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Effect of treatment of Wool Fabrics with Pentaeryltheritol on its dyeability and its clothing

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Abstract: Wool fabric was treated with pentaerythritol solution. Then pretreated fabric was dyed with acid dyes. The possibility of reducing the temperature of conventional wool dyeing with an acid dye using ultrasonic radiation was studied in order to reach exhaustion values comparable to those obtained with the conventional dyeing, obtaining dyed samples of good quality. The colour intensity of dyed fabrics using ultrasonic radiation was determined as well as fastness properties. Some properties of untreated and treated wool fabrics such as moisture regain %, roughness, pilling, tensile strength and elongation % were evaluated. The bending stiffness, bending stiffness seam fabrics and sewability of untreated and treated wool fabrics were assessed. Elemental analysis, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) of untreated and treated wool fabrics were performed. This study was determined also, to optimize the effect of treatment with pentaerythritol on wool fabrics in garment manufacturing.
Key Words: Wool fabrics, pentaerythritol, ultrasonic dyeing, fastness properties, sewability, garment appearance.

1. Introduction

Chemical and physical treatments were carried out to enhance the shrinkage and felt- resisting of wool fibres, such as treatments with glycerol polyglycidyl ether (GPE), polyamide epichlorohydrin and dichlorodicyanuric acid. [1-3] Wool fibres were treated with enzymes –based process to obtain felt- resist fabrics. Enzymes treatments were carried out on the surface of the fibres with little loss in both weight and tensile strength. [4, 5] Pentaerythritol used as cross linking agents to improve the pilling and antistatic charge. [6, 7] Ultrasound is sound of a frequency that is above the threshold of human hearing. [8, 9]The use of ultrasound technology has established an important place in different industrial processes such as the medical field, and has started to revolutionize environmental protection. [10] The use of ultrasound energy for dyeing textiles as a cleaner production is very well known in the literature**.** [11, 12] Ultrasound can enhance a wide variety of chemical and physical processes, mainly due to the cavitation effect in liquid medium (that is the growth and explosive collapse of microscopic bubbles, which causes a large increase in the pressure and temperature) that enhances the rate of dyeing process. [13]

The construction and the sewability of garment as well as the effect of some treatments on performance properties of garment were studied. The fabric mechanical properties tests were measured using FAST tests. [14] In apparel industry, the sewing process is one of the critical processes in the determination of productivity and the quality of the finished garment. Fabrics with very high bending rigidity values may lead to sewing and handling problems as they are too stiff to be manipulated and controlled. [15] Resistance to bending or flexural rigidity is called stiffness in textile test methods. The longer the bending length, the stiffer is the fabric. [16]

Bending stiffness is a property of fabrics that can influence fabric drape and therefore the aesthetic characteristics of garments, and clothing comfort as well. The fabric stiffness is a feature having important effect on converting flat fabric into 3-D garment, influencing aesthetic appearance of the garment. Additionally, fabric stiffness affects fabric hand. Sewing needle penetration forces and fabric deformation during sewing are effective factors for seam performance. [16] The sewing needle penetration force is one of the most significant technical parameters in the sewing process affected by various factors such as: type, number of layers of the sewing material, and needle size. The penetration force of a sewing needle is mostly based on the friction occurring between the sewing work pieces and sewing needle, and the highest penetration force of the sewing needle occurs at the moment when the sewing needle penetrates the sewing material, which should be taken into consideration. [17, 18]

The aim of this study carried out to find the effect of treatments with pentaerythritol on many physical and mechanical properties. Using ultrasonic radiation was studied in order to reach exhaustion values comparable to those obtained with the conventional dyeing with acid dyes and performance properties of garment. Also, this study, showed the effect of treatment on both bending and sewability properties of woolen garment. The obtained results will surely help to identify the design criteria for clothing so as to produce high quality garments.

2. Experimental

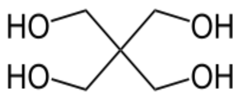
2.1. Materials

2.1.1. Fabrics

Wool fabrics were supplied by Misr El Mahalla Co., for Spinning and Weaving, Egypt. The fabrics were soaped with (2 g/L) nonionic detergent solution (Hostapal CV., from Clariant, Egypt) with a liquor ratio 1 : 25, at 45ºC, for 30 min, then rinsed twice with cold tap water, and dried at room temperature.

2.1.2. Chemical

Pentaerythritol 98%, is an organic compound with the formula (2, 2 – Bis (hydroxymethyl -1, 3 – propanediol) supplied from (A Johnson Matthey Company – Germany). Acetic acid are of laboratory grade.

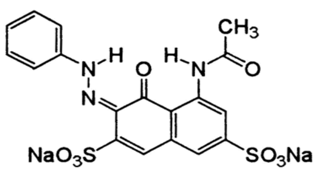
$C_5H_{12}O_4$ 

(2, 2 – Bis (hydroxymethyl -1, 3 –propanediol)

2.1.3. Dye

The colour index and chemical structures of the acid dyes used in this study is C.I. Acid dye1.

(Fast Red EG)



C.I. Acid dye 1

2.2. Treatment

The Wool fabric was treated in solutions of (3% - 10% w/v) pentaerythritol with a liquor ratio 1: 25 for 1hr. at 85°C. One series of the samples were cured at 150ºC for 10 min. The samples were rinsed with warm water then rinsed with tap water, and finally air dried.

2.3. Dyeing

Dyeing was carried out by two methods, namely conventional and ultrasonic- assisted dyeing.

2.3.1. Conventional dyeing

Dyeing process was carried out at 60ºC, L.R 1:50. Dyeing bath contains 2% (w.o.f) acid dye, pH was adjusted from 4 to 4.5 then the fabric was immersed in dyeing bath and the temperature was raised to 90ºC in water bath for 60 min. At the end of dyeing, the dyed samples were rinsed with tap water and washing in a bath containing 2g/L non-ionic detergent (Triton x-100) at 45 °C for 30 minutes The fabrics were then rinsed and air dried.

2.3.2. Ultrasonic Dyeing

Untreated and treated wool fabrics were dyed with (2% (o. w. f) acid dyes) at liquor ratio 1:50, for 1h., pH 4 - 4.5 using ultrasonic radiation bath at different dyeing temperature 60°C, 70°C, 80°C and power levels at (300,400,500 W). The dyed samples were then thoroughly rinsed with cold water and washed for 30 min., in bath containing 2g/l of nonionic detergent (Triton x-100) at 45°C. Finally, the fabrics were rinsed with warm and cold water and air-dried. (**Ultrasound equipment)** A CREST Ultrasonic, TRU-SWEEPTM ultrasonic bench top cleaner bath, model 575 D with a capacity 5.75 L, was used. The experimental setup used was composed of an electrical generator at a frequency of 38.5 kHz and power ranging from 135W average/500 W. The output power levels are from 50 up to 500 W, and are supplied by three transducers at the bottom of the tank. Precise digital control of time (0– 90 min), thermostatically controlled heater (ambient to 80 ºC), and power level and degas functions. The internal dimensions of the tank were 11.5 x 6 x6depth and 292 x 152 x 152 mm.

2.4. Measurements

2.4.1. Roughness

Surface roughness of treated and untreated fabrics was measured according to JIS-94 standard using surface roughness tester Model SE 1700∞ (Kosaka Laboratory Ltd. Japan).

2.4.2. Infrared Spectra

Infrared spectra were recorded on FT-IR Nicolet 5 DX Spectrophotometer. The samples were examined as 1.5% KBr pellets.

2.4.3. Moisture Regain %

Measurements of moisture regain of the fabrics are performed according to the standard ASTM method 2654–76.[) 19)] Moisture regain of the samples are calculated according to the following equation:

$$\text{Moisture Regain \%} = \frac{W_1 - W_2}{W_2} \times 100$$

Where W_1: Weight of sample (gm.) after saturation in the standard humidity atmosphere.

W_2: Constant weight (gm.) of dry sample.

2.4.4. Elemental analysis

The amount of carbon, nitrogen, hydrogen, and sulphur, in the untreated and selected treated wool fibers were assessed using Elementary CHNS Analyzer, Model Vario EL III, Germany.

2.4.5. Colour intensity (K/S)

The colour strength of the dyed samples was evaluated by a light reflectance technique using the Perkin–Elmer, UV–Vis Spectrophotometer (Model, Lambda 3B). The relative colour strength (K/S values) was assessed using the Kubelka–Munk equation. [20]

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

Where: R is the reflectance of colored samples and K and S are the absorption and scattering coefficients, respectively. Summation method (Σ (K/S) λ) was used for an equally spaced selection of wavelength in range of 350–750 nm.

2.4.6. Fastness Testing

The colour fastness to washing was determined in accordance to ISO standard methods. The specific standard tests were: ISO 106-CO2 (1993) for wash fastness, ISO 105-X12 (1987 colour fastness to rubbing and ISO 105-EO4 (1989) for fastness to perspiration.

2.4.7. Tensile Strength and Elongation %

The tensile strength and elongation of fabric before and after treatment are evaluated using a Instron Tensile Tester (USA) according to ASTM D 76 Standard Specification for Textile Testing Machines. The average is taken for 10 samples (5x 20 cm).

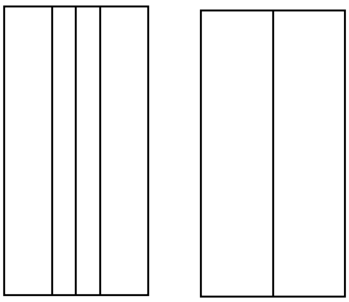
2.4.8. Bending Stiffness test

The samples were stitched using sewing yarn (40/2 Ne ring spun stitches number per unit length 10 per inch)**, needle size 14\90 (as** cloth)**.** The samples were conditioned in the standard testing temperature (20C° ± 2C°) and humidity (65% ± 2 RH) for 24 hour according to (ASTM) standards before conducting any test. [21] Fabrics are tested on bending stiffness according to BS 3356 the samples were tested in warp and weft direction. Bending stiffness, G was calculated according the following equation (1)

$$G = 0.1 \cdot M \cdot C^3 \text{ (mg. cm)} \qquad (1)$$

Where: M - fabric weight (g/m^2),

C - Bending length mean value in warp and weft direction (cm)



(a) (b)

Figure1. Fabric strip with vertical seam, (a) back side of fabric with seam allowance, (b) right side of fabric with a plain seam at the center

2.4.9. Scanning Electron Microscope

The untreated and treated ultrasonic dyed wool fabric were examined using a SEM Model Philips XL 30 with an EDX Unit attached, with accelerating voltage of 30 kV and magnification between 10 and 400.000. All the samples were coated with gold before SEM testing.

2.4.10. Sewability

Using the sewability tester (based on US patent 3979951, 1976), a device used in many studies on the needle penetration force. This equipment simulates a sewing machine by penetrating the tested fabric with an unthreaded needle, at a rate of 100 penetrations per min., with needle count 90, 110. [22]

2.4.11. Pilling test

Pilling tester is used to determine the pilling resistance of all kinds of textile structures. Sample was rubbed against the same material of sample at low pressures and the amount of pilling is compared against standards parameters. Pilling test was carried out according to standards ASTM standard D4970 (pilling), all samples and standard fabrics should be conditioned in the standard atmosphere for testing (20^0C - + 2 and 65% RH - , + 2%). The specimens assed are using the 5 point scale.

3. Results and Discussion

3.1. Tensile strength, elongation % roughness, and pilling resistance

The tensile strength and elongation %, roughness and pilling resistance of treated and untreated wool fabrics with pentaerythritol were illustrated in table 1. The treatment led to limited decrease in both tensile strength, and roughness, this decrease depended on the concentrations of pentaerythritol. This decrease may be attributed to effect of pentaerythritol on fibres which led to disappear the scales of wool as acceptable with results of surface morphology as shown in figures 2. It is observed that the increase in the concentrations of pentaerythritol increase both elongation % and pilling resistance. The treatment led to improvement pilling resistance from 1-2 for untreated fabric to 3-4 for treated fabric with 10 % pentaerythritol.

Table 1: Tensile strength, elongation %, Roughness and pilling resistance of treated and untreated wool fabrics with pentaerythritol

Samples	Tensile strength Kg f/mm^2	Elongation %	Roughness µ	Pilling 1000 (cycle)
Untreated	0.9146	16.6	28.96	1-2
Treated with 3 %	0.956	18.0	28.01	2-3
Treated with 6 %	0.9897	26	27.5	3
Treated with 10 %	0.9543	30.1	25.41	3-4

Condition of treatments: (3- 10) % w/v pentaerythritol, L: R 1: 25 for 1hr. at 85°C,
then cured at 150ºC for 10 min.

3.2. Moisture regain %, Loss in weight % and Colour strength

Moisture regain %, loss in weight % and colour strength of treated and untreated wool fabrics with pentaerythritol were shown in table 2. The results shows that the weight of treated fabrics was limited decrease than untreated one, and more decrease in case of treatment with 10% pentatheratiol. The treatment enhancement the colour strength of dyed fabrics. Also, moisture regain % results of treated fabrics give increase than untreated one. Both colour strength and moisture regain results of treated fabrics depended on the concentrations of pentaerythritol.

Table 2: Moisture regain %, loss in weight % and colour strength of treated and untreated wool fabrics with pentaerythritol

Samples	Moisture regain %	Loss in weight %	K/S value
Untreated	9.7	0	21.65
Treated with 3 %	9.9	1.4	24.65
Treated with 6 %	9.8	6.7	27.8
Treated with 10 %	10.1	8.1	27.9

Condition of treatments: (3- 10) % w/v pentaerythritol L: R 1: 25 for 1hr. at 85 °C, then cured at 150ºC for 10 min.
Dyeing condition: 2% (o. w. f) C.I. Acid Red 1, L: R 1: 50 for 1hr. at 70 °C power level400w, pH 4 - 4.5

3.3. Colour strength and fastness properties

The colour strength of treated and untreated wool fabrics with pentaerythertiol using ultrasonic dyeing method illustrated in Table 3. It can be observed that increasing the temperature of ultrasonic dyeing, increased colour strength values at different power levels. The results indicate also, that as the US power increased the colour strength values were also increased at a higher power levels (300–500W), studying the possibility of reducing the temperature of conventional wool dyeing with an acid dye using ultrasound in order to reach colour strength values comparable to those obtained with the conventional procedure at 90°C and to compare the results. The results of colour strength values of treated fabrics with 3 % w/v pentaerythritol give 24.65 by conventional dyeing but give same result (24.3) at 80°C with (300W) by ultrasonic dyeing method. Also, it was found that colour strength value increased to be 26.4 at 70°C with (400W) by ultrasonic dyeing method. Power ultrasound can enhance a wide variety of chemical and physical processes, mainly due to the phenomenon known as cavitations' in a liquid medium that is the growth and explosive collapse of microscopic bubbles. Sudden and explosive collapse of these bubbles can generate ‘‘hot spots’’, i.e., localized high temperature, high pressure, shock waves and severe shear force capable of breaking chemical bonds. [9] High temperature and pressures resulting from the collapse of the transient cavitation bubbles are responsible for all the observed effects of ultrasound. [11] The ultrasonic dyeing method saved more energy than conventional dyeing method.

Table 3: Colour strength of conventional dyeing and ultrasonic dyeing methods on wool fabrics with pentaerythritol

Samples	K/S of Conventional Dyeing	K/S of Ultrasonic Dyeing								
		60°C			70°C			80°C		
		300w	400w	500w	300w	400w	500w	300w	400w	500w
Untreated	21.65	14.32	17.26	19.29	22.19	23.6	25.9	22.8	25.5	24.8
Treated with 3 %	24.65	18.46	18.9	19.91	22.79	26.4	26.7	24.3	27.4	26.6

Condition of treatments: 3 % w/v pentaerythritol, L: R1: 25 for 1hr. at 85°C, then cured at 150ºC for 10 min.
Dyeing condition: 2% (o. w. f) C.I. Acid Red 1, L: R 1: 50 for 1hr. at (60, 70, 80) °C, (300, 400, 500) W power level, pH 4 - 4.5

The results of these fastness tests are shown in Table 4 describe the normal fastness grades crocking, perspiration, washing and light fastness grades respectively. The results of the show that the treated wool fabric exhibited better fastness test to crocking , perspiration as well as to washing fastness than untreated wool fabric, as the same light fastness is obtained for treated and untreated fabric.

Table 4: Fastness properties of dyed of untreated and treated wool fabric with pentaerythritol with acid dyes

Samples	Fastness properties											
	Washing			Perspiration						Crocking		Light
	W	C	A	Acid W	Acid C	Acid A	Alkali W	Alkali C	Alkali A	Dry	wet	
Untreated	2- 3	2- 3	2- 3	3	2- 3	3- 4	3	3	3	3	4	5
Treated	4	4	4	3-4	4	4	4	3- 4	4	4	4	5

Where: A = alteration, C = staining on cotton, W = staining on wool
Condition of treatments: 3 % w/v pentaerythritol L: R 1: 25 for 1hr. at 85°C, then cured at 150ºC for 10 min.
Dyeing condition: 2% (o. w.f) C.I. Acid Red 1, L: R 1: 50 for 1hr. at 70°C, power level 400w, pH 4 - 4.5

3.4. Elemental analysis

Elemental analysis Carbon, hydrogen, nitrogen, and sulfur of the untreated as well as the treated wool fabrics were summarized in table 5. It can be observed that there is little increase in carbon content where as little decrease in nitrogen content of the treated wool in comparison with the untreated fiber. A decrease in hydrogen and sulfur contents of treated wool were observed than the untreated sample, and more decrease in case treatment with 10% pentatheratiol. This decrease may be attributed to the effect pentatheratiol as on wool surface.

Table 5: Elemental analysis Carbon, hydrogen, nitrogen, and sulfur

Sample	Nitrogen N %	Carbon C %	Sulfur S %	Hydrogen H %
Untreated	14.82	44.07	2.52	4.30
Treated with 3 %	14.54	44.16	2.45	3.88
Treated with 6 %	14.46	44.32	2.37	3.55
Treated with 10 %	14.22	44.49	2.11	3.31

Condition of treatments: (3- 10) % w/v pentaerythritol, L: R 1: 25 for 1hr. at 85°C,
then cured at 150ºC for 10 min.

3.5. Surface morphology

Changes of the surface morphology of wool fibers after treatment with penterythritol were investigated using SEM as shown in figures 2 (a-d). The SEM analysis of surface morphology reveals changes which occur on the surface of wool fibers as a result of modification with penterythritol. The treatment make then cover on the scales of the treated wool fabric, this led to disappear the scales on wool fabrics. The more effect happens with increasing the concentration penterythritol to 10%. This result illustrates improvement in pilling resistance property of treated wool fabrics as compared with untreated one.

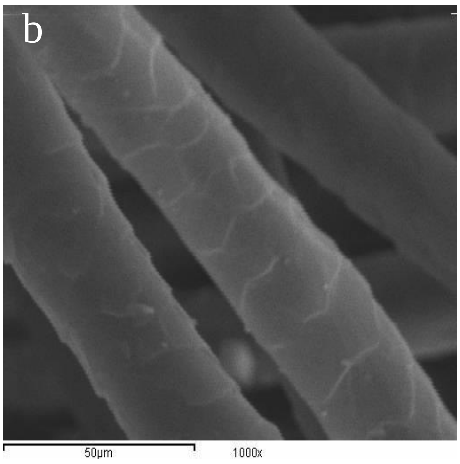
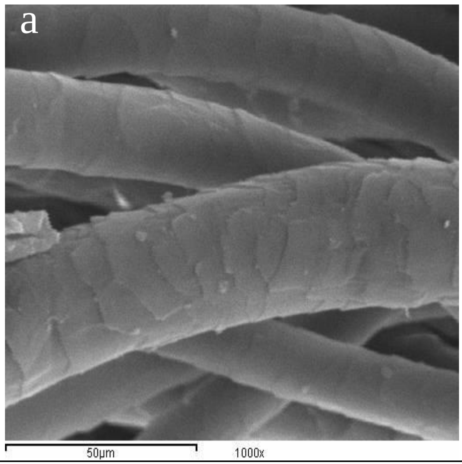
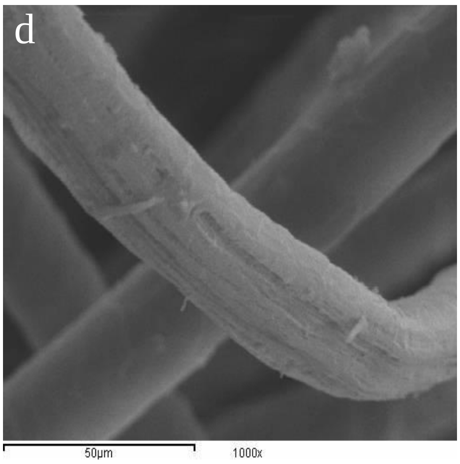
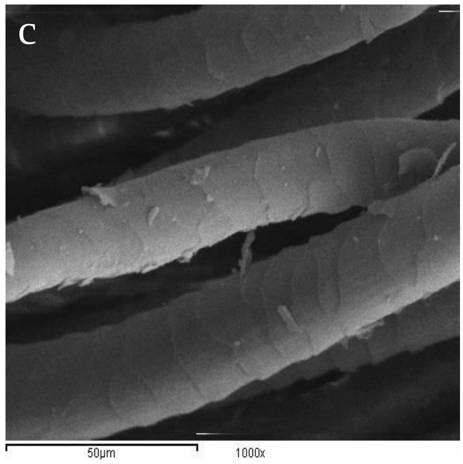
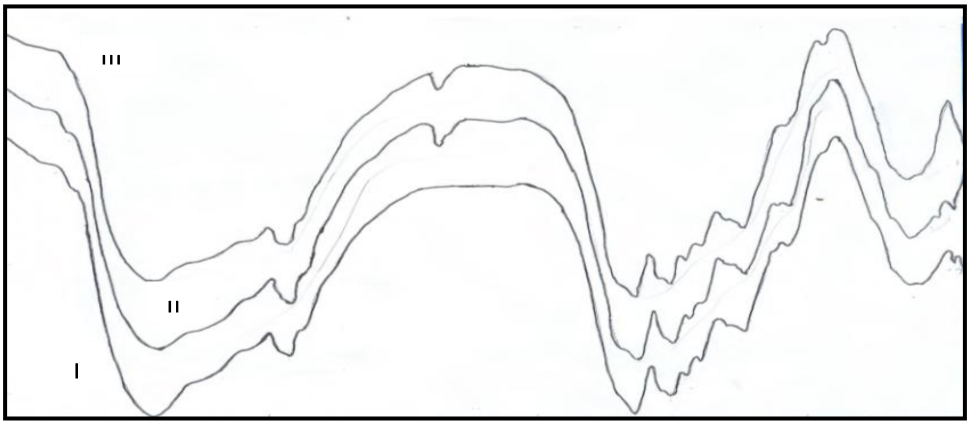
	
Treated wool fabric 3% pentaerythritol	Untreated wool fabric
	
Treated wool fabric 10% pentaerythritol	Treated wool fabric 6% pentaerythritol

Figure 2: Surface morphology of untreated and treated wool fibers with pentaerythritol

3.6. Infrared Spectra

Infrared spectra of the treated and untreated wool fabrics with pentaerythritol are shown in figure 3. Plot I show spectra of the untreated and plots II and III show the spectra of the treated wool fabrics with 3% and 10% pentaerythritol respectively. The treatments gave some changes in the absorption bands between 3650-3200 cm-1 in both plots II and III which may be attributed to free $–NH_2$ valence vibration in amide group.



4000 3000 2000 1000 400

Wave length (cm $^{-1}$)

Figure 3: Infrared spectra of untreated and treated wool fabric with pentaerythrit

3.7. Fabric Stiffness, seam fabric Stiffness, thickness and Sewability

Table 6 illustrated stiffness, stiffness seam fabric, and thickness of untreated and treated wool fabrics. The treatment led to decrease the thickness of fabrics as a result to loss in weight. From this the bending stiffness of treated fabrics were improved as well as stiffness of treated seam fabrics.

Table 6: fabric stiffness, seam fabric stiffness and thickness

Sample	Weight g/m^2	Fabric stiffness	Seam fabric stiffness	Thickness mm.
Untreated	252	1020.807	5093.7468	0.755
Treated with 3 %	238	1005..1949	5827.297	0.859
Treated with 6 %	221	860.21991	5003.3393	0.8995
Treated with10 %	201	822.51129	6375.3115	0.85

Condition of treatments: (3- 10) % w/v pentaerythritol, L: R1: 25 for 1hr. at 85°C, then cured at 150ºC for 10 min.

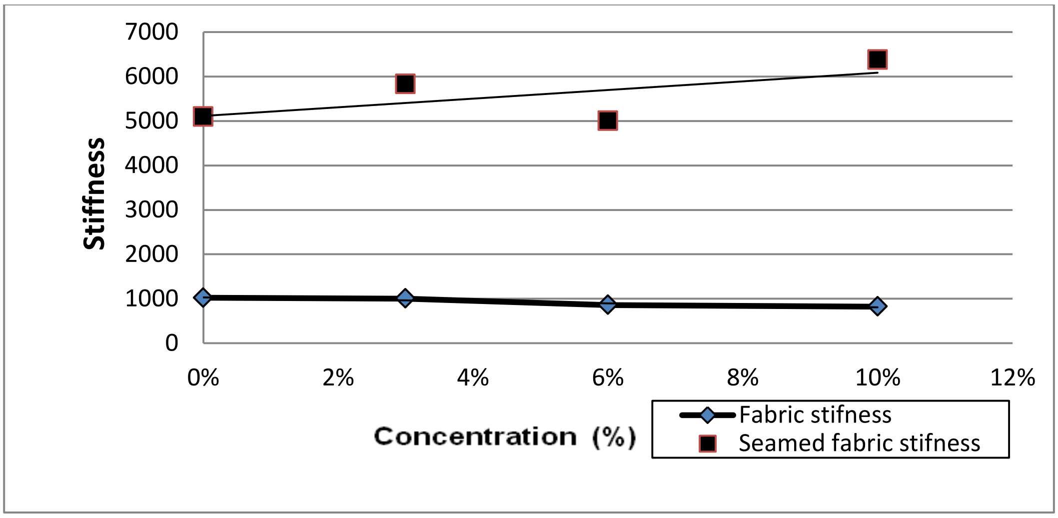


Figure 4: The relation between stiffness and concentration of pentaerythritol

Table 7: Quality Factor

Samples	QF %
Untreated	64.8
Treated with 3 %	71.5
Treated with 6 %	87.7
Treated with 10 %	85.6

Condition of treatments: (3- 10) % w/v pentaerythritol,
L: R 1: 25 for 1hr. at 85°C, then cured at 150ºC for 10 min.

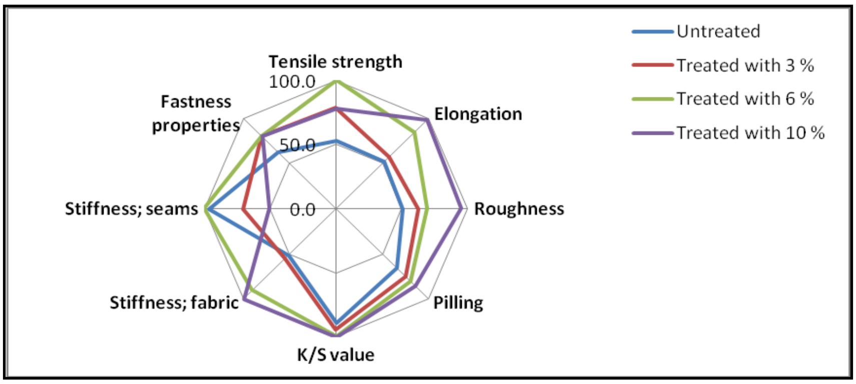


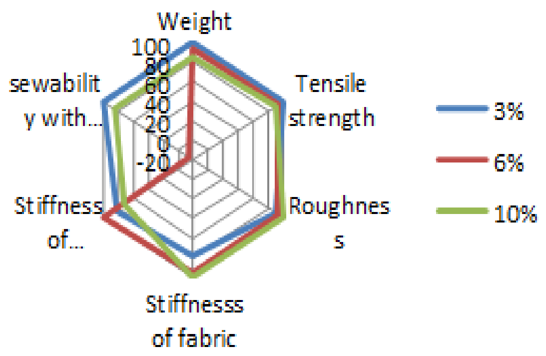
Figure 5: Radar charts of treated wool fabrics

Figure 5 shows radar charts of untreated, treated wool fabrics with pentaerythritol showed that the sample with concentration 6% gave the best results followed by 10% ,sample with concentration 3% and sample untreated, which gave the lowest evaluation. Quality Factor were given 87.7% for treatment with 6% pentaerythritol, 85.6% for treatment with 10% pentaerythritol , 71.5% for treatment with 3% pentaerythritol ,and 64.8% for untreated sample as shown in table 7. The increase in Quality Factor of treatment with 6% pentaerythritol may be attributed to decrease in weight and thickness.

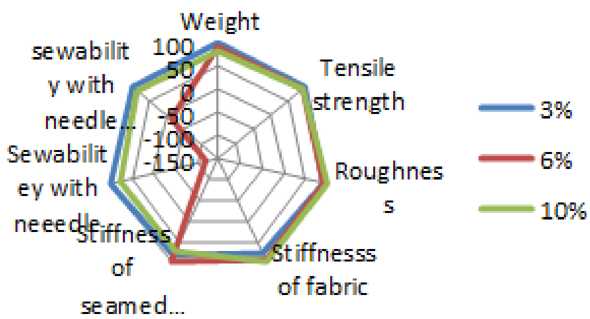
Table 8: Radar area for a, b

Sample	**Radar area for a**	**Radar area for b**
Treated with 3 %	23714.924	21999.646
Treated with 6 %	9253.3561	5223.635
Treated with10 %	20760.85	19951.21

Condition of treatments: (3- 10) % w/v pentaerythritol, L: R 1: 25 for 1hr. at 85°C, then cured at 150ºC for 10 min.



a: Sewability with 1 needle



b: Sewability with 2 needle

Figure 6: Radar charts of treated wool fabrics with sewability

Figure 6 shows radar charts of treated wool fabrics with pentaerythritol and sewing with two needle the first at 90 and the second at 110. Radar charts shows that the sample with concentration 3% is the best in results followed by sample with concentration 10% then sample with concentration 6% which gave the lowest evaluation because of its lower sewability. Radar area were given 23714.924 for treatment with 3 % pentaerythritol, 9253.3561 for treatment with 6% pentaerythritol and 20760.85 for treatment with 10 % pentaerythritol as shown in table 8. The decrease in values of treatment with 10 % pentaerythritol may be attributed to decrease in weight and thickness

4. Conclusion

The treatments with pentaerythritol led to significant enhancement in anti-pilling behavior of treated wool fabric than untreated fabrics as well as elongation %.

The ultrasonic dyeing method saved more energy than conventional dyeing method. It was found that colour strength value increased at 70°C with PL 400W by ultrasonic dyeing methods as compared with conventional dyeing method at 90°C.

The treatments led to decrease in carbon, hydrogen, nitrogen, and sulfur contents of the treated wool fabrics. The treatment make then cover on the scales of the treated wool fabric, this led to disappear the scales on wool fabrics.

The treatment led to decrease the thickness of fabrics as a result to loss in weight. The bending stiffness of treated fabrics were improved as well as stiffness of treated seam fabrics. It was found the treatments with 3 % penterythritol as optimum condition for bending stiffens of wool garment.

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