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POTENTIAL USE OF BIO-DYES FOR GREEN COLORING OF MEDICAL TEXTILES

COCÎRLEA Maria Denisa[1], COMAN Andrei[2], POPOVICI Lucia-Florina[1], OANCEA Simona[1], COMAN Diana[3*]

[1] "Lucian Blaga" University of Sibiu, Faculty of Agricultural Sciences, Food Industry and Environmental Protection, 550012, 7-9 Dr. I. Ratiu Street, Sibiu, Romania, E-mail: denisa.cocirlea@ulbsibiu.ro, luciaflorina.popovici@ulbsibiu.ro, simona.oancea@ulbsibiu.ro

[2] Medical Practice Comosan SRL, Sibiu, Romania, E-mail: dr.comanandrei@gmail.com

[3] "Lucian Blaga" University of Sibiu, Romania, Faculty of Engineering, 550024, 10 Victoriei Blvd, Sibiu, Romania, E-mail: diana.coman@ulbsibiu.ro

Corresponding author: Coman Diana, E-mail: diana.coman@ulbsibiu.ro

Abstract: *Cotton is a textile material frequently used in the medical system. The possibility of using natural extracts for coloring biotextiles is a current way of giving sustainability attributes to articles used in medicine. The influence of five different classical mordants and biomordants (citric acid, tannic acid, ferrous sulfate II, copper sulfate and ferrous sulfate with oxalic acid), by meta-mordanting and dyeing by exhaustion and sonication methods with Rhus typhina L. fruit extract, was evaluated. The investigation was carried out by measuring the chromatic coordinates, water and dry rubbing resistance, as well as measuring the FT-IR absorption spectra. The mixture between ferrous sulfate and 4% oxalic acid produced, according to the ATR-FTIR analysis, the greatest changes in the cotton structure, but significantly reduces the color changes resulting from the simple use of sulfate. The promising obtained results such as low color changes when using biomordants, good resistance to water and friction, encourage to continue the research regarding the application of this extract in the friendly technology of cotton dyeing.*

Key words: *cotton fabrics, Rhus typhina L., biomordant, exhaustation, ultrasonication, ATR-FTIR*

1. INTRODUCTION

In recent years there has been an increasing interest in textile dyes obtained from natural resources, which present a significantly lower risk for human health and the environment than synthetic dyes [1].

In 2023, Karadag [1] proposed the introduction of a new standard named the Natural Organic Dye Standard (NODS) for naturally dyed textiles, which also comprised a list of dye plants among which the woody species *Rhus typhina* L. was included. *R. typhina* or staghorn-sumac is an alien species found in the flora of Romania, which was introduced as an ornamental species [2]. Its bright red fruits are rich in antioxidant and antimicrobial compounds such as vitamin C [3], flavonoids, phenols and anthocyanins [4]. The fruits of different sumac species were previously used in a mixture with other tinctorial plants to obtain a red dye [5], but the staghorn-sumac fruit itself would produce a crimson dye [6]. Moreover, tannins identified in the leaves make them a good alternative for fixing pigments in dyed fabrics [7].

In order to eliminate the release from textile supports of some toxic compounds on the skin with exposure to allergies or other dermatological conditions, attemps are made to use natural dyes for medical articles as well. The scientific world has become more and more captivated by the use of plant extracts to obtain textile materials with antimicrobial properties [8]. According to the study of Secareanu et al. [9], cotton could represent an important functionalized material for medicine, with the potential to limit the spread of acne-causing bacteria [9], or much stronger infections, such as those caused by *Klebsiella pneumoniae* and *Staphyloccocus aureus* [10], this being a material frequently found in hospitals, in medical suits and bed sheets [11].

Plant-based dyeing represent a complex process defined by the characteristics of textile material, how the fabric is treated with different mordants, how the vegetable substrate is collected, processed and then extracted, but also what values are selected for the actual dyeing parameters [12, 13]. Many researches have been conducted using the eco-dyeing solution with polar solvents such as distilled water [13], ethanol [13,14] and methanol [15]. Moreover, a plant extract used as a natural dye was presented as being more valuable when it has the lowest possible content of heavy metals and a wide palette of shades obtained by varying the mordant [14]. In different studies, natural dyeing improved cotton characteristics such as the ultraviolet protection factor and the tensile strength [16].

The aim of the present study was the evaluation of two different dyeing techniques of a cotton fabric with extracts of *R. typhina* fruits, using standard mordants and biomordants, in order to identify the most effective conditions that provide a good color and resistance quality, but also of the painting during the use of the product/fabric.

EXPERIMENTAL RESEARCH

2. MATERIALS AND METHODS

2.1. Plant material and extract preparation

R. typhina fruits were collected from Popești, Vâlcea county, in November 2022. These were dried at room temperature, in the shadow, until a moisture content below 6%, then were ground and sieved through a 0.71 mm mesh sieve. Distilled water brought at 40°C was used as a solvent to obtain the coloring extract. The maceration took place at room temperature for 24 h with occasional stirring, followed by centrifugation at 8000 rpm, at 4°C (Universal 320, Hettich, Germany), for 10 min.

2.2. Analysis of anthocyanin content

The anthocyanin content of the extract was evaluated using the method of Giusti and Rodriguez-Saona (1999) [17] usig the spectrophotometer (Analytik Jena, Specord 200 Plus UV-Vis, Germany). The results were expressed as „mean ± SD", in mg/100 g cyanidin-3-O-glucoside.

2.3. Material used and dyeing of the textile support

The textile material is constituted by 100% cotton, fabric weighing 120 g/m^2, with a canvas binding, and a warp and weft density of 20 yarns/cm. The chemicals used come from the company

Scharlau S.L. and were of analytical grade, with purities over 98%. Dyeings by exhaustation and ultrasonication were done on samples of cotton fabrics, in presence and absence of biomordants (citric and tannic acids), and standard mordants (copper sulfate and iron sulfate).

For each dyeing experiment, two concentrations of mordants were used: 3% and 5%, and in addition to the mentioned solutions, a mixture of iron sulfate with oxalic acid 4% was used, related to the amount of textile material.

The exhaustation dyeing method (80°C) and sonication procedure (40°C) using the Elmasonic E Ultrasonic bath, were applied as coloring methods, each being performed for 15 min to 25:1 liquid ratio.

2.4. Chromatic characterization in CIELAB system of dyed cotton and fastness properties

The CIELAB analysis of cotton samples dyed with *Rhus typhina* L. fruit extract was realised spectrophotometrically (Datacolor 110 LAV reflection spectrophotometer) by using the Tools II Plus software, under the D65 /10^0 illumination/observer conditions, in order to evaluate the chromatic parameters: L* (luminosity), a* (redness), b* (yellowness), C* (chroma) and h (hue/tone) and calculate by their differences compared to the reference sample, also the total colour change (ΔE*).

The experimental evaluation of the color was done by comparing it with the samples dyed without mordant.

As medical articles are generally disposable, we consider their characterization by resistance of paints to water and friction. Color fastness to water and dry rubbing were performed based on specific standards [18,19], for the latter using Crockmaster equipment. The color resistance of samples was analyzed before and after meta-mordanting treatment.

2.5. ATR-FTIR analysis

The changes in the chemical structures of colored cotton were studied by attenuated total reflection-FTIR analysis, operated at a resolution of 4 cm^{-1}, performed with an ATR-FTIR spectrometer (Bruker, Germany), with the combined ZnSe ATR and QuickSnapTM modules.

3. RESULTS AND DISCUSSIONS

The total content of anthocyanins extracted from *R. typhina* fruits was 18.29±1.35 mg/100g DW.

Cotton is a textile material that is difficult to undergo natural coloring without the use of a mordant, compared to other textile materials such as silk, ferrous sulfate and copper sulfate being often used in its dyeing, while tannic acid is more considered a pretreatment mordant that increases the affinity of the material for mordants [20].

3.1 Chromatic characterization and resistance testing of dyed cellulosic support

The evaluation of color changes is carried out to appreciate the influence of the mordants used and then anticipating the various treatments to which the colored materials are subjected. The results are presented in table 1.

Figures 1-2 are suggestive for the interpretation of changes in color, brightness, hue and saturation, as well as the variation of the most common colorfastness.

Table 1: *Colour measurements and colour fastness values for exhaustion dyed cotton samples with Rhus typhina L. extract*

No.	Dyeing procedure / Mordant used	ΔL*	Δa*	Δb*	ΔC*	ΔH*	ΔE*	Water fastness	Rubbing fastness
	EXHAUSTION METHOD								
1.	Reference sample (REH)	75.86	21.46	2.31	21.59	6.14	-	2-3	3
2.	3% Citric acid (CA3)	-1.28	1.09	2.33	1.44	2.13	2.87	3-4	3-4
3.	5% Citric acid (CA5)	-1.02	0.31	1.92	0.59	1.85	2.19	4-5	4
4.	3% Tannic acid (TA3)	-0.64	-0.23	1.60	0.01	1.62	1.74	3-4	4
5.	5% Tannic acid (TA5)	-0.33	-1.96	1.15	-1.78	1.41	2.29	4	4-5
6.	3% $FeSO_4$ (iron sulfate) (IS3)	-18.52	-14.68	-6.88	-13.41	-9.11	24.61	4	4
7.	5% $FeSO_4$ (iron sulfate) (IS5)	-25.57	-16.50	`-7.94	-14.07	-11.71	31.45	4-5	5
8.	3% $CuSO_4$ (copper sulfate) (CS3)	-1.35	-2.92	3.05	-2.29	3.55	4.44	5	4-5
9.	5% $CuSO_4$ (copper sulfate) (CS5)	-1.40	-4.08	5.47	-2.55	6.33	6.97	5	4-5
10.	3% $FeSO_4$ + 4% Oxalic acid (IO3)	-1.59	-3.13	4.12	-2.16	4.71	5.42	4-5	4-5
11.	5% $FeSO_4$ + 4% Oxalic acid (IO5)	-1.02	-4.77	2.62	-4.19	3.48	5.54	4-5	4-5
ULTRASONICATION METHOD									
1.	Reference sample (RUS)	75.56	22.00	1.36	22.05	3.53	-	2-3	3
2.	3% Citric acid (CA3)	-1.04	1.54	1.37	1.65	1.23	2.31	4	3-4
3.	5% Citric acid (CA5)	-0.34	0.68	0.59	0.72	0.54	0.96	5	4
4.	3% Tannic acid (TA3)	-0.12	0.21	1.41	0.34	1.38	1.43	4-5	4
5.	5% Tannic acid (TA5)	-1.34	-1.96	0.90	-1.87	1.06	2.53	4-5	4-5
6.	3% $FeSO_4$ (iron sulfate) (IS3)	-14.18	-13.82	-4.82	-13.16	-6.41	20.38	4	4-5
7.	5% $FeSO_4$ (iron sulfate) (IS5)	-19.64	-15.77	-6.33	-14.07	-9.53	25.97	4-5	5
8.	3% $CuSO_4$ (copper sulfate) (CS3)	-1.46	-1.31	2.99	-0.90	3.13	3.57	5	4-5
9.	5% $CuSO_4$ (copper sulfate) (CS5)	-1.84	-1.77	4.09	-1.10	4.32	4.83	5	4-5
10.	3% $FeSO_4$ + 4% Oxalic acid (IO3)	-1.79	0.44	3.52	0.92	3.43	3.98	4-5	5
11.	5% $FeSO_4$ + 4% Oxalic acid (IO5)	-4.76	-7.55	-0.76	-7.58	-0.36	8.95	5	5

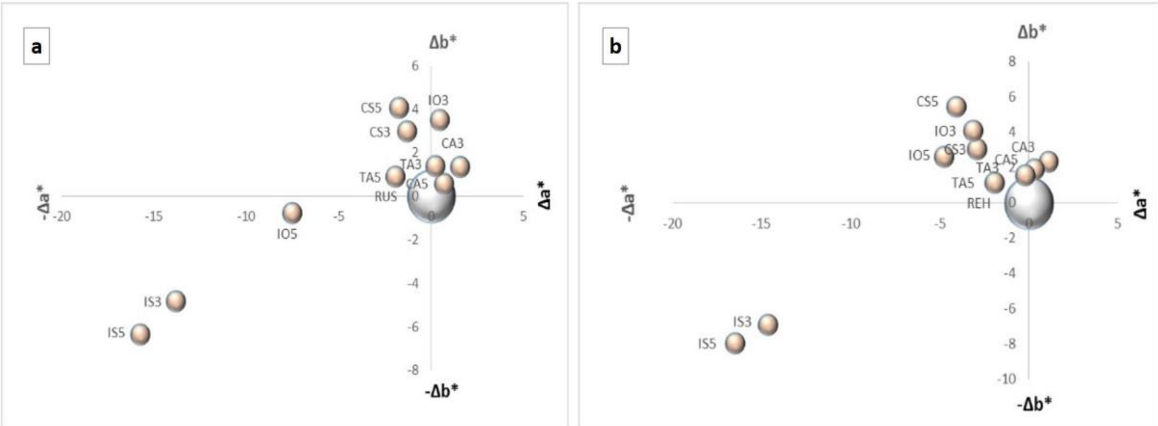

***Fig. 1**: Distribution of colour attributes of simultaneously mordanted and dyed samples, through the exhaustion procedure (a) and sonication procedure (b).*

Cotton fabrics dyed with extract changed slightly from pinkish-slightly purple by adding classic mordants or biomordants. Some differences were easily perceptible, others more sensitive, >3 units being evaluated negatively. High differences > 6 units were observed in samples treated with iron sulfate 3 and 5% and those with copper sulfate 5%. The samples treated with citric acid kept the pink-violet color with positive a* (redness) and b* (yellowness) coordinates, when dyeing by exhaustion, and even the use of tannic acid changed the hue relatively little towards pink-yellow, except for the samples colored in greenish-blue where iron sulfate was used and the samples with copper sulfate, iron sulfate and oxalic acid colored pinkish-yellow-light greenish.

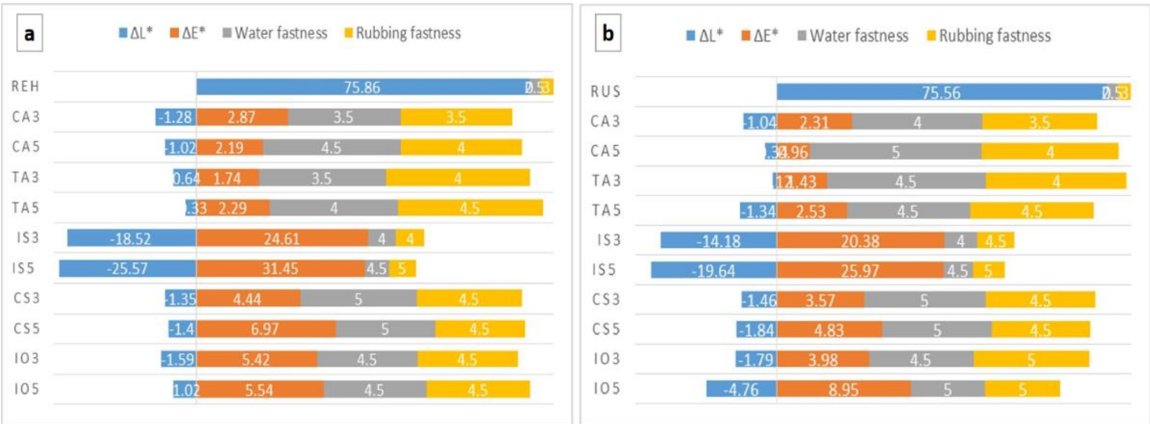

***Fig. 2**: Graphical representation of the difference in luminosity, colour and fastness values of samples dyed by the exhaustion method (a) and ultrasonication method (b).*

Compared to untreated samples, the brightness differences are reduced in case of using tannic acid, citric acid and iron sulfate+oxalic acid, at 3 and 5%, for both applied dyeing methods.
From table 1 and figure 2(a) it can be seen that the water fastness of the cotton mordanted with citric and tannic acid was slightly (1/2 unit) lower than the resistance of the samples dyed and mordanted with copper and iron sulfate, while the rubbing fastness is comparable to these mordants.

Through ultrasonication dyeing, color differences were low, suggesting that a temperature of around 40^{0}C does not significantly change the shade of the paints. Samples treated with iron sulfate 3-5%, but also iron sulfate with oxalic acid 5%, were negative. The color of the samples is somewhat stable for dyeing assisted by biomordants for both 3 and 5% concentrations, close to the shade of the untreated sample, in pink-slightly orange tones, but for the classic $FeSO_4$ mordant it changes to greenish-blue.

An increase in water and rubbing resistance for the samples dyed using ultrasounds in the presence of 5% tannic acid, but also iron sulfate with oxalic acid, with the exception of citric acid at low concentration, was noticed.

3.2. ATR-FTIR analysis of dyed cotton samples

The results of the ATR-FTIR analysis for control and cellulosic samples dyed by exhaustation and ultrasound method are reported in Figure 3 and Figure 4.

The assignment of absorption peaks in the ATR FT-IR spectra was done based on the literature [21,22,23,24,25,26,27,28,29]. The absorption peaks at 3271.06 cm^{-1} (E) and 3270.06 cm^{-1} (US) are due to the alcoholic/ phenolic −OH stretching hydroxyl group of cellulose and tannins present in natural extract The sharp peaks in this region can also be associated with a NH stretching [23]. The peaks at 2849.85 and 2897.89 cm^{-1} (E) and at 2849.19 and 2894.74 cm^{-1} (US) are assigned to the asymmetric and symmetric stretching vibrations of CH_2. The peak at 1646.23 cm^{-1} are attributed to carbonyl group (C=O) stretching vibration. The peaks at 1453.64 cm^{-1} for exhaustation (E) and 1450.17 cm^{-1} for ultrasonication (US) indicates the presence of methylene groups (CH_2). The peaks at 1203 cm^{-1} may be attributed to C-O-C-functions of ethers and esters. The peak at 1028 cm^{-1} was associated with C–O–C pyranose ring skeletal vibration of cellulose. The range below 1000 cm^{-1} is considered the fingerprint area, providing information about the substitution of aromatic rings. The peak at 897 is specific for β-glycosydic bonds of cellulose.

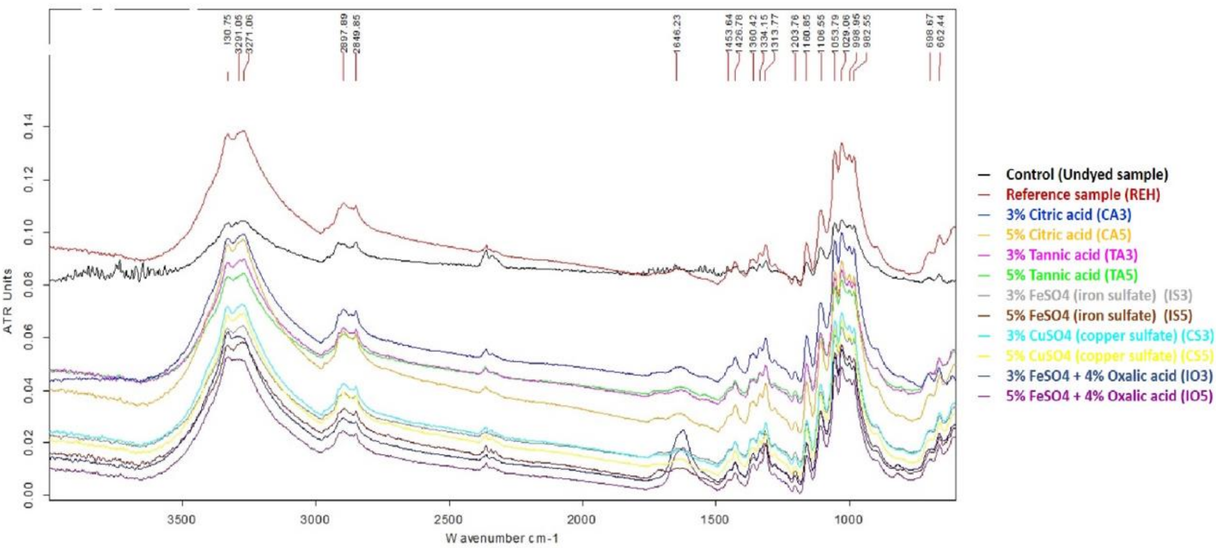

***Fig. 3**: ATR-FTIR spectra of investigated cotton samples, dyed by exhaustation with R. typhina extract in the presence and absence of mordants.*

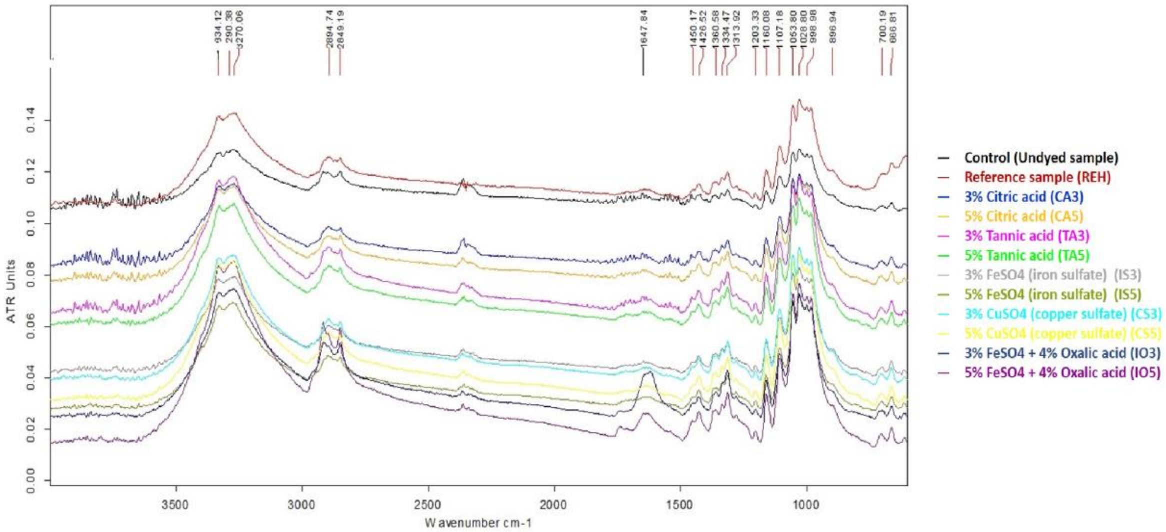

Fig. 4*: ATR-FTIR spectra of investigated cotton samples, dyed by ultrasonication with R. typhina extract in the presence and absence of mordants.*

The use of mordants, did not significantly change the structure of the cellulosic fibres support and did not bring differences compared to the structure of the sample dyed without mordant, respectively REH and RUS. Decrease of intensities of several absorption peaks were observed in samples dyed by both methods compared to the undyed cotton fabric, except for the ones dyed by ultrasonication technique using TA3 and CA3. In the study by Rosu et al. (2021) [24], after the dyeing of cotton with triphenodioxazine dye, the absorption bands of the FTIR spectra specific to the groups present in its structure decreased in intensity, a fact that was less visible in case of dyeing with staghorn sumac extract. On the contrary, some absorption peaks, at ≈3270 cm^{-}1, were intensified. The main difference was that US dyeing preserved a structure similar to that of the control than in the case of the E method. The mixture between ferrous sulfate and 4% oxalic acid produced the greatest changes in the cotton structure support.

5. CONCLUSIONS

The present study highlights the potential of the *R. typhina* extract to provide pleasant shades of pink-purple when applied on cellulosic support by conventional and modern dyeing methods.

The most efficient treatment with a biomordant (citric or tannic acid) simultaneously with dyeing, in addition to the relatively small color changes, can also lead to acceptable resistance to water and rubbing. The use of biomordants best preserved the cotton structure after dyeing, especially by ultrasonication. Based on the obtained results, iron sulfate is not recommended as a standard mordant for dyeing cotton with the *R. typhina* fruit extract, as it considerably changed the color of the materials and affected their resistance by combining with oxalic acid. However, the addition of oxalic acid keeps the color of the textile dyes close.

The ecodyeing with the *Rhus typhina* L. extract and assisted by citric acid gives the cellulosic support stability of the color shade and the possibility of use for health products.

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