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Semen Litchi Extract Dyeing on Silk Fabrics

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Abstract. The extraction of natural dye from semen litchi was studied, and the stability and dyeing ability of the dyestuff for the silk fabrics were also investigated. The results showed that the extraction efficiency using ethanol and sodium hydroxide was better than other solvents. The results indicated that the stability of the semen litchi stuff was good. The optimum direct dyeing process is at 90 ^{o}C for 45 min with pH 4. Meanwhile, the color yield and color fastness of dyed fabrics can be improved by mordant dyeing.

Introduction

Natural dyes are a kind of traditional dyes in China. However, with the development of chemical industry, synthetic dyes are widely used for its bright and colorful luster and excellent color fastness. In recent years, it was found that more than 100 varieties of common synthetic dyes may produce carcinogenic substances, and the waste water in the process of printing and dyeing is harmful to environment. With the trends of global green revolution, the world has begun to pay more attention to natural dyes [1]. China has the biggest cultivated area and output of litchi in the world. The cultivation area and annual production of litchi account for 80% and 75% of the world's total area and production, respectively. It's by-products such as peel and seed reach more than 70 tons [2]. However, with high yield of litchi, most stones of litchi cannot be put to good use except that some are used as traditional Chinese medicine. Moreover, semen litchi contain flavonoid phenolic compounds, anthraquinone compound and total terpene lactones with strong bactericidal anti-inflammatory effect [3], thus, it can aid in healing skin disease with fabrics dyed with natural dyes from semen litchi.

The paper aimed to develop the semen litchi pigment as natural dye and study its dyeing properties. Semen litchi dye can be considered as the substitute of some synthetic dyes for waste minimization, which has an important significance on the development of litchi industry and ecological textiles.

1 Experimental

1.1 Materials, reagents and apparatuses

Materials: semen litchi (Guangdong China), silk habotai was provided by Hangzhou HSDP Group Co., Ltd (Hangzhou China). Reagents: ethanol, copper sulphate, ferrous sulphate, alums, NaOH, soap flakes and sodium carbonate were purchased from the local market. All chemicals were of reagent grade and used without further purification. Apparatuses: Nicolet 5700 FT-IR spectrophotometer, IR-12S infrared dyeing machine, SF600 pH meter, ROACHES rubbing fastness tester and UV2550 UV-Vis spectrometer.

1.2 Extraction

A set of 4 samples of crushed powder of semen litchi weighing 5 gm, was dissolved in 100 ml water, 100 ml 20% ethanol, 100 ml 1% NaOH, 1g NaOH with 20% ethanol and heated at 80 °C for 1 hour, respectively. After filtration, constant volume of extract reaches to 250 ml. After being diluted by 12.5 times, the absorbances were measured by UV-Vis spectrometer.

1.3 Dye properties analysis

1.3.1 Effect of pH on dye stability

Each 1ml extract by alkaline alcohol extraction was diluted by 12.5 times with buffer solutions with pH of 3.04, 5.06, 6.98 and 9.01, respectively. The absorbances were measured by UV-Vis spectrometer.

1.3.2 Effect of temperature on dye stability

The 1 ml extract by alkaline alcohol extraction was diluted by 12.5 times with buffer solutions with pH of 4.01, 6.08 and 8.01, then divided into 5 parts, and heated at the temperature of 20 °C, 40 °C, 60 °C, 80 °C, 100 °C for 1 hour respectively. The absorbance of the solutions was measured to show the effect of temperature on absorbance.

1.4 Extracts component analysis

1.4.1 Qualitative chemical analysis

The dye extracts were tested by solution of 1% $FeCl_3$, gelatin solution, solution of 1% $AlCl_3$ and ethanol solution of 1% $FeCl_3$.

1.4.2 Infrared spectroscopic analysis

FTIR-KBr spectra of the products were collected on Nicolet 5700. In a typical experiment, 32 scans at resolution of 2 cm^{-1} were collected.

1.5 Dyeing

1.5.1 Direct dyeing

The dye bath containing 30 ml of the dye extract had a liquor ratio of 50:1 The dye bath was heated at different temperatures (from 60 to 90 °C), at pH 3~8.

1.5.2 Mordant dyeing

The metallic mordant used for simultaneous mordanting and dying were $FeSO_4$, $CuSO_4$ and $KAl(SO_4)_2 \cdot 12H_2O$. The dye bath contained 30ml of the dye extract and 1% (o.w.f) mordant at a liquor ratio of 50:1. The dye solution was heated at 90 °C at pH 3.

1.6 Testing

The color fastnesses were determined based on the standards: GB/T3921.1-1997. GB/T3920-1997. GB/T8426-1998.

2 Results and Discussion

2.1 Pigment extraction

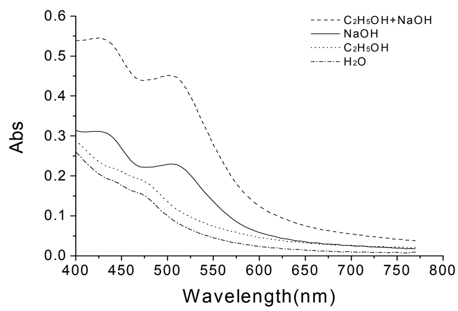


Fig. 1 Absorption spectra of different extraction method

From Fig. 1, the absorbance of ethanol and NaOH extract were more than that of other solvents extracts, which was due to higher solubility of pigment extracted by alkaline and ethanol.

2.2 Litchi extracts stability

2.2.1 Effect of pH on dye stability

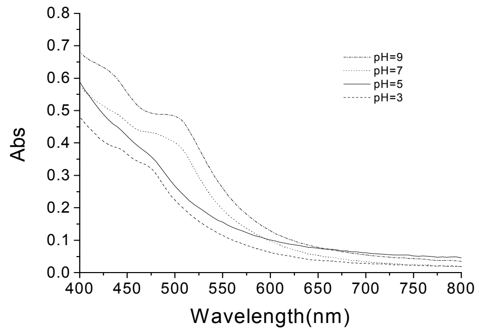


Fig. 2 The effect of pH value on dye stability

As shown in Fig. 2, when pH value was in the range of 3~5, the dye absorbances were stable. However, when pH value was more than 7, the color shade of litchi extracts changed from pale yellow to dark red, and the maximum absorption wavelength had a red shift. It was probably because that red 2-hydroxy chalcone was generated by ring-opening reaction of the extraction of flavonoids.

2.2.2 Effect of temperature on dye stability

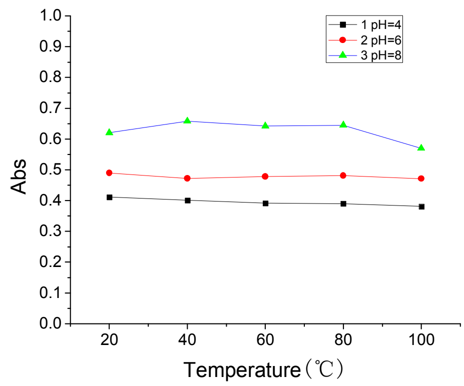


Fig. 3 The effect of temperature on dye stability

As shown in Fig. 3, with the increasing of temperature, the dye absorbance had little changes, which illustrated that semen litchi dye can keep stable at higher temperatures.

2.3 Extracts component analysis

2.3.1 Qualitative chemical analysis

Table 3 The chemical analysis of semen litchi extract

Analysis	Method	Experimental phenomena
Tannins	1% $FeCl_3$ solution	dark green precipitate
	gelatin solution	precipitate
Flavonoids	1% $AlCl_3$ solution	yellowish-brown precipitate
	ethanol of 1% $FeCl_3$	the color of dark green

2.3.2 Infrared spectroscopic analysis

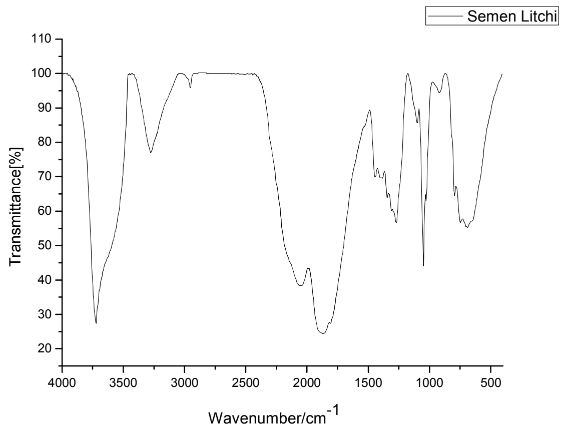
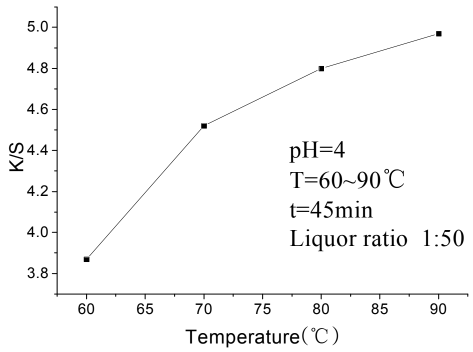


Fig. 4 The infrared spectrum of semen litchi extract

Tannins and flavones are both natural polyphenols with common molecular structure containing hydroxyl, ester, aromatic ring and glycosidic bond [6]. Fig. 4 shows that the strong absorption peak near 3570 cm^{-1} is -OH stretching vibration peak, which indicated that there are a lot of hydroxyls in the extract. The absorption peak at 2932.74 cm^{-1} was attributed to -CH antisymmetric stretching vibration. The skeletal vibration of corresponding benzene ring at 1578.31 cm^{-1} and 1458.08 cm^{-1} indicated that there are benzene rings in the extract. The absorption peak near 1054.45 cm^{-1} was the vibration of glycosidic bond, which further confirmed that semen litchi extract contains polyphenols.

2.4 Dyeing

2.4.1 Direct dyeing



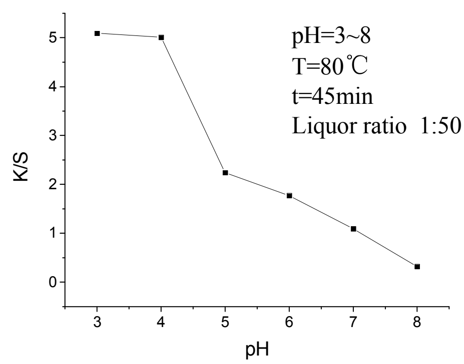


Fig. 5 The impact of dyeing temperature on K/S values of silk fabrics

Fig.6 The impact of pH on K/S values of silk fabrics

Fig. 5 shows the effect of temperature on the color strength obtained from the dyed fabrics. It is clear that the color strength increased with the increase of dyeing temperature and arrived at a maximum value at 90 ^{o}C.

Fig. 6 shows that the pH values of dye bath had considerable effects on the dyeability of silk fabrics dyed by semen litchi extract and the highest color strength was achieved at pH 3. The effect of the dye bath pH was attributed to the correlation between dye structure and silk fabric. Since the dye used is sparingly soluble in water and contains -OH groups, it would thus interact ionically with the protonated terminal amino groups of silk fibers at acidic pH via ion exchange reaction due to the acidic character of the -OH groups. The anion of the dye had complex characteristics, and when it was bound on the fiber with ionic forces, the ionic attraction would increase the dyeability of the fiber as clearly observed in Fig.6. Under acidic condition, amine groups of silk become positively charged, allowing anionic dye molecules to form ionic bonds with the silk fiber.

2.4.2 Effect of mordant dyeing on K/S and color fastness

Table 4 The K/S values and color fastness of fabrics and fastness after different mordant dyeing

		Original sample	$KAl(SO4)_2 \cdot 12H_2O$	$FeSO_4$	$CuSO_4$
Crocking	dry	3~4	4	4	4~5
fastness	wet	3	3~4	4	4
Staining on cotton		5	5	5	5
Staining on silk		4	4~5	5	5
color change		2~3	2	3	2~3
light fastness		2	3	4~5	3~4

Table 4 shows silk fabrics dyed by ferrous sulphate mordant had good color fastnesses.

3 Conclusions

The results showed that the optimal extraction solvents are ethanol and NaOH. The litchi extract has better thermal stability. The optimal dye process is as follows: pH=4, 90 ^{o}C for 45 min. The fabric has high color fastnesses except the color change which can be resolved by mordant dyeing.

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