

Enhancing Dyeing of Wool Fabrics with Natural Kamala Dye via Bio-Treatment with Safflower Extract

M.F. Shahin and H.S. El-Khatib

Textile Printing, Dyeing and Finishing Department, Faculty of Applied Arts, Helwan University, Cairo, Egypt

Copyright © 2016 ISSR Journals. This is an open access article distributed under the **Creative Commons Attribution License**, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

ABSTRACT: In this work an attempt has been made to optimize the process of dyeing wool fabric with Kamala natural dye, firstly by enhancing the colour strength obtained via treating wool with Safflower enzyme extract.

The noticeable increasing in colour strength as a result of bio-treatment with Safflower extract is attributed to the enzyme extract which contains mainly lipase and protease enzymes which improve the dyeability of wool fabrics. All the parameters that may affect the bio-treatment process were studied in details, also the dyeing parameters such as dye concentration, pH value of the dyeing bath; both dyeing temperature and time were studied to determine the optimum conditions for both the treatment and dyeing processes.

In order to develop an eco-friendly natural dyeing process that is compatible with the environment, fixation process was made by using pomegranate as a natural mordant for the Kamala natural dye instead of the artificial ones. The factors affecting the fixation process with pomegranate natural mordant were investigated to conclude the optimum conditions of fixation stage.

It is obvious from this study the possibility of having bright deep colour from Kamala natural dye on the bio-treated wool fabric with good fastness properties that will match with the recent demands of using eco-friendly materials to be attuned with the environment.

KEYWORDS: Wool, Kamala natural dye, dyeing, bio-treatment, lipase, protease enzyme, pomegranate.

1 INTRODUCTION

Green chemistry using biotechnology has joined incredible importance in the textile wet processing industry. The search for new ,efficient and eco-friendly alternatives have increased interest in using green catalysts i.e., enzymes. Enzyme are organic protienaceous catalysts producing by all living cells .They obtained from three sources i.e., plant ,animal and microbial source like bacteria.[1] There are some researches related enzymes extracted from natural sources such as fungal source (brewer's yeast) [2-4] and plant source (seeds of safflower).[5] In general the use of enzymes leads to a reduction in water and energy consumption . In addition, they can often replace toxic chemical agents and be recovered from the wastewater and reused ,satisfying both environment and economic requirements.[6] Enzymes have been used for desizing, scouring ,polishing , washing, degumming and bleaching of fabrics as well as for decolouring of dyehouse wastewater.[1,7-10]

Wool fibres are one of the most popular natural biopolymers and they are commonly used for high grade textiles. However , existence of characteristic scales on the surface of wool fibres produces some undesired effects such as diffusion difficulty for dye molecules and felting propensity , thus hindering the process and application of wool products .[11] Several studies reported that biotreatment of wool improved antishrinkage properties , removed impurities and increased subsequent dyeing affinity were determined .[12-15]

The increasing awareness of health and pollution hazards of chemical dyestuffs has increased the interest in using natural dyes. Natural dyes have better biodegradability and generally have higher compatibility with the environment. They are nontoxic, non-allergic to skin, non-carcinogenic, easily available and renewable. [16] The lack of colour reproducibility is no

longer of importance but the issue of regarding light fastness and dye uptake remain. While metal salts mordants may offer good results, they do not satisfactorily address environmental concerns.[1] The usage of natural mordants is important to advance an eco-dyeing process. From this point of view, usage of natural mordants is with great importance to advance an eco-dyeing process, as using of Tamarindus Indica L. seed coat tannin as a natural mordant. [17] Also the powdered rind, or skin, of the pomegranate (*Punica granatum*) can be used as a tannin mordant, as well as a dye. [18]

The aim of this work is to use enzymes extract from plant(safflower) in order to develop an environmentally friendly alternatives to chemical processes and to examine the efficiency of safflower enzyme extract and its action on the woolen fabrics and subsequently on its dyeability when dyeing with Kamala natural dye. As well as to develop a complete eco-dyeing system that matches with the worldwide demands to use green substances through using natural dyes along with natural mordants

2 EXPERIMENTAL

2.1 MATERIALS

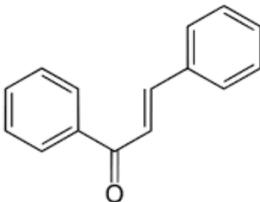
2.1.1 FABRIC

Scoured and bleached wool fabric (~ 154 g/m²) was kindly supplied by Misr Helwan Co. for Spinning and Weaving Helwan, Egypt.

2.1.2 DYES

Kamala natural dye (extracted powder) was produced by “Table Rock Llamas Fiber Arts Studio, Inc.”, Colorado, USA. The structure of Kamala dye was introduced in Table(1)

Table (1) : Structure of Kamala dye [19]

Common name	Botanical name	Part used	Chemical structure	C.I No.
Kamala	<i>Mallotus phillipensis</i>	Fruits	Chalcone structure “Open chain analogues of flavonoids” 	Natural orange 2

2.1.3 ENZYMES

Safflower seeds(*Carthamustinctorius*) were used to obtain enzymes extract for biotreatment of wool fabric .The seeds of Safflowers were obtained from local market.

2.1.4 MORDANTS

Pomegranate (*Punica granatum L*) dried powder “used as a natural mordant” was obtained from “Table Rock Llamas Fiber Arts Studio, Inc.”, Colorado, USA.

2.1.5 CHEMICALS AND AUXILIARIES:

Sodium carbonate and acetic acid of laboratory grade were used. Dispersing agent (Ebcasperse N) and non ionic detergent: (Chromatech 3QJ) supplied by Chromatech Co. Egyptian British Co.

2.2 METHODS

2.2.1 PREPARATION OF THE SAFFLOWER ENZYME EXTRACT

The safflower seeds were soaked in water for 12 hours, and then they were germinated for 3 days after which they were grinded, then water was added ($1000\text{cm}^3 / 500\text{gm}$ of seeds) while stirring. Finally the extract was filtrated and stored in the refrigerator.

2.2.2 BIO-TREATMENT OF WOOL FABRICS

Wool fabrics were bio-treated with safflower extract solution at different concentrations ranging from 25 to 100% according to the volume of treatment bath.

The treatment process was carried at different pH values (4-8), for different durations ranging from 30 to 120 minutes and at temperatures from 40°C - 80°C . The pretreated samples were rinsed with cold water and then with hot water at 80°C for 10 minutes. Finally the samples were rinsed with cold water and air-dried.

After which the samples were dyed with kamala natural dye at L.R 1:100, 6% (o.w.f) dye and 2g/L dispersing agent at pH= 5.5. The dyeing process started at about 40°C then the temperature was gradually raised to 80°C during 15 minutes, after which the dyeing process continued for 60 min., then washing process was performed with 2g/L detergent at 50°C for 10 minutes.

2.2.3 DYEING OF WOOL FABRIC WITH KAMALA DYE

The biotreated wool fabrics(treated at the optimum condition) were dyed with Kamala natural dye at different dye concentrations ranging from 2 to 10% (o.w.f) at pH levels (4-8) for different durations (15-75 minutes) at temperatures ranging from 40°C - 100°C using 2g/L dispersing agent.

The dyed samples were then washed as mentioned earlier. The dyeing process is illustrated in **Fig. (1)**

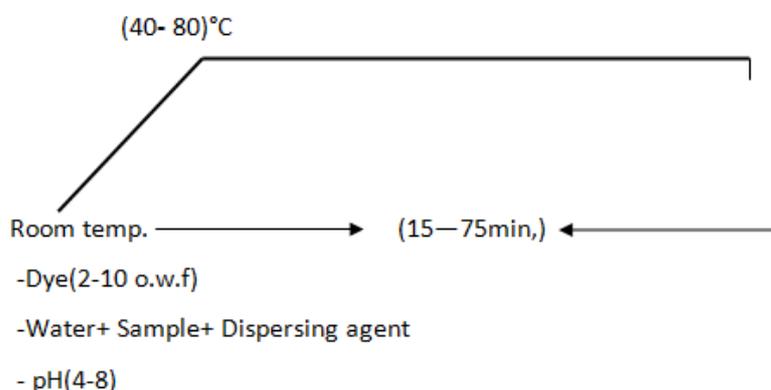


Fig. (1): Dyeing process curve

2.2.4 FIXATION OF KAMALA DYE USING POMEGRANATE NATURAL MORDANT:

After reaching the optimum conditions of the dyeing process ,pre-mordanting process were carried out and factors affected the fixation statement were determined. The pretreated wool samples were subjected to fixation process using pomegranate as a natural mordant, at bath L R 1:100, using 4% - 10 % (o.w.f) pomegranate solution, the pH value was adjusted at (4-8) , the temperature from 40°C to 100°C , time: 15-60 minutes , then the optimum dyeing was applied Finally the washing process takes place after mordanting as mentioned before.

2.3 MEASUREMENTS AND TESTING

2.3.1 COLOUR MEASUREMENTS

The dyed samples were subjected to colour measurement by using reflection spectrophotometer model Optimatch 3100, SDL Company, England. The K/S values were obtained directly according to Kubelka Munk equation:

$$K/S = (1 - R)^2 / 2R$$

Where K and S are the absorption and scattering coefficient respectively, and R is the reflectance of the dyed fabric.

2.3.2 DETERMINATION OF THE WETTABILITY

A standard method for measuring wettability was used to determine the wettability of the wool samples treated with different concentrations of safflower enzyme extract.

2.3.3 TENSILE STRENGTH OF FABRIC

This test was carried out in order to determine the change of the tensile strength of wool fabric after treating with enzymes extracted from safflower seed.

This Test was carried out on a tensile strength testing apparatus (QMat 5.37) according to ISO text method "EN ISO 13934-1; 1999 Maximum Force & Elongation- Strip Method".

2.3.4 COLOUR FASTNESS

Fastness properties of untreated and treated wool fabrics dyed with kamala natural dye and mordanted with pomegranate were evaluated according to ISO standard methods. The specific tests were: colour fastness to washing: ISO-X12(1987), and colour fastness to perspiration: ISO 105-E04. The samples were also subjected to light fastness standard test by AATCC test method 16-1993.

2.3.5 SCANNING ELECTRON MICROSCOPE ANALYSIS

Fiber samples for treated and untreated wool fabrics were viewed under scanning electron microscope (JE 100 s), at magnification=6000 x.

2.3.6 ENZYMATIC ANALYSIS OF THE SAFFLOWER SEEDS EXTRACTION

LIPASE ASSAY

Lipase activity was determined colorimetrically by p-nitrophenyl palmitate (pNPP) method.[20] The assay mixture contained 500 µL of the assay reagent [1 mL of pNPP (90 mg in 30 mL 2-propanol) and 9 mL of (Triton-X 100, 2.0 and gum Arabic, 0.5 g in 450 mL Tris/HCl buffer, 50 mM, pH 7.0). and 500 µL of enzyme solution. After incubation of the enzyme solution with substrate in water bath for 25 min at 40°C, the liberated p-nitrophenol was measured at 410 nm. One unit of enzyme was defined as the amount of enzyme that releases 1 µmol of p-nitrophenol per min.

PROTEASE ASSAY

The protease activity was determined using casein in 0.1 M phosphate-buffer, pH 7.0. The casein solution (500 µL) and 500 µL of enzyme solution was incubated in water bath at 40 °C for 25 min. The reaction was stopped by an addition of 0.5 mL of 10% TCA and the soluble peptides were separated by centrifugation for 10 min at 3000 rpm. The absorbance of the TCA-soluble peptides in the supernatant was measured at 280 nm). [21] The enzyme activity was calculated using tyrosine standard curve. One unit of alkaline protease was defined as one µmol of tyrosine liberated per min.

3 RESULTS AND DISCUSSION

3.1 OPTIMIZATION OF PRETREATMENT PROCESS

Wool is just one member of a group of proteins called keratin. Wool fibers are composed of two types of cell, namely, the cuticle cells and the cortical cells. The epicuticle of wool is strongly hydrophobic and forms a resistant barrier to the penetration of dyes. The cortex of wool fibers has a bilateral structure and can be subdivided into two parts, the orthocortex and the paracortex. The orthocortex has a more open structure and is more accessible to dyes and more reactive chemically than the paracortex. [22]

Enzymes are natural protein molecules that act as highly efficient catalysts in biochemical reactions.[23] At present, the applications of pectinases, lipases, proteases, catalases, xylanases etc., are used in textile processing.[24] However enzyme treatment with different concentrations wool fabric showed improvement in softness, slight weight loss, smoother surface scales, improved absorbency and drapability.[23]

Protease is a class of enzyme, that active only on protein macromolecules and their activity beings with hydrolysis of covalent peptide bond that link successive amino acid residues in a polypeptide chain and this process is termed proteolysis.[1] In particular, both anionic and non-ionic surfactants partially deactivate proteases but also have a pronounced inhibition of lipolysis. The effect of the lipase treatments, in general, is to reduce the fatty acid and other lipid signal intensities.[14]

In this study wool fabric was treated with safflower seeds extract which contained lipase and protease enzymes in order to improve its absorbance, hence enhancing the dyeability of wool with kamala natural dye.

3.1.1 ACTIVITY OF LIPASE AND PROTEASE IN SAFFLOWER SEEDS EXTRACT

In order to determine the activity of lipase and protease in the safflower seeds extract, the received extract was subjected to a biological analysis and the obtained results are illustrated in Table (2).

Enzyme	Activity
Protease	167 U/m167 U/ml
Lipase	82 U/ml

From the previous results we can conclude that there is an appreciable activity of both lipase and protease enzymes in the safflower seeds extract.

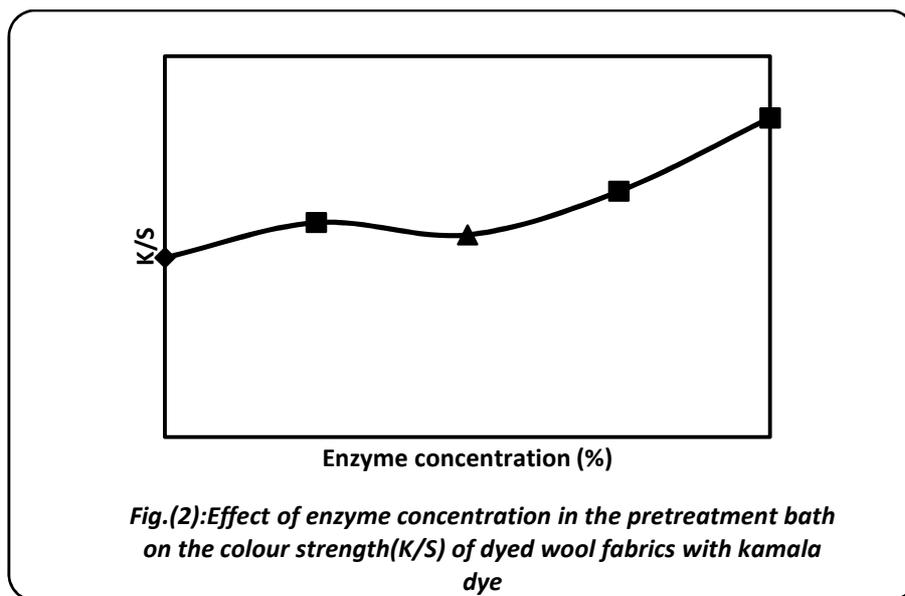
3.1.2 EFFECT OF ENZYME EXTRACT CONCENTRATION

Wool fabrics were subjected to a bio-treatment by using the safflower seeds enzymatic extract at different concentrations, in order to study its effect on the dyeability of wool to kamala dye.

Wool fabrics were bio-treated with safflower extract solution at different concentrations ranging from 25 to 100% according to the volume of treatment bath.

The pretreatment process was carried out at L.R 1:50, at pH value=7, and 70° C for 60 min. Then the dyeing operation were carried out with kamella dye as mentioned above

The colour strength of the dyed samples was measured and the results are illustrated in **Fig. (2)**.



Pretreatment: L.R: 1:50, at pH=7, and at 70° C for 60 min.

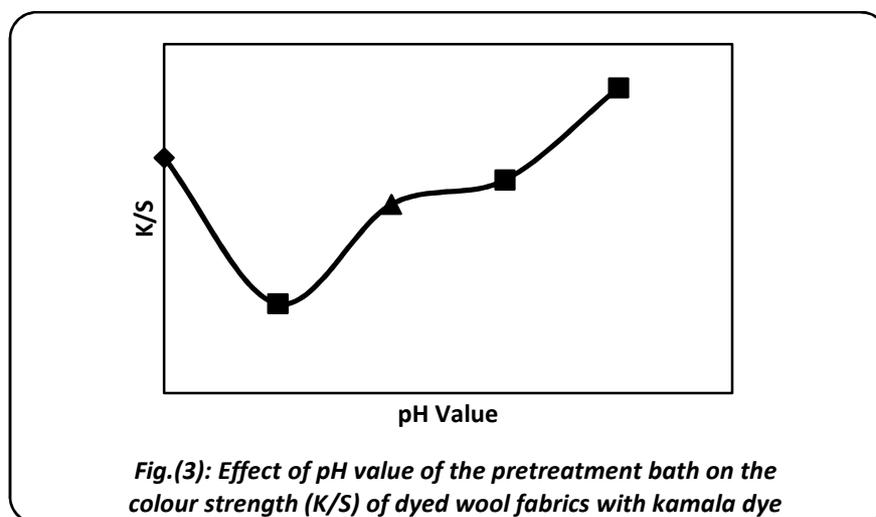
Dyeing: L.R 1:100, 6% natural dye, 2 g/L dispersing agent and at 80°C for one hour.

From the previous figure, it can be noticed that K/S is increased by increasing the enzyme concentration till it reaches its maximum value at the highest enzyme concentration of the treatment bath. increasing of the amount of enzymes and hence more effectiveness in the treatment of wool fabrics resulting in improving the dyeability with kamala natural dye. It is of great value to notice that the colour strength percentage (K/S %) was improved to 77.7% at 100% enzyme conc. in comparison with the untreated one.

Since wool fibre is mainly composed by polypeptides and small amount of lipids, so it is an ideal substrate for protease and lipase enzymes.[1] The action of lipase which is the main enzyme is to partially remove lipids from wool keratin surface and subsequently enhance the fibres absorbance and wettability.[14] As for protease enzyme, it causes the scales of wool to be flattened, also improves other physical properties including absorbency, dyeability and colour fastness properties, which proteases treatment helped to remove some of the hydrophobic compounds on wool surface.[23]

3.1.3 EFFECT OF PH VALUE OF TREATMENT BATH

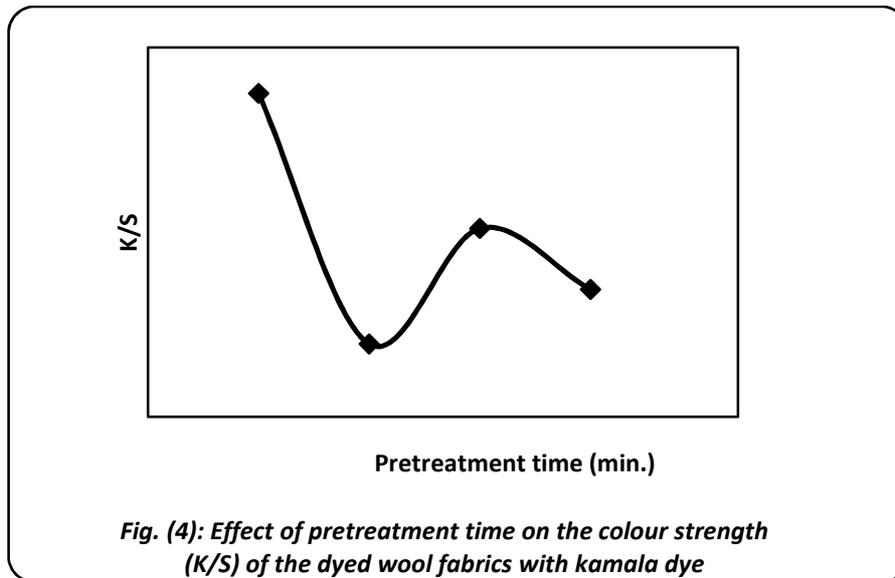
The pH value of the treatment bath plays a great rule as it controls the efficiency of the enzymes performance. In order to determine the effect of the pH value of the pretreatment bath on the colour strength of wool fabrics dyed with kamala dye, the wool specimens were treated at different pH values and the results are plotted in **Fig. (3)**.



From the figure, it can be concluded that the best pH value of the treatment bath to achieve the most efficient enzyme performance is at pH= 8 and pH= 4 .The activity of an enzyme strongly depends on the pH and temperature. Below the optimum pH and temperature, the enzyme, a protein, is denatured or inactivated, which decreases enzyme activity.[7]

3.1.4 EFFECT OF PRETREATMENT BATH TIME

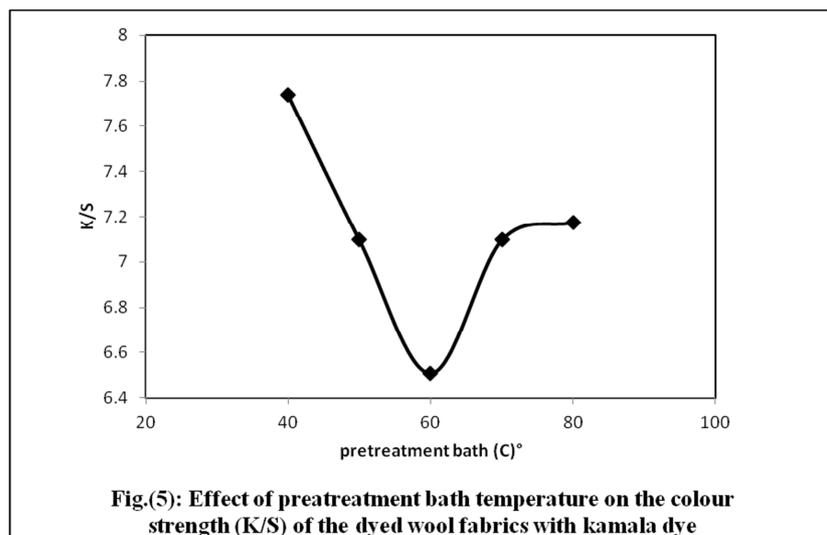
Wool fabrics were bio-treated with the safflower extract at different durations ranging from 30 to 120 minutes then they were dyed with kamala dye.The results are shown in **Fig. (4)**.



It is obvious from the former figure that performing the pretreatment process for 30 minutes gives the maximum colour strength. It is evident from the figure that lasting the time of pretreatment ends in decreasing of the K/S obtained. This may leads to a decreasing in the activity of the mixed enzymes in the extracted solution.

3.1.5 EFFECT OF PRETREATMENT BATH TEMPERATURE

Temperature of the enzymatic pretreatment bath is one of the most important factors that affect and determine to great extent the enzyme activity. Therefore to find out the most suitable pretreatment temperature, wool fabrics were bio-treated with the safflower extract at various degrees of temperature and then they were dyed with kamala dye.



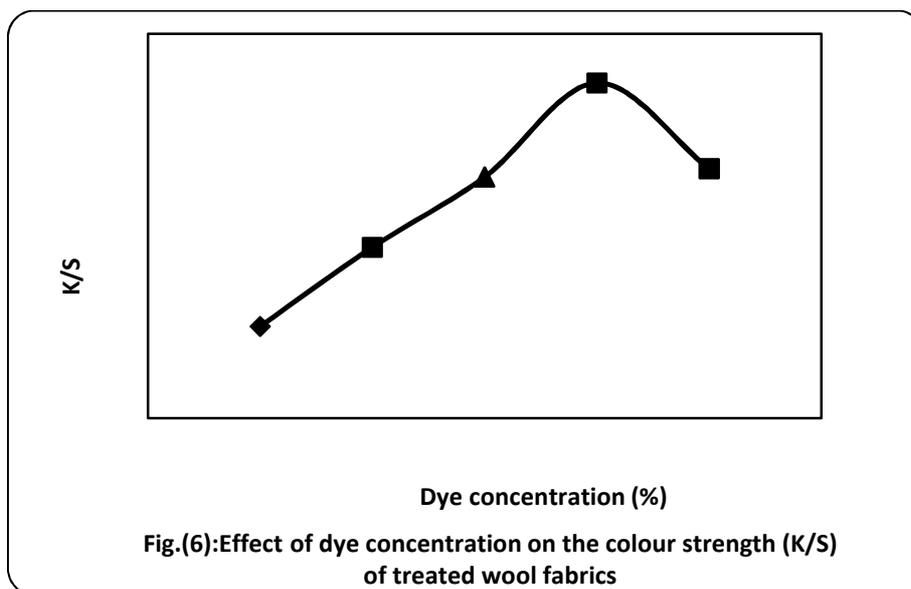
The results indicate that the most suitable temperature which establishes the maximum efficiency of the enzymes is at 40°C. This result because the enzyme is very sensitive to higher temperatures. The gap in hydrolytic activity was bigger depending on pH levels than on temperature values. [7]

3.2 OPTIMIZATION OF DYEING PROCESS

Treated wool fabrics were dyed with kamala natural dye. The factors that may affect the dyeing process were studied in details in order to figure out the optimum conditions to apply kamala to the treated wool fabrics.

3.2.1 EFFECT OF DYE CONCENTRATION

The treated wool fabrics were subjected to kamala dye bath at various dye concentration e.g. (2, 4, 6, 8, 10% o.w.f) L.R 1:100, pH= 5.5, 2 g/L dispersing agent and at 80°C for one hour . The measured colour strength results are plotted in Fig. (6)

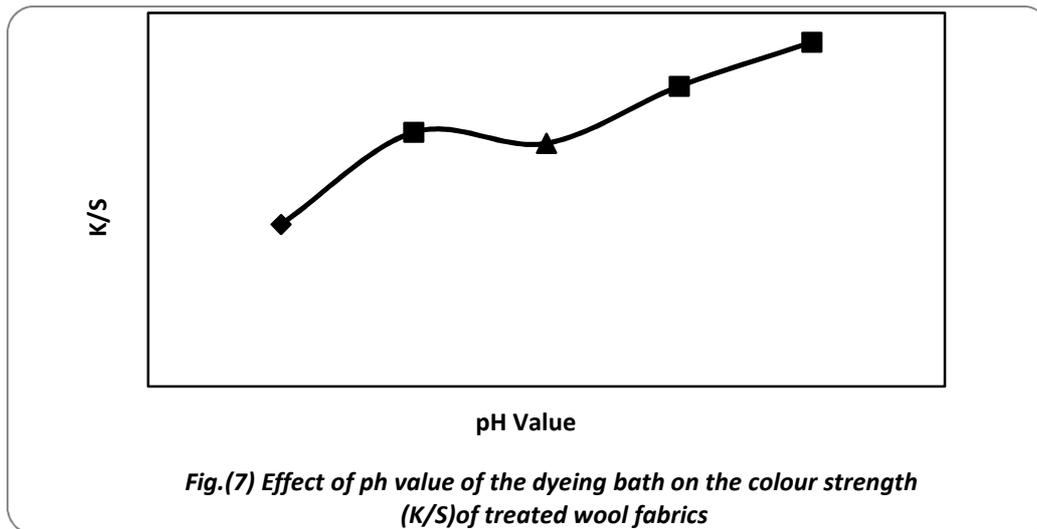


From the previous figure, it is obvious that the K/S increase as increasing of Kamala natural dye concentration and the maximum K/S can be achieved at dye concentration (8%). After which there is a slight decrease in K/S.

One of the parameters affecting the rate of dye uptake of wool fibers is the cuticle layer.[25] during the enzymatic processes the bonds between macromolecules are reduced and new groups (amino) which dyestuffs can react with could be formed and the cuticle layer became thinner , and so the dye uptake became easier.[22] The used natural Kamala dye is plant (CI Natural orange 2) which produces a yellow colour, have chalcone structures which can be considered as open chain analogue of flavonoids. Flavonoids are water-soluble compounds with molecules derived from 2-phenyl-1,4-benzopyrone.[26] As generally known anionic dyes such as natural dyes, form electrostatic forces with the positively charged protonated amino groups (—NH_3) of protein fibers. Furthermore van der Waals forces and hydrophobic interactions also involved between these dyes and protein fibers.[25] kamala powder can be used for dyeing wool and silk to bright orange-yellow and golden-yellow colors.[19]

3.2.2 EFFECT OF DYEING PH VALUE:

The pH value of the dyeing bath plays a great role especially when using natural dyes, so the dyeing process of the treated wool fabrics was carried out at various pH values ranging from 4 to 8. The results are shown in Fig.(7)

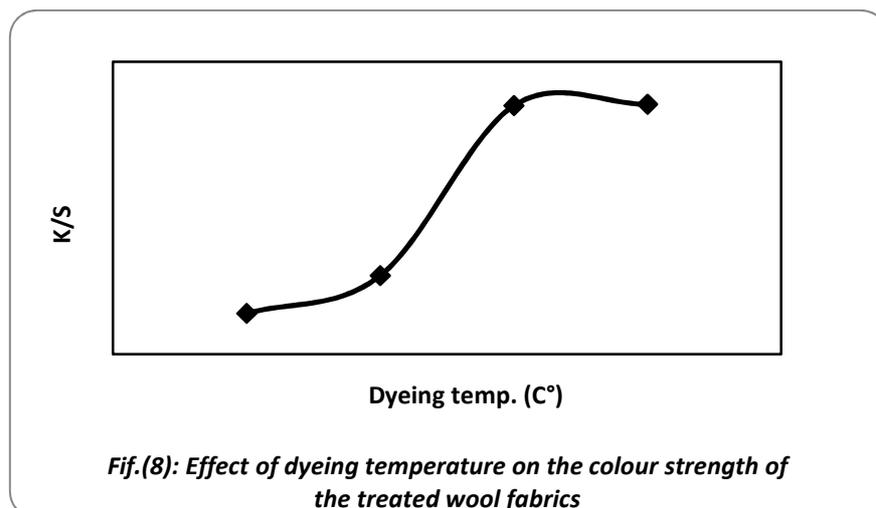


From the prior figure, we can conclude that maximum K/S was recognized at pH=8 and this may be attributed to the nature of the dye, which is insoluble in water and became dispersed by adding alkali. The higher the pH value, the higher the amount of alkali added to the bath. Hence the more soluble is the dye.

It is evident from **Fig.(7)** that this natural dye has been higher colour strength on wool fiber in alkaline dyebath and the maximum K/S was recognized at pH=8. This may be due to effect of alkali on the chemical structure of the dye molecules and shift in the adsorption of dye molecules in alkaline media.[27] where the solubility of the dye increased as the natural of the dye. Flavones and flavonols have yellowish colours. The colours are sensitive to pH. The yellow becomes much deeper in solutions of high pH .[26]

3.2.3 EFFECT OF DYEING TEMPERATURE

Dyeing temperature is one of the most important factors that affect dyeing. In order to find the most suitable temperature to carry out the dyeing process at, the dyeing process was applied at various degrees of temperature from 40°C to 100°C. The results are illustrated in **Fig.(8)**.

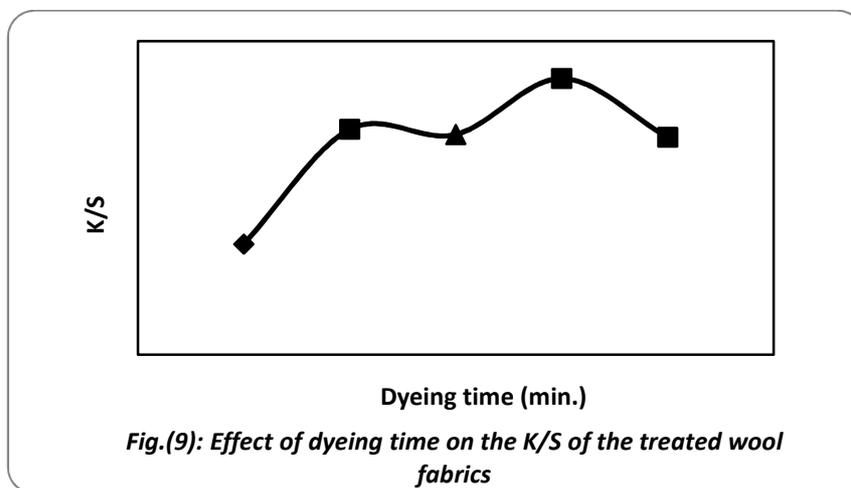


We can notice from the previous figure that the K/S value of the treated wool fabrics dyed with kamala was increased by increasing the dyeing temperature until reaching its highest value at 100°C. Although, the dyeing process can be applied at 80°C because no much difference between K/S values at these two points.

The increasing in the colour strength which followed with increasing of the dyebath temperature is due to the breakdown of the dye aggregates, increase in the dye kinetic energy and swelling of the fiber at elevated temperatures.[27]

3.2.4 EFFECT OF DYEING TIME

The dyeing process was carried out on the treated wool fabrics by using 8% kamala dye, at pH= 8 and at 80°C for different durations e.g. (15, 30, 45, 60, 75) and the results are shown in Fig. (9).



From the above figure, we can conclude that maximum K/S can be achieved when dyeing for 60 minutes at 80 °C. There will be a decrease when continue dyeing for longer than 60 min. and this may be attributed to the non stability of the dye molecules at long durations getting out from the fabric leading to that decrease in the K/S. Hence, it is preferable to dye at 80°C for one hour.

3.3 OPTIMIZATION OF MORDANTING PROCESS

Mordant is a substrate which is used to improve the colour quality of natural dyes because mordant can be bonded with dyes molecule and fiber, called the colour lake region that increases the colorfastness quality.

Tannin is an astringent vegetable product found in a wide variety of plant parts such as bark, wood, fruit, fruit pods, leaves, roots and plant galls. Tannins are defined as naturally occurring water soluble polyphenolic compounds of high molecular weight containing phenolic hydroxyl groups to enable them to form effective crosslinks between proteins and other macromolecules. The use of tannin as mordant in dyeing with natural dyes catches a lot of interest in the natural dyes to increase the durability of the colour after washing and enhancing the quality of the natural dyed fabrics.[28] Tannin is largely employed in the dyeing, Tannin substances are used on account of their lower prices in mordanting.[29]

Tannins may be in the form of tannic acid or vegetable-tannin-containing substances such as myrobolan (Harda, Terminalia chebula), oak galls, sumac, or pomegranate rind may be used for mordanting. Rinds of pomegranate (Punica granatum) fruits are rich in tannins and are used for mordanting purposes.[19]

After figuring out the optimum conditions for dyeing treated wool with kamala dye, the treated wool fabrics were subjected to premordanting with a natural mordant (pomegranate). The mordanting factors that may affect the colour strength of the natural kamala dye on the bio-treated wool e.g. (mordant concentration, pH value of the mordanting bath, mordanting temp. and mordanting time) were studied in details in order to determine the optimum conditions to apply (pomegranate) on biotreated wool fabric before dyeing at L.R : 1:100, 8% dye, 2g/L dispersing agent at pH=8, and at 80° C for 60 min. the optimum condition. The result of these factors are showed in the following table.

Table (3): Effect of mordanting factors on the colour strength of dyeing biotreated wool fabric with kamala natural dye (pre-mordanting)

Factors studied		K/S (W.L 400)
mordant concn.	Without mordant	6.4
	4%	6.5
	6%	6.8
	8%	7.1
	10%	7.1
pH value of mordanting bath	pH=4	9.2
	pH=5	7.3
	pH=6	7.9
	pH=7	8.1
	pH=8	8.4
mordanting temperature	40°C	7.8
	60°C	8.7
	80°C	9.9
	100°C	9.2
mordanting time	15min.	10.6
	30min.	11.2
	45min.	11.6
	60min.	12.0

From the previous table, the K/S values were found to be improved with increasing the natural mordant concentration, and the (8%) is the best concentration of the mordant to be applied on the treated wool fabrics as maximum K/S can be achieved.

The pH=4 is the best pH value of the mordanting bath that can achieve the maximum K/S of the dyed wool fabrics. Also the best temperature to carry out the mordanting process at is 80°C after which there will be a slight decrease in the K/S value. It is clear that by increasing the mordanting time from 15 to 60 minutes, the higher the K/S we can get to reach its maximum value at 60 min.

3.4 TENSILE STRENGTH TEST

Tensile strength testing was applied on both untreated (48 Kg/cm²) and treated wool fabrics (46 Kg/cm²) with (100%) enzymatic extract. By comparing the results, it is obvious that there is a slight decrease in tensile strength in the treated sample. This is result due to modifying morphological structure of wool fabric due to the hydrolysis of protein chains occurring with concentrations of proteases. With higher hydrolysis, the length of polypeptide chains in the proteins of wool decreases, and a lower energy is needed for the separation of chains and breakage of the fabric.[23]

3.5 SURFACE MORPHOLOGY OF WOOL FABRICS

Surface morphology of the untreated and treated wool samples is investigated by electron microscopy. The SEM micrographs are shown in **Fig. 10(a-b)**. The images demonstrate that the enzymatic treatment of wool samples accompanied with abrasion in the scales of wool surface leading to better absorption hence better dyeability.

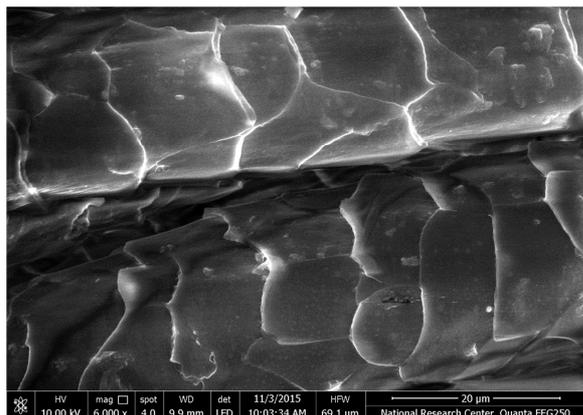


Fig.(10a): Untreated wool

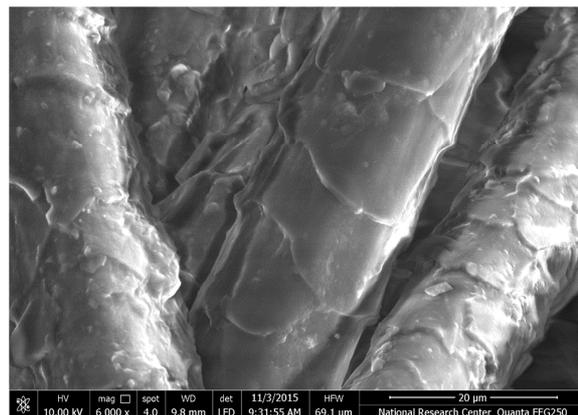


Fig.(10b): Treated w

SEM images showed that untreated wool fibre was rough and scales were sharp. The surface scales of wool fibre got blunt and the smoothness of surface was found enzymatic treatment blunted the sharp edge of the cuticle layer of angora fibers. As a result, the cuticle layer became thinner compared to the untreated one, and so the dye uptake became easier.[22]

3.6 DETERMINATION OF THE WETTABILITY OF WOOL FABRICS

To assess the effect of the enzymatic treatment on absorbability of wool fabric, the wettability of the treated wool fabric with different concentrations (25-100%) of safflower extract were carried out comparing the results with the blank. The data are illustrated in table (4).

Table (4): Effect of safflower extract concn. on the wettability of wool fabrics

Safflower extract concentration(%)	Time of disappearance of water drop
0	28 min.
25	41 sec.
50	31 sec.
75	28 sec.
100	16 sec.

It was observed from the table that the untreated sample showed no absorbency within 28 minutes and after enzymatic treatment it was improved with increasing the concentration of safflower extract , reached to 16 seconds. Table clearly reveals that once the percent of concentration has increased, the amount of time for the water drop to be absorbed decreased indicating that proteases treatment helped to remove some of the hydrophobic compounds on wool surface.[23]

3.7 FASTNESS PROPERTIES

The colour fastness properties of untreated and treated wool fabrics dyed with kamala natural dye and mordanted with pomegranate are evaluated in terms of fastness to washing , perspiration and light ,the results are illustrated in **Table(5)**. The fabrics are compared with grey scale to obtain the colour change compared with fabric before testing. Grey scale has scale from 1 to 5 that scale 1 indicates the most colour difference and scale 5 means no color difference.

Table (5): Colour fastness properties of untreated and treated wool fabrics dyed with kamala dye and mordanted with pomegranate

Samples	Washing			Perspiration						Light
	Alt.	Staining		Acidic			Alkaline			
		*	**	Alt.	Staining		Alt.	Staining		
					*	**		*	**	
1-Untreated wool dyed with kamala	4/5	4/5	4/5	3	4	4	3/4	4	4	3
2- Untreated wool dyed with kamala and mordanted	4/5	5	4/5	3/4	4	4	3/4	4	4	4
3- Treated wool dyed with kamala	4/5	4/5	4/5	3	4	4	3/4	4	4	3
4- Treated wool dyed with kamala and mordanted	4/5	5	4/5	3/4	4	4	4	4	4	4

N.B: - (*) refers to staining on cotton fibres and (**) refers to staining on wool fibres.

-The light fastness was evaluated according to the grey scale i.e. (5 grades only).

From the previous table, it can be concluded that all samples show very good colour fastness properties especially for washing fastness compared to perspiration which showed a slight decrease. As for light fastness, one can notice that mordanting with pomegranate which contain tannins accompanied with an increase in the fastness for both untreated and treated wool fabrics. Mordanting not only increase the dye uptake for fabric but also form metal dye complex which is more stable than colouring component itself. [30]

In general tannins form three types of bonds with proteins (such as wool and silk). These are:

- hydrogen bonds between the phenolic hydroxyl groups of the tannins and both the free amino and amido groups of the protein,
- ionic bonds between suitably charged anionic groups on the tannin and cationic groups on the protein,
- covalent bonds formed by the interaction of any quinone or semiquinone groups that may be present in the tannins and suitable reactive groups in the protein.[22]

4 CONCLUSION

This study illustrate the possibility of extracting a mixture of enzymes from safflower seeds (protease – lipase) and use this enzymatic extract successfully in treating wool fabrics leading to an improvement in absorption hence in dyeability towards kamala natural dye. The optimum of enzymatic treatment summarized (6% conc. enzymes extract, pH 8, 30 minutes at 40°C), Also dyeing process (8% kamala natural dye, pH 8 , 60 minutes at 80 °C).

Pre-mordanting with a natural mordant (pomegranate) is achieved with 8% conc.at pH 4 for one hour at boiling. Accordingly there will be a great enhancement when dyeing wool with kamala natural dye and by using pomegranate as an eco-friendly mordant for better fixation of the natural dye; this will develop an entire eco-friendly system (beginning from pretreatment through dyeing and ending with mordanting) that matches with the environment demands. And the fastness properties improved according to this eco-friendly system.

REFERENCES

- [1] Ammayappan, L . , " Application of enzyme on woolen products for its value addition: An Overview", Journal of textile and apparel technology and management , vol.8,no.3 ,p.1-12, (2013).
- [2] H.S.El-Khatib and G.M.Shokry , Dyeing of cellulosic fabrics pretreated with brewer's yeast filtrate using natural casuarinas plant, journal of Science &Arts , vol.18,no.4,p.25-40 , (2007).
- [3] H.S.El-khatib, Dyeing of Bio-Treated Cotton / Wool Blend Fabric with New Natural Dye, 13th International Conference of The Faculty of Applied Arts,p.1-15,(2012).
- [4] H.S.El- Khatib, Dyeing ofWool/Nylon Blend Fabric with Camphor Plant After Bio-Treatment with Brewers 'Yeast Suspension, International Design Journal,no.12,p.143-152,(2012).
- [5] H.S.El- Khatib , Bio- Treatment of Cellulosic Fabrics (Cotton – Linen) Using Natural Enzyme Extracted from Safflower Seeds , 4 International Conference of Textile Reseach Division , April 15-17 , p.364 – 369, (2007).

- [6] Wimonrat S., Preechaya R., Wanvichit P. and Siwapol P., "Effect of enzymatic treatment on the dyeing of pineapple leaf fibres with natural dyes", *Science Asia*, vol. 35, p.31-36,(2009).
- [7] So H. L and Wha S. S., "Modification of polylactic acid fabric by two lipolytic enzyme hydrolysis ",*Textile Research Journal*, vol. 83,no.3, 229-237, (2013).
- [8] Nina S. and Petra F. T., "Complete enzymatic pre-treatment of cotton fabric with incorporated bleach activator ", *Textile Research Journal*, vol. 83: 566, (2013).
- [9] Elias A., Karl-H. R., Georg M., et al, "Enzymatic Decolorization of Textile Dyeing Effluents", *Textile Research Journal*, vol. 70: 409, (2000).
- [10] Graça M. B., Soares M., Teresa P., Amorim M. L. and Maria C.F., "Pilot-scale Enzymatic Decolorization of Industrial Dyeing Process Wastewater", *Textile Research Journal*, vol. 76: 4, (2006).
- [11] Jiugang Y., Qiang W., Xuerong F. and Ping W., "Enhancing Dye Adsorption of Wool Fibers with 1-butyl-3-methylimidazolium Chloride Ionic Liquid processing", *Textile Research Journal*, vol. 80: 1898, (2010).
- [12] Riva A., Algaba J. and Prieto R., "Dyeing Kinetics of wool fabrics pretreated with protease" *Colouration Tehnology*, vol. 118:2 ,p.59-63 (2002).
- [13] Kantouch A., Rslan W.M and El Sayed H., "Effect of Lipase pretreatment on the dye ability of wool fabric " *Journal of Natural fibres*, vol.2,p.35-48 (2005).
- [14] Mall J.K., Sims P. and Carr C. M., "Surface chemical analysis of Lipase enzyme treatment on wool and mohair" *The Journal of The Textile Institute*, vol.93:1,p.43-51(2002).
- [15] Monlleo D., Julia M., Pinazo A., Erra, P., "Use of Lipase for wool modification ", *Melliand Text.*, vol.5,p.402-405(1994).
- [16] Kanlayanee B. and Sunan S., "Influence of silk surface modification via plasma treatments on adsorption kinetics of lac dyeing on silk", *Textile Research Journal*, vol.83:3,p. 288–297 (2013).
- [17] Prabhu K.H. and Teli M.D., "Eco-dyeing using Tamarindus indica L. seed coat tannin as a natural mordant for textiles with antibacterial activity", *Journal of Saudi Chemical Society*, vol.18,p.864-872, (2014).
- [18] <http://www.motherearthnews.com>
- [19] Sujata Saxena and A. S. M. Raja, *Natural Dyes: Sources, Chemistry, Application and Sustainability Issues*, Springer Science+ Business Media Singapore, p.37-79, (2014).
- [20] Vorderwuelbecke T., Kieslich K. and Erdmann H., "Comparison of lipases by different assays", *Enzyme Microb Technol.*, 14, P. 631-639, (1992).
- [21] Amara, A. A., Hassan, M. A., Abulhamd, A. T. and Haroun B. M., "Non-mucoid *P. aeruginosa* aiming to safe production of protease and lipase", *Inter. Sci. and Invest. J.*, 2(5), (2013).
- [22] E. Perrin Akçakoca Kumbasar, Riza Atavand M. Ibrahim Bahtiyari Effects of Alkali Proteases on Dyeing Properties of Various Proteinous Materials with Natural Dyes, *Textile Research Journal*, Vol 79(6): 517–525,(2009).
- [23] Pooja, Ekta Sharma, Nargis Fatima Quality Improvement of Wool Fabric Using Protease Enzyme Environment and Ecology Research 2(8): 301-310, (2014).
- [24] Kiro D. Mojsov, Trend in Bio-processing of Textile: A Review, *Advanced Technologies*, 3(2) 135-138, (2014).
- [25] Riza Atav and Fatih Tu`rkmen, Investigation of the dyeing characteristics of alpaca fibers (Huacaya and Suri) in comparison with wool *Textile Research Journal*, Vol. 85(13) 1331–1339,(2015).
- [26] ACTIVITY BRIEF, Extracting and testing a natural plant dye, *advanced applied science*, p.1-19, (2008).
- [27] Aminoddin Haji, *Journal of Biodiversity and Environmental Sciences*, OPEN ACCESS Natural dyeing of wool with *Arnebia euchroma* optimized by plasma treatment and response surface methodology Vol. 5, No. 2, p. 493-498,(2014).
- [28] M. D. Teli, Javed Sheikh, Maruti Kamble, Ecofriendly Dyeing and Antibacterial Finishing of Soyabean Protein Fabric Using Waste Flowers from Temples, *Textiles and Light Industrial Science and Technology (TLIST) Volume 2 Issue 2, April(2013)*.
- [29] Thet Thet Aung and New Yee Win, Extraction of Naturale Mr 2o0r0d8a)n t from Plant Source for Dyeing Application, *International Conference on Sustainable Development: Issues and Prospects for the GMS, 12-14 Nov. (2008)*.
- [30] Priti B. Tayade, Ravindra V. Adivarekar, Dyeing of Silk Fabric with *Cuminum Cuminum L* as a Source of Natural Dye, *International Journal of Chem.Tech. Research*, Vol.5, No.2, p. 699-706,(2013).