
Microwave-Assisted Natural Dye Extraction from *Pterocarpus soyauxii*

T. Thangabai and K. Kalaiarasi*

Department of Textiles and Clothing,
Avinashilingam Institute for Home Science and Higher Education for Women,
Coimbatore, Tamil Nadu, India

ABSTRACT

The present study aimed at the extraction of dye from *Pterocarpus soyauxii* (padauk wood) saw dust using both conventional and microwave assisted extraction techniques. Various factors such as extraction time, temperature, pH, dye source concentration and irradiation power influencing the dye yield were studied. The extracted dye was applied to cotton fabric using padding mangle. UV Protection property and antibacterial activity of the dyed fabrics were also determined. The results of the present study showed that 2 % dye source concentration with extraction time of 120 sec and irradiation power of 360W at pH 10 resulted in maximum dye yield in microwave assisted dye extraction whereas 6 % dye source concentration at pH 10 with extraction time of 180 min at 90°C resulted in maximum dye yield in conventional extraction method. The cotton fabrics dyed with extracted dye showed antibacterial activity against *S.aureus* and *E.coli*. The fabric dyed with microwave assisted dye extract showed very good UV protection when compared with the fabric dyed with conventional dye extract. The dyed fabrics were assessed for colour fastness to washing, rubbing, sunlight and pressing and found to be good to excellent. The results indicated that microwave-assisted extraction method proved to be an excellent alternative technique for the extraction of natural colourant compared to conventional extraction method.

KEY WORD: Natural dye; Padauk wood saw dust; Microwave assisted extraction; UV protection factor.

1. INTRODUCTION

Natural dyes are used for colouring food, leather and textile fibres, like wool, silk and cotton [1]. Natural dyes are colourants that are obtained from natural sources without any chemical processing [2]. They are biodegradable, less toxic and allergenic than synthetic dyes and hence they are considered to be environmental friendly [3]. Also natural dyes provide functionality to fabrics such as antibacterial activity [4, 5] and ultraviolet protection [6].

Growing consumer interest in purchasing 'green' products and safe environmental conservation has led to the revival of natural colourants into the modern markets [2]. In recent years, an increasing interest has been developed for the potential use of plant waste as source of natural dye [7]. One such potential source is *Pterocarpus soyauxii* (Padauk) which belongs to the family Fabaceae. On exposure to light, the padauk wood red at first becomes brown colored. It is a multipurpose tree which produces timber, dye and has traditionally been used as a medicine. It can be used to treat various diseases like hypertension, diabetes, intestinal parasites, renal and skin diseases.

Bark extract is used in herbal medicine to treat fungal infections. *P. soyauxii* bark contains various compounds such as biflavonoids (santalins A, santarubins A and B) isoflavonoids (pterocarpin, formononetin and prunetin), an isoflavone quinine (claussequinone), isoflavanes (vestitol and mucronulatol), tannins, ascorbic acid, glucosides, triterpenes and xanthenes [8, 9].

Textile dyers use the aqueous extraction method to extract natural dyes from plant sources. Plants generally give less amount of coloring matter on extraction with water, as the coloring component is tightly bound to the cell wall. Also, this method takes several hours of extraction time. It is therefore important to utilize most efficient extraction method in textile dyeing [10]. Microwave assisted extraction (MAE) has received

increasing attention as a potential alternative to traditional solid-liquid extraction methods mainly due to considerable saving in processing time and solvent consumption [11].

Only limited work has been reported on the studies comprising the effect of microwave radiation on extraction of dye from plants. Hence to explore the potential use of microwave irradiation on dye extraction, the present research was carried out to investigate the feasibility of the application of microwave in the extraction of dye from padauk wood saw dust and optimize the conditions for its extraction. The antibacterial efficacy and UV protection property of the dyed fabrics were evaluated.

2. MATERIALS AND METHODS

2.1 Selection of dye source

Application of waste materials as source of natural dyes can assist in the preservation of the environment and also decrease the cost of natural dyeing. Hence padauk wood saw dust was selected as dye source. Padauk wood saw dust was collected from Sri Balaji Timber Mart, Mettupalayam Road, Coimbatore, Tamil Nadu. The collected padauk wood saw dust was washed under flowing water to remove dust particles and shade dried at room temperature and grinded to powder form in an electrically operated grinder.

2.2 Fabric

Cotton fabric of plain weave was purchased from Murugan mills, National Textile Corporation Ltd., Coimbatore, Tamil Nadu. Before dyeing, the fabric was desized, scoured and bleached.

2.3 Conventional extraction

Conventional extraction was carried out using 1g of padauk wood saw dust in 100ml of water. The solution was boiled for 1 h at 100°C. The dye extract was filtered. Different extraction parameters such as dye source concentration, extraction time, pH and temperature were optimized. In order to study the effect of dye source concentration, extraction was carried out at different concentrations of powdered padauk wood saw dust (1-10%). The optimum extraction time was determined by carrying out the dye extraction at different time intervals such as 30, 60, 90, 120, 180, 210, 240, 270 min. The effect of temperature on dye yield was investigated by extracting the dye at different temperatures such as 30°C, 40°C, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C. To determine the optimum pH for dye extraction, the pH of the solvent was adjusted to 5-12 using acetic acid or sodium carbonate. The extracted dye from Padauk wood saw dust was filtered and the colour yield was measured using UV Visible Spectrophotometer at 517 nm (maximum absorption wave length).

2.4 Microwave assisted extraction

The process of microwave assisted extraction method was performed in experimental microwave equipment. Padauk wood saw dust (1g) was weighed and transferred into 500ml microwave container and 100 ml distilled water was added. The container was exposed to microwave for 2 min. The colour intensity of the dye extract was measured in UV-Visible Spectrophotometer at 517 nm. To determine the optimum concentration of the dye source, different amounts of powdered padauk wood saw dust (1-10%) was taken individually in a microwave container and dye was extracted for 2 min. To study the effect of time on dye yield, extraction was carried out at different time intervals (30, 60, 90, 120, 180, 210, 240, 270 sec) and exposed to different powers (180, 360, 540, 720, 900W). To determine the optimum pH, the pH of the solvent was adjusted to 5-12 using acetic acid or sodium carbonate. The extracted dye from Padauk wood saw dust was filtered and the colour yield was measured using UV Visible Spectrophotometer at 517 nm.

2.5 Mordanting

Cotton fabric was treated with 5% on weight of fabric of alum at 90°C for 1 h, keeping the material to liquor ratio of 1:30.

2.6 Dyeing method

Dyeing of the selected cotton fabric using dye solution extracted by conventional and microwave assisted extraction was done using padding mangle. After dyeing, the fabrics were rinsed with cold water and air-dried.

2.7 Antibacterial Activity

Sterile Muller Hinton agar medium was prepared and swabbed with *Staphylococcus aureus* and *E. coli* separately. The original and dyed fabric samples were placed on the above medium and incubated at 37°C for overnight. After incubation, the zone of inhibition formed against the tested bacteria was measured.

2.8 UV Protection ability

The UV protection factor (UPF) and UV transmittance of the original and dyed cotton fabrics were measured using a Labsphere UV -1000F UV Transmittance Analyzer in the range of 280-400 nm. The UPF value of each fabric was determined from the total spectral transmittance based on AATCC 183:2010 test method, as follows.

$$UPF = \frac{\sum_{\lambda=290}^{400} E_{\lambda} S_{\lambda} \Delta_{\lambda}}{\sum_{\lambda=290}^{400} E_{\lambda} S_{\lambda} T_{\lambda} \Delta_{\lambda}}$$

Where

E_{λ} - is the relative erythemal spectral effectiveness (unitless)

S_{λ} - is the solar Ultraviolet radiation (UVR) spectral irradiance in $W.m^{-2}.nm^{-1}$

T_{λ} – is the measured spectral transmission of the fabric

Δ_{λ} – is the bandwidth in millimeter

λ - is the wavelength in nanometer

The UVR band consists of three regions: the UV-A band (320 nm to 400 nm), the UV-B band (290 nm-320 nm) and UV-C band (200 nm-290 nm) [12]. UV-A causes little visible reaction on the skin but has been shown to decrease the immunological response of skin cells. UV-B is most responsible for the development of skin cancers. Therefore, transmittance of UVR (UV-A and UV-B) through the fabrics was evaluated in this experiment. Fabrics with a UPF Value in the range of 15 to 24 are defined as providing “good UV protection”, 25 to 39 as “very good UV protection” and 40 or greater as excellent UV protection. There is no rating assigned if the UPF value is greater than 50 [13].

2.9 Evaluation of colour fastness and physical properties of the dyed fabrics

Laboratory tests like fabric weight, strength, thickness, stiffness and colour fastness were carried out for original and the dyed fabrics. All experiments were carried out in triplicates, and average values are reported.

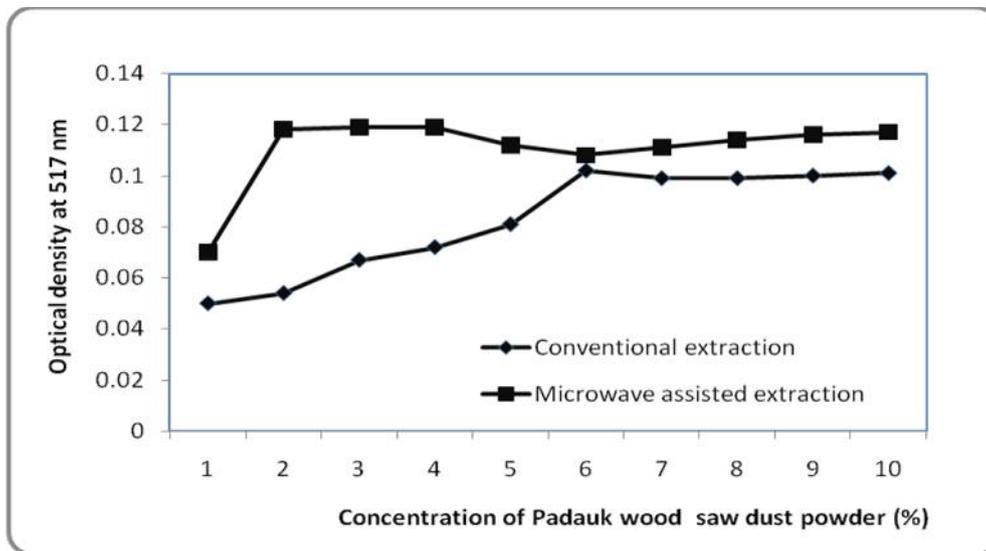
3. RESULTS AND DISCUSSION

3.1 Optimization of various parameters for dye extraction

Effect of dye source concentration

In conventional extraction method, dye yield increased with increase in dye source concentration and reaches maximum at 6 per cent, whereas in microwave assisted extraction method 2 per cent dye source concentration resulted in maximum dye yield (Fig 1).

Fig. 1 Effect of dye source concentration on dye yield

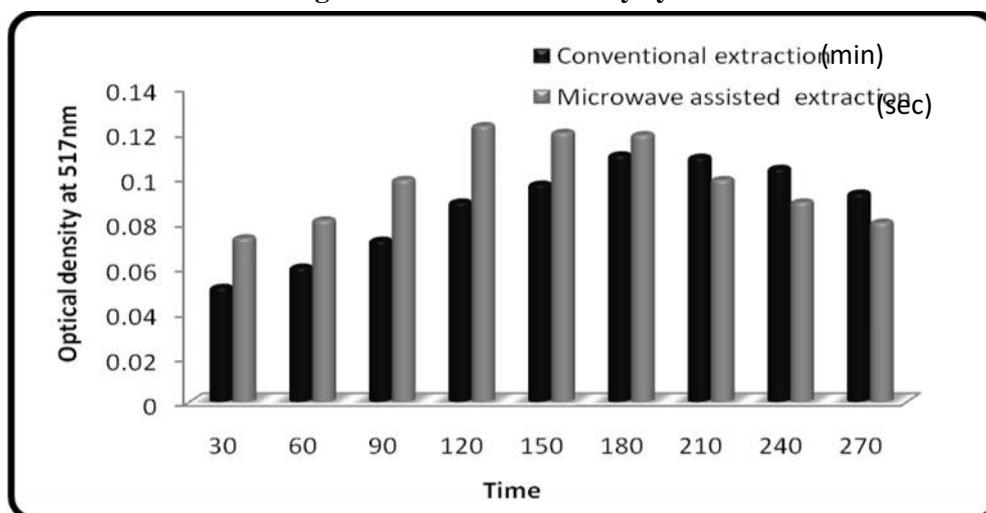


Increase in dye source concentration above 6 per cent in conventional method and 2 per cent in microwave assisted extraction method does not found to show appreciable increase in dye yield. The results indicate that microwave assisted extraction method requires lesser amount of dye source concentration as compared to conventional extraction method. This might be due to microwave irradiation which loosens the cell wall matrix and leads to severing of the parenchymal cells [14]. Also the tissues are rapidly and extensively opened up by the microwave leading to increased interaction between extracting agent and source material in the extraction process, thereby enhancing the yield of natural dye [15].

Effect of extraction time

The effect of time on dye extraction was studied and the results clearly indicate that the dye yield increased with increase in time up to 180 min in conventional method whereas in microwave assisted extraction 120 sec was found to produce maximum dye yield (Fig.2). Further increase in time does not increase the dye yield which may be due to degradation of the dye molecule. Hence 180 min and 120 sec was selected as optimum time for conventional and microwave assisted extraction of the dye respectively. The obtained result was in concurrence with Sinha et al. [16] who have reported 3 hrs as optimum time for conventional extraction and 2 min as optimum time for the microwave assisted extraction of dye from pomegranate rind.

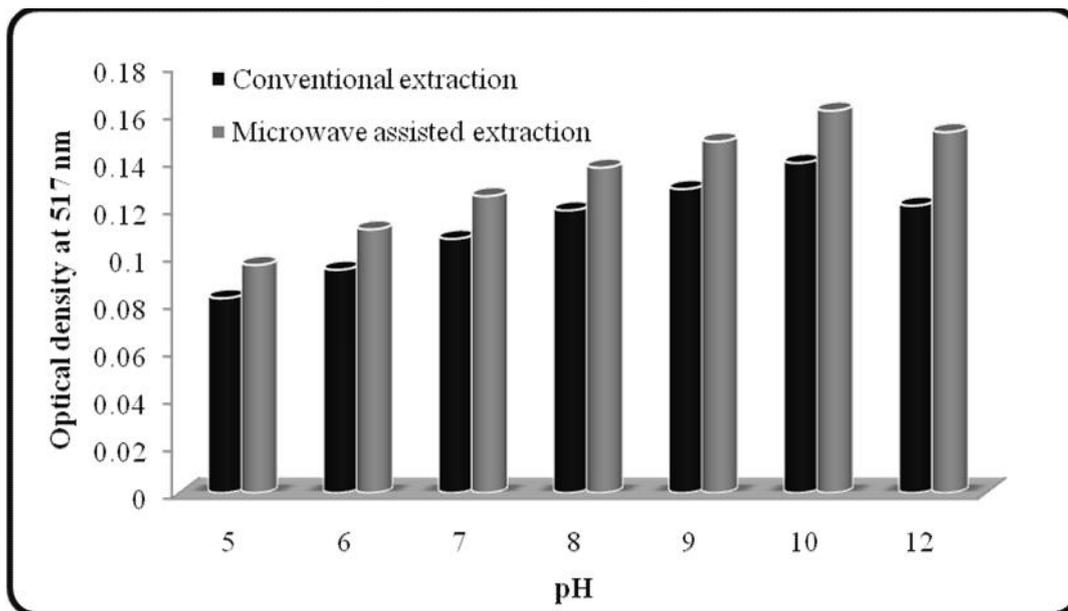
Fig.2 Effect of time on dye yield



Effect of pH

The pH of the extraction medium is an important process parameter because it can exert great impact on the stability of natural dyes. It is observed from Fig.3, that the colour intensity of dye extracts attained a maximum at pH 10 in both conventional and microwave assisted extraction methods. Beyond this pH, the colour intensity decreased. Hence pH 10 was selected as optimum pH for both conventional and microwave assisted extraction methods. The dye yield was found to be maximum at alkaline pH due to the increased ionization of hydroxyl groups in alkaline medium. The obtained result was in concurrence with Tiwari *et al.* [17] who have reported pH 10 as optimum for the extraction of dye from Pomegranate rind.

Fig. 3 Effect of pH on dye yield



Effect of extraction temperature and microwave power

Extraction of natural dyes in an aqueous solution is temperature dependant. Results of this study indicates a linear increase in dye yield from 30- 90°C after which it showed a decreasing pattern in conventional method. High temperature is beneficial to diffusion but too high temperature may result in the decomposition of the dye components. In microwave assisted extraction 360W was found to produce maximum dye yield. Hence 90°C was selected as optimum temperature for conventional method and 360W was selected as optimum power for microwave assisted dye extraction. The obtained result was in concurrence with Mongkholrattanasit *et al.* [1] and Ramadevi and Kalaiarasi [18] who have reported 90°C as optimum temperature for the extraction of dye from eucalyptus leaves and *Ricinus communis* leaves respectively. However, highest colorant yield in mesocarp and exocarp of *Cocos nucifera* (coconut palm) was reported to be at 100W and 500W, respectively [19].

3.2 Antibacterial activity

Natural dyeing with padauk wood saw dust powder increased the antibacterial activities of cotton fabrics against *E.coli* and *S.aureus*. The fabric dyed with microwave assisted dye extract exhibited a zone of inhibition of 6mm for *S.aureus* and 8mm for *E.coli* respectively whereas the fabric dyed with conventional dye extract exhibited a zone of inhibition less than 2mm for both *S.aureus* and *E.coli*. The antimicrobial activities of natural dyes may be due to their chromophores and the functional groups present in their molecules [20]. Similar such antibacterial activities were observed in natural yellow dyes curcumin [21] and tamarind seed coat tannin dyed fabrics [22] against *E.coli* and *S.aureus*.

3.3 UV Protection ability

Natural dyes due to its UV absorption characteristics can impart a certain degree of UV protection to the dyed fabrics. In the present study the UV Protection property of the dyed samples was found to be higher when compared to control fabric as shown in Table 1.

The UV Protection factor was found to be higher in sample dyed with microwave assisted dye extract as compared to sample dyed with conventional extract. The results indicate that the color yield and dye uptake was higher with microwave assisted dye extract when compared to conventional extract. The UV protection property of fabrics is evaluated as “good” when the UV transmittance is less than 5 per cent [12]. The UV transmittance of the original fabric was found to be 5.22 per cent in UV-A band and 5.93 per cent in the UV-B band indicating that the resistance of undyed fabric to Ultra violet rays was good.

Table 1. UPF values and Percentage UV transmission

Sl.No	Sample	UPF	Percentage UVA transmission	Percentage UVB transmission	UV protection class
1.	Original cotton	17	5.22	5.93	Good
2.	Cotton dyed with conventional dye extract	28	3.85	3.35	Very Good
3.	Cotton dyed with microwave assisted dye extract	39.32	3.07	2.41	Very Good

UPF-Ultraviolet Protection Factor

The UV transmittance of the conventional extract dyed fabric was found to be 3.85 per cent in UV-A band and 3.35 per cent in the UV-B band indicating that the resistance of the dyed fabric to Ultra violet rays is very good. The UV transmittance of the microwave assisted extract dyed fabric was found to be 3.07 per cent in UV-A band and 2.41 per cent in UV-B band indicating that the resistance to UV rays was better when compared to conventional extract dyed fabric. A high correlation exists between the UPF and fabric porosity. The decrease in the percentage of UV- A and UV-B transmission values and increase in Ultraviolet Protection Factor can be ascribed to reduction in fabric porosity due to dye absorption. Salah [23] reported good UV protection property of banana peel dyed cotton fabric.

3.4 Fabric analysis

The fabric weight, strength, thickness and stiffness of the dyed samples in warp and weft directions increased after dyeing when compared to the original fabric. The dyed samples showed good fastness to sunlight. With regard to pressing and crocking, all the samples were found to have good to excellent fastness. All the dyed samples were found to have excellent washing fastness.

CONCLUSION

The study reveals that microwave assisted extraction of padauk wood is more efficient as compared to conventional extraction methods. Extraction time, concentration of the dye source, temperature and pH of the solution markedly influenced the dye extraction. Application of microwave irradiation in the extraction of dye from dried padauk wood powder dramatically reduced extraction time as well as improved the yield of dye. The colorant from padauk wood has antibacterial and very good UV Protection properties. Sun protective clothing is an excellent sun protection measure as it provides a physical block that doesn't wash or wear off and can protect the skin from UV radiation. Hence padauk wood extract dyed fabrics can be used for protective textiles. It could be concluded that the microwave assisted extraction technique is highly effective in terms of saving the processing time and energy. Findings of the paper reveal that the natural dyes extracted out of superficial barks, otherwise wasted, can be a sustainable alternative to synthetic dyes in developing countries like India in the long run.

REFERENCE

- [1]. Mongkholrattanasit, R., Krystufek, J., Wiener, J., and Vikova, M. 2011. Dyeing, fastness and UV Protection properties of silk and wool fabrics dyed with eucalyptus leaf extract by the exhaustion process. *Fibres and Textiles in Eastern Europe Journal*, **19**: 94-99.
- [2]. Bhargava, D., and Shahnaz, J. 2013. Microbial dyes: A new dimension to natural dyes. *Colourage*, **60**:42.
- [3]. Mongkholrattanasit, R., Krystufek, J., and Wiener, J. 2010. Dyeing and fastness properties of natural dye extracted from eucalyptus leaves using padding techniques. *Fibers and Polymers*, **3**: 346–50.
- [4]. Yusuf, M., Ahmad, A., Shahid, M., Khan, M.I., Khan, S.A., Manzoor, N., and Mohammad, F. 2012. Assessment of colorimetric, antibacterial and antifungal properties of woollen yarn dyed with the extract of the leaves of henna (*Lawsonia inermis*). *Journal of Cleaner Production*, **27**: 42-50.
- [5]. Bin, Z., Lu, W., Liangfei, L., and Martin, W.K. 2014. Natural dye extracted from Chinese gall – the application of color and antibacterial activity to wool fabric. *Journal of Cleaner Production*, **80**: 204-10.
- [6]. Hou, X.L., Chen, X.Z., Cheng, Y.X., Xu, H.L., Chen, L.F., and Yang, Y.Q. 2013. Dyeing and UV protection properties of water extract from orange peel. *Journal of Cleaner Production*, **52**: 410-19.
- [7]. Singh, S.B., Jyotsnarani, J., Das, T., and Das, N.B. 2013. Role of cationic and anionic surfactants in textile dyeing with natural dyes extracted from waste plant materials and their potential antimicrobial properties. *Industrial Crops and Products*, **50**:618-24.
- [8]. Betti, J.L. 2004. An ethno-botanical study of medicinal plants among the Bakapymies in the Dja biosphere reserve, Cameroon. *African Study Monographs*, **25**(1):1-27.
- [9]. Tchamadeu, M.C., Dzeufiet, P.D.D., Nana, P., Kouambou, N.C.c., Ngueguim, A.F., Allard, B.J., Siagat, N., Zapfack, R.L., Girolami, J.p., Tack, I., Kamtchouing, P., and Dimo, T. 2011. Acute and sub-chronic oral toxicity studies of an aqueous stem bark extract of *P. Soyauxii* Tauab (Papilionaceae) in rodents. *Journal of Ethnopharmacology*, **133**:329-35.
- [10]. Eskilsson, C.S., and Bjorklund, E. 2000. Analytical-scale microwave assisted extraction. *Journal of Chromatography A*, **902**:227-50.
- [11]. Minoo, D., Shabnam, S., Alireza, G, Ali, R and Mohammad, T. 2005. Optimization of microwave assisted extraction for alizarin and purpurin in Rubiaceae plants and its comparison with conventional extraction methods. *Journal of Separation Science*, **28**:387-96.
- [12]. Feng, X.X., Zhang, L.L., Chen, J.Y., and Zhang, J.C. 2007. New insights into solar UV-protectives of natural dye. *Journal of cleaner production*, **15**:366-72.
- [13]. Sarkar, A.K. 2004. An evaluation of UV protection imparted by cotton fabric dyed with natural colourants. *BMC Dermatology*, **4**: 1-8.
- [14]. Bagherian, H., Ashtiani, F.Z., Fouladitajar, A., and Mohtashamy, M. 2011. Comparisons between conventional; microwave and Ultrasound-assisted methods for extraction of pectin from grape fruit. *Chemical Engineering and Processing*, **50**:1237-43.
- [15]. Sinha, K., Chowdhury, S., Saha, P.D., and Datta, S. 2013. Modeling of microwave-assisted extraction of natural dye from seeds of *Bixa orellana* (Annatto) using response surface Methodology (RSM) and artificial neural network (ANN). *Industrial Crops and Products*, **41**:165-71.
- [16]. Sinha, K., Saha, P.D., and Datta, S. 2012. Response surface optimization and artificial neural network modelling of microwave assisted natural dye extraction from pomegranate rind. *Industrial Crops and Products*, **37**:408-14.
- [17]. Tiwari, H., Pratibhasingh, C., Mishra, P.K., and Srivastava, P. 2010. Evaluation of various techniques for extraction of natural colorants from pomegranate rind-Ultra sound and enzyme assisted extraction. *Indian Journal of Fibre and Textile Research*, **35**: 272-76.
- [18]. Ramadevi, S., and Kalaiarasi, K. 2015. Optimization of microwave assisted extraction of natural dye from *Ricinus communis* L leaves. *International Journal of Advanced Science and Engineering*, **2**:298-302.
- [19]. Zulrushdi, N.A.F., Hassan, R.M., and Yusoff, A.M. 2016. Microwave-assisted extraction of natural colorant extracted from mesocarp and exocarp of *Cocos nucifera* (coconut palm). *European Journal of Biotechnology and Bioscience*, **4**: 1-5.
- [20]. Shahid, M., and Shahid-ul-Islam Mohammad, F. 2013. Recent advancements in natural dye applications: a review. *Journal of Cleaner Production*, **53**:310-31.
- [21]. Zhou, Y., Zhang, J., Tang, R., and Zhang, J. 2015. Simultaneous dyeing and functionalization of silk with three natural yellow dyes. *Industrial Crops and Products*, **64**:224-32.
- [22]. Prabhu, K.H., and Teli, M.D. 2014. Eco-dyeing using *Tamarindus indica* L. seed coat tannin as a natural mordant for textiles with antibacterial activity. *Journal of Saudi Chemical Society*, **18**:864-72.
- [23]. Salah, S.M. 2013. Antibacterial activity and ultraviolet (UV) protection property of some Egyptian cotton fabrics treated with aqueous extract from banana peel. *African Journal of Agricultural Research*, **8**:3994-4000.