Application of Some Plant Extracts as Biocolorants for Leather During Finishing Process

by

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Abstract

In this research, chrome-tanned bovine crust leathers were dyed during the finishing process using plant extracts: walnut shell (*Juglans regia*), oak bark (*Quercus cortex*), and onion peel (*Allium cepa*). In this study, the standard recipe applied by the factory was used in the leather finishing process. For this recipe, various plant extracts were used instead of pigments, and groups that did not contain pigment and any dyestuff were formed as the control groups.

After these processes, color measurement analyzes were performed on Konica Minolta CM 3600d spectrophotometer. In order to investigate the effects of walnut shell (*Juglans regia*), oak bark (*Quercus cortex*), and onion peel (*Allium cepa*) on the other performance properties of leather, dry and wet rub fastness test according to standard method TS EN ISO 11640 (2001) was performed. The results of the study were statistically evaluated using the NCSS method (Number Cruncher Statistical System). As a result of the study, it was noticed that, depending on the extract, different colors were obtained. It was found that the dry and wet fastness of leathers treated with plant extracts improved.

Introduction

The concept of sustainability has gained importance in leather industry applications as well as in all industrial production.[1] Formally referred to as sustainable production and consumption; the notion concerns with the production and consumption of products, services, and resources in a manner that is environmentally benign, economically viable, and socially beneficial. Within the scope of sustainable production, the use of herbal products in various stages of leather processing have gained importance.[2-10] In addition, research on the use of waste herbal products has intensified and it has been scientifically proven to have many advantages such as elimination of free formaldehyde or reduction of hexavalent chromium formation etc.[11-14] In this way, the use of waste plant products is very important for sustainable production, both to prevent environmental pollution and to create economic value.

The finishing process can give the leathers many different properties, as well as the coloring process for dyed or undyed leathers. The desired surface properties (color, dull or glossy appearance, burning effects, etc.) to the leather by the finishing process and the performance characteristics that provide protection against external factors (light fastness, rubbing fastness, resistance to solvents, etc.) become available for sale.[15] The oak tree has a special importance from the point of view of tanners, compared to other trees, the history of which has been going on for many years.[16] To protect the environment, it is necessary to investigate a more environmentally friendly leather treatment.[17]

In this study, onion peel, oak bark and walnut shell were taken and extracted, and the prepared extract was used instead of pigment in the finishing process. In this way, its usability as a dyestuff was investigated. As far as our research on literature, this work has not yet been studied and has no analogues, so research in this area is important.

Experiments

Materials and Methods

For the study, undyed chrome-tanned bovine leathers processed by the Turan-Skin factory, located in Kazakhstan in the city of Shymkent, were used.

The plants picked from various regions of Kazakhstan were first dried and then ground.

100 grams of dried plant material were mixed with 3000 grams of distilled water and boiled for 3 hours over low heat. The resulting liquid was cooled and filtered. The amount of pH in a liquid dye solution is shown in Table I.

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Table I

pH value of extracts and chemical pigment

№	Dye Solution	pH
1	Walnut shell (*Juglans regia*)	5
2	Oak bark (*Quercus cortex*)	3.5
3	Onion peel (*Allium cepa)*	4.5
4	Chemical pigment	6.5

Plant extracts were used during the finishing. During the finishing the look and function of the leather was defined, its surface was protected. This made it a delicate and important phase, where we decided to invest in order to develop an increasingly eco-sustainable production. During the finishing, binders, waxes, pigments and dyestuffs were applied to the grain of the leather in order to impart the color, give a good surface and other organoleptic, physical and commercial characteristics. The finishing was carried out by spraying, the process consisted of two stages. The first stage was coating dyeing, the second stage was fixing the coating with lacs and dyestuffs. The finishing process was carried out using different dyestuff options:1) natural extracts (walnut shell, oak bark, onion peel); 2) chemical pigments. The finishing recipe is shown in Table II.

The experiment was carried out three times.

As a result, 5 types of samples were used for the study, of which 3 were made with plant extracts (walnut shell, oak bark, onion peel) and 2 control samples were made with chemical pigment and water.

All leathers were conditioned for reproducible testing in the laboratory under the same conditions (20±2°C, 65±2% RH).

Color Measurement Tests of Leather Samples

Determination of color fastness was carried out on a Konica Minolta CM 3600d spectrophotometer. The measurements were carried out by reading 5 points (4 corners and 1 middle) from the surface of the

Table II

Finishing recipe used in application

Chemicals	Application Coat (gramme)	Explanations
Stage 1		
CPT 2350	150	Acrylic Binder(Alpa Chemistry)
CPT 2345	150	Binder (Alpa Chemistry)Acrylic Polymer
CPU 1641	150	Polyurethane Binder (Stahl)
CRE 1036	200	Acrylic Binder(Alpa Chemistry)
CST 6760	200	StukoWax(Alpa Chemistry)
CW 171	50	Synthetic Wax(Alpa Chemistry)
CW 159	50	StukoWax (Stahl)
CST HD	50	Polyurethane Binder (Stahl)
Dyestuff	2000	Plant Extracts (walnut shell, oak bark, onion peel) or chemical pigment or water
1) 3× spray – RotoPress (80°C, 150 Bar) – 3× spray – RotoPress(80°C, 70 Bar) – 3× spray (RotoPress 80°C, 70 Bar)		
Stage 2		
CK 1622	150	Polyurethane lacs (Stahl)
Dyestuff	300	Plant Extracts (walnut shell, oak bark, onion peel) or chemical pigment or water
1) 2× spray – RotoPress (90°C – 70 Bar)		

Table III

Color Measurement Test Results

	Data Name	L*(D65)	a*(D65)	b*(D65)	DL*(D65)	Da*(D65)	Db*(D65)	DE*ab(D65)
Control group								
1	water 1	81.46	-0.05	4.4	52.59	0.05	5.01	52.83
	water 2	79.75	0.04	4.64	50.88	0.14	5.26	51.15
	water 3	80.07	-0.41	4.32	51.19	-0.31	4.93	51.43
2	chemical pigment 1	78.61	-0.07	12.64	49.74	0.04	13.25	51.47
	chemical pigment 2	79.29	-0.16	12.68	50.41	-0.06	13.29	52.14
	chemical pigment 3	78.83	-0.09	12.8	49.95	0.01	13.41	51.72
Experimental group								
1	oak bark 1	70.55	5.79	23.9	41.67	5.9	24.51	48.71
	oak bark 2	69.09	6.28	24.36	40.21	6.38	24.97	47.76
	oak bark 3	69.99	5.99	24.1	41.11	6.1	24.71	48.36
2	onion peel 1	74.54	3.55	36.7	45.67	3.65	37.31	59.08
	onion peel 2	74.34	4.05	37.72	45.46	4.16	38.33	59.61
	onion peel 3	74.78	3.89	37.71	45.9	3.99	38.33	59.93
3	walnut shell 1	74.86	1.5	15.81	45.98	1.6	16.42	48.86
	walnut shell 2	74.64	1.61	15.65	45.76	1.72	16.27	48.6
	walnut shell 3	74.44	1.83	16.46	45.56	1.93	17.08	48.7

samples in the reading zone of the device. The results were evaluated by averaging these 5 measurement points.

Cut samples of 15 × 15 cm were used for the aging test. Color measurements were made in Minolta before the samples were aged. Before starting the measurement on the machine, black or white standards were read as a reference. Since the leather samples used for this experiment were close to white in color, the measurement was made based on the white standard.

Over time, discoloration and aging occurs on the surface of the leather under the influence of environmental factors. The purpose of the various aging methods described in this international standard is to obtain results indicating changes that may occur when exposed to the leather in a certain environment over a long period of time. According to ISO 17228-7B (2005) standard, leather samples were subjected to aging processes under 60°C and 90% humidity conditions for 24 and 96 hours in a UV cabinet whose temperature and humidity can be adjusted. Color measurement test results are shown in Table III.

The results in Table III show that the magnitude of a*(D65), Da*(D65) increased compared to the control group of the experimental group, approaching red. The indicator b*(D65), Db*(D65) moved in the direction of more yellow than the control group. Around L*(D65), DL*(D65), there is only a small difference between the control group and the experimental group.

Color Measurement Test Results after the aging process under 60°C and 90% humidity conditions for 24 hours in a UV cabinet are shown in Table IV.

Table IV

Color Measurement Test Results after aging process (24 hours)

	Data Name	L*(D65)	a*(D65)	b*(D65)	DL*(D65)	Da*(D65)	Db*(D65)	DE*ab(D65)
Control group								
1	water 1	80.35	-0.09	4.52	51.47	0.02	5.14	51.73
	water 2	78.8	0.59	4.3	49.93	0.69	4.92	50.17
	water 3	78.19	0.8	4.74	49.31	0.91	5.35	49.61
2	chemical pigment 1	78.44	-0.03	12.62	49.56	0.08	13.24	51.3
	chemical pigment 2	78.67	-0.01	12.63	49.8	0.09	13.24	51.53
	chemical pigment 3	78.52	-0.09	12.66	49.64	0.02	13.27	51.38
Experimental group								
1	oak bark 1	69.68	6.08	23.87	40.81	6.18	24.48	47.99
	oak bark 2	69.81	5.49	24.13	40.94	5.59	24.74	48.16
	oak bark 3	70.2	5.74	24.16	41.32	5.84	24.78	48.53
2	onion peel 1	74.11	4.85	37.93	45.23	4.95	38.54	59.63
	onion peel 2	73.71	4.46	37.48	44.83	4.57	38.09	59.01
	onion peel 3	75.16	4.02	36.75	46.28	4.12	37.36	59.63
3	walnut shell 1	72.77	2.09	17.31	43.9	2.19	17.92	47.46
	walnut shell 2	73.14	1.83	16.28	44.26	1.94	16.89	47.42
	walnut shell 3	74.07	1.53	15.69	45.19	1.63	16.31	48.07

Table V

Color Measurement Test Results after ageing process (96 hours)

	Data Name	L*(D65)	a*(D65)	b*(D65)	DL*(D65)	Da*(D65)	Db*(D65)	DE*ab(D65)
Control group								
1	water 1	76.84	1.50	5.12	47.96	1.61	5.73	48.33
	water 2	76.83	1.51	5.12	47.95	1.61	5.74	48.32
	water 3	76.74	0.31	7.06	47.87	0.42	7.67	48.48
2	chemical pigment 1	78.46	-0.14	12.66	49.58	-0.04	13.27	51.33
	chemical pigment 2	79.31	-0.08	13.03	50.43	0.03	13.64	52.24
	chemical pigment 3	78.64	-0.05	12.71	49.76	0.05	13.32	51.51
Experimental group								
1	oak bark 1	70.53	6.00	24.77	41.66	6.10	25.39	49.16
	oak bark 2	69.11	6.28	24.38	39.24	6.39	24.99	46.96
	oak bark 3	70.03	5.76	24.29	41.15	5.86	24.90	48.45
2	onion peel 1	75.66	3.40	35.12	46.78	3.50	35.73	58.97
	onion peel 2	75.50	3.31	35.31	46.62	3.42	35.92	58.95
	onion peel 3	76.33	3.11	36.25	47.45	3.22	36.87	60.18
3	walnut shell 1	73.91	1.75	16.62	45.04	1.86	17.23	48.26
	walnut shell 2	74.14	1.93	16.49	45.26	2.03	17.10	48.43
	walnut shell 3	73.54	1.97	17.41	44.67	2.08	18.03	48.21

There was no significant difference between the before aging process of leather and after the aging process of leather under 60°C and 90% humidity conditions for 24 hours in color.

Color Measurement Test Results after the aging process under 60°C and 90% humidity conditions for 96 hours in a UV cabinet are shown in Table V.

No significant difference was found between the before aging process of leather and after the aging process of leather under 60°C and 90% humidity conditions for 96 hours in color.

Dry and Wet Rubbing Fastness Analyses

The leather samples were made from black and white felts and tested for dry and wet fastness test according to TS EN ISO 11640 (2001) from the surface of the sample. An Otto Specth Bally Finish Tester was used for analysis. The change in color of the leather and felt samples was evaluated in accordance with A02 (1996) and ISO 105 A03 (1996) and ISO 105 with a gray scale.

Statistical Evaluation of Results

When evaluating the results of the study, statistical analysis was used NCSS (Number Cruncher Statistical System) 2022 Statistical Software (NCSS LLC, Kaysville, Utah, USA). descriptive statistics (mean, standard deviation, median, frequency and ratio).

Normal distribution of parameters between groups One-way Anova test and Bonferroni test to detect group differences; Student's t-test was used in their evaluation according to the two groups.

Between groups of abnormal distribution of parameters Wallis test and Dunn's test in identifying differences between groups; the Mann Whitney U test was used in the evaluation according to the two groups.

When comparing qualitative data, the Fisher Freeman Halton test was used. Results were evaluated at 95% confidence interval and $p<0.05$ significance level.

Results and Discussion

Results of the Appearance of Leather

The leathers were subjected to visual evaluation after application. Figure 1 shows the difference in leather samples after finishing process.

Dry and Wet Rubbing Fastness Results

In accordance with the standard method, the analysis of rub fastness of the leather to dry friction was carried out using black felt, for wet friction of the leather white felt was used. The results were evaluated on a gray scale. Dry rub fastness test results are shown in Table VI.

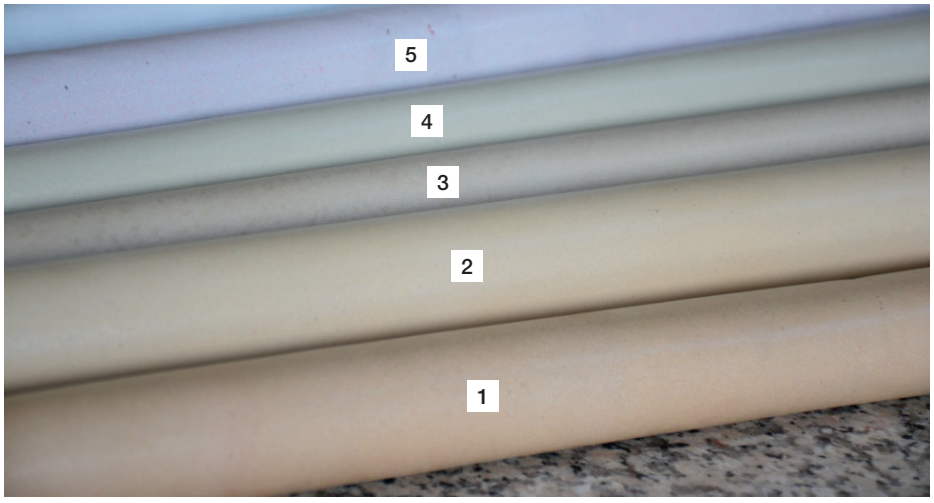

Figure 1. Color change observed in leather samples after finishing process
1- walnut shell; **2**- onion peel; **3**- oak bark; **4**- chemical pigment; **5**- water

Table VI

Dry rub fastness test results.

	Type of leather	Leather	Felt
Control	water 1	4	4
	water 2	4	4
	water 3	4	4
	chemical pigment 1	3/4	3/4
	chemical pigment 2	3/4	3/4
	chemical pigment 3	3/4	3/4
Experimental	walnut shell 1	4	4
	walnut shell 2	4	4
	walnut shell 3	4	4
	oak bark 1	4/5	4/5
	oak bark 2	4/5	4/5
	oak bark 3	4/5	4/5
	onion peel 1	4/5	4/5
	onion peel 2	4/5	4/5
	onion peel 3	4/5	4/5

Evaluation of dry rub fastness test results are shown in Table VII.

The results of evaluation of fastness to dry friction were higher in the experimental group. The tests were carried out three times. The leather treated with water instead of chemical pigment and the walnut shell showed a score of 4 three times. The leather treated with chemical pigment was rated at 3/4. Dry rub fastness of walnut shell higher than chemical pigment. Dry rub fastness was significantly higher in the oak bark and onion peel than chemical pigment, water and walnut shell and showed three times 4/5.

Wet rub fastness test results are shown in Table VIII.

Evaluation of wet rub fastness test results are shown in Table IX.

Even in a wet fastness test study, the experimental samples showed good results compared to the control sample. There were 4 score in the control group, exactly in water and chemical pigment. Onion peel and oak bark showed good results among herbal extracts, 4/5 score tree times. There were 4 in leather of walnut shell and 4/5 in felt.

Color measurement analysis results

Color measurements were evaluated according to the DE * ab (D65) results obtained from L * a * b color area measurements. Table X shows the results of color measurement evaluation.

In this study a*(D65), b*(D65), da*(D65), db*(D65) levels were considerably higher in the experimental group. There was considerably significant difference between control and experimental groups regarding to L*(D65), dL*(D65), dE*ab(D65) levels.

Table VII

Evaluation of dry rub fastness test results.

		Control		Experimental		
		water n(%)	chemical pigment n(%)	walnut shell n(%)	oak bark n(%)	onion peel n(%)
Leather	3/4	0 (0)	3 (100)	0 (0)	0 (0)	0 (0)
	4	3 (100)	0 (0)	3 (100)	0 (0)	0 (0)
	4/5	0 (0)	0 (0)	0 (0)	3 (100)	3 (100)
Felt	3/4	0 (0)	3 (100)	0 (0)	0 (0)	0 (0)
	4	3 (100)	0 (0)	3 (100)	0 (0)	0 (0)
	4/5	0 (0)	0 (0)	0 (0)	3 (100)	3 (100)

Table VIII

Wet rub fastness test results

	Type of leather	Leather	Felt
Control	water 1	4	4
	water 2	4	4
	water 3	4	4
	chemical pigment 1	4	4
	chemical pigment 2	4	4
	chemical pigment 3	4	4
Experimental	walnut shell 1	4	4/5
	walnut shell 2	4	4/5
	walnut shell 3	4	4/5
	oak bark 1	4/5	4/5
	oak bark 2	4/5	4/5
	oak bark 3	4/5	4/5
	onion peel 1	4/5	4/5
	onion peel 2	4/5	4/5
	onion peel 3	4/5	4/5

Table IX

Evaluation of wet fastness test results.

		Control		Experimental		
		water n(%)	chemical pigment n(%)	walnut shell n(%)	oak bark n(%)	onion peel n(%)
Leather	4	3 (100)	3 (100)	3 (100)	0 (0)	0 (0)
	4/5	0 (0)	0 (0)	0 (0)	3 (100)	3 (100)
Felt	4	3 (100)	3 (100)	0 (0)	0 (0)	0 (0)
	4/5	0 (0)	0 (0)	3 (100)	3 (100)	3 (100)

Table X

Evaluation results of color measurements

	Control		Experimental	
	Median (min-max)	Ort+SD	Median (min-max)	Ort+SD
L*(D65)	78.8(78.6/81.5)	79.7±0.57	69.9(69.1/74.9)	73.03 ±0.57
a*(D65)	-0.1 (-0.4 /-0.1)	-0.1±0.37	3.5(1.5/6.3)	3.8±0.38
b*(D65)	8.5(4.4/12.8)	8.58±0.19	24.4(15.8/37.7)	25.8±0.19
dL*(D65)	50.8(49.7/52.5)	50.8±0	41.1(40.2/-45.9)	44.15±0
da*(D65)	-0.01(-0.06/0.1)	-0.02±0.19	4.2(1.6/6.4)	3.94±0.19
db*(D65)	9(4.9/13.4)	9.2±0.38	24.7(16.2/38.3)	26.44±0.38
dE*ab(D65)	51.7(51.4/52.8)	51.8±0.57	52(48.3-59.9)	52.18±0.57

Table XI

Evaluation results of color measurements after aging process (24 hours)

	Control		Experimental	
	Median (min-max)	**Ort+SD**	**Median (min-max)**	**Ort+SD**
L*(D65)	78.5(78.5/80.3)	78.8±0.57	72(69.7/75.2)	72.5 ±0.57
a*(D65)	0.1 (-0.1 /0.8)	0.19±0.37	4(1.5/6.1)	4.01±0.38
b*(D65)	8.6(4.3/12.7)	8.6±0.19	25(15.7/37.9)	25.9±0.19
dL*(D65)	49.6(49.3/51.5)	49.9±0	43(40.8/-46.3)	43.64±0
da*(D65)	0.3(0.02/0.9)	0.3±0.19	4(1.6/6.2)	4.11±0.19
db*(D65)	9(4.9/13.3)	9.1±0.38	26(16.3/38.5)	26.57±0.38
dE*ab(D65)	51.7(49.6/51.7)	50.9±0.57	51(48.2-59.6)	51.77±0.57

Table XI shows evaluation results of color measurements after aging process under 60°C and 90% humidity conditions for 24 hours in a UV cabinet.

No significant difference was found between evaluation results of color measurements in Table X and Table XI in color.

Evaluation results of color measurements after aging process under 60°C and 90% humidity conditions for 96 hours in a UV cabinet are shown in Table XII.

Evaluation results of color measurements after aging process under 60°C and 90% humidity conditions for 96 hours in a UV cabinet showed that temperature and humidity did not negatively affect the color measurements of the leather.

Table XIII shows evaluation results of color measurements according to type of plants.

Color measurements of water and chemical pigment are almost the same. No significant difference was found between water and oak bark, walnut shell ($p < 0.01$). The dE*ab(D65) color value obtained in those using water was significantly lower than those onion peel ($p < 0.01$). Color measurements obtained by using onion peel were found to be significantly higher than chemical pigment ($p < 0.01$). There was no significant difference between chemical pigment and oak bark ($p < 0.01$). The dE*ab(D65) color value obtained in those using oak bark was significantly lower than those onion peel ($p < 0.01$). In our study, minor significant difference between walnut shell and oak bark color measurements. Delta E*ab(D65) color measurements were significantly higher in onion peel than walnut shell, oak bark. The results show that color measurements were minor significant difference between chemical pigment and oak bark. Color measurements were not significantly changed after the aging process of leather under 60°C and 90% humidity conditions for 24 and 96 hours.

Table XII

Evaluation results of color measurements after aging process (96 hours)

	Control		Experimental	
	Median (min-max)	**Ort+SD**	**Median (min-max)**	**Ort+SD**
L*(D65)	77.5(76.7/79.3)	77.8±0.57	73.9(69.1/75.7)	73.19 ±0.57
a*(D65)	0.5 (-0.1 /1.5)	0.5±0.37	3.7(1.7/6.3)	3.7±0.38
b*(D65)	9.2(5.1/13.0)	9.2±0.19	26(16.5/36.2)	25.6±0.19
dL*(D65)	49.1(47.9/50.4)	48.9±0	44(39.2/-47.5)	44.2±0
da*(D65)	0.5(-0.04/1.6)	0.6±0.19	4(1.9/6.4)	3.8±0.19
db*(D65)	9(5.7/13.6)	9.9±0.38	26(17.1/36.9)	26.24±0.38
dE*ab(D65)	50(48.3/52.2)	50.0±0.57	52(48.3-60.2)	51.95±0.57

Table XIII

Evaluation results of color measurements according to type of plants

		n	dE*ab(D65) Median (min-max)	Ort+SD	Test Value	p
Group	**Control**	**180**	50.7(50.5/51.5)	50.9±0.5	**25.16**	**a0.001****
	1. water	**30**	51.4(51.1/52.8)	51.8±0.7		
	2. water (24 hours)	**30**	50.2(49.6/51.7)	50.5±0		
	3. water (96 hours)	**30**	48.3(48.3/48.5)	48.4±0.7		
	4. chemical pigment	**30**	51.7(51.4/52.1)	51.8±0.57		
	5. chemical pigment (24 hours)	**30**	51.4(51.3/51.5)	51.4±0.57		
	6. chemical pigment (96 hours)	**30**	51.3(51.5/52.2)	51.7±0.57		
	Experiment	**270**	51.9(51.5/52.3)	51.9±0.28		
Plant extract	7. oak bark	**30**	48.3(47.8/48.7)	48.3± 0.52	**39.47**	**b0.001****
	8. oak bark (24 hours)	**30**	48.2(47.9/48.5)	48.3± 0.39		***Post Hoc;***
	9. oak bark (96 hours)	**30**	48.4(46.9/49.2)	48.2± 0.26		***1-4 p:0.001***
	10. onion peel	**30**	59.6(59.1/59.3)	59.5± 0.12		***1-7 p:0.001***
	11. onion peel (24 hours)	**30**	59.6(59.1/59.6)	59.4± 0		***1-10 p:0.001***
	12. onion peel (96 hours)	**30**	59 (58.9/60.2)	59.4± 0.13		***1-13 p:0.001***
	13. walnut shell	**30**	48.7(48.6/48.9)	48.7± 0.26		***4-7 p:0.001***
	14. walnut shell (24 hours)	**30**	47.5(47.4/48.1)	47.7± 0.39		***4-11 p:0.001***
	15. walnut shell (96 hours)	**30**	48.3(48.2/48.4)	48.3± 0.52		***4-15 p:0.001***

[a]Student t-test [b]Oneway Anova test&post hoc Bonferroni test **p<0.01

Conclusion

In this study, the possibility of using plant extracts as a dye in the finishing process of the leather was investigated. The recipe was provided according to the finished formula, replacing the chemical pigment with plant extracts, using oak bark, walnut shell and onion peel. For this reason, chrome-tanned crust leathers were dyed in the finishing process. These processes were repeated 3 times and compared with the control example of the leather, which is made according to the main recipe, where the chemical pigment and water were used. In these comparisons, visual evaluation, color measurement, dry and wet rubbing fastness were analyzed according to standard methods. The color measurement evaluation results showed that there was no significant difference between control and experimental groups regarding dE*ab (D65) levels.

According to the dry rub fastness test results, the experimental group showed excellent results, especially the leather finished by onion peel and oak bark extracts showed 4/5 score, when water and chemical pigment from control group were rated at 4 score. During the wet rubbing fastness, a good color quality was observed in the experimental group. The results of the study showed that in all experiments the experimental group showed a good result and plant extracts can be used as biocolorants during the finishing process. The finishing formulation used in this research belongs to the factory itself. The proportions of the materials used may vary according to the desired properties. For this reason, it may be appropriate to try plant extracts in different formulations.

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