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Extraction of Natural Dye from Saffron Flower Waste and its Application on Pashmina fabric

A. S. M. Raja*[1], P. K. Pareek[1], D. B. Shakyawar[1], Sarfaraz. A. Wani[2], F. A. Nehvi[2] and Asif H. Sofi[2]

[1]*Division of Textile Manufacture and Textile Chemistry, Central Sheep and Wool Research Institute, Avikanagar, Rajasthan, India*
[2]*Sher-e- Kashmir University of Agricultural Science and Technology of Kashmir, Srinagar, India*

ABSTRACT

After harvesting the stigma from saffron flower, the petal part of the flower, which is violet in colour, is thrown as a waste. Pashmina shawl is a very delicate material and requires mild chemical treatments for dyeing. In the present study, an attempt has been made to utilize the petal part of the saffron flower to extract dye for application on the Pashmina shawl. The saffron flower waste was dried and ground into powder form. The natural dye was extracted by aqueous method at boiling conditions. The extracts were then applied on Pashmina wool at two different pH namely pH 4-5 and pH 7-8 with and without the use of mordant. The results showed that saffron flower waste extracts was able to dye the Pashmina shawl satisfactorily with very good washing and light fastness properties. It is also proposed to give an alkaline detergent treatment after dyeing to avoid tone variation of dyed fabric. Saffron flower extract dyed fabric at acidic pH without mordant showed zone of inhibition for the growth against Staphylococcus aureus.

Keywords: Natural Dye, Saffron Flower, Mordanting, Pashmina, Dyeing, Antimicrobial.

INTRODUCTION

Saffron is a perennial plant with the botanical name of *Crocus Sativus Linn* belongs to *Iridaceae*. In Sanskrit, it is named as *Keshara, Kunkuma, Aruna, Asra* and *Asrika* and in Hindi it is called as *Zaffran and Kesar*. This plant is cultivated in Jammu & Kashmir and Himachal Pradesh for the production of flowers. A spice made from the dried stigma of the flowers is considered as the world's most expensive spice. The spice has culinary and medicinal properties [1]. The red stigma of the flower is harvested in the morning when the flowers are open. The collected stigma is then dried and converted into powder. Approximately 1 kg of flower is required to produce 12g of dried saffron spice. After harvesting the stigma from saffron flower, the petal part of the flower which is violet in colour is thrown as a waste. In present study, an attempt has been made to utilize the petal part of the saffron flower which is considered as a waste to extract dye for application on Pashmina. The utilization of the saffron flowers waste will add the value to the saffron growers. The Pashmina which is the costliest fibre [2] also require value addition through dyeing using natural colour. Hence, it is attempted to dye the Pashmina shawl using the dye extracted from saffron flower. In this study, attempts have also been made to the see anti microbial properties of saffron dyed Pashmina fabric.

MATERIALS AND METHODS

Collection of Flowers

The saffron flower after harvesting their stigma was collected from Srinagar, Jammu & Kashmir. The flowers were dried in shadow and then ground in to powder using lab model grinding machine.

Extraction of Dye

The 100g of powder was soaked in 2 liters of water and left for overnight. Then, the extract was boiled for 1 hr to get a water soluble dye solution. The extract was filtered and used as such for dyeing of Pashmina shawl.

Pashmina Shawl

The shawl used for present study was made of pure hand spun Pashmina yarn with ends/inch-55 and picks/inch 66. The areal density of shawl fabric was 95 g/ m^2. The shawl was mild scoured using 0.5% non-ionic detergent before dyeing.

Dyeing and Mordanting

The dyeing of Pashmina fabric with saffron flower extract was carried out in two different pH conditions. In the first method, the dyeing was conducted at pH 4-5 and the second at pH 7-8 using the following recipe.

Dye	*5%*
Acetic acid	*3 gpl (Method I)*
Sodium carbonate	*2 gpl (Method II)*
Temperature	*85°C*
MLR	*1:30*
Time	*1 hr*

All the dyeing experiments were carried out by exhaustion method in a water bath, keeping material to liquor ratio to 1:30 as per the standard procedure [3]. The scoured Pashmina shawl was introduced into dye bath containing 5% dye at room temperature and temperature was increased to 85°C with gentle stirring. The dyeing was then continued for one hour. The mordanting with different salts viz: Aluminum sulphate, Stannous chloride and Ferrous sulphate of 3% concentration was carried out in the same bath after exhaustion of the dye. The temperature of the bath was allowed to cool below 50°C and the mordant solution of 3% concentration on the weight of the material was added to the bath. The temperature was then raised to 85°C and dyeing continued for another one hour. After mordanting, the samples were taken out of the bath and thoroughly washed with water, followed by washing with detergent.

Colorimetric and K/S value

The colorimetric values of dyed samples were evaluated using a JAYPAK4802 Colour matching system (Jay Instruments Ltd, Mumbai, India) at D65 illuminate/ 10^o observer. The L*, a* and b* of dyed materials were studied with the help of Computer Colour Matching Software [4]. Colour strength values (K/S) were calculated of the dyed shawls using the spectrophotometer as per standard procedure [5]. L* value indicates Lightness- Darkness index of the sample. The L* value of pure white fabric is 100. The a* indicates Redness- Greeness index. If a* is lesser than zero, it is classified as greener in shade and if the value more than zero, it is classified as redder. The b* value indicates Yellowness- Blueness index. If b* value less than zero, then it is classified as blues and if the value more than zero, it is classified as yellower.

Evaluation of Fastness Properties

The washing fastness of the dyed samples was evaluated as per standard procedure ISO 105-A02 [4]. The light fastness of the dyed material was determined using Q-Lab Xe-1-S light fastness tester using standard procedure. For both washing and light fastness the ratings are given from 1 to 5. The fastness rating of 5 indicates that the dyed materials are possessing excellent fastness and the rating of 1 indicates that the fastness is very poor.

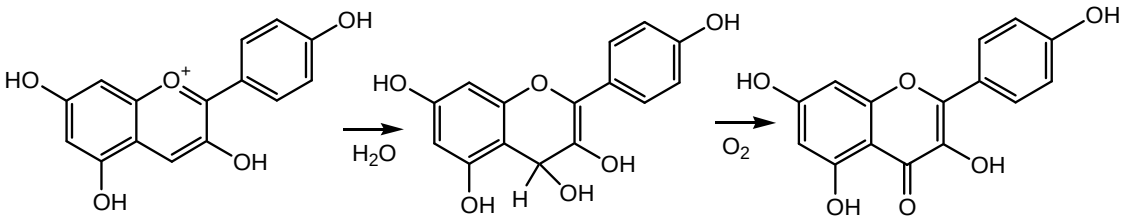
Evaluation of Antimicrobial Efficacy

The antimicrobial efficacy of the saffron waste flower extract dyed pashmina fabric was determined using qualitative Agar Diffusion Method (SN 195920) against gram positive and gram negative bacteria viz. *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E.coli*) respectively [6-8]. In this method, the evaluation was made on the basis of absence or presence of an effect of bacteria in the contact zone under the specimen and the

possible formation of a zone of inhibition around the test specimen. The area of inhibition zone in mm was a measure of antimicrobial effectiveness.

RESULTS AND DISCUSSION

The aqueous extraction of saffron flower waste is yielded 12% colourant by aqueous extraction method. The colour of the extract is dark yellow. The aqueous extract of the saffron flower dyed the Pashmina wool satisfactorily with and without the use of mordants. The stigma of the saffron plant contains chemicals such as saffranal, crocin, crocetin, dimethylcrocetin and carotenoid [9-10]. The petal parts contain anthocyanidin and kaemferol (flavonol). The chemical structure of the saffron petal extract is given in Fig.1. However, there is no detailed investigation on the chemical constitution of the flower petals. The colour obtained on the Pashmina fabric using saffron dye extract is given Table 2.



Pelargonidin
(Anthocyanidin)
Purple

Kaempferol
(Flavonol)
Yellow

Colorimetric Coordinates of the Dyed Materials

Dyed at Acidic pH

The colour coordinate values of the dyed fabrics at acidic pH are given in Table 1. The lightness L* value of the dyed fabrics were reduced after mordanting with ferrous sulphate and stannous chloride mordants . However, there was no significant change in the L* value due to aluminium sulphate mordanting. During mordanting, the natural dyes present in the fabric formed coordinate bonds with the metal ions and form insoluble complexes. The formation of dye-metal complexes alters the original colour of the dye generally to dark colours [11]. This was the reason for the reduction in lightness value of the mordanted fabrics. From the results, it was evident that the stannous chloride and ferrous sulphate mordants darkened the original shade produced on Pashmina fabric without mordant. The change in shade in the aluminium sulphate mordant treated fabric was negligible. From the results, it was also evident that L* value of the all the dyed fabrics increased after washing with detergent. This may attributed to intensive complex formation between mordant and fabric after washing. The a* value of the dyed fabric without mordant showed that it was yellowish green in colour. The a* value of the dyed samples was reduced after mordanting with all mordants compared to dyed fabrics without mordant. From the result, it was observed that addition of mordants increased the greenness in the fabrics. The increases in greenness were higher in aluminium sulphate and stannous chloride mordanted fabrics compared to ferrous sulphate mordanted fabrics. The b* value of the dyed fabric indicated that the samples were more yellower in tone. The aluminum sulphate and stannous chloride mordanted fabrics were also more yellower in shade compared to dyed fabric without mordant. However, the ferrous sulphate mordanted fabrics showed reduction in yellowness compared to other dyed fabrics. The flavonoid colorant present in the saffron flower extract which has the potential to improve the yellowish hue after coordinate bonding with Al^{3+} , Sn^{2+} may be the reason for more yellowness in the mordanted fabric. There was no significant change in the b* values among washed fabrics.

Dyed at Alkaline pH

The colour coordinate values of the dyed fabrics at alkaline pH are also given in Table 1. The L* value of the dyed fabrics showed that the fabrics were lighter in colour compared to acidic pH dyed fabrics. The a* values indicated that the dyed fabrics were slightly reddish in shade compared greener shades of acidic pH dyed fabrics. However, the ferrous sulphate mordanted fabric was redder in colour. The b* values showed that in general all the dyed and mordanted fabrics were yellower in shade compared to acidic pH dyed ones.

K/S Value

K/S values of Pashmina fabric dyed with saffron flower waste extract at acidic and alkaline pH are shown in Table 1 and Fig.1. The K/S value of dyed fabrics indicates the dye uptake of the fabrics. From the results, it is observed that the dye uptake on pashmina at pH 4-5 was higher compared to the dye uptake at alkaline pH. The dyed fabrics showed 27%, 13%, 32% and 8% dye uptake in the dyed fabric without mordant, aluminum sulphate mordanted, stannous chloride mordanted and ferrous sulphate mordanted fabrics respectively compared to corresponding alkaline pH dyed fabrics. In general, the K/S value of the dyed fabrics decreased after detergent wash. The reduction of K/S value also was higher in alkaline pH dyed fabrics compared to acidic pH dyed fabrics.

Over all, it was observed that the saffron flower extract dyed the Pashmina wool with bright greenish yellow and green colours at acidic pH, compared to the dyeing light reddish brown shades at alkaline condition. However, the shade produced on Pashmina fabrics during acidic pH changed to redder hue during washing with detergent. The colour change was attributed to the presence of anthocyanidin in the saffron extract. The anthocyanidin constituents has the inherent property of exhibiting bathochromic shift due to the deprotonation of phenolic and carboxylic acid groups present in the dye extracts when their pH changed from acidic to alkaline. Hence, it is very important to give a soaping treatment for the saffron dyed pashmina fabrics at acidic pH to maintain the colour during usage.

Fastness Properties

The light and washing fastness properties of dyed and mordanted fabrics at both acidic and alkaline pH are given in Table-2. The washing fastness result showed that all the dyed samples of saffron extract in acidic condition had good to very good fastness properties on Pashmina fabric with and without the use of mordants was falling between 4 to 5. It is also observed that shades obtained are not only bright and attractive but also quite fast to a large extent for washing. The alkaline pH dyed samples showed only moderate washing fastness is falling between 3 to 4, which is satisfactory. The light fastness results were also found good to very good in acidic condition with and without the use of mordants.

Antimicrobial Efficacy of Dyed Fabrics

The results of antimicrobial testing indicated that saffron flower extract dyed fabric at acidic pH without mordant showed 20 mm of zone of inhibition against *Staphylococcus aureus* bacterium. The corresponding mordanted samples as well as alkaline pH dyed fabrics did not show any antimicrobial efficacy against both *Staphylococcus aureus* as well as *Escherichia coli*.

Table 1: Spectra of Pashmina shawl using saffron colorant

S. No	Particulars	L*	a*	b*	K/S
Saffron at pH 4-5					
1	Without mordant	67.88	0.90	14.15	1.638
2	Washed sample	70.86	1.12	13.31	1.635
3	Aluminium sulphate	67.85	-3.74	18.24	1.668
4	Washed	72.62	-2.72	20.97	1.60
5	Stannous chloride	64.77	-4.57	17.75	1.986
6	Washed	70.67	-2.74	21.85	1.780
7	Ferrous sulphate	60.21	0.58	10.73	1.798
8	Washed	61.63	0.15	10.52	1.848
Saffron at pH 7-8					
1	Without mordant	77.76	0.20	20.27	1.191
2	Washed sample	78.00	0.78	16.89	0.958
3	Aluminium sulphate	77.78	-1.37	28.34	1.451
4	Washed	78.77	-2.30	17.83	0.872
5	Stannous chloride	77.66	0.81	26.81	1.337
6	Washed	77.98	-0.38	22.52	1.095
7	Ferrous sulphate	63.98	4.26	15.92	1.650
8	Washed	69.00	3.85	17.42	1.394

Table 2: Colour and washing fastness properties of Pashmina shawl dyed with saffron flower

S. No	Particulars	Colour Obtained	Washing Fastness	Light Fastness
	Acidic			
1	Saffron	Pale greenish yellow	4	3-4
2	Saffron +Aluminum sulphate	Green	4 -5	3-4
3	Saffron+ Stannous chloride	Dark green	4 -5	3-4
4	Saffron + Ferrous sulphate	Dull green	4	3-4
	Alkaline			
1	Saffron	Light yellow	3- 4	3
2	Saffron +Aluminum sulphate	Yellow	3- 4	3
3	Saffron+ Stannous chloride	Bright yellow	3- 4	3
4	Saffron + Ferrous sulphate	Light brown	3- 4	3

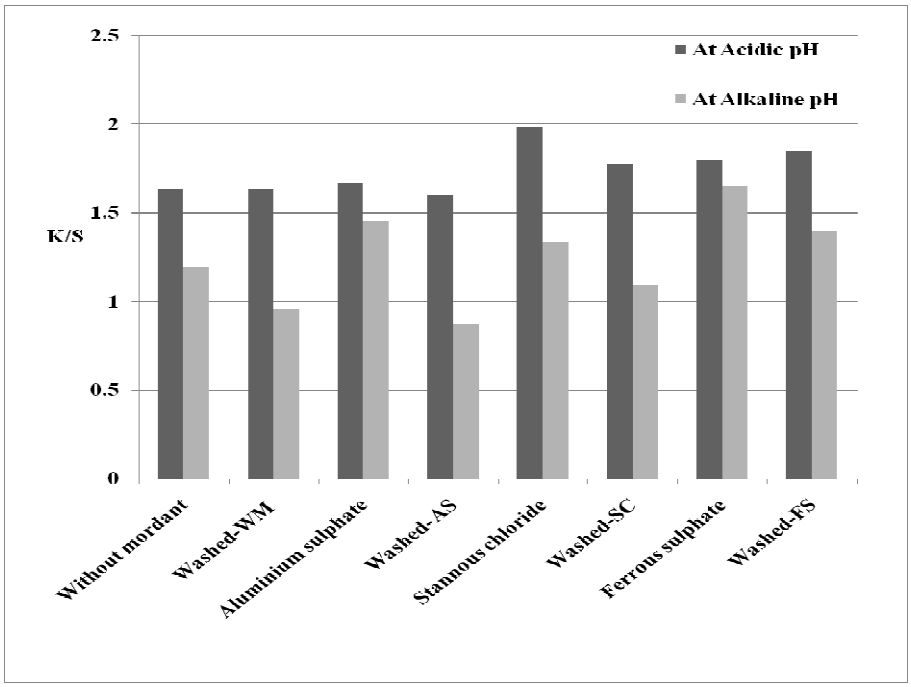


Fig.1. K/S of Dyed and mordanted fabrics at acidic and alkaline pH

CONCLUSION

The saffron flower waste after harvesting stigma can be used as dye to dye the Pashmina fabric. The extract produced bright greenish yellow and green colours on Pashmina fabrics at acidic pH. The light and washing fastness of saffron dyed Pashmina fabric at acidic condition was satisfactory. The dyed pashmina fabric at acidic pH without mordant was showed antimicrobial efficacy against *Staphylococcus aureus*. Hence, this process can be considered as a commercially viable process of dyeing Pashmina fabrics with antimicrobial efficacy. The process enhanced the value of saffron as well as Pashmina wool fabric due to waste utilization and eco friendly dyeing with hygiene textiles point of view.

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