Statistical study of the effect of metallic mordants on tensile strength of wool

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REZUMAT – ABSTRACT

Studiul statistic al efectului mordantului metallic asupra rezistenței la tracțiune a lânii

În ciuda varietății mari de studii în domeniul efectului diferiților mordanți asupra proprietăților de vopsire și de rezistență a culorii lânii cu coloranți naturali, nu există o investigație aprofundată a efectului mordantului metallic asupra proprietăților de tracțiune a lânii. În acest studiu s-au aplicat cinci tipuri diferite de săruri metalice pe lână: sulfatul de potasiu, de aluminiu, clorura de staniu, dicromatul de potasiu, sulfatul de cupru și sulfatul feros, cu concentrații cuprinse între 1 %owf și 20 %owf. Rezistența la tracțiune a probelor a fost măsurată și a fost utilizat software-ul SPSS pentru a evidenția efectul diferitelor concentrații de mordanți asupra rezistenței la tracțiune a firelor de lână, comparativ cu proba netratată. Rezultatele au arătat că sulfatul de potasiu, de aluminiu și sulfatul feros nu au avut un efect semnificativ statistic asupra rezistenței firelor, în timp ce clorura de staniu a prezentat cel mai mare effect și a redus în mod semnificativ rezistența firelor. Sulfatul de potasiu, de aluminiu și sulfatul feros au redus tenacitatea la maximum 4,2 %owf și respectiv 4,4 %owf, în timp ce proba tratată cu clorură de staniu a fost complet distrusă atunci când s-a aplicat mai mult de 5 %owf din mordant.

Cuvinte-cheie: mordant, tenacitate, lână, formare complex, alaun, SPSS

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Despite the vast variety of studies in the field of effect of different mordants on dyeing and fastness properties of wool with natural dyes, there is no thorough investigation on the effect of metal mordants on tensile properties of wool. In this study, five different metallic salts namely aluminum potassium sulfate, tin chloride, potassium dichromate, copper sulfate, and ferrous sulfate were applied on wool with concentrations ranging from 1 %owf to 20 %owf. The tenacity of the samples was measured and SPSS software was employed to investigate the effect of different concentrations of various mordants on tensile strength of woolen yarn compared with raw sample. The results showed that aluminum potassium sulfate and ferrous sulfate had no statistically significant effect on the tenacity of the yarns while tin chloride showed the highest inverse effect and lowered the yarn strength significantly. Aluminum potassium sulfate and ferrous sulfate reduced the tenacity for maximum of 4.2 %owf and 4.4 %owf respectively while the tin chloride treated sample was completely destroyed when applying higher than 5 %owf of the mordant.

Keywords: mordant, tenacity, wool, complex formation, alum, SPSS

INTRODUCTION

Metal mordants are usually used in combination with various synthetic and natural mordant dyes in order to improve the fastness and depth of shade or obtain different hues when using a single dye [1–2]. Various transition metals can act as mordants and their salts can be applied on wool by three different application routes namely, pre-mordanting, meta-mordanting and after-mordanting depending on whether the mordant is applied before, together with or after the dyeing procedure. However, the use of mordant dyes has declined in recent years, owing to their negative environmental impacts and eco-toxicity [1, 3–4].

Recently, a great tendency to the use of natural products has been arisen specially when speaking about the coloration of textiles. This renewed interest is mainly due to the increased awareness of the environmental and health risks that synthetic dyes produce in the synthesis, processing and application stages [5–6]. Most of the natural dyes possess low affinity towards the textile fibers, therefore high amounts of the dyeing material and prolonged dyeing times are usually needed to dye a textile product using natural dyeing plants satisfactorily. To improve the exhaustion of natural dyes onto textile fibers, different techniques have been employed. Several pretreatments like cationization [7], plasma treatment [6, 8–9], enzyme treatment [10], gamma treatment [11–12], and microwave treatment [13] are examples of techniques which have been studied to overcome this drawback.

However, the most usual way to enhance the dyeing of textile fibers with natural dyes is still mordanting with metal salts. Examples of the most common mordants are the salts of chromium, tin, iron, copper and aluminum and several studies have been published on the optimization of natural dyeing and mordanting of fibers with different mordants [14–19]. When applying mordants on wool fibers, the main action of mordanting is to increases the interaction between the amine groups of protein molecules of wool fibers and hydroxyl and carbonyl groups of dye molecules. Figure 1 shows the mechanism of complex formation between wool protein, aluminum ion, and juglone as a model natural dye molecule [20–21].

Recently the use of binary and ternary metal salt combinations has been reported with the aim of obtaining new shades using annatto and walnut bark as natural dyesand their colorimetric and fastness properties have been studied [22–23]. The main concern in the previous studies have been about the improvement of exhaustion, color strength and fastness properties besides achieving different shades or

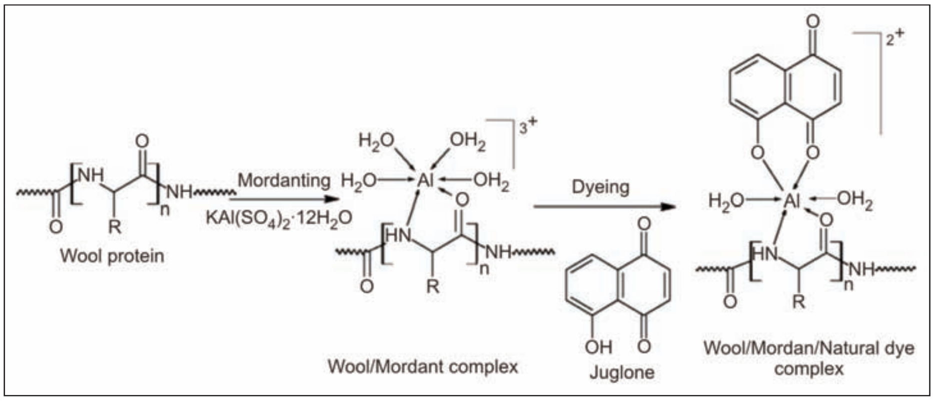

Fig. 1. Mechanism of complex formation between wool, aluminum mordant and juglone dye [20–21]

functional properties when using a specific natural dye [20, 24–30]. However, there is no research published on the effect of different mordants on physical properties of fibers. In this study the effect of mordanting process with several common mordants in various concentrations on tensile strength of wool fibers have been studied and compared with the raw sample.

EXPERIMENTAL WORK

Materials and methods

Woolen yarn (Nm = 400, 2 ply) was purchased from a local spinning mill and used for the experiments after scouring and drying [1% non-ionic detergent (Triton X-100, Sigma-Aldrich, USA), 50 ºC, for 30 min]. All other chemicals used in this study were analytical grade reagents obtained from Merck, Germany.

Mordanting: The mordanting bath was prepared using the required amount (1, 2, 5, 10, 20 %owf) of mordant (aluminum potassium sulfate, tin chloride, potassium dichromate, copper sulfate, and ferrous sulfate) according to the experimental design. The liquor to goods ratio (L:G) was 50:1 and the mordanting was done at boil temperature for 1 hour.

Tensile strength measurement: The tenacity of raw and different mordantedwoolen yarns was measured according to ASTM D 2256 test method. Gauge length was 25 cm and crosshead speed was 30 cm/min. The samples were chosen randomly and the average of five measurements was reported for each sample. To evaluate the difference between the tensile strength of samples mordanted with various amounts of each mordant, the test results were analyzed for significant differences using one way analysis of variance (ANOVA) and the Tukey post hoc test at a 95% level of confidence using SPSS software version 16.0 (IBM, USA).

The hypotheses to be tested were determined as follows:

H_0: There is no significant difference between average tenacity of mordanted yarns with different amounts of mordant.

H_1: There is a significant difference between average tenacity of mordanted yarns mordanted with different amounts of mordant.

Independent samples t-test for equality of means was performed to compare the average tenacity of samples mordanted with various amounts of different mordants with the raw sample. The hypotheses to be tested were determined as follows:

H_0: There is no significant difference between average tenacity of mordanted and raw yarns.

H_1: There is a significant difference between average tenacity of mordanted and raw yarns.

RESULTS AND DISCUSSION

Effect of mordants on tensile strength of yarns

Figures 2–6 show the effects of various amounts of different mordants on the tenacity of the woolen yarn. The mean tenacity of the raw woolen yarn was 8.53 cN/Tex. The highest effect on the tenacity of yarns was observed in the case of stannous chloride mordant specially when using concentrations higher than 2 %owf. $SnCl_2$ is a reducing agent and causes breaking of disulfide bonds which are present between wool protein chains and are sensitive to reducing agents. These covalent bonds are very important for

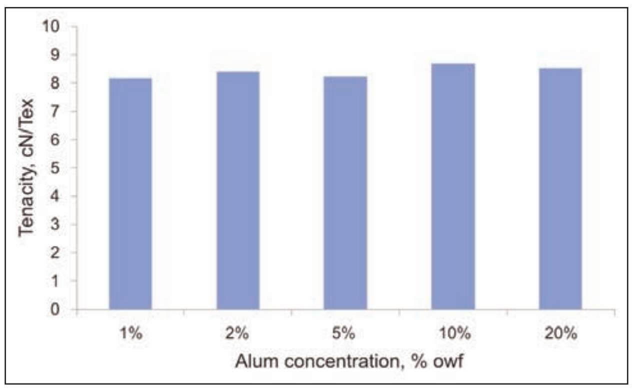

Fig. 2. The effect of concentration of alum on tenacity of woolen yarn

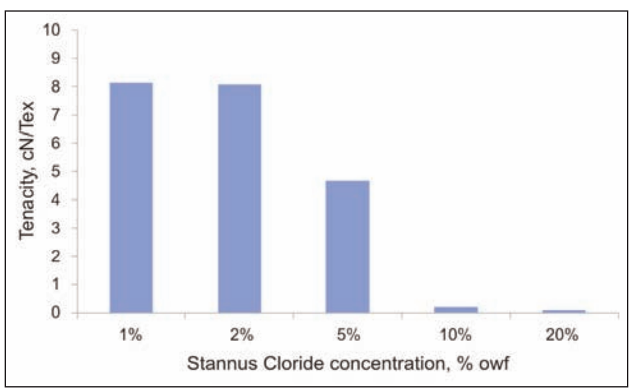

Fig. 3. The effect of concentration of stannous chloride on tenacity of woolen yarn

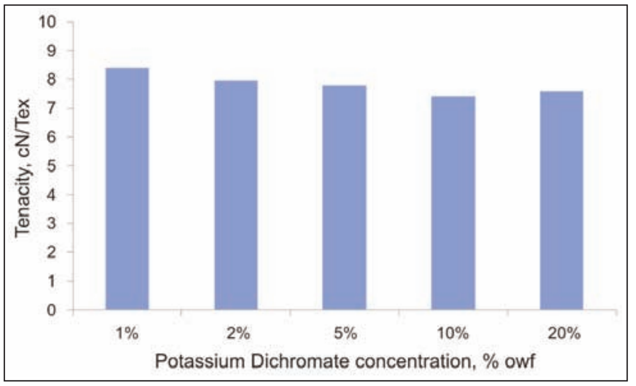

Fig. 4. The effect of concentration of potassium dichromate on tenacity of woolen yarn

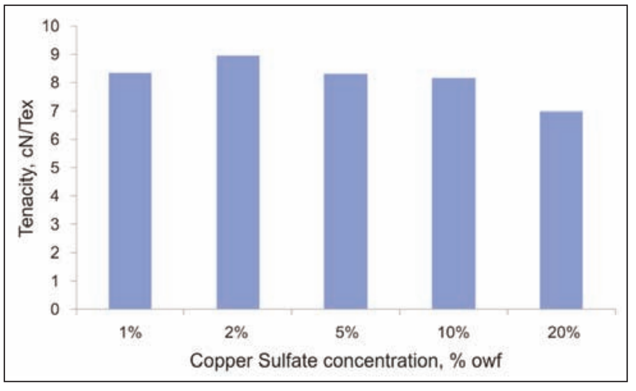

Fig. 5. The effect of concentration of copper sulfate on tenacity of woolen yarn

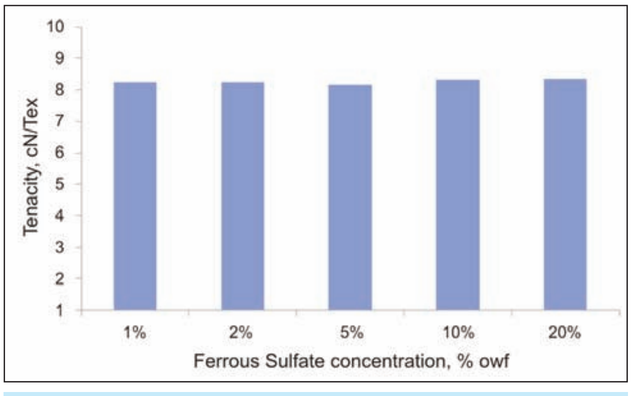

Fig. 6. The effect of concentration of ferrous sulfate on tenacity of woolen yarn

strength of wool fibers as they are the only offered covalent bonds between protein chains of wool and therefore the decrease in tensile strength due to mordanting with stannous chloride has been observed. The changes of the tenacity of alum and ferrous sulfate mordanted samples were negligible. More detailed discussion will be made in the statistical analysis section.

Statistical analysis

The one-way analysis of variance (ANOVA) is used to determine whether there are any statistically significant differences between the means of two or more independent groups. One-way ANOVA requires the homogeneity assumption which states that the population variances are equal for all groups. Table 1 shows the results of the Levene's test with the null hypothesis of "All compared groups have similar population variances". It can be concluded that the variances are not equal if "Sig." < 0.05. As can be seen in table 1, Levene's test showed that the variances of the tenacity of all compared groups are equal.

Table 1

TEST OF HOMOGENEITY OF VARIANCES

	Levene statistic	df1	df2	Sig.
Al	.735	4	20	.579
Sn	1.186	3	16	.346
Cr	.040	4	20	.997
Cu	.273	4	20	.892
Fe	1.031	4	20	.416

Table 2 shows the output of the ANOVA analysis. If "Sig." < 0.05 for a specific mordant, it means that there is a statistically significant difference in the tenacity between the samples treated with different concentrations of that mordant. Here it can be seen that the tenacity of samples mordanted with Al and Fe salts, does not significantly change when the amount of the mordant was varied between 1 %owf up to 20 %owf but the tenacity of samples mordanted with Sn, Cr and Cu salts, significantly change when

Table 2

ANOVA RESULTS

		Sum of squares	df	Mean square	F	Sig.
Al	Between groups	.839	4	.210	.545	.705
	Within groups	7.696	20	.385		
	Total	8.535	24			
Sn	Between groups	198.282	3	66.094	199.503	.000
	Within groups	5.301	16	.331		
	Total	203.583	19			
Cr	Between groups	4.154	4	1.038	6.467	.002
	Within groups	3.212	20	.161		
	Total	7.365	24			
Cu	Between groups	10.242	4	2.561	11.515	.000
	Within groups	4.447	20	.222		
	Total	14.690	24			
Fe	Between groups	.123	4	.031	.071	.990
	Within groups	8.574	20	.429		
	Total	8.697	24			

applied on wool in this range of concentration. Multiple Comparisons table which contains the results of the Tukey post hoc test shows which of the specific groups differed (tables 3, 4, and 5).

As can be seen in table 3, only the tenacity of samples mordanted with 1 %owf and 2 %owf of stannous chloride are equal and the other samples showed statistically significant difference in tensile strength (“Sig.” < 0.05).

According to the data presented in table 4, the tenacity of samples mordanted with 1 %owf and 2 %owf of sodium dichromate statistically differ with samples mordanted with 10 %owf and 20 %owf of the same mordant. There is no significant difference between

Table 3

MULTIPLE COMPARISONS FOR DIFFERENT CONCENTRATIONS OF $SNCL_2$

(I) $SnCl_2$ %	(J) $SnCl_2$ %	Mean difference (I–J)	Std. error	Sig.	95% Confidence interval	
					Lower bound	Upper bound
1	2	.06000	.36403	.998	–.9815	1.1015
	5	3.43000*	.36403	.000	2.3885	4.4715
	10	7.69000*	.36403	.000	6.6485	8.7315
2	1	–.06000	.36403	.998	–1.1015	.9815
	5	3.37000*	.36403	.000	2.3285	4.4115
	10	7.63000*	.36403	.000	6.5885	8.6715
5	1	–3.43000*	.36403	.000	–4.4715	–2.3885
	2	–3.37000*	.36403	.000	–4.4115	–2.3285
	10	4.26000*	.36403	.000	3.2185	5.3015
10	1	–7.69000*	.36403	.000	–8.7315	–6.6485
	2	–7.63000*	.36403	.000	–8.6715	–6.5885
	5	–4.26000*	.36403	.000	–5.3015	–3.2185

* The mean difference is significant at the 0.05 level.

Table 4

MULTIPLE COMPARISONS FOR DIFFERENT CONCENTRATIONS OF $K_2Cr_2O_7$

(I) $K_2Cr_2O_7$ %	(J) $K_2Cr_2O_7$ %	Mean difference (I–J)	Std. error	Sig.	95% Confidence interval	
					Lower bound	Upper bound
1	2	.06000	.25344	.999	–.6984	.8184
	5	.62000	.25344	.144	–.1384	1.3784
	10	1.02000*	.25344	.005	.2616	1.7784
	20	.82000*	.25344	.030	.0616	1.5784
2	1	–.06000	.25344	.999	–.8184	.6984
	5	.56000	.25344	.217	–.1984	1.3184
	10	.96000*	.25344	.009	.2016	1.7184
	20	.76000*	.25344	.049	.0016	1.5184
5	1	–.62000	.25344	.144	–1.3784	.1384
	2	–.56000	.25344	.217	–1.3184	.1984
	10	.40000	.25344	.527	–.3584	1.1584
	20	.20000	.25344	.931	–.5584	.9584
10	1	–1.02000*	.25344	.005	–1.7784	–.2616
	2	–.96000*	.25344	.009	–1.7184	–.2016
	5	–.40000	.25344	.527	–1.1584	.3584
	20	–.20000	.25344	.931	–.9584	.5584
20	1	–.82000*	.25344	.030	–1.5784	–.0616
	2	–.76000*	.25344	.049	–1.5184	–.0016
	5	–.20000	.25344	.931	–.9584	.5584
	10	.20000	.25344	.931	–.5584	.9584

* The mean difference is significant at the 0.05 level.

Table 5

MULTIPLE COMPARISONS FOR DIFFERENT CONCENTRATIONS OF $CuSO_4$						
(I) $CuSO_4$ %	(J) $CuSO_4$ %	Mean difference (I–J)	Std. error	Sig.	95% Confidence interval	
					Lower bound	Upper bound
1	2	–.60000	.29824	.296	–1.4924	.2924
	5	.05000	.29824	1.000	–.8424	.9424
	10	.18000	.29824	.973	–.7124	1.0724
	20	1.36000*	.29824	.002	.4676	2.2524
2	1	.60000	.29824	.296	–.2924	1.4924
	5	.65000	.29824	.228	–.2424	1.5424
	10	.78000	.29824	.105	–.1124	1.6724
	20	1.96000*	.29824	.000	1.0676	2.8524
5	1	–.05000	.29824	1.000	–.9424	.8424
	2	–.65000	.29824	.228	–1.5424	.2424
	10	.13000	.29824	.992	–.7624	1.0224
	20	1.31000*	.29824	.002	.4176	2.2024
10	1	–.18000	.29824	.973	–1.0724	.7124
	2	–.78000	.29824	.105	–1.6724	.1124
	5	–.13000	.29824	.992	–1.0224	.7624
	20	1.18000*	.29824	.006	.2876	2.0724
20	1	–1.36000*	.29824	.002	–2.2524	–.4676
	2	–1.96000*	.29824	.000	–2.8524	–1.0676
	5	–1.31000*	.29824	.002	–2.2024	–.4176
	20	–1.18000*	.29824	.006	–2.0724	–.2876

* The mean difference is significant at the 0.05 level.

the tenacity of the samples mordanted with 5 %owf of sodium dichromate with samples treated with the lower or higher amounts. However the tenacity of the samples mordanted with 10 %owf and 20 %owf of sodium dichromate differ with the samples treated with 1 %owf and 2 %owf and is statistically equal with the tenacity of the samplemordanted with 5 %owf of sodium dichromate.

Table 5 shows that when using copper sulfate as a mordant on wool, there is no statistically significant difference between the samples mordanted with 1 %owf, 2%owf, 5 %owf and 10 %owf, but the tenacity was changed significantly when 20 %owf of $CuSO_4$ was used.

Table 6 displays the results of the independent samples t-test. In the "Levene's Test for Equality of Variances" column, Sig. is the p-value corresponding to this test statistic. If "Sig." < 0.05 we should look at the "Equal variances not assumed" row for the t-test results. Sig (2-tailed) is the p-value corresponding to the given test statistic and degrees of freedom. Mean Difference is the difference between the sample means and is calculated by subtracting the mean of the second group from the mean of the first group. The group means are statistically significantly different if the value in the "Sig. (2-tailed)" row is less than 0.05. As seen in table 6, the equality of the variances is assumed for all samples. So the "Sig. (2-tailed)" of the first row will be considered for comparison of each mordanted sample with the raw sample.

As can be seen there is no significant difference between the mean tenacity value of all samples mordanted with alum or ferrous sulfate and the raw wool sample. It means that thesemordants can be applied on wool without any significant change in the tensile strength of the yarns.

About the samples mordanted with stannous chloride, there is no significant decrease in tenacity when using 1 %owf and 2 %owf of the mordant but the tenacity significantly changes when using higher amounts of the stannous chloride mordant. When using 20 %owf of this mordant, the fibers were completely destroyed and it was impossible to measure the tenacity of the yarn.

About the samples mordanted with chromium, the tenacity significantly changes when using higher than 5 %owf of the mordant, but the decrease in the tenacity of the chromium mordant is much lower than the stannous chloride mordanted samples.

When mordanting with copper sulfate, there was a statistically significant change in the tenacity compared with the raw sample only when 20 %owf of the mordant was applied.

CONCLUSION

In this study, the effect of five different mordants on wool tensile strength was statistically analyzed using "independent samples t-test" and "one-way ANOVA" by SPSS software. The concentration of all mordants

Table 6

INDEPENDENT SAMPLES TEST										
		Levene's test for equality of variances		t-test for equality of means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean difference	Std. error difference	95% Confidence interval of the difference	
									Lower	Upper
Al 1%	Equal variances assumed	.022	.885	–1.214	8	.259	–.36200	.29821	–1.04968	.32568
	Equal variances not assumed			–1.214	7.892	.260	–.36200	.29821	–1.05132	.32732
Al 2%	Equal variances assumed	.018	.896	–.449	8	.666	–.12000	.26756	–.73699	.49699
	Equal variances not assumed			–.449	7.925	.666	–.12000	.26756	–.73800	.49800
Al 5%	Equal variances assumed	.102	.758	–.938	8	.376	–.30000	.31980	–1.03747	.43747
	Equal variances not assumed			–.938	7.591	.377	–.30000	.31980	–1.04444	.44444
Al 10%	Equal variances assumed	.000	1.000	.490	8	.638	.14000	.28596	–.51943	.79943
	Equal variances not assumed			.490	7.987	.638	.14000	.28596	–.51961	.79961
Al 20%	Equal variances assumed	1.269	.293	–.041	8	.968	–.02000	.48637	–1.14158	1.10158
	Equal variances not assumed			–.041	5.532	.969	–.02000	.48637	–1.23497	1.19497
Sn 1%	Equal variances assumed	.179	.684	–1.661	8	.135	–.41000	.24679	–.97911	.15911
	Equal variances not assumed			–1.661	7.382	.138	–.41000	.24679	–.98751	.16751
Sn 2%	Equal variances assumed	.132	.726	–1.431	8	.190	–.47000	.32838	–1.22725	.28725
	Equal variances not assumed			–1.431	7.450	.193	–.47000	.32838	–1.23709	.29709
Sn 5%	Equal variances assumed	.825	.390	–8.459	8	.000	–3.84000	.45395	–4.88682	–2.79318
	Equal variances not assumed			–8.459	5.784	.000	–3.84000	.45395	–4.96090	–2.71910
Sn 10%	Equal variances assumed	1.334	.281	–37.249	8	.000	–8.10000	.21746	–8.60146	–7.59854
	Equal variances not assumed			–37.249	5.568	.000	–8.10000	.21746	–8.64225	–7.55775
Cr 1%	Equal variances assumed	.002	.963	–.437	8	.674	–.12000	.27480	–.75369	.51369
	Equal variances not assumed			–.437	7.987	.674	–.12000	.27480	–.75387	.51387
Cr 2%	Equal variances assumed	.109	.750	–.729	8	.487	–.18000	.24679	–.74911	.38911
	Equal variances not assumed			–.729	7.382	.488	–.18000	.24679	–.75751	.39751
Cr 5%	Equal variances assumed	.012	.915	–2.751	8	.025	–.74000	.26896	–1.36021	–.11979
	Equal variances not assumed			–2.751	7.942	.025	–.74000	.26896	–1.36101	–.11899
Cr 10%	Equal variances assumed	.053	.824	–4.398	8	.002	–1.14000	.25922	–1.73776	–.54224
	Equal variances not assumed			–4.398	7.778	.002	–1.14000	.25922	–1.74074	.53926
Cr 20%	Equal variances assumed	.001	.974	–3.304	8	.011	–.94000	.28453	–1.59613	–.28387
	Equal variances not assumed			–3.304	7.993	.011	–.94000	.28453	–1.59623	–.28377
Cu 1%	Equal variances assumed	.004	.949	–.627	8	.548	–.18000	.28725	–.84241	.48241
	Equal variances not assumed			–.627	7.981	–.548	–.18000	.28725	–.84268	.48268
Cu 2%	Equal variances assumed	.006	.942	1.441	8	.188	.42000	.29151	–.25222	1.09222
	Equal variances not assumed			1.441	7.954	.188	.42000	.29151	–.25289	1.09289
Cu 5%	Equal variances assumed	.072	.795	–.749	8	.476	–.23000	.30727	–.93857	.47857
	Equal variances not assumed			–.749	7.780	.476	–.23000	.30727	–.94207	.48207
Cu 10%	Equal variances assumed	.142	.716	–1.128	8	.292	–.36000	.31924	–1.09617	.37617
	Equal variances not assumed			–1.128	7.600	.294	–.36000	.31924	–1.10297	.38297
Cu 20%	Equal variances assumed	.549	.480	–6.574	8	.000	–1.54000	.23426	–2.08020	–.99980
	Equal variances not assumed			–6.574	6.746	.000	–1.54000	.23426	–2.09819	–.98181
Fe 1%	Equal variances assumed	.043	.841	–.935	8	.377	–.29000	.31031	–1.00559	.42559
	Equal variances not assumed			–.935	7.737	.378	–.29000	.31031	–1.00985	.42985
Fe 2%	Equal variances assumed	1.172	.311	–1.397	8	.200	–.31000	.22198	–.82189	.20189
	Equal variances not assumed			–1.397	5.914	.213	–.31000	.22198	–.85507	.23507
Fe 5%	Equal variances assumed	.105	.754	–1.196	8	.266	–.38000	.31766	–1.11252	.35252
	Equal variances not assumed			–1.196	7.625	.267	–.38000	.31766	–1.11884	.35884
Fe 10%	Equal variances assumed	1.129	.319	–.401	8	.699	–.22000	.54801	–1.48371	1.04371
	Equal variances not assumed			–.401	5.177	.704	–.22000	.54801	–1.61436	1.17436
Fe 20%	Equal variances assumed	.001	.973	–.643	8	.538	–.18000	.27975	–.82509	.46509
	Equal variances not assumed			–.643	8.000	.538	–.18000	.27975	–.82510	.46510

was varying between 1 %owf and 20 %owf. The results showed that alum and ferrous sulfate had no significant effect on the tensile strength of wool and there was no statistically significant difference between the tenacity of samples mordanted with different concentrations of these mordants. Samples mordanted with 20 %owf of sodium dichromate and copper sulfate exhibited statistically significant decrease in tensile strength while the sample mordanted with 20 %owf of stannous chloride was completely destroyed. The loss of tensile strength for samples mordanted with stannous chloride having concentrations higher than 2 %owf was statistically significant.

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