



Evaluation And Optimization of Seidlitzia Rosmarinus (Ashnan) Extract in Washing Historical Textiles

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ABSTRACT

Contamination is an unwanted threat, and always affects the health and artistic features of a piece of textile. One of the most important concerns in the arena of the conservation of historical woven fabrics is the presentation and use of safe materials and methods for the historic works and conservators. So far, many researchers have been working on this field and have caused a lot of changes. The purpose of this research is to apply Ashnan extract to the cleaning and washing of historical cotton fabrics. For this purpose, the plant was collected from the Aqdah deserts in Ardakan, Yazd, and was extracted in hydroalcoholic medium using Soxhlet method. The extract was applied to the prepared contaminated samples in the washing step as detergent. In the washing process, all constant independent variables were concerned constant and only three factors such as concentrations of extract, washing frequency and times were considered in the design of the test using a central composite design (CCD). The detection of delta DE* by spectrophotometer as a dependent variable expresses the effect of extract cleansing rate. The results show that extract concentrations, washing frequency and time are most effective in cleaning the contamination, respectively. However, the adopted washing process and the applied materials had the least impact on reducing the strength of the fabric.

Key Words: Ashnan, Seidlitzia, historical textiles, clearing, washing, cotton, CCD

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INTRODUCTION

Ashnan is a kind of short shrub, 60 cm to 1.5m in height [1]. This plant is known as the "Seidlitzia" in honor of Sultys [2]. The Seidlitzia is from the Salsoleae tribe and from the Spirolobeae family and the Chenopodiaceae spinena. Ashnan is resistant to drought and are often found in saline and alkaline areas of deserts and salt-marsh in the Dasht-e-Kavir, and Kavir-e-Lut as an adaptive species. This plant has

spiral embryo, horizontal seed, shoot strap, fluffy round the winged fruit, and the flower has 5 flora and its fluffy has 5 pieces in Ashnan which is the important plant [1]. *S. rosmarinus*, plant, which is called Ashnan or Ashnun in general language, is spread in the saline areas of Iran and the best way to replicate it is through seeds [3]. Nowadays, the ash of this plant is a source of alkaline materials (known as Ghalyab or Kalyab in Iran) and it is used in soap and detergent industries.

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The ash of Ashnan has a lot of sodium and potassium carbonate. These carbonates are also present in ashes of plant material are rich in organic salts. Nowadays, 26 tones of alkaline (Ghalyab) are produced annually from Ashnan saline soil in Qom province in Iran [4]. The annual rainfall is generally less than 100 mm in the area of Ashanan. In contrast, the level of underground water reservoir in the lands located in the areas of Ashnan is high. So this kind of plant gets part of its water through the high humidity of the soil under the influence of underground water reservoir. Soil salinity and the level of the groundwater are two of the most prominent land characteristics for growing Ashanan [5]. Despite the large application of this plant in the past for washing textiles, today in Iran, one of the origins of Ashnan lack of attention is given it.

Literature review

The presence of dirt in the long run will attract pollutant and dust, while distorting the visual beauty of the work and covering the information on textiles such as the color and texture, it also results in localized hardening and leads to break the fibers [6]. Given that the cleaning step is irreversible, different methods are presented and used. The washing or wet cleaning is one of the applicable methods which is used according to the conditions and the type of contamination that is not met by other methods [6]. washing leads to the reduction of dirt, fiber tension free, removal of wrinkle and shiny dye cloth [7]. If the fabrics are contaminated with fat, the use of detergent will be necessary [8]. Bleaching solutions that are oxidants or chlorinated compounds reduce the fabric's strength, as well as a hot, dilute, or cooled acid solutions decompose the cotton [9].

Common detergents do not have all of the above features, but are powerful and valuable in some special fields. For example, it may sound good in terms of cleaning power, but in other respects this is not desirable. According to researchers, the use of Synpronc N is prohibited in Sweden, due to its non-degradability [10]. It is still used as a detergent in today's clearing of historical textiles [7]. Nonylphenols are introduced as estrogenic compounds that endangers conservators health, which has been evaluated in reports the risks of its use and the problems associated with the absorption of these substances [11] and the dangers of using it have been evaluated [12]. "Embree" suggested the use of soap tree (Saponin) for washing [13]. Soaked soap herb, called 'radicula' was used for cleaning the wool by the ancient Romans and Greeks (Cam, 1983). A report on the use of Saponin to wash silk was presented [14] and some experiments with Saponin DAB9 based on Quillaja saponaria and Saponin's roots were performed. This is a non-ion material that reduces water surface tension from $73 \frac{mN}{m}$ to $20 \frac{mN}{m}$ in $20^{\circ}C$ with $1/5 \frac{gr}{Lit}$ [15]. Saponins are classified as steroids and Tri-tryptone. In these cases, water-loving saccharides (glucose, galactose, rhamnose, exiliosis, pentose, etc.)

are attached to water- soluble saccharides and triterpenes. The new method presented for cleaning the historic fabric in which CO_2 is used as a solvent. In this method, initially the fabric is wetted with the aid of solvent and then placed in a CO_2 container. These conditions for various colored and raw cotton and silk samples were taken that showed 53% to 97% clearing. [18], but the effects of its alkaline environment and its impact during time have not been reported. One of the other results is that washing conditions are better in the lower pH (near neutral) and buffer does not have positive effect on washing oprations. "Azadi et al" have studied on the use and optimizing the mixture of Ashanan and Arabic gum as a natural detergent in cleaning up textiles and demonstrated the effect of these two biodegradation agents on cleaning the contaminated cotton fabric, as well as the best washing conditions at ambient temperature of 30 minutes, which is important both for the conservator and artifact [16]. A researcher suggested that either the whole or part of the foams can be substituted in shampoo formulation with natural Saponins

METHODOLOGY

This paper is a scientific and applied research. The The data were analyzed through historical and descriptive studies, content analysis, and experimental method. For the purpose of sample preparation, a cloth with a cotton cellulose structure is selected. But before any action, for removing the possible contamination and increase the hydrophilic property, the fabric was washed in a bath with content of 0.5 grams per liter anionic detergent and 1 g / liter of NaOH for 30 minutes at $75^{\circ}C$ and then rinsed and samples were dried under dry conditions.

Table 1. The characteristics of a cloth

Kind of swirling weft	Kind of swirling warp	Weight	Warp type	Weft type	Tread grade weft	Tread grade warp	Weft Density	Warp Density	Texture
z	z	90 g/m	cotton	cotton	30nc	40Nc	22.5	33	Taffeta

To produce the contamination the compositions of table 2 was used. After preparing the material, to uniformize the particle size, these materials (from Mesh 200) were passed through a sieve and then, according to the magnitude of tables 2 and 3, they were completely mixed to achieve black and greasy powder.

Table 2. Historical dirt compound

Materials	Percentage distribution (%)
Swollen moss	38
Cement	18
Kaolin Clay	18
Silica	18
Mineral oil (Nujol)	6.2
Furnace Black	1.5
Red oxide iron	0.3
Total	100



The fabric was contaminated with the same and equal amount of contamination by silkscreen printing technique. After undergoing the accelerated ageing tests, the samples were cut into 10× 10 cm and prepared for testing.

Extraction

The degreasing Ashnan powder was extracted in a Soxhlet apparatus with Methanol in 600 minute time intervals. The obtained Methanolic extract was filtered by Wattman paper number 1 and then with the use of rotary evaporator (STEROGLASS Model 202/102, Italy), the solvent was removed under vacuum and the extract was condensed at 70oC until it has reached a relatively viscous liquid with the brown color. The condensed methanolic extract was dissolved in distilled water twice. Then with ethyl acetate solution, which was previously saturated with distilled water, mixed in a separatory funnel and separated by aqueous solution. Aqueous solution was mixed with butanol in several steps and butanaytic phase was separated in the separatory funnel. The obtained butanyl phase in drying condensed conditions and finally the existing Saponin was deposited and separated by using 5 times the volume of ethylene ether [17]. The obtained material is collected in a completely dry condition and kept in the containers with lid in cool place for further studies.

FINDINGS

Design

The range of independent variables is determined during initial tests. Tests were based on Central Composite Design (CCD) and factors of washing times, time and concentration of Ashnan extracts independent variables and delta DE* as an associated variable were considered in which independent variables, according to the preliminary tests are defined in table 3.

Table 3. Range of variables in the experiment based on CCD

Factors	min	max
Time/min	5	30
Repeat washing	1	3
Concentration of extract solution	0.1	0.5

For the washing process, the treated samples which are encoded, were soaked in distilled water for up to 15 seconds and transferred to the corresponding containers. In this situation, creating motions that are rotated and reversed with slow and steady speed by Rossari Labtech Model pc-03, variable speed (2010 India) was given to the dishes, only the washing solution and samples were moving and the least practical mechanical operation was applied to the samples at ambient temperature. All samples were tested according to the test design and factors such as time, repeated washing, and the amount of detergent. After washing, the samples was rinsed in order to rinse into three baths, containing distilled water and

rinse with gentle motion. In the following the sample was then placed between several layers of paper towels and placed on them a weight of one kilogram for a minute. In the last step, in order to follow the principles of washing and completing, samples were dried with a Black & Decker hair dryer (model px5, made in England 2007) with cool air and medium speed at a distance of 20 cm.

Spectrophotometer

In order to evaluate the efficiency of the conditions and the degree of cleanliness, the amount of differences (DE*), by the reflective spectrophotometer made in Germany, which is repeated three times and was measured according to the following equation (1).

$$DE^* = (\Delta a^* + \Delta b^* + \Delta l^*) 1/2 \tag{1}$$

DE* is delta values for wash detergent for the used samples in different detergents with repeated washing times shown in table 4.

Table 4. Value of DE* (Delta) based on the designed conditions with Ashnon

Run	Tim/min	Repeat	Extract %	DE*
1	26	1	0.4	4.262
2	30	2	0.3	5.452
3	18	2	0.3	3.456
4	26	3	0.2	5.907
5	18	2	0.1	4.037
6	18	2	0.3	4.376
7	18	3	0.3	7.271
8	9	3	0.4	6.620
9	18	2	0.3	5.101
10	5	2	0.3	4.266
11	18	2	0.5	4.774
12	18	1	0.3	3.716
13	9	1	0.2	3.394

Figure 1 shows the curve of DE response based on variables of time and frequency of repeated washing:

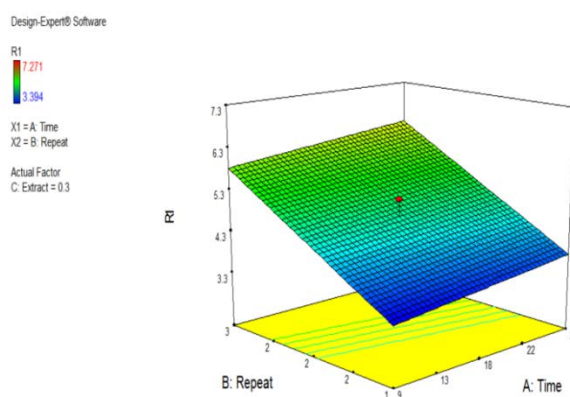


Figure 1. 3 D curve, shows the DE* value based on the variations of two variables, percentage of time and frequency of washing.



In this image, the values of the extract percentage variable were considered as 0.3% steadily. This curve shows the high effect of the frequency of repetitions of washing to the time in increasing DE*.

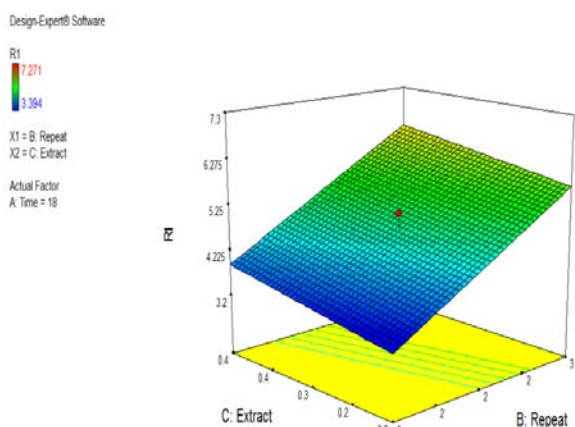


Figure 2. Curve of DE* response based on variables of extract and frequency of repeated washing

The figure 2, shows the 3D curve, value of DE* based on the variations of the two variables, the percentage of extract and the frequency of repeated washing. In this image the values of the time variable are set to 18 minute steadily. This curve indicates the significant effect of the frequency of washing repetitions variable on the percentage of extract in increasing DE*.

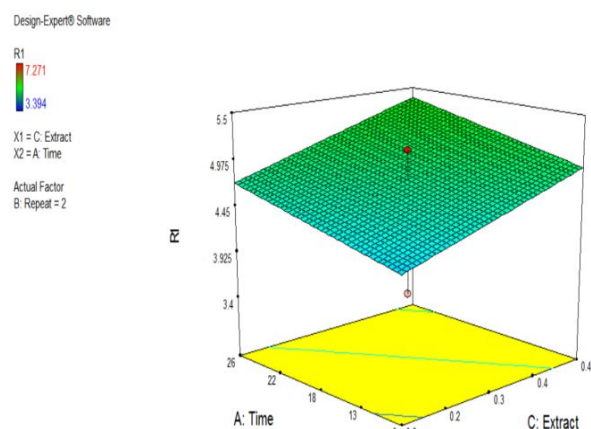


Figure 3. Curve of DE response based on variables of extract and time

In figure 3, 3D curve, the value DE* is indicated based on the variation of two variables, the percentage of time and percentage of the extract. In this image, the variable values of the frequency of repeated washing are considered constant twice. This curve expresses the effect of both variables, the percentage of extract and time in increasing DE*. By statistical analysis (ANOVA), the obtained significance level of the equations is shown in the table 5.

Table 5. Analysis of variance for reproduction level by activated cotton samples with Ashnan extract

Source	Sum of Squares	df	Mean Square	F Value	P-value Prob> F	
Model	13.53	3	4.51	10.34	0.0028	Significant
A-Time	0.42	1	0.42	0.96	0.3522	
B-Repeat	12.25	1	12.25	28.10	0.0005	
C-Extract	0.86	1	0.86	1.97	0.193	
Residual	3.92	9	0.44			
Lack of Fit	2.56	7	0.37	0.54	0.7744	Not significant
Pure Error	1.36	2	0.68			
Total	17.45	12				

To ensure the evaluation of strength of the sample after the washing process, the sample is checked physically.

DISCUSSION AND CONCLUSION

Analysis of variance (ANOVA) and the quadratic model for DE*:

The 26 washing processes in 13 steps according to the central compound design was carried out by taking into account time, repeat washing and concentration of the extract variables for the cotton sample presented in table 4. This design includes three variables such as time, rinse repetition and extract concentration, as well as response variables including DE* values for treated cotton samples. Analysis of variances for each response level was done by taking into account certain levels (table 5). If the P-value (the probability of a predetermined value of P) (F> F0) for a quadratic model is less than 0.1, 0.05 and 0.001, then the resulting model percentage by using these levels will be 10%, 5% and 1% respectively is significant. The relationship between the variables and the response is based on three independent variables for the obtained values. This model is shown in equation 2:

$$DE^* = 0.16913 + 0.025912 \text{ Time} + 1.74983 \text{ Repeat} + 2.31867 \text{ Extract} \quad (2)$$

In order to obtain the model DE*, for samples that have been tested with Ashnan, F-value is equal to 10.34, in this case according to the analysis of variance, the resulting model is at a level of 1% with the significance level of 99% in the desired range (table 5). The P-value is 0.0028 or it is lower than 0.05. The results were obtained by using the RSMS (Response Surface Methodology) statistical method. The statistical model was optimized so that the efficiency of the washing operation is related to quadratic equations for repeat washing, time and concentration of the extract factors.



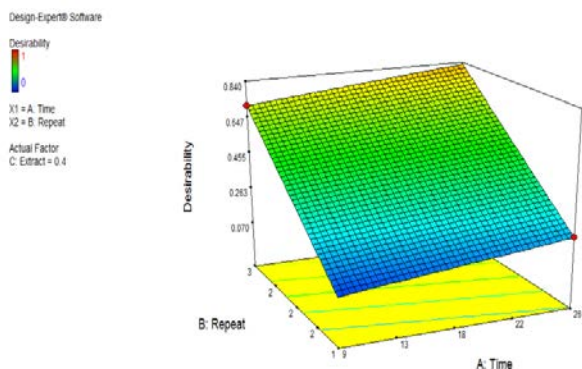


Figure 4. Curve response procedure of optimal values of DE

According to obtained results in CCD and the use of (Design-Expert software) and statistical analysis, the conditions (DE*) based on three variables of time, repeat washing and concentration of the extract, are calculated, and favorable results are shown. In this case, the highest response rate is obtained in relation to the quantity and type of each variable. Table 6 shows the best optimal state of DE* with the extract of Ashnan.

Table 6. Optimum conditions for DE response with Ashnan extract

Time	Repeat	Extract	DE*	Desirability
26	3	0.4	6.61199	0.830

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