

Evaluation and Optimization of Acanthophyllum Extract in Washing of the Historical Textiles

Mohammad Mahdi Karim-Nejad^a, Mehrnaz Azadi Boyaghchi^{b*}, Behzad Zolfaghari^c

^aConservation PhD Scholar, Isfahan Art University, Iran and Faculty Member of Department of Conservation and Restoration, Yazd Branch, Islamic Azad University, Yazd, Iran

^bAssistant Professor at Conservation Department, Conservation Faculty, Art University of Isfahan, Isfahan, Iran

^cDepartment of Pharmacognosy, Faculty of Pharmacy and Pharmaceutical Sciences, Isfahan University of Medical Sciences, Isfahan, Iran

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Abstract

Contamination is an unwanted threat, which affects the health and the artistic features of a piece of textile. One of the most important concerns in the area of protection of historical woven fabrics is the presentation and use of safe materials and methods for the historic works. So far, many researchers have been working on this field and have caused a lot of changes in this field. The purpose of this research is to apply Acanthophyllum extract to the cleaning and washing of historical cotton fabrics. For this purpose, after preparing the Acanthophyllum, using the Soxhlet method, the extract of hydro-alcoholic was obtained. The extract was applied, as a detergent, to the prepared contaminated samples in the washing step. In the washing process, all constant independent variables were concerned, and only three 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. However, the adopted washing process and the applied materials had the least impact on reducing the strength of the fabric.

Keywords: Acanthophyllum; Historical Textile; Clearing; Washing; Cotton; CCD

* Corresponding author. Tel: +98-9395783562.

E-mail address: mehrnaz_azadi@yahoo.com.

1. Introduction

Acanthophyllum is a shrub belonging to the clove family and the *Acanthaphillum* species. There are 61 species of this genus in the world, of which 33 species can grow in Iran, and 23 species are native to this region (Ghaffari, 2004). Based on available sources, most of these species are identified in eastern parts of Iran (Khorasan province) and its adjacent regions (Afghanistan and Turkmenistan) (Schiman, 1988). The lower parts of the plant are entirely woody with white flowers, the length of the plant is 20-25, and the petals 5 cm, with white tip and red bottom (Ghahraman, 1990). The root of *Acanthophyllum* plant is a source of Saponin compounds, which are the most important and active compounds (Mir-heyder, 1996); and effective in reducing surface tension and on chemical detergents. Most of the research was carried out with the focus on identifying the structure and determining the physicochemical and biological characteristics of this plant (Gaidi, 2004; Lacaille, 1993). On the other hand, the plant has features such as affordability, accessibility, low and natural complications and bio-degradability (Azadbakht, 2005). Despite the plant's large application in the past for washing textiles, today in Iran, not enough attention is given to the challenges of the *Acanthophyllum*.

2. Literature Review

The presence of dirt in the long run will attract pollutants and dust, while distorting the visual beauty of the work and covering the details of textiles, such as the color and texture, and it also results in localized hardening and leads to breaking of the fibers (Eastop, 2005). Given that the cleansing step is irreversible, different methods are presented and used. The flushing is one of the methods applicable to the conditions and the type of contamination (Eastop, 2005). Flushing leads to the reduction of dirt and free flowing stress, wrinkle removal and dye cloth (Dash Harby et al., 2001). If the fabrics are contaminated with fat, the use of detergent is necessary (Plenderleith & Werner, 1971). Bleaching solutions that are oxidants or chlorinated compounds reduce the fabric's strength, and hot, dilute, or cooled acid solutions decompose the cotton (Haji Sharifi, 2009).

Common detergents do not have all the above features, but are powerful in some special fields. For example, they may sound good in terms of the cleaning power, but in other respects, they are not desirable. According to researchers, the use of Synpronin N in Sweden is prohibited, due to the fact that they are not decomposable (Dash Gentle & Muller, 1995). It is still used as a detergent in today's clearing of textiles (Harby, 2011). Nonylphenols are introduced as estrogenic compounds that jeopardize the health of the preservative. Reports have been provided of problems associated with absorption (Moda Hayward & Allen, 1992) and the dangers of using it have been evaluated (Weeks, 1996). "Embree" suggested the use of soap tree (Saponin) for washing (Embree, 1995). A 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 (Shahoua, 1990) 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 to 20 in 20°C with 1/5 Gr Lit (Czerwinski, 1997). Saponins are classified as steroids and Tri-tryptone. In these cases, water-soluble saccharides (glucose, galactose, rhamnose, exiliosis, pentose, etc.) are attached to water-saturated and tertiary terphenic saccharides (Tisuji, 1998).

A new method was presented for cleaning the historic fabric, in which CO₂ is used as a solvent. In this method, initially the fabric is wetted with the aid of solvent and then placed in a CO₂ container. In these conditions, various colored and raw cotton and silk samples showed 53% to 97% clearing. (Costas Panayiotou et al., 2013), but the effects of its alkaline environment and its impact

have not been reported. One of the other results is that washing conditions are better in the lower pH (near neutral) and that buffer for operation wash has a positive effect (Antje Potthast et al., 2011). "Azadi et al" have studied the use and optimization of the mixture of *Acanthophyllum* and Arabic gum as a natural detergent in cleaning up textiles and demonstrated the effect of this biodegradation biomass on purifying the contaminated fabric, as well as the best washing conditions at ambient temperature and in 30 minutes, which is important both for protection and work (Azadi et al., 2013). A researcher suggested that either the whole or part of the foams can be substituted in shampoo formulation with natural Saponins (Aghel, 2007).

3. Methodology

This paper is a scientific and applied research. For obtainment of the information, historical and descriptive method, content analysis, and experimental method were used. For the purpose of sample preparation, a cloth with cotton cellulose structure was selected. But before any action was taken, for removing the possible contamination and increasing the hydrophilic property, the fabric was washed in a bath with a content of 0.5 grams per liter anionic and 1 g / l of oil for 30 minutes at 75°C, and then rinsed and lastly, samples were dried under dry conditions.

Table 1 Characteristics of a cloth

Kind of swirling (Weft)	Kind of swirling (Warp)	Weight	Stuff (Weft)	Stuff (Warp)	Tread grade (Weft)	Tread grade (Warp)	Density (Weft)	Density (Warp)	Texture
z	z	90 g/m	cotton	cotton	30Ne	40Ne	22.5	33	Taffeta

To produce the contamination, the composition 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

Material	Percent
Peat moss	۳۸
Cement	۱۸
Kaolin soil	۱۸
Silica	۱۸
Mineral oil	۶,۲
Red oxide iron	5.1
Soot	3.0
Total	۱۰۰

The fabric is contaminated with the same amount of contamination by silk screen method. After undergoing the rapid ageing steps, the samples were cut in 10×10 cm and prepared for testing.

3.1. Extraction

The degradation of *Acanthophyllum* powder was extracted by Soxhlet device, with Methanol at 600-minute intervals. The obtained Methanolic, extract by Wattman paper number 1, was filtered, and later with the use of condenser spinning under vacuum (STEROGLASS Model 202/102, Italy),

the solvent was removed and the extract was condensed at 70°C until it had turned into a relatively viscous liquid with the brown color. The condensed methanolic extract was dissolved in distilled water twice. Then, with ethyl acetate solution, which had been previously saturated twice with distilled water, was mixed in a decantation funnel and separated by aqueous solution. Aqueous solution was mixed with butanol in several steps and extracted in butanaytic phase decantation funnel. The obtained butanyl phase in drying condensed conditions and finally Saponin was deposited and depleted by using 5 times the amount of ethylene ether (Moghimi Pour & Khalili, 2007; Aghel et al., 2007). The obtained material was collected in a completely dry condition and kept in containers with doors in a cool place for further studies.

4. Findings

4.1. Extraction

Afterwards, the range of independent variables was determined during initial tests. Tests based on Central Composite Design (CCD) and factors of washing times, time and concentration of *Acanthophyllum* extract as independent variables, as well as delta DE* as an associated variable were considered. The 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 wash	1	3
Concentration of extract	0.1	0.5

For the rinse process of the samples which are coded, they were soaked in distilled water twice, for up to 15 seconds, and then 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 (Sacht 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 design and factors such as washing time and the amount of detergent. After the washing step, the sample for rinsing was twice transferred to three baths containing distilled water and gently rinsed. The sample was continuously put between a multi-layer paper towels and pressed with 1-kilogram weight for a minute. At the last step, in order to follow the principles of washing and completing, samples were dried with a Black & Decker hair dryer (model px5 Sachs England 2007) with cool air and medium speed from a distance of 20 cm.

4.2. Spectrophotometer

To evaluate the performance and the degree of cleaning, the amount of color correction (DE*) was measured three times, by a German reflective spectrophotometer, according to equation (1).

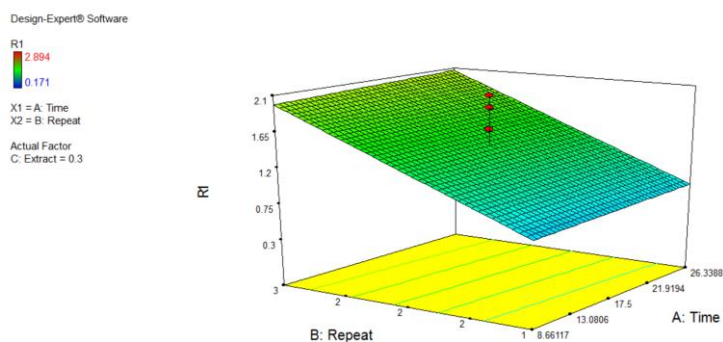
$$DE^* = (\Delta a^* + \Delta b^* + \Delta l^*) / 2 \quad (1)$$

DE* is the 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 Acanthophyllum

Run	Tim/min	Repeat	Extract %	DE*
1	26	1	0.4	1.329
2	30	2	0.3	1.404
3	18	2	0.3	2.041
4	26	3	0.2	1.527
5	18	2	0.1	0.266
6	18	2	0.3	1.622
7	18	3	0.3	1.747
8	9	3	0.4	2.894
9	18	2	0.3	1.894
10	5	2	0.3	1.037
11	18	2	0.5	2.085
12	18	1	0.3	0.324
13	9	1	0.2	0.171

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


Fig 1 3D 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 are considered as 0.3% steadily. This curve shows the high effect of the frequency of repetitions of washing to the time in increasing DE*.

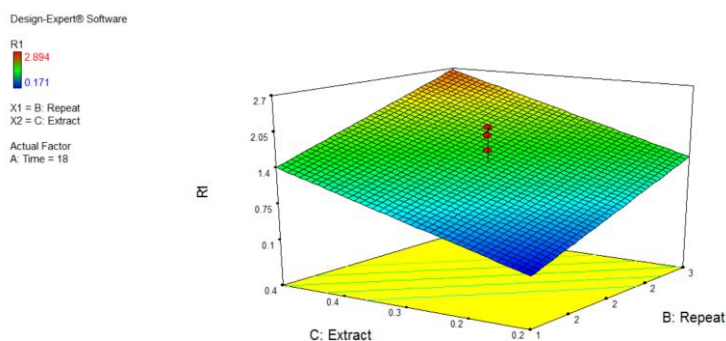

Fig 2 Curve of DE* response based on variables of extract and frequency of repeated washing

Fig 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 on the percentage of extract in increasing DE^* .

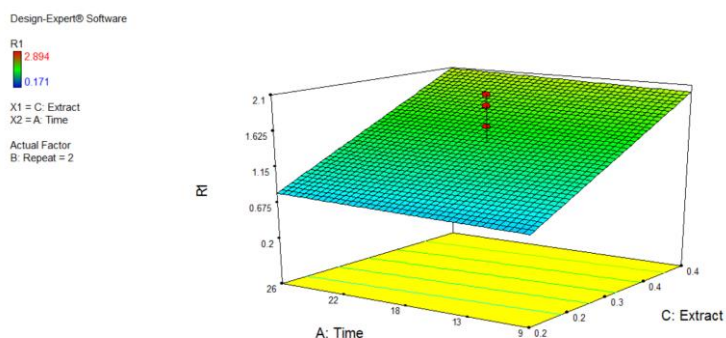


Fig 3 Curve of DE response based on variables of extract and time

In fig 3, 3D curve, the value DE^* is indicated based on the variation of two variables, the percentage of time and that of the extract. In this image, the variable values of the frequency of repeated washing are considered twice. This curve expresses the effect of both variables, the percentage of extract and time in increasing DE^* .

Via statistical analysis (ANOVA), the significance level of the equations was obtained, and is shown in table 5.

Table 5 Analysis of variance for reproduction level by activated cotton samples with *Acanthophyllum* extract

Source	Sum of Squares	df	Mean Square	F Value	P-value Prob>F	
Model	6.30	3	2.10	14.23	0.0009	Significant
A-Time	0.012	1	0.012	0.081	0.7819	
B-Repeat	3.04	1	3.04	20.60	0.0014	
C-Extract	3.25	1	3.25	21.99	0.0011	
Residual	1.33	9	0.15			
Lack of Fit	1.24	7	0.18	3.92	0.2185	Not significant
Pure Error	0.090	2	0.045			
Total	7.63	12				

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

5. 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 of time, rinse repetition and extract concentration, as well as response variables, including DE^* values on sample. 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) ($F > F_0$) 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% significant, respectively. 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:

$$DE^* = -1.7711 \times 10^{-3} E - 300 \text{ Time} + 0.87211 \text{ Repeat} + 4.50556 \text{ Extract} \quad (2)$$

In order to obtain the model DE^* , for samples that have been tested with *Acanthophyllum* extract, F-value is equal to 14.23, 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 0.0009 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 was related to repeat washing, time and concentration of the extract factors.

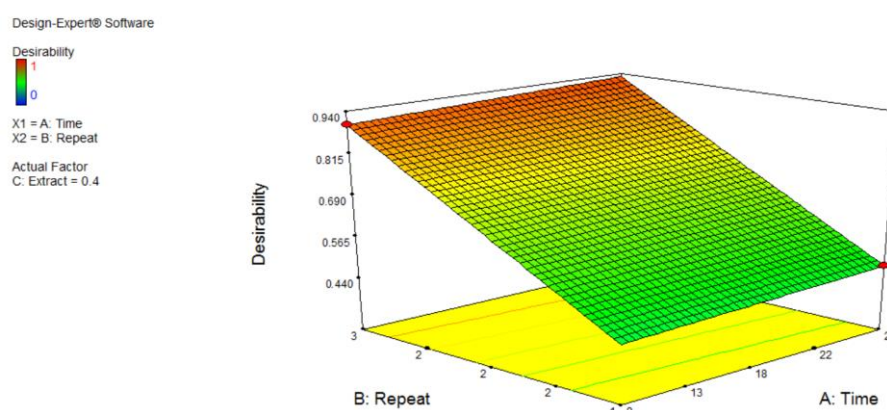


Fig 4 Curve response surface optimal values

According to obtained results in CCD and the use of Design-Expert Software and statistical analysis, the conditions (DEA) for three variables of time, repeat washing and dipping extract were calculated, and desirable results were shown. In this situation, in terms of quantity and type, each variable has the highest response value. Table 6 shows the best optimal state of DE^* with the extract of *Acanthophyllum*.

Table 6 Optimum conditions for DE response with *Acanthophyllum* extract

Time	Repeat	Extract	DE*	Desirability
26	3	0.4	6.61199	0.830

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