معالجة هذه الأقمشة فى حمامات تجهيز مختلفه تحتوى على عديد فينيل البيروليدون و جزيئات ثانى اكسيد التيتانيوم النانونيه أو كبريتات النحاس كعامل حفاز فى ظروف تفاعل مختلفه و قد أدت التجارب العمليه الى النتائج الأتيه:

1- يؤدى امرار أقمشه الكتان فى حمامات تجهيز تحتوى على تركيزات مختلفه من الرابط العرضى DMDHEU (50-75 جم/لتر) إلى زيادة فى المحتوى النيتروجينى و زوايا الانفراج من التجعد و معامل الاصفرار للأقمشة المجهزة مصحوبا ذلك بنقص فى قوه شد و إبتلالية هذه الأقمشة.

2- تؤدى معاملة أقمشه الكتان فى حمامات تجهيز تحتوى على تركيزات مختلفه من الرابط العرضى DMDHEU (50-75 جم/لتر) و عديد فينيل البيروليدون بتركيز 4% الى زيادة المحتوى النيتروجينى و قوه شد و معامل الاصفرار للأقمشة المجهزة مصحوبا ذلك بنقص فى زوايا الانفراج من التجعد و إبتلالية هذه الأقمشة.

3- تؤدى معاملة أقمشه الكتان فى حمامات تجهيز تحتوى على تركيزات مختلفه من الرابط العرضى حمض الستريك أو حمض البيوتان رباعى الكربوكسيل وعديد فينيل البيروليدون بتركيز 4% الى زيادة المحتوى النيتروجينى و قوه شد و معامل الاصفرار للأقمشة المجهزة مصحوبا ذلك بنقص فى زوايا الانفراج من التجعد و إبتلالية هذه الأقمشة.

4- تؤدى معاملة أقمشه الكتان فى حمامات تجهيز تحتوى على الرابط العرضى DMDHEU و عديد فينيل البيروليدون بتركيز 4% و جزيئات ثانى اكسيد التيتانيوم النانونيه أو كبريتات النحاس كعامل حفاز الى زيادة خواص الأقمشة المقاومه للبكتريا و الأشعه فوق البنفسجيه.

تثبت خواص الأقمشة المقاومه للبكتريا و الأشعه فوق البنفسجيه بالغسيل المتكرر حتى 10 دورة غسيل.