Asian Journal of Chemistry; Vol. 29, No. 10 (2017), 2279-2284

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2017.20761

Study on Tencel Fabric Dyeing with Pomegranate Peel Natural Dyes

FAISAL REHMAN[1], TAYYAB NAVEED[2], MUHAMMAD NADEEM[1] and WANG WEI[1,3,*]

[1]College of Chemistry, Chemical Engineering and Biotechnology, Donghua University, Shanghai 201620, P.R. China
[2]College of Textile Science and Engineering, Donghua University, Shanghai 201620, P.R. China
[3]Saint Year Holding Group Co. Ltd., P.R. China

*Corresponding author: Fax: +86 21 67792456; Tel: +86 13311700563; E-mail: wangv@dhu.edu.cn

Received: 5 May 2017; *Accepted*: 30 June 2017; *Published online*: 31 August 2017; AJC-18534

Textile industries have initiated consideration of using natural dyes, for dyeing and printing of textile goods, to provide the eco-friendly environment. In this research, dyestuff extraction from the pomegranate peel was applied to dye tencel fabric by the solvent extraction process. Ferrous sulphate and copper sulphate were used as mordant agents and three different mordanting methods as pre-mordanting, simultaneous-mordanting and post-mordanting were executed. The dyeing results were accessed through colour fastness properties in accordance with the ISO standards. The color shade (K/S: 4.72), washing fastness, light and rubbing fastness in all dyed samples were excellent (4-5 grade) through pre-mordanting dyeing method. Ferrous sulphate mordant was more effective than the copper sulphate mordant while samples dyed with pomegranate peel. Perspiration fastness outcomes (4-5 grade) were similar through both mordant agents in all the methods. In general, the best results of fastness were obtained by ferrous sulphate mordant using pre-mordanting method.

Keywords: Natural dyes, Tencel, Pomegranate peel, Colour fastness.

INTRODUCTION

In recent years, there is a continuous intensification in the environmental contaminations due to the discharge of toxic and non-biodegradable effluents from the textile manufacturing industries, particularly the colouration industry [1]. The commercially used synthetic dyes (manmade through chemicals) have jeopardized the worldwide environment [2]. In fact, this concern causes serious public health care issues, as well, in marine livings [3]. Therefore, the roles of synthetic dyes have been criticized widely [4]. In these aspects, the environmentalists have made renaissance attentions, to look promptly for eco-friendly qualified products, which are produced from the natural resources [5].

Natural dyes are alternately obtained from natural resources (animal or plant matter) [6]. These are biodegradable, anti-allergic and non-toxic [7]. Since bronze age, dyeing was existed and practiced [8]. However, the uses of natural dyes were declined, since 1856, with the great influence of synthetic dyes, which have moderate-to-excellent colour fastness properties, an economical price and widely available [9]. During the decade of the 1990s, synthetic dyes, in view of non-sustainable aspects has been restricted and the natural dyes, even though with of limited existence, technical drawbacks (dying process complexities, blending issues, colour yield, limited colour shades and reproducible results, *etc.*) and lack of standardizations are gaining the significant importance [10]. Therefore, many textile industries have started looking at the possibilities of using natural dyes for dyeing and printing of textile goods [11]. However, natural dyes when often applied without mordants have poor fastness properties [12]. To overcome the affinity and colour fastness problems between the dyes and the textile fibers, mordants are generally used [13]. A mordant is a component which creates chemical reaction between the dye and the fibers [14]. Some of the commonly mordants used are alum, potassium dichromate, chrome, stannous chloride, stannic chloride, copper sulphate, ferrous sulphate, *etc.* [15].

A number of research efforts [16-19], have been accomplished with the aim of improving colour strength and fastness properties of natural dyes with textiles. Most of them have focused mainly on wool, cotton and silk fibers with the use of different mordants, application of various extraction processes (*i.e.* aqueous extraction, solvent extraction, ultrasonic extraction and super critical fluid extraction) and techniques/methods (pre-mordanting, simultaneous mordanting and post mordanting) [20-22]. These processes and techniques varied a lot. Amongst all, aqueous extraction and solvent extraction processes were frequently used because of satisfactory outcomes [23,24]. They applied these extraction processes with and without mordants for dyeing of cotton, silk, wool and flax textiles.

Pomegranate peel powder is reported for different color shades on textile fabric and for many health benefits. Adeel *et al.* [25] applied aqueous extraction for dyeing of cotton fabric using pomegranate peel without mordants. However, Kulkarni *et al.* [26] examined cotton dyeing with pomegranate peel through solvent extraction process. Pomegranates are important natural sources for producing yellow shades on the textile fabrics [27]. Pruthi *et al.* [28] have used aqueous extraction process for dyeing of silk with varied mordant concentrations through all mordanting methods. Alternatively, Win and Swe [29] have analyzed the silk dyeing and wool dyeing together by using the solvent extraction process for their results. Likewise, Goodarzian and Ekrami [30] investigated wool dyeing with pomegranate peel through both aqueous and solvent extraction process. In fact, the outcomes in literature assessment seems virtuous but having lack of standardization in procedures, commingle complexities and impediments which causes abstruse and confusions. Also, the literature review indicates that there have been relatively no research works on investigating the dyeing of tencel fabric with natural dye and any of the above mentioned techniques and methods.

This research, in the view of above, tencel (regenerated cellulosic fiber) fabric is investigated with natural dyes extracted from pomegranate peel by using different techniques as pre-mordanting, meta-mordanting (simultaneous mordanting) and after-mordanting (post mordanting). Furthermore, the standardization procedure of extraction (in solvent extraction phase) is also recommended for regeneration of results to reduce the complexities and confusions. The aim of the present work is to examine the appropriateness of pomegranate to provide the sufficient information about the extraction, dyeing behaviour and colour fastness properties in textile field.

EXPERIMENTAL

100 % Tencel fabric of weight 160 g/m^2 (plain weave, warp-30, weft-30, fabric density 128 X 82) was obtained from Xiaoshen Hangzhou, China. *Punica granatum* (Pomegranate Peel) was purchased from a domestic market of Shanghai, China. Ferrous(II) sulphate and copper(II) sulphate which are laboratory grade metallic salts were used as a chemical mordants.

Preparation of raw material: The peels were cut and removed from the pomegranates. They were further cleaned with water to remove the dirt and impurities. After drying them in the tray at temperature (26 °C) for 48 h, the pomegranate peels (brown coloured) were crushed and converted into powder form by using grinder machine. Fig. 1 shows the preparation steps of pomegranate powder (powder and granules). The dry granules and powder were separated and passed through a standard test sieve (BSS-45) for a uniform (equal graded) particle size. The powder was stored in an opaque airtight plastic bottle for further experimentations.

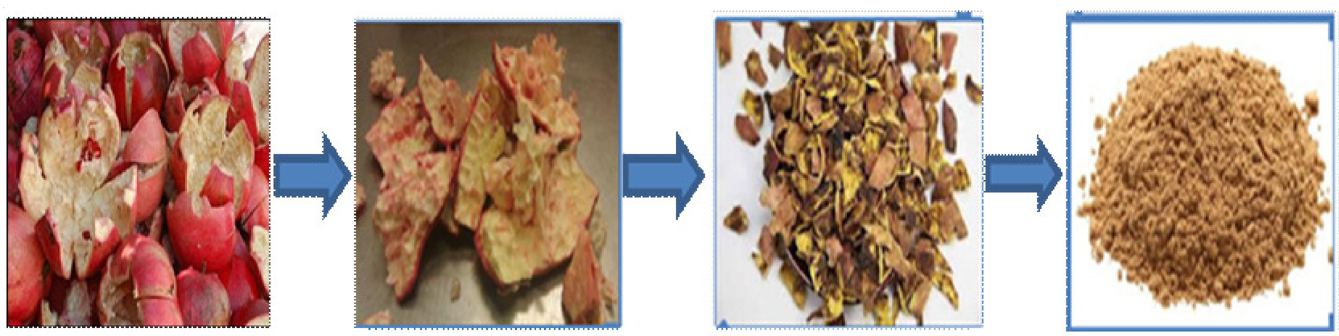
Method: The dried pomegranate powder (5 g) was taken in thimble (sample container) of apparatus. The solvents 120 mL (ethanol) and 80 mL of distilled water with a liquor ratio 1:40, in a round bottom flask (solvent container) were heated to 95 °C. The vapours were passed through the tube and raised up into the condenser. At the top, the vapours were condensed and dripped down into the thimble. The thimble was drained by the suction effect, when the condensed solvent was reached at the top level of siphon. The extracted material flew back into the round bottom flask and started mixing with the clean solvent. The working of the apparatus was continued for 100 min. The extract was purified through rotary evaporator and subsequently the filtrate dye solution was used for dyeing the samples of tencel fabric. It was observed that the colour of the dye extracted was brownish yellow. The weight of the extracted dye was 120 mL.

Dyeing process: The fabric samples were mordant prior to dyeing. Mordanting is a procedure that fixes the dyestuff to the textile materials. Without mordanting the dye uptake ability decreases. Three different methods were used for mordanting of tencel fabric samples. The differences between these three methods are summarized in Table-1.

After mordanting, dyeing process was carried out to add colours to the sample fabrics. The Tencel fabric was dyed by exhaust method with extracted dye from pomegranate peel. Tencel fabrics were dyed for 1 h with material to liquor ratio (MLR) 1:40 in water shaker dyeing machine, at 90 °C. After dyeing process, the dyed fabric samples were treated with soaping agents and washed with water. Subsequently, they were dried in an oven at 26 °C temperature for 24 h.

RESULTS AND DISCUSSION

In three different mordanting methods, pomegranate peel was used with ferrous sulphate and copper sulphate for dyeing tencel fabric samples at 90 °C. Characterization of dyed samples were performed to investigate different properties like colour strength, washing fastness (ISO 105-C01), light fastness (ISO



(a) Peels in fresh cut (b) Peels in washed form (c) Peels in dried form (d) Peels in powder form

Fig. 1. Preparation of pomegranate peel powder

TABLE-1
DIFFERENT METHODS USED FOR MORDANTING OF TENCEL FABRICS

Methods of mordanting	Chemicals treatment	pH	Temp. (°C)	Liquor ratio	Time (min)	Rinsed
Pre-mordanting method	Fabric samples were first mordanted with ferrous and copper sulphate and then used for dyeing	Neutral	Boiling	1:40	60	Dyed samples were rinsed with water and dried at room temperature
Simultaneous-mordanting method	The mordants and dye were added to the dyeing bath simultaneously	Neutral	Boiling	1:40	60	Dyed samples were rinsed with water and dried at room temperature
Post mordanting method	(i) Fabric samples were first dyed with pomegranate dye (ii) After-dying process, samples were carried out with mordants	Neutral	Boiling	1:40	60	Dyed samples were rinsed and dried at room temperature

105-B02), rubbing (dry and wet) (ISO 105-X12) and perspiration (ISO 105-E04). Colour strength of the dyed fabric samples is measured through absorption coefficient (K) and scattering coefficient (S) values. K/S values are determined through Kubelka-Munk equation which is shown below.

$$K/S = (1-R)\ 2/2R \qquad (1)$$

where K = absorption coefficient, S = scattering coefficient and R = reflectance.

Colour measurement and dye absorption: Table-2, shows the colouring effect on dyed tencel fabrics with mordants ferrous sulphate and copper sulphate through pre-mordanting, simultaneous-mordanting and post-mordanting methods at 90 °C. Spectra Flash-Data Colour (SF-600) was used to measure the dye absorption concentration on the surface of Tencel fabric by using K/S values. Range of colours on tencel fabrics were also measured by using L*, a*, b*, C*, h*.

Table-3 shows L*, a*, b*, C* and h* values of dyed tencel fabrics. CIE L*, a*, b* (CIE Lab) is a colour space standard, specified by the international commission on illumination. L*, a* and b* are the three axis of CIE-Lab system. The L* value shows luminance in CIE-Lab colour space and its values from 0 (black) to 100 (white). It explains that lower value of L* has higher darker shades and higher value of L* has lighter shades for tencel fabric. The +a* value indicates red and -a* values indicates green colour, while +b* values indicates yellow and -b* values indicates blue colour [31]. Due to chemical mordants, the highest colour value (K/S = 4.72) was found with ferrous sulphate and lowest colour value (K/S = 1.57) with copper sulphate.

Fig. 2 displays the results of K/S values of dyed tencel fabrics through three mordanting methods at 90 °C. In all the mordanting methods, K/S values with ferrous sulphate are represented through pink colour bars while K/S values with copper sulphate are represented with grey colour bars. K/S values were found more with ferrous sulphate as compare to copper sulphate mordant. Also the higher values of K/S (4.72) through pre-mordanting method depicts that ferrous sulphate

TABLE-2
SAMPLES OF DYED TENCEL FABRICS

Methods of mordanting	Ferrous sulphate	Copper sulphate	Original raw material
Pre-mordanting method			
Simultaneous-mordanting method			
Post mordanting method			

TABLE-3
CIE L* a* b*C* h* AND K/S VALUES OF DYED TENCEL FABRIC

Mordant	Mordant Methods	K/S	L*	a*	b*	C*	h*
$FeSO_4$	Pre-mordanting	4.72	44.95	0.55	1.10	1.23	63.41
	Simultaneous mordanting	1.95	78.59	3.71	15.63	16.06	76.63
	Post-mordanting	2.45	76.63	6.57	21.52	22.50	73.01
$CuSO_4$	Pre-mordanting	3.07	54.51	0.84	3.46	3.56	76.42
	Simultaneous mordanting	1.57	86.55	0.86	5.94	6.01	81.51
	Post-mordanting	2.05	82.16	4.13	18.17	18.64	77.20

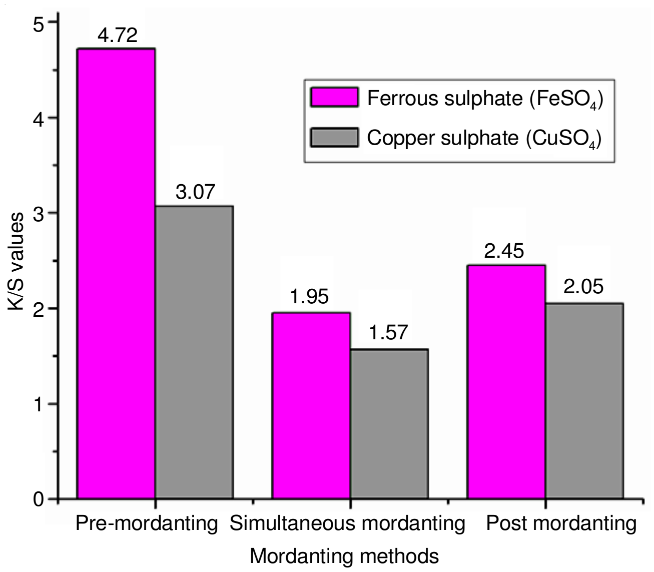


Fig. 2. Colour strength (K/S values) of dyed tencel fabrics through mordanting methods

has better coordination complexes between the dye and fibers in comparative to other methods.

Colour fastness properties of dyed tencel

Light fastness (ISO 105-BO2): Fig. 3 reveals the results of light fastness values of tencel fabric through ISO 105-BO2 of three mordanting methods. The pink colour bar indicates the usage with ferrous sulphate mordant while the grey colour bar shows the treatment with copper sulphate mordant. It is perceived that the light fastness with ferrous sulphate mordant

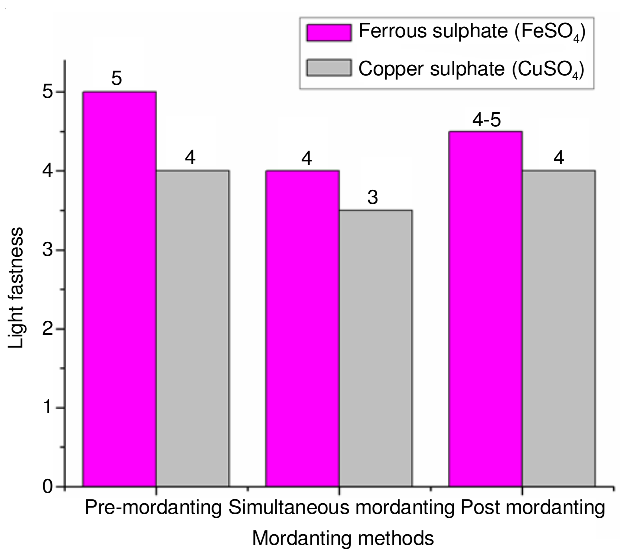


Fig. 3. Light fastness values of dyed tencel fabric through mordanting methods

is greater than the copper sulphate mordant in all the methods. The differences observed are (5:4, 4:3 and 4-5:4).In the figure, it is obvious that dyed tencel fabrics have good light fastness (4-5 grade) with mordant ferrous sulphate whereas copper sulphate mordant shows fair light fastness (3-4 grade) results. It is also observed that the results through pre-mordanting method are better quality. It is due to the fact that the samples show no change in colour or no colour fading.

Washing fastness (ISO 150-C01): The washing fastness with ferrous sulphate is represented by pink colour bars while the copper sulphate is signified with grey colour bars (Fig. 4). It is also displayed that pre-mordanting method is superior in comparative to other mordanting methods. For the outcomes, ISO 150-C01 was used. The results of washing fastness property through simultaneous-mordanting and post mordanting methods were similar. The quality grade of the washing fastness through ferrous sulphate is 4-5 whereas through copper sulphate, the quality grade is 4 in all mordanting methods and it showed a slight change in colour.

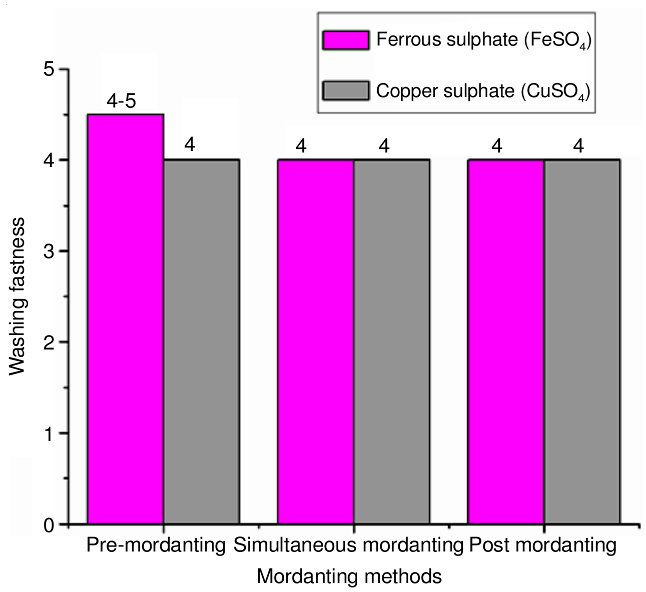


Fig. 4. Washing fastness values of dyed tencel fabric through mordanting methods

Rubbing fastness (dry and wet) (ISO 105-X12): Fig. 5 illustrates the rubbing fastness results of pomegranate peel dyed tencel fabric samples (dry and wet). The standard ISO 105-X12 was used for achieving the results. Pink colour and light pink bars indicate the dry and wet rubbing test results of dyed fabric samples with ferrous sulphate while the grey colour and light grey colour bars specify the dry and wet rubbing results of dyed fabric samples with copper sulphate in all mordanting

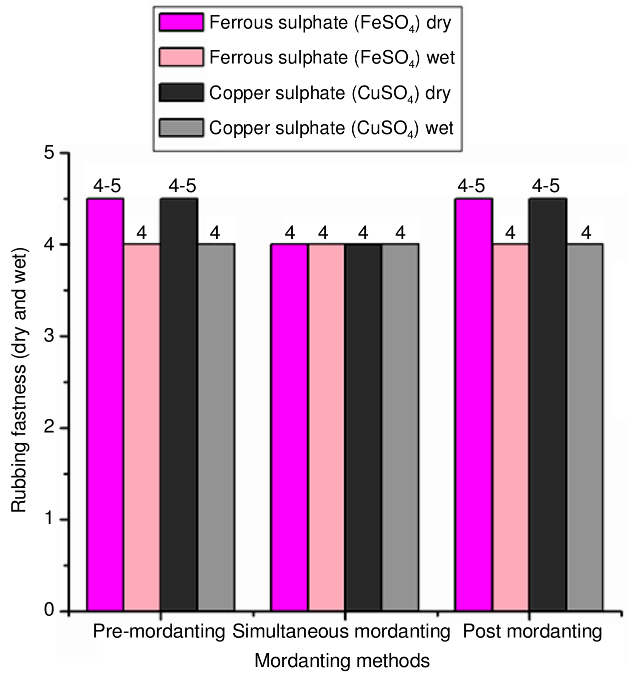


Fig. 5. Rubbing fastness (dry and wet) values of dyed tencel fabrics through mordanting methods

methods. It is found that the results of dry and wet rubbing fastness with ferrous sulphate and copper sulphate mordants are similar in each method *i.e.* ferrous sulphate (4-5: 4), copper sulphate (4-5: 4) in pre-mordanting method and post-mordanting method and in simultaneous-mordanting method the ferrous sulphate and copper sulphate has 4 grades. However, dry rubbing test results display good to excellent grade (4-5) in all methods while in the wet rubbing test results is equal to 4 grades in all mordanting methods.

Perspiration fastness (ISO 105-E04): Fig. 6 shows the perspiration fastness results of dyed tencel fabric through all mordanting methods. The pink colour bars represents the ferrous sulphate mordant and grey colour bars denotes the copper sulphate mordants. The standard ISO 105-E04 was applied. It is shown that the tencel fabric dyed with peel of pomegranate have good to excellent (4-5 grade) perspiration fastness (acidic and alkali) in all mordanting methods.

Conclusion

In this paper, the extracted dye from pomegranate peel was used as a potential source of synthetic dye. It is a first step to investigate the tencel fabric samples through pomegranate peel dye. The important results of the study under discussion can be summarized as follows:

• Mordants ferrous sulphate and copper sulphate both are active for colour fastness properties. However, ferrous sulphate is more effective than the copper sulphate mordant.

• Experimental results show that pre-mordanting method is the most effective for better quality in comparison to the other methods (simultaneous mordanting and post-mordanting) for dyeing tencel fabric samples.

• Ferrous sulphate mordant is better in light fastness, washing fastness and rubbing fastness properties through pre-mordanting methods. Conversely, the perspiration fastness is equivalent in with copper sulphate mordant in all mordanting methods.

Tencel fabric dyeing though pomegranate peel is biodegradable, supportive to environmental safety and protection determinations for the surrounding community. Thus, mordant ferrous sulphate is a good choice for dyeing tencel fabric through pre-mordanting method. In addition, it is low-cost than copper sulphate mordant and therefore cost effective. It has good colour strength (4.72), colour fastness to light (4-5), washing (4-5), rubbing (4-5) and perspiration (4-5) properties on tencel fabric dyed with extracted dye from pomegranate peel.

ACKNOWLEDGEMENTS

The research was supported by the Fundamental Research Funds for the Central Universities (No. 17D310503) and National Natural Science Foundation of China (No. 51403032).

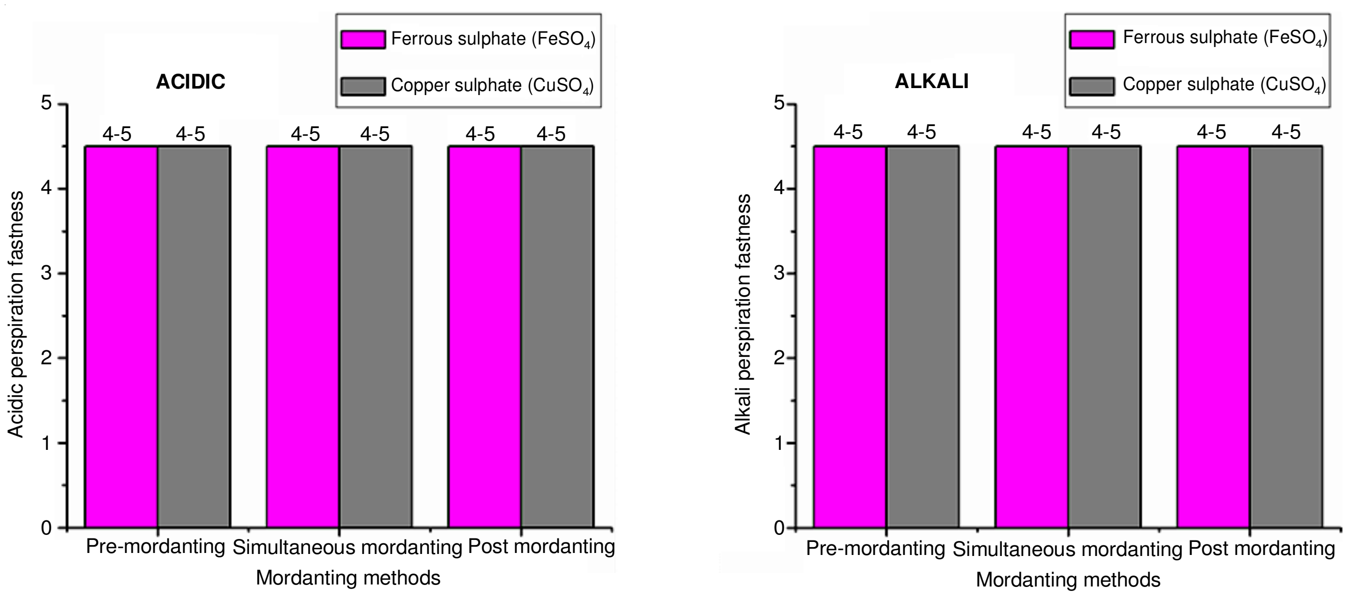


Fig. 6. Perspiration fastness results of dyed tencel fabrics through mordanting methods

REFERENCES

1. V.K. Gupta and Suhas, *J. Environ. Manag*., **90**, 2313 (2009); https://doi.org/10.1016/j.jenvman.2008.11.017.
2. H.C. Tiwari, P. Singh, P.K. Mishra and P. Srivastava, *Indian J. Fibre Text. Res*., **35**, 272 (2010).
3. Y.H. Lee, *J. Appl. Polym. Sci.*, **103**, 251 (2007); https://doi.org/10.1002/app.25221.
4. A. Kechi, R. Chavan and R. Moeckel, *Text. Light Ind. Sci. Technol*., 2, 137 (2013).
5. R. Shanker and P.S. Vankar, *Dyes Pigments*, **74**, 464 (2007); https://doi.org/10.1016/j.dyepig.2006.03.007.
6. A. Davulcu, H. Benli, Y. Sen and M.I. Bahtiyari, *Cellulose*, **21**, 4671 (2014); https://doi.org/10.1007/s10570-014-0427-8.
7. A.K. Samanta and P. Agarwal, *Indian J. Fibre Textile Res*., **34**, 384 (2009).
8. D. Jothi, *AUTEX Res. J.*, **8**, 49 (2008).
9. K. Prabhu and A.S. Bhute, *J. Nat. Prod. Plant Resour.*, **2**, 649 (2012).
10. A. Shams-Nateri, A. Hajipour, E. Dehnavi and E. Ekrami, *Cloth. Text. Res. J.*, **32**, 124 (2014); https://doi.org/10.1177/0887302X14525658.
11. Shahid-ul-Islam, M. Shahid and F. Mohammad, *J. Clean. Prod.*, **57**, 2 (2013); https://doi.org/10.1016/j.jclepro.2013.06.004.
12. A. Farooq, S. Ali, N. Abbas, N. Zahoor and M.A. Ashraf, *Asian J. Chem.*, **25**, 5955 (2013); https://doi.org/10.14233/ajchem.2013.14202.
13. R. Singh and S. Srivastava, Silk Dyeing with Natural Dye Extracted from Spice, International Conference on Inter Disciplinary Research in Engineering and Technology, New Delhi, India (2016).
14. R. Mongkholrattanasit, J. Krystufek, J. Wiener and M. Viková, *Fibres Text. East. Eur.*, **19**, 94 (2011).
15. R. Jihad, *Int. J. Sci. Eng. Res.*, **5**, 809 (2014).
16. R.R. Mahangade, P.V. Varadarajan, J.K. Verma and H. Bosco, *Indian J. Fibre Text. Res*., **34**, 279 (2009).
17. P.S. Vankar, R. Shankar and S. Wijayapala, *J. Text. Apparel Technol. Manag*., **6**, 1 (2009).
18. D. Cristea and G. Vilarem, *Dyes Pigments*, **70**, 238 (2006); https://doi.org/10.1016/j.dyepig.2005.03.006.
19. R. Prabhavathi, A. Devi and D. Anitha, *IOSR J. Polymer Textile Eng.*, **1**, 21 (2014).
20. M. Kamel, H. Helmy and N. El Hawary, *J. Nat. Fibers*, **6**, 151 (2009); https://doi.org/10.1080/15440470902958041.
21. V. Sivakumar, J. Vijaeeswarri and J.L. Anna, *Ind. Crops Prod.*, **33**, 116 (2011); https://doi.org/10.1016/j.indcrop.2010.09.007.
22. S. Saxena and A.S.M. Raja, Natural Dyes: Sources, Chemistry, Application and Sustainability Issues, In: Roadmap to Sustainable Textiles and Clothing, Springer, pp. 37-80 (2014).
23. L. Janani, L. Hillary and K. Phillips, *Int. J. Sci. Res. Publ.*, **4**, 1 (2014).
24. P. Lokesh and M. Swamy, *Der Chemica Sinica*, **4**, 111 (2013).
25. S. Adeel, S. Ali, I.A. Bhatti and F. Zsila, *Asian J. Chem.*, **21**, 3493 (2009).
26. S. Kulkarni, A.V. Gokhale, U.M. Bodake and G.R. Pathade, *Univ. J. Environ. Res. Technol.*, **1**, 135 (2011).
27. J.U. Lloyd, *Punica granatum*, Engelhard, Chicago (1897).
28. N. Pruthi, G.D. Chawla and S. Yadav, *Nat. Prod. Rad.*, **7**, 40 (2008).
29. Z.M. Win and M.M. Swe, *World Acad. Sci. Eng. Technol.*, **22**, 536 (2008).
30. H. Goodarzian and E. Ekrami, *World Appl. Sci. J.*, **8**, 1387 (2010).
31. K. Maha-In et al., *Dyeing Silk Fabric with Natural Dye from Longan Leaves Using Simultaneous Mordanting Method*. in *Materials Science Forum*. 2016. Trans Tech Publ.