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Determination of Colour Strength on Cationized Cotton Fabric Dyed with Waste Wine Extract (Grape Pomace)

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Abstract: *The main aim of this research is to study the effect of cationic agents (pentafix AQL, alkyl ether ammonium carbonate) on cotton fabric dyed with waste wine extract (grape pomace). Firstly, the cotton fabric is modified with cationic agents namely pentafix AQL and alkyl ether ammonium carbonate. The concentrations of cationic agents are varied with 6%, 12% and 18% on weight of fabric. And then, natural dye is extracted from grape pomace by alkaline extraction method. The phytochemical test is carried out to confirm the presence or absence of colouring compounds in the grape pomace extract. And then, dyeing of cotton fabrics (unmodified and modified) with grape pomace extract is performed with material to liquor ratio of 1:15 by using jigger. Finally, colour value and colour strength of dyed cotton fabrics are determined. It is clearly seen that cationization improves the colour strength of cotton fabric as compared to unmodified fabric. Treated fabric sample modified with 18% pentafix AQL (C_{13}) gives the highest colour strength and colour saturation among the dyed fabrics.*

Key Words: *Grape pomace, Cationic agent, Cotton fabric, Phytochemical test, Colour strength.*

1. INTRODUCTION:

Colour has always fascinated humankind, for both aesthetic and social reasons. For most of the thousands of years, dyeing has been used by humans to decorate clothing by giving a desired hue or fashion shade. Dye is a substance that is used to impact colour to textiles, paper, leather and other materials [1]. Generally, dyes are classified into two main groups: synthetic and natural. Natural dyes are derived from mineral, animal, or plant sources and generally are complex mixtures of materials [2]. The ancient people exclusively used natural dyestuffs, all easily obtained in their own vicinity before the discovery of synthetic dyes.

For ready availability of pure synthetic dyes, most of textile manufacturers shifted towards use of synthetic colourant. However, most of the synthetic dyes are synthesized from petrochemical sources through hazardous chemical processes. Synthetic dye sometimes causes skin allergy and other harmfulness to human body. In the recent years, there has been a trend to revive the art of natural dyeing. This is mainly because in some aspects, natural colourants are advantageous against synthetic dyes.

The shades produced by natural dyes are usually soft, lustrous and soothing to the human eye. Unlike non-renewable basic raw materials for synthetic dyes, the natural dyes are usually renewable, being agro-renewable/vegetable based and at the same time biodegradable. Some of its constituents are anti-allergens, hence safe for skin contact and are mostly non-hazardous to human health [3]. However, most natural dyes have poor substantivity and fastness properties for fibres such as wool and cotton. Cationic agents can be used to enhance cotton dyeing with anionic natural dyes [4].

Cationization is the chemical modification of cellulose to produce cationic (positively charged) dyeing sites in place of existing hydroxyl (-OH) sites at which negatively charged dye can attach. In addition, the strong dye-fibre interactions resulting from cationizing allow dyeing with no added electrolytes [5].

In this research, pentafix AQL and alkyl ether ammonium carbonate are used as cationic agents to study the effect of these cationic agents on cotton fabric dyed with waste wine extract (grape pomace).

2. EXPERIMENTAL PROCEDURE:

2.1 Collection of Raw Materials

The bleached cotton fabric, cationic agents (pentafix AQL and alkyl ether ammonium carbonate) and sodium hydroxide are collected from local market. Grape pomace is provided by Golden Bee Wine Industry which is located in Thandaung Township, Kayin State. After the collection of grape pomace, they are dried out of the sunlight.

2.2 Fibre Identification Test

To prove that the fabric sample is made of cotton fibre, fibre identification tests such as burning test, microscopic examination and solubility test are carried out according to the procedure of ASTM D 276-87 at the Fibre Microscopy Laboratory of the Department of Textile Engineering, Yangon Technological University.

2.2.1 Burning Test

The burning test is a sample identification test based on the knowledge of how particular fibres burn. One end of the fibre is held by the tweezer and taken near the flame. And then, the burning behaviours are observed when (1) approaching flame, (2) in flame, and (3) after removal of flame. The results described in Table 2.1 show that the sample fabric is made of cotton.

Table 2.1. Reaction of Fibre Sample to Flame

Approaching Flame	In Flame	After Removal of Flame	Odour	Typical Ash (Characteristics)
Ignites readily	Burns quickly; Yellow flame	Continues to burn without melting; has afterglow	Like paper burning	Light, Feathery, Small, Fluffy grey ash

2.2.2 Microscopic Examination

A fibre is pulled out from the sample fabric and observed under the microscope. It resembles a collapsed, spirally twisted tube with a rough surface. The thin cell wall of the fibre has natural twists or convolutions. The fibre appears flat, twisted, and ribbon-like, with a wide inner canal (the lumen) and a granular effect. When zinc chloro-iodine is put on the fibre, the fibre changes into brick red colour. This means that the sample fabric is made of cotton.

2.2.3 Solubility Test

The reagents used in this work are sulphuric acid, acetic acid and hydrochloric acid. Fibre pulled out from the sample fabric is placed in the desired solvent and after a few seconds, the fibre is checked. The results are shown in Table 2.2 and it is observed that the sample fabric is made of cotton.

Table 2.2. Solubility Test Results

Reagent	Remark
Sulphuric acid	Dissolved
Acetic acid	Dissolved
Hydrochloric acid	Dissolved

2.3 Modification of Cotton Fabric with Cationic Agents

Before the dyeing process, the cotton fabric is modified with cationic agents namely pentafix AQL and alkyl ether ammonium carbonate. The concentrations of cationic agents are varied with 6%, 12% and 18% respectively based on the weight of fabric. Cationization is performed at a temperature of 50°C for 60 minutes by using jig dyeing machine. After the treatment, the fabric is squeezed to remove excess water and then air-dried at room temperature.

2.4 Extraction of Natural Dye from Grape Pomace

Natural dye is extracted from grape pomace by alkaline extraction method. The dried grape pomace (70 g/l) is added into water containing sodium hydroxide (0.13M) and boiled at 80°C for 70 minutes. Then, the dyed solution is cooled and filtered. Finally, the required grape pomace extract as shown in Figure 2.1 is achieved.

Figure 2.1. Grape Pomace Extract

2.5 Phytochemical Examination of Grape Pomace Extract

The phytochemical tests are carried out to confirm the presence of colouring compounds in the grape pomace extract according to the procedure described in the Text Book of Pharmacognogy, Pharmacopoeia of India, Phytochemical Methods; A Guide to Modern Techniques of Plant Analysis. The tests are implemented at the Research Department of Myanma Pharmaceutical Industrial Enterprise, Ministry of Industry. The test results are shown in Table 3.1.

2.6 Dyeing of Cotton Fabric with Grape Pomace Extract

In order to perform the dyeing process, the dye bath is firstly prepared. The extracted dye solution is added into the jigger shown in Figure 2.2 with material to liquor ratio of 1:15. Then, unmodified and modified cotton fabrics are dyed separately with grape pomace extract at 100°C for 55 minutes. At the end of the dyeing, the fabric is removed and rinsed well under water until the water becomes clear. Then, the dyed fabrics are dried at room temperature and kept out of the sunlight. The designation of the dyed cotton fabrics is shown in Table 2.3.

Figure 2.2. Dyeing of Fabric with Jigger

Table 2.3. Designation of Dyed Cotton Fabrics

Sr. No.	Sample Code	Cationic Agent	Cationic Agent Conc. (o.w.f)	Cationization		Dyeing	
				Temp. (°C)	Time (min)	Temp. (°C)	Time (min)
1	C_{00}	(Unmodified)	-	-	-	100	55
2	C_{11}	Pentafix AQL	6%	50	60	100	55
3	C_{12}		12%	50	60	100	55
4	C_{13}		18%	50	60	100	55
5	C_{21}	Alkyl Ether Ammonium Carbonate	6%	50	60	100	55
6	C_{22}		12%	50	60	100	55
7	C_{23}		18%	50	60	100	55

2.7 Measurement of Colour

The colour of the dyed fabrics is measured with X-rite spectrophotometer. X-rite instrument is first calibrated using standard tiles (white, black, green). The colour measurement is performed randomly at five different locations along the front side and back side of the fabric.

After completing the measurement, colour value, colour difference and K/S values are achieved. The test is carried out at the Laboratory of Textile Testing and Quality Control of Department of Textile Engineering, Yangon Technological University. The test results are shown in Table 3.2.

3. RESULTS AND DISCUSSIONS:

3.1 Phytochemical Examination of Grape Pomace Extract

The phytochemical test is carried out to confirm the presence or absence of colouring compounds in the grape pomace extract. According to the test results shown in Table 3.1, the grape pomace extract contains carbohydrate, glycoside, phenol, α-amino acid, tannin, flavonoid, terpenoid and reducing sugar. Among these compounds, glycoside, phenol, tannin and flavonoid are regarded as colouring matters.

Flavonoids constitute a major class of natural yellow dyes. The basic flavonoid chromophore is susceptible to phytochemical attack and probably leads to the formation of quinones. Consequently, the yellow colour turns to a dull brown. Tannin are very complex compound and it has a slight negative charge. Tannin gives yellow or pale brown colour on cotton fabric.

Table 3.1. The Results of Phytochemical Examination of Grape Pomace Extract

No.	Type of Compound	Extract	Reagent Used	Observation	Results
1	Alkaloid	1% HCL	Mayer's reagent	No ppt.	-
			Wagner's reagent	No ppt.	
			Dragendorff's reagent	No ppt.	
			Hager's reagent	No ppt.	
2	Carbohydrate	H_2O	10% α-naphthol and $H_2SO_{4(Conc:)}$	Red ring	+
3	Glycoside	H2O	10% Lead acetate soloution	White ppt.	+
4	Phenol	H2O	5% FeCL3 solution	Black ppt.	+
5	α-amino acid	H2O	Ninhydrin reagent	Pink colour	+
6	Saponin	H2O	H2O	No persistent foam	-
7	Tannin	H2O	1% Gelatin and 10% NaCL solution	White ppt.	+
8	Flavonoid	70% EtOH	Mg ribbon and Conc; HCL	Pink colour	+
9	Steriod	Petroleum ether	Acetic anhydride and Conc; H2SO4	-	-
10	Terpenoid	Petroleum ether	Acetic anhydride and Conc; H2SO4	Pink	+
11	Reducing sugar	H2O	Fehling's solution	Brick red ppt.	+
12	Starch	H2O	Iodine solution	Reddish brown ppt.	-
13	Cyanogenic glycoside	H2O	H2O, Conc; H2SO4, Sodium picrate paper	No colour change	-

(+) = presence (-) = absence

3.2 Effect of Cationic Agents on Colour Value, Colour Difference and Colour Strength of Dyed Cotton Fabrics

According to the resultant data, the dyed samples modified with pentafix AQL are yellower and higher colour saturation than unmodified sample. Cationization with alkyl ether ammonium carbonate causes the samples to be lower yellowness and colour saturation compared to the unmodified sample.

According to h° value, the hue angle of dyed fabrics ranges from 51° to 58° implying that all samples give the same angle range of orange. In terms of ΔE* value, C_{12} has the range between 1 and 2, and thus it is perceptible through close observation. The other samples are perceptible at a glance because their ΔE* values are between 2 and 10. The results of the colour value, colour difference and colour strength of dyed cotton fabrics are shown in Table 3.2.

Table 3.2. Summary of the Test Results of Colour Value, Colour Difference and Colour Strength

Sr. No.	Sample	CIELAB					ΔE*	K/S
		L*	a*	b*	C*	h°		
1	C_{00}	77.19	7.14	10.87	13.01	56.70	0.00	0.36
2	C_{11}	71.37	7.57	11.90	14.10	57.55	5.92	0.59
3	C_{12}	75.98	7.12	11.42	13.46	58.06	1.33	0.40
4	C_{13}	70.66	8.12	11.70	14.25	55.23	6.66	0.63
5	C_{21}	70.90	7.02	9.74	12.00	54.22	6.39	0.58
6	C_{22}	71.85	6.56	8.64	10.85	52.81	5.81	0.52
7	C_{23}	71.09	7.15	9.15	11.61	51.99	6.34	0.56

The value of colour strength (K/S) of the dyed fabrics is taken at the wavelength of 480 nm. According to K/S values, modification of cotton fabric gives higher colour strength as compared to the unmodified fabric. This is due to the fact that the introduction of positively charged sites to cotton fabric enables the formation of an electrostatic attraction (ionic bond) between the fibres and the negatively charged dye molecules. Among the modified fabrics, C_{13}

induces the highest colour strength and C_{12} gives the lowest colour strength. It is also observed from the results that the dyed fabrics modified with 12% concentration of cationic agents produce the lowest colour strength in both reagents.

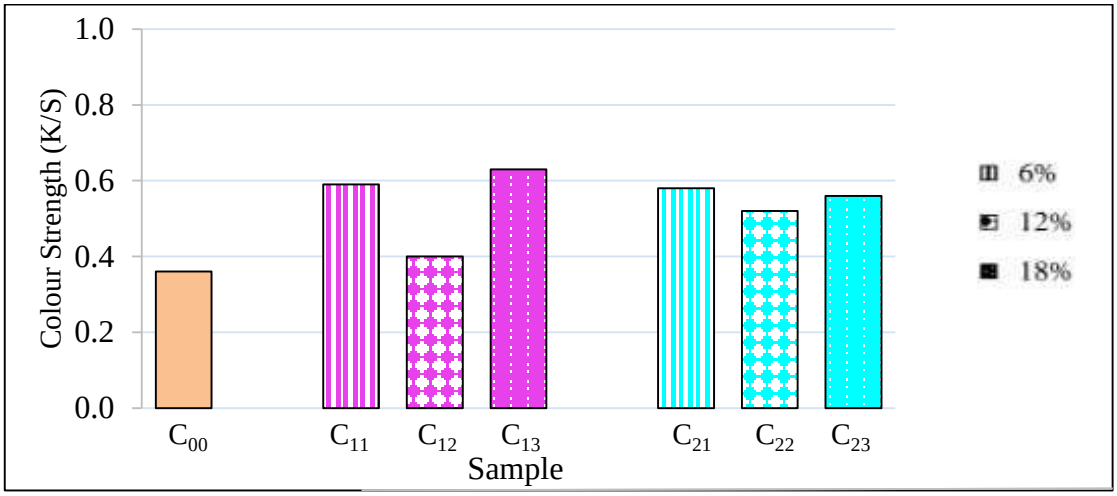

Figure 3.1. Effect of Cationic Agents on Colour Strength of Dyed Cotton Fabrics

4. CONCLUSIONS:

In this research, the cotton fabric is modified with different concentrations of cationic agents (pentafix AQL, alkyl ether ammonium carbonate), natural dye is extracted from waste wine (grape pomace) and unmodified and modified cotton fabrics are dyed with waste wine extract. According to the phytochemical test results, the grape pomace extract contains the natural colouring compounds such as glycoside, phenol, tannin and flavonoid. Due to the presence of these compounds, the waste wine extract is suitable to use as natural dye.

Regarding with the result of K/S values, cationization increases the colour strength of cotton fabric as compared to unmodified fabric. Among the dyed cotton fabrics, C_{13} induces the highest colour strength. It can be concluded that cationization of cotton fabrics with pentafix AQL and alkyl ether ammonium carbonate enhances the dye uptake properties of cotton fabric towards anionic dyes due to the addition of cationic sites.

5. RECOMMENDATIONS:

As for the future work, it is suggested to study the dyeing properties of waste wine extract on other fibres such as wool and silk. In addition, the other extraction method of grape pomace should be studied. Then, colour value, colour strength and physical properties of dyed fabrics should be investigated at various dye concentration, dyeing time and dyeing temperature. Furthermore, an attempt should be made to modify the cotton fabric with natural cationic agents or other modification methods to increase the colour yield of the fabric.

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