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Cationic Pretreatment of Cotton and Dyeing with *Fallopia Japonica* Leaves

Kationska predobdelava bombaža in barvanje z listi japonskega dresnika (Fallopia Japonica)

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Abstract

This work examines the possibility of using leaves from the invasive plant species *Fallopia japonica* (Japanese knotweed) as a source of dye for the natural dyeing of cotton. To achieve a higher uptake of extracted dye, a cationic agent instead of a classical mordant was used to treat the cotton prior to dyeing. Distilled water and 0.5 M NaOH were used as extraction mediums to produce natural dyebaths with different concentrations (10, 20 and 50 g/L) of *Fallopia japonica* leaves. The colorimetric measurements revealed that a higher concentration of extract, the extraction of leaves in NaOH and a cationic pretreatment of cotton yield a dark-brown-coloured cotton with good wash stability.
Keywords: *Fallopia japonica* leaves, cotton, dyeing, cationic pre-treatment, wash stability

Izvleček

Raziskana je možnost uporabe listov invazivne rastlinske vrste Fallopia japonica (japonski dresnik) kot vir barvila za naravno barvanje bombažne tkanine. Za povečanje adsorpcije ekstrahiranega barvila na bombaž je bila tkanina namesto s klasično čimžo predobdelana s kationskim sredstvom. Kot ekstrakcijski medij za barvilo sta bila uporabljena destilirana voda (H_2O) in 0,5 M natrijev hidroksid (NaOH). Barvalne kopeli so bile pripravljene v treh koncentracijah listov japonskega dresnika, tj. 10, 20 in 50 g/l. Rezultati refleksijske spektrometrije so pokazali, da višja koncentracija listov, ekstrakcija v NaOH in kationska predobdelava vodijo v temnejša rjava obarvanja bombaža z zelo dobro obstojnostjo pri pranju.
Ključne besede: listi, japonski dresnik, barvanje, kationska predobdelava, obstojnost pri pranju

1 Introduction

Invasive alien plant species (IAPS) cause damage to European ecosystems and economies. The “APPLAUSE” project aims to use IAPS as a resource for the development of new products (e.g., dyes for textiles). The rhizome and leaves of *Fallopia japonica* (*F. japonica*) have the potential to be used as dyes for textiles. To date, no research has been published on the subject, except for a study on the dyeing of plasma-treated cotton and bamboo rayon with *F. japonica* rhizome extract [1], where it was found that plasma-treated textile samples had a higher adsorption of dye and, consequently, better antibacterial properties. The traditional pretreatment of textiles, when dyeing with natural dyes, is mordanting. Mordants are metal salts that enable a higher adsorption of dye onto textiles, and can be used to achieve different colours with the same natural dye [2]. Alternatives to the classical mordants are plasma, chitosan, and cationic agent pretreatments [3–5]. Plasma is a partially ionised gas that enables the formation of new functional groups on the fibres’ surface and/or their micro- to nano-etching [6]. It is an

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ecologically benign form of textile modification. Natural dyes extracted from curcumin and green tea were shown to have a higher adsorption onto cotton fabric if the fabric was pretreated with ammonia plasma [3]. In contrast, the adsorption of natural dyes was shown to decrease if the cotton samples were pretreated with oxygen plasma. It was found that plasma pretreatment did not influence the change in the bathochromic or hypsochromic shift of the absorption spectra of dyed cotton. The pretreatment of wool with oxygen plasma was shown to enable a better adsorption of dendrimer with amine end groups, and consequently a better adsorption of cochineal dye [7]. A great amount of dye was absorbed due to the electrostatic attraction between the negatively charged dye molecules and positively charged dendrimers as well as the positively charged amine groups of wool fibres. Chitosan is poly-cationic amino polysaccharide and has acquired great importance as a new functional material for textile applications mainly because of its biocompatibility and non-toxicity [8]. When it is used to pretreat textiles for dyeing with natural dyes, it serves as a "biological" mordant that enhances the adsorption of the dye, e.g., between wool and tea [9], wool and henna [10] and cotton and black tea [11].
The purpose of this research was to examine the potential of *F. japonica* leaves as a source of textile dye and to use a cationic agent instead of metal mordants to pretreat cotton.

2 Experimental

2.1 Material

For the research bleached 100% cotton fabric (Tekstina d. o. o., Ajdovščina) was used. The Japanese knotweed leaves were collected and delivered by SNAGA d. o. o., washed with water to remove the dirt, air dried at room temperature and grounded into powder.

2.2 Preparation of extracts

The extracts were prepared in two extraction media, i.e., deionised water (H_2O) and 0.5 M NaOH. The weighted leaves powder (at concentrations of 10, 20 and 50 g/L) was put in cold extraction medium and heated to boiling. The extraction of the dye took 60 minutes. Afterwards, the mixture was filtered. The extract was further used as a dyeing bath.

2.3 Cationic pretreatment

Prior to dyeing, cotton samples were treated with 5% Denimcol FIX-OS (CHT, Switzerland) as a cationic agent. The cationic bath included 2.5 mL/L of 32% NaOH. The cationic pretreatment was performed at a liquor-to-goods ratio of 10:1, at 50 °C, for 20 min. The cotton fabric was neutralised afterwards with 5 mL/L of 30% CH_3COOH at room temperature for 5 min.

2.4 Dyeing

Dyeing of untreated and cationic pretreated samples was performed in a GyroWash laboratory machine (James Heal, Great Britain), which simulates exhaustion dyeing. Dyeing was performed in the prepared extracts at the liquor-to-goods ratio of 20:1, at 60 °C, for 60 min. After dyeing, the samples were washed with water and air-dried at room temperature.

2.5 Wash test

A wash fastness test of dyed samples was performed in a GyroWash laboratory machine according to the ISO 105-C06:2010 standard method. The samples were washed 10 times at 40 °C for 45 min. The washing solution contained 4 g/L of Standards Development Committee (SDC) standard detergent. After washing, the samples were rinsed twice in distilled water at 40 °C and then air-dried at room temperature.

2.6 Colour measurements

The colour (CIELAB values) of dyed samples was measured on a reflectance spectrophotometer (Datacolor Spectraflash SF 600 PLUS-CT). All measurements were performed using four layers of fabric with a 9 mm aperture, wherein the specular component was under D65 illumination and a 10° standard observer. An average of 10 measurements was recorded for each sample.

3 Results and discussion

The results of the colour measurements (Table 1) show that the colour is dependent on three parameters, i.e., the initial concentration of *F. japonica* leaves for extract preparation, the extraction medium (H_2O or NaOH), and the cationic pretreatment of cotton.

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Table 1: CIELAB values and photos of cotton samples dyed with dye extracted from F. japonica leaves

Extraction medium	c_{extr} [g/l]	Cotton pretreatment	*L**	*a**	*b**	Photo
H_2O	10	Without	83.92	0.96	20.68	
		Cationic	70.98	1.80	10.80	
	20	Without	79.97	2.28	11.91	
		Cationic	64.59	1.39	11.14	
	50	Without	76.23	2.20	14.04	
		Cationic	60.66	2.68	13.20	
0.5 M NaOH	10	Without	72.18	6.14	16.54	
		Cationic	46.71	6.23	18.38	
	20	Without	65.76	7.25	19.40	
		Cationic	43.49	7.41	17.55	
	50	Without	60.05	9.13	18.35	
		Cationic	40.17	9.91	19.20	

Increasing the concentration of *F. japonica* leaves for extract preparation caused the dyed sample to become darker (CIE *L** decreases), redder (CIE *a** increases) and yellower (CIE *b** increases), regardless of the medium used for extract preparation or cationic pretreatment. The samples that were dyed in extract prepared in NaOH medium were darker and redder. The value on the yellow–green axis differed according to the concentration of the used extract; i.e., at a lower concentration (10 g/L) the samples were less yellow (CIE *b** decreases) and at higher concentrations (20 and 50 g/L) the samples were more yellow. In the case where the samples were pretreated, the samples that were dyed in NaOH extract were darker, redder and yellower for all used extract concentrations. The cationic pretreatment of cotton influenced the colour of dyed samples mostly on the lightness axis (CIE *L**), as the pretreated samples were much darker than the untreated samples. The effect was even more pronounced for those samples that were dyed in extract prepared in NaOH medium. The dye that is present in *F. japonica* is emodin [1]. Figure 1 presents the reaction of emodin with NaOH.

The newly formed compound is red in colour, water soluble and more substantive to cellulose. The dye was ionized to yield a negatively charged anion dye with positively charged sodium cations [12]. The negative potential of the cellulose (cotton fibers) can repulse the anions in the dye; however, the large amount of sodium ion content ensures that the negative potential of the fibers is overcome, so it is not necessary to put any additional common salt in the dyeing bath [12]. The cationic pretreatment (which is a reactive polyammonium compound) of cotton ensures that the fibers have cationic sites onto which anionic dyes can be more easily adsorbed; therefore, the dyeability of the cotton is increased [5].

Table 2 presents the measured CIELAB values after 10 repeated washings. In the case of untreated cotton, the samples almost completely lost their color, while the color remained in the cationic pretreated samples. Therefore, the cationic pretreated cotton, which was dyed with natural dye extracted from *F. japonica* leaves, had very good wash stability. In the case where extract was prepared in NaOH medium, the samples became lighter, less red and less yellow after washing. An interesting effect after washing was observed for cationic pretreated samples dyed with extract prepared in H_2O medium. The value on the lightness axis was decreased, meaning that the samples became darker after washing. The same effect was also observed in a study on dyeing cotton with Goldenrod extract, where the fabric was precationised [5].

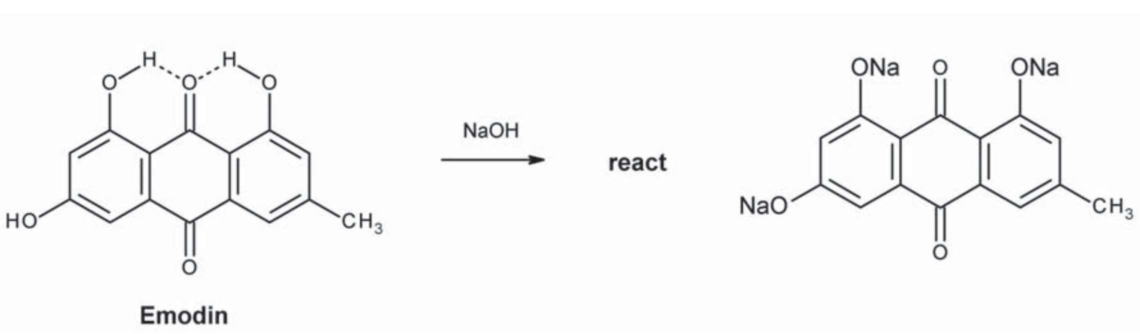

Figure 1: Reaction of emodin with NaOH

Table 2 CIELAB values and photos of cotton samples dyed with dye extracted from F. japonica leaves after ten repetitive washings

Extraction medium	c_{extr} [g/l]	Cotton pretreatment	*L**	*a**	*b**	Photo
H_2O	10	Without	87.20	1.01	5.99	
		Cationic	62.08	3.11	19.89	
	20	Without	88.81	1.19	5.65	
		Cationic	50.87	2.31	12.29	
	50	Without	84.46	1.16	5.94	
		Cationic	48.22	2.07	14.26	
0.5 M NaOH	10	Without	84.86	1.33	9.12	
		Cationic	56.59	4.83	16.14	
	20	Without	83.77	1.77	8.76	
		Cationic	50.58	6.28	18.66	
	50	Without	78.58	3.99	11.10	
		Cationic	45.28	9.21	20.25	

4 Conclusion

The results of this study show that extract from *F. japonica* leaves has great potential to be used for the dyeing of cotton in different brown shades (from light to very dark), depending on the amount of leaves used for the extraction and cationic pretreatment of cotton. The cationic pretreatment, which rendered good wash stability, can be used in the natural dyeing of cotton instead of traditional mordants. The samples that were not pretreated with the cationic agent were found to have very poor wash stability.

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