(REFEREED RESEARCH)

USE OF DIFFERENT NATURAL DYE SOURCES FOR PRINTING OF COTTON FABRICS

PAMUKLU KUMAŞLARIN BASILMASI İÇİN FARKLI DOĞAL BOYARMADDE KAYNAKLARININ KULLANILMASI

M. İbrahim BAHTİYARİ[1] Hüseyin BENLİ[2] Arzu YAVAŞ[3] Candan AKCA[4]

[1]Department of Textile Engineering, Erciyes University, Kayseri, Turkey
[2]Mustafa Çıkrıkçıoğlu Vocational School, Erciyes University, Kayseri, Turkey
[3]Department of Textile Engineering, Pamukkale University, Denizli, Turkey
[4]Salihli Vocational School, Celal Bayar University, Manisa, Turkey

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ABSTRACT

In the study it was aimed to print cotton fabrics with natural dye sources to respond the demands on the natural products and cleaner production. For this purpose, bleached, bleached and mercerized cotton fabrics were colored by pigment printing method with the use of the extracts obtained from the five different natural dye sources “pomegranate peel, nutshell, orange tree leaves, alkanet roots and dyer's chamomile". By this way it was planned to show the coloration of cotton with natural dye sources. For the environmentally friendly production, no mordanting has been applied but good colors with sufficient fastnesses were observed from the printed fabrics nonetheless. In printing process, binder was used instead of mordanting agents for the fixing of the dye stuff. Like in other dyeing and printing processes, it was found that pretreatment processes had an effect on the printability of cotton with natural dyes. By mercerization higher color efficiencies, darker shades and meanwhile increases in light fastnesses and decreases in washing and perspiration fastnesses were generally obtained.

Keywords: Cotton, Natural Dyes, Printing, Pretreatment, Mercerization

ÖZET

Bu çalışmada pamuklu kumaşların doğal boyarmadde kaynakları ile basılması sağlanarak doğal ürünlere ve daha temiz üretime olan talebe cevap verilmesi amaçlanmıştır. Bu amaçla, ağartılmış, ağartılmış ve merserize edilmiş pamuklu kumaşlar, "nar kabuğu, fındık kabuğu, portakal ağacı yaprakları, havacıva bitkisinin kökü ve papatya" olmak üzere beş farklı doğal boya kaynağından elde edilen ekstraktlar kullanılarak pigment baskı yöntemi ile renklendirilmiştir. Bu sayede, pamuğun doğal boyarmadde kaynakları ile renklendirilebileceğinin gösterilmesi planlanmıştır. Çevre dostu üretim için, mordan maddesi kullanılmamış fakat yinede baskılı kumaşlardan yeterli haslık değerlerine sahip iyi renkler elde edilmiştir. Baskı işleminde boyarmaddenin bağlanması için mordan maddeleri yerine binder kullanılmıştır. Diğer boyama ve baskı işlemlerinde olduğu gibi, ön terbiye işlemlerinin pamuklu kumaşların doğal boyalar ile basılabilirliğine etkisinin olduğu tespit edilmiştir. Genel olarak merserizasyon işlemi ile yüksek renk verimlilikleri, daha koyu renk tonları ve aynı zamanda ışık haslıklarında artış, ve yıkama ve ter haslıklarında düşüş elde edilmiştir.

Anahtar Kelimeler: Pamuk, Doğal Boyalar, Baskı, Ön terbiye, Merserizasyon

Corresponding Author: Hüseyin Benli, hbenli@erciyes.edu.tr

1. INTRODUCTION

Cotton can be used in every type of garment and house hold fabrics (1). It has to go through several chemical processes to obtain properties suitable for further dyeing and use (2). In the coloration of cotton extensive number of dye classes can be used (3). On the other hand there is renewed interest in rediscovering and improving vegetable-based dyeing of fabrics but they are not important in textile markets (4). Natural dyes are mostly non-substantive and must be applied on textiles by the help of mordants, usually a metallic salt (5). Beyond this, printing of natural dyes via binders can be an alternative for the fixing of the dyes on the fabrics (6). The pigment printing process is the easiest printing method (7). In a study, the applicability of madder by using printing method has been investigated and the effects of dye and urea concentration, type of fixation, fixation temperature and time, effect of mordant type and

mordanting methods has been examined (8). In another study, marigold petals were used in printing of cotton and the effect of different mordants has been studied (9). Differently El-Hennawi et al., (2012) were used laccase enzyme instead of harmful mordants for fixation of different natural dyes in printing of fabrics (10). In the light of our previous study, in which madder, buckthorn, walnut bark and indigo were used as natural dyes in printing of cotton and wool fabrics (6), in this study we have planned to show the usability of dye extracts from pomegranate peel, nutshell, orange tree leaves, alkanet roots and dyer's chamomile in printing of cotton fabrics and additionally the effect of mercerization on printability of cotton fabrics were investigated too. There are studies on the use of these tested natural dye sources but they are generally related with the dyeing processes.

Pomegranate (*Punica granatum* L.) peels has been presented as the suitable source of colorant for textile industry (11) and there are different studies available on the use of pomegranate peels. For example, Kulkarni et al. (2011) reported that wide range of shades with good fastnesses can be obtained with the natural dye extracted from pomegranate peels (12). Adeel *et al.* (2009) have showed the usability of pomegranate aqueous extract in dyeing of cotton fabrics as well (13). The use of nutshell in dyeing of textile materials has also been investigated. In a study, the nutshell extract solutions were used in dyeing of cotton and wool fabrics and the extract residues of nutshell were then used as adsorbent for the color removal of a dye effluent (14). The other tested natural dye source was the dyer's chamomile. It (*Anthemis tinctoria* L.) was popular in Turkey for the manufacture of carpets and kilims. In the flowers of this, yellow dye sources the glycosides of luteolin 45 and apigenin 46 can be found as well as quercetagetin and patuletin (15). The total flavonoid content of commercially available flower-head dry dyer's chamomile (calculated as quercetin) ranged between 1.64-2.43 % (16). It was reported that after dyeing of cotton fabric on a jet dyeing machine with an extract of dyer's chamomile, brilliant yellow color shade can be achieved, but after drying light/dark strips were observed (17). The root of alkanet or dyer's bugloss, Alkanna tinctoria (L.) Tausch (Boraginaceae) which contains a purple dye (18) were also studied previously. Zarkogianni *et al.* (2010) have investigated nine different natural dyes consisting also alkanna in dyeing of cotton and wool with different mordants and showed the usability of these natural dyes and the effect of mordanting agents (19). Rekaby *et al.* (2009) have studied the printing of wool, silk, cotton and flax with the natural dyes from alkanet and rhubarb (20). The other investigated natural dye source is the orange tree leaves. As known, oranges are grown in tropical and subtropical climates throughout the world. Brazil and the United States are the top-producing countries and Turkey is the other principal country that grows oranges (21). In the study the orange tree leaves from Turkey has been used as natural dye source too.

The aim of this study is to show the usability of different plantal natural dye sources in printing of the cotton fabrics and also to emphasize the effect of pretreatment processes on the obtained colors.

2. EXPERIMENTAL

In the study, CMC sized cotton woven fabrics weighing 220 g/m^2 were used. Prior to the printing process the fabrics were desized, scoured, bleached and bleach cleaned up continuously in a laboratory type bleaching machine (Ataç trade mark). For this aim firstly the fabrics were impregnated with a bath detailed in Table 1. Then the impregnated fabrics were steamed at 101^{o}C for 10 minutes and subsequently washing/rinsing were carried out. These whole steps were conducted in a laboratory type continuous bleaching machine. The production velocity was 2m/min in this laboratory type machine. Then to see the effect of mercerization the scoured and bleached fabrics were mercerized with 26°Be NaOH at room temperature in a laboratory type mercerization machine (Ataç trade mark) in which washing and neutralization was also carried out continuously. The production velocity was 4m/min and the dwelling in alkali was nearly 15 seconds. All the pretreatment processes have been carried out in Kayseri, Turkey, Vocational and Technical Education Centre (METEM).

Table 1. Recipe of the scouring/bleaching process

40 mL/L NaOH (47°Be) 45 mL/L H_2O_2 (50%) 8 mL/L Stabilizing agent 4 mL/L Wetting agent 2 mL/L Sequestering agent Impregnating with a Pick-up of 80%
Steaming at 101°C
Bleach clean up

After pretreatment processes the scoured/bleached and scoured/bleached and mercerized fabrics were printed with a recipe detailed in our previous study (6) and it was given in table 2. This printing process is similar to a pigment printing process but natural dye extracts were used as dyestuff instead of pigment dyes.

Table 2. Printing paste recipe

Printing paste	
Synthetic Thickener (Ammonium salt of carboxylic acid polymers)	4 g
Thermally Crosslinkable Polyacrylate Binder	15 g
Extract	81 g
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	100 g

For the use of printing five different natural dye extracts; pomegranate peel, nutshell, orange tree leaves, alkanet roots, dyer's chamomile were prepared. For extraction of these natural dye sources, the extraction equipment in Figure 1 was used and the dye base of the natural dye source was extracted by water.

Before extraction the whole natural dye sources were grinded and then from these grinded natural dye sources 20 g were taken into extraction period and the final volume of extract was adjusted to 200 ml by evaporation of water. From this 200 ml extract 81 g were used for preparation of the printing paste as detailed in Table 2. Then fabrics were

printed with 6 m/min at 4 pres on a laboratory-type printing machine (Ataç trade mark). 10 mm in diameter doctor blade was used two times. Printed fabrics were dried and cured at 100^0C/3 min and 150^0C/5 min, respectively.

Figure 1. Extraction equipment

The printed fabrics were then analyzed in terms of CIE *L* a* b** values and color efficiencies *(K/S* values) in Minolta 3600d spectrophotometer. The other important parameter for the evaluation of the printing process is the fastnesses of the samples. So the fabrics were also tested in terms of washing fastness with ISO 105-C10 standard in test condition of Test A (1) (22), light fastness with ISO 105-B02 standard (23), rubbing fastness with ISO 105-X12 standard (24), perspiration fastness with ISO 105-E04 standard (25) and dry cleaning fastness with ISO 105-D01 standard (26). Moreover for the saliva fastness DIN 53160-1 standard (27) was used. In the experiment of the saliva fastness test, the filter paper soaked with the solution detailed in standard was put on the surface of the printed fabrics and color changes of the fabrics and staining on filter papers were evaluated with the gray scale.

3. Results and discussion

➢ Colors of the printed fabrics

Color efficiency is an important parameter for the evaluation of textile printings as well dyeings. The fabrics differently pretreated and printed with different dye extracts were analyzed in terms of color efficiencies (Fig.2).

The results obtained from the color efficiencies showed that use of different plantal extracts caused different color efficiencies. It was observed that the dyer's chamomile and pomegranate peel showed the highest color efficiencies while the nutshell extract based prints gave limited. Even so nutshell can be an alternative for the coloration of cotton by printing as seen from the figure 3. It was thought that these differences obtained from different plantal extract based prints could be related with the obtained colors, color shades, the dye content of the natural dye sources and also the extraction yield.

The interesting point is that in all printings beyond the obtained colors, the pretreatment process showed great importance in terms of color efficiencies. In other words, applying a mercerization after bleaching prior to printing process has caused a significant increase in color efficiencies. For example in printing with dyer's chamomile the color efficiencies of bleached and bleached + mercerized fabrics were 3.38 and 3.95 respectively. This tendency was valid for all tested natural dye sources in different grades. For example, in nutshell based prints the fabrics bleached and mercerized prior to printing had showed different color efficiencies such as: 0.5 in printing after bleaching and 0.57 in printing after bleaching and mercerization. As a result it could be told that mercerization prior to printing caused an increase in color efficiencies but this increase could be varied according to the natural dye source used. This effect of mercerization is well known. It helps in increasing the dye uptake ability (28). By this study it was exhibited that this effect of mercerization is valid for the printing with natural dyes too.

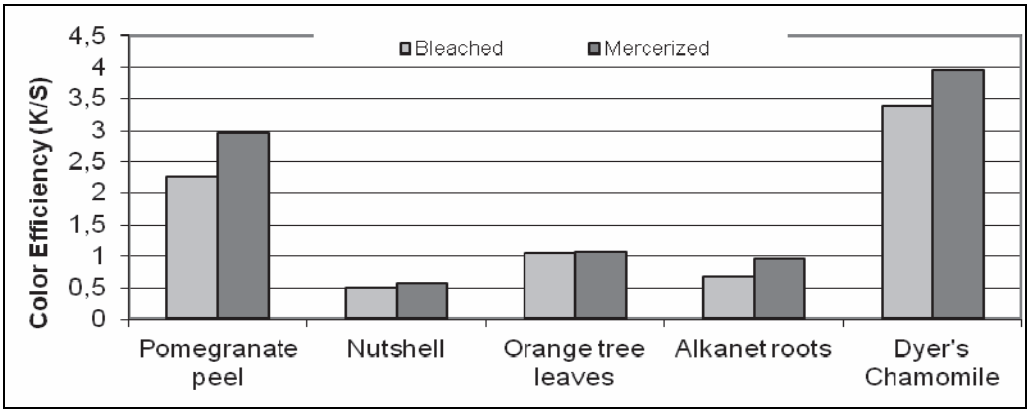


Figure 2. Color efficiencies of printed fabrics

The printed fabrics' colors and color shades were also analyzed. Fabrics' CIE *L*a*b**, chroma (*C**) values and hue angles were investigated and collected in Table 3.

*L** indicates the lightness; the perfect reflecting diffuser has *L**=100 and the perfect black has *L**=0. The colors with *a**>0 represent redness and with *a**<0 greenness; *b**>0 means yellowness and *b**<0 blueness (29).

The fabrics differently pretreated have shown different color values like in color efficiencies. This situation has already known for the fabrics colored conventionally but addition to this, it was presented that this common belief is valid for the fabrics printed with natural dye sources too. For all tested natural dyes the *L** values of the fabrics were changed with the change in the pretreatment process. In all cases the fabrics mercerized and then printed has shown lower *L** values than the one previously not mercerized but only bleached. In other words, the colors of the mercerized fabrics were darker. For instance in pomegranate peel based prints the previously bleached one had the 82.03 *L** value while it was 79.19 in the fabric printed after mercerization. This is valid for all tested natural dye based prints but limited in some cases. For instance in Orange tree leave based prints this difference was observed too but it was not dominant like in color efficiencies (Fig.2). The *L** value of the printed fabric previously bleached and bleached mercerized were 88.17 and 87.18 respectively. It was also found that in general the C* chroma values of the printed fabrics were affected from the pretreatment processes. The fabrics previously mercerized had higher chroma values than the fabrics bleached and printed with the same natural dye.

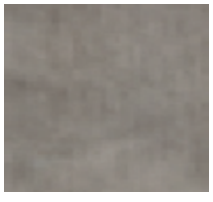
As a result it can be concluded that by applying a mercerization prior to printing of natural dyes, higher color efficiencies, higher chroma values and lower *L** values in other words darker colors and higher saturations can be obtained for the same natural dye based printings.

In figure 3 the photographs of the printed fabrics were presented to exhibit the color differences between the printed fabrics. As expected, use of different natural dye sources in printing of the fabrics cause different colors in the fabrics. The color values seen from the table 3 and the photos of the prints (Figure 3) supported this argument.

Table 3. CIE *L*a*b*C** and *h°* values of fabrics printed with various plantal extracts

Natural Dyes	Pretreatment Processes	*L**	*a**	*b**	*C**	*h°*
Pomegranate peel	Bl.	82.03	3.99	22.89	23.23	80.1
	Mer.	79.19	4.91	24.84	25.32	78.81
Nutshell	Bl.	80.19	6.9	13.61	15.26	63.1
	Mer.	78.77	6.88	13.72	15.35	63.38
Orange tree leaves	Bl.	88.17	0.03	17.68	17.68	89.91
	Mer.	87.18	0.47	17.51	17.52	88.45
Alkanet roots	Bl.	83.31	3.02	15.69	15.98	79.09
	Mer.	80.88	3.44	17.12	17.46	78.65
Dyer's Chamomile	Bl.	84.28	1.58	26.22	26.26	86.55
	Mer.	82.79	1.15	26.92	26.94	87.56

Bl. means the fabric has been desized, scoured and bleached
Mer. means the fabric has been desized, scoured and bleached and finally mercerized

Processes	Natural Dyes				
	Pomegranate peel	***Nutshell***	***Orange tree leaves***	***Alkanet roots***	***Dyer's Chamomile***
Bl.					
Mer.					

Bl. means the fabric has been desized, scoured and bleached
Mer. means the fabric has been desized, scoured and bleached and finally mercerized

Figure 3. Photographs of fabrics printed with various plantal extracts

For example, the hue angles (*h*°) which were related with the region in CIE Lab color space where the color existed in the color space were varied with the use of different natural dye sources. As well the colors' *a** and *b** values were differed in different based prints. In pomegranate peel and dyer's chamomile based prints, the *b** values were higher and so the colors were more in yellow shade when compared with the other prints. In nutshell based prints *a** values were comparatively high (6.9 for bleached and 6.88 for mercerized one) and *b** values were lowest among the tested printings so the obtained color is in both red and yellow shade with different amounts and the *h*° was nearly 63^{o}.

- **Fastnesses of printed fabrics**

The other important parameter for the investigation of natural based prints is the fastnesses of the fabrics. For this reason, all the fabrics differently pretreated and printed with different natural dyes were analyzed. In the study addition to the common fastness tests, different fastness properties (saliva, dry cleaning) of the samples were analyzed too.

The rubbing fastness which is critical for the evaluation of the binder based prints, has been found very good generally. 5 grade of dry rubbing fastness has been achieved in all cases, in wet state the fastnesses were good as well but not 5 grades have been achieved in all studied cases but still the values were very good. Minimum 4 grades were obtained in wet rubbing fastness in printing with pomegranate peel extract on bleached cotton. In terms of washing fastness staining on cotton was found very limited but color change after washings were observed in some prints. The excellent washing fastness values in terms of color change was observed from the samples printed with pomegranate peel and nutshell extracts but in alkanet roots and dyer's chamomile based prints the color change grade was limited and it was nearly 3. Likewise in fastnesses to acidic and alkaline perspiration in terms of staining on cotton was found 4/5 and 5 that means these printed fabrics had excellent fastnesses in terms of staining on cotton during acidic and alkaline perspiration. However except pomegranate peel and nutshell based prints, the color change during the perspiration fastness test was observed limited as seen from the Table 4. The interesting thing should be examined here was that the mercerization after bleaching prior to printing caused a slight decrease in fastnesses to washing and perspiration in terms of color change of the fabrics.

The other tested fastness was the saliva fastness of the printed fabrics. As told before, today the demand on natural based products is increasing and the people is more aware of the selection of goods especially for their children. In this respect these fabrics printed with the use of natural sources were tested against saliva test to show the usability for child and baby garments. It was observed that excellent fastnesses were obtained against both acidic and alkaline saliva. Except alkanet root based prints, in which the fastness values were 4-5, the fastnesses were all 5 grade.

Dry cleaning fastness of the fabrics was also tested. It was found that the dry cleaning fastness in terms of color change was 5 grades in all tested printed fabrics. In terms of staining on cotton, 5 grades were also obtained from the fabrics printed with pomegranate peel, nutshell, alkanet roots and orange tree leaves however in the printing of dyer's chamomile 4 grades which is also a good fastness value was observed.

Table 4. Fastnesses of fabrics printed with various plantal extracts

		Rubbing		Washing		Perspiration				Saliva		Dry Cleaning		Light
						Acidic		Alkaline						
Natural Dyes	**Processes**	**Wet**	**Dry**	**Sta.[a]**	**C.C.[b]**	**Sta.[a]**	**C.C.[b]**	**Sta.[a]**	**C.C.[b]**	**Sta.[c]**	**C.C.[b]**	**Sta.[a]**	**C.C.[b]**	
Pomegranate peel	***Bl.***	4	5	5	5	4/5	4/5	4/5	4/5	5	5	5	5	6
	Mer.	4/5	5	5	5	4/5	4	4/5	3/4	5	5	5	5	6
Nutshell	***Bl.***	5	5	5	5	5	5	5	5	5	5	5	5	2
	Mer.	4/5	5	5	5	5	5	5	5	5	5	5	5	2
Orange tree leaves	***Bl.***	5	5	5	4	5	3	5	3/4	5	5	4	5	4
	Mer.	5	5	5	3	5	3	5	3	5	5	5	5	5
Alkanet roots	***Bl.***	5	5	5	3	4/5	3	5	3	4/5	4/5	5	5	5
	Mer.	5	5	5	2/3	5	2/3	5	2/3	4/5	4/5	5	5	6
Dyer's Chamomile	***Bl.***	4/5	5	4/5	3	4/5	3	4/5	3/4	5	5	4	5	5/6
	Mer.	5	5	4/5	3	4/5	3	4/5	3	5	5	4	5	6

a. Staining on cotton b. Color Change c. Staining on filter paper

Light fastness is the other important fastness should be analyzed to shape the usability of a dyestuff and dyeing process for a fiber. As a result of this, the printed fabrics' light fastnesses were tested. It was observed that except nutshell based prints sufficient light fastness values were obtained. It is known that there is a close relationship between the chemical structure of a dye and its light fastness and addition to it; the inherent photo stability of the dye molecule, the concentration of the dye, the nature of the fiber, the wavelength distribution of the incident radiation and the composition of the atmosphere are the other parameter can effect the light fastness (30). So the differences in fastness of the fabrics could be related with the chemical structure of dye extract and the dye concentration transferred to the fiber since the other parameters are all same for the printed fabrics. The pomegranate peel and dyer's chamomile based prints gave the higher light fastness values (5/6 and 6) in which the higher color efficiencies were obtained (Fig.2). However the nutshell based printed fabrics were exhibited the lowest color efficiencies and light fastness grades. Moreover the mercerization process had been generally caused better light fastness grade than the bleached and printed fabrics. As told in Fig.2 and Table 3 this process has caused higher color efficiencies with higher saturation and darker shades. So it can be told that the obtained light fastness values were particularly related with the dye concentration on the fabric as well the chemical structure of the natural dye.

4. CONCLUSIONS

In this study the usability of pomegranate peel, nutshell, orange tree leaves, alkanet roots and dyer's chamomile as natural dyes in cotton printing has been investigated. For printing of the extracts pigment printing method has been carried out. On the other hand, the effect of pretreatment on the printability of cotton fabrics was analyzed. It was observed that different colors can be obtained with the use of these vegetative extract by printing without use of any mordanting process. Moreover the mercerization process was found important in terms of the color shades and color efficiencies. The mercerized and then printed fabrics displayed higher color efficiencies and darker shades as expected.

Several fastnesses of the prints were examined and pomegranate peel based printed fabrics showed the best fastnesses in all tested standards. Beside this, the tested fabrics showed sufficient fastnesses in general. Fastness of the prints has been found somewhat related with the mercerization process. In some natural dyes, mercerization cause decreases in washing and perspiration fastnesses and increases in light fastnesses but these changes were limited.

Consequently, the specificity and the main findings of this study can be summarized as;

- Introduction the usability of pomegranate peel, nutshell, orange tree leaves, alkanet roots and dyer's chamomile in printing of cotton fabrics with binder.
- Introduction the effect of different pretreatments on natural dye based printings of cotton fabrics.
- Beyond the common fastness tests, the saliva and dry cleaning fastnesses, which are rarely used, were also determined for the natural dye based prints.

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