Dyeing of Wool/Nylon Blend Fabric with Camphor Plant After Bio-Treatment with Brewers 'Yeast Suspension

H.S.El-khateeb*

Textile Printing , Dyeing &Finishing Dept.,Faculty of Applied Arts,Helwan University,Giza,Egypt.

Abstract :

As the present trend throughout the world is shifting towards the use of eco-friendly and biodegradable commodities, the demand for natural dyes is increasing day by day.

Single- bath dyeing and of wool/nylon blend fabric was carried out using new natural dye which was extracted from the bark of camphor tree (*Eucalyptus citriodora*) after bio-treatment process by using brewer's yeast suspension which contain many enzymes .The effect of enzymes conc. , pH ,temperature and time which may affect the enzymatic treatment process were studied and various parameters that may affect the dyeing process were investigated such as dye conc., pH, temp. and time and addition of salt . Different tests and analysis were studied such as wettability and color strength The effect of post-mordanting using alum different metal salts on the colur strength and were determined . Also colour fastness properties to washing ,perspiration and light have been also assessed

Key words: Wool/Nylon blend fabric –Brewer's yeast suspension – Camphor tree - Dyeing – Wettability -Mordanting -Fastness properties.

Introduction:

The population explosion and the environmental pollution in the recent years have forced researchers to find new health and hygiene related products for the well being of mankind.[1] Nowadays, there is a growing interest in the revival of natural dyes in textile dyeing; arguments based around keywords such as sustainability, green chemistry, improved eco-balances and thereby leading to niche products for special markets [2].

Also enzymes are gaining an increasing role in textile wet processing due to their proven flexibility, reliability, and concerns about safety, energy and water conservation, and environmental responsibility. [3] Enzymatic treatments cause changes in mechanical properties of substrates and may also lead to unique textile effects or ecological improvements in existing procedures [4]

Microfibres are used in sport fashion and functional clothing due to their water repellence and air permeability properties . Due to the above properties polyamide microfibre alone or in blends with natural fibres will have a very important place in textiles in the very near future. So the dyeing of polyamide and blends become very important .[5]

Although nylon 6,6 was commercially introduced over 60 years ago, demand for the textile has been enlivened in recent years by the introduction of nylon 6,6 microfibre which is increasingly being used for apparel, sportswear and high performance functional fabrics due to its superior handle, good drapeability and excellent luster.[6,7]

It is well known that the presence of amino end groups (AEG) in nylon fibres imparts substantivity towards various classes of anionic dye, namely acid dyes, direct dyes, mordant dyes and reactive dyes; of these dye classes, acid dyes, predominate commercially ; in addition, disperse dyes also are substantive .[6]

It is widely held that the substantivity of such anionic dyes, under acidic conditions, towards nylon 6,6 is based mainly on electrostatic forces of interaction operating between anionic (typically sulfonate) groups in the dye and the protonated, terminal amino groups in the fibre; the adsorption of anionic dyes on nylon is thus considered to be site-specific . [8]

The aim of this study was applying one-bath dyeing of wool/nylon by using new natural dye (camphor plant) , after bio- treatment with brewer's yeast suspension which contains many enzymes [protease , lipase , amylase] . Many tests and analysis were carried out to determent the change of hydrophobic/hydrophilic properties and the improving in absorbency of blend towards natural dye .

2- Experimental:

2.1- Materials :

2.1.1- Fabric:

Wool/Nylon blended fabric : (65 / 35) were kindly supplied by Misr/ Helwan for Spinning and Weaving Co., Cairo, Egypt .

2.1.2-Dyestuff:

Natural dye from vegetable source was extracted from the bark of camphor tree (*Eucalyptus citriodora*) .

2.1.3- Enzymes :

The suspension of active brewer's yeast {(fung) saccharomyces uvarum and saccharomyces cerevisiae}was used in the present work . Yeast was obtained from the Egyptian Starch, Yeast and Detergents Campany, Egypt.

2.1.3- Chemicals and auxiliaries:

Sodium carbonate , acetic acid , Alum (Aluminum Sulfate) , Ferrous sulfate , sodium chloride , were used in the present work .

2.2- Methods:

2.2.1- Preparation of Enzyme Suspension:

The suspension of active yeast fungus was prepared by pasting the brewer's by adding such amount of sugar (100g of sugar to 126 g of dry basis) then water was added to the paste of yeast while well stirring to complete the total volume to one litre. Finally the solution was filtrated and freezed.

2.2.2- Preparation of plant material:

The plant materials were dried in dry air crisp at 35 ^oC for 24 hours till the plant become after which they were grinded.

2.2-3-Extraction of Natural Dyes:

The plant materials were soaked in water for 24 hours, using 10 g of plant materials in 100 ml water and then the colouring matter was extracted at the boil for 30 minutes after which the solution was filtered and stored under refrigerator.

2.2-4- Pretreatment of Wool/Nylon blended fabrics:

Wool/Nylon blended fabric was treated by using the filtrated brewer's yeast suspension after diluted it with water to obtain aqueous suspension of different conc. (25-100%) . the treatment was carried out at pH values from (5-8) for times (30-90) and temperature from (40-80^o C) . After that the blended fabrics were rinsed with cold and hot water . Then the enzymatic pretreated blended fabrics were soaped with 2 g/l non-ionic detergent at 70 ^oC for 15 min. and rinsed after washing with cold water

2.2-5- Dyeing of Wool/Nylon blended fabrics with natural dye:

The pretreated wool/nylon blended fabrics were dyed in one-bath by using the extract of the bark of camphor tree with different conc. (25-100 %) at pH from (5-8) for (30-90 min.) and the temperature were (40-100 ^OC) at L:R 1.50

2-2-6-Mordanting process:

The post- mordanting process was carried out after the optimum dyeing on wool/nylon blended fabrics by using 5 g/l of different mordants (Alum , Ferrous , copper) . the process was continued for 60 min. at boiling , after which the samples were soaped with 2 g/l non- ionic detergent for 15 min at70 ^oC .

2.3-Measurements and testing:

2.3.1- Colour measurements:

The dyed samples were subjected to colour measurement by using reflection spectrophotometer model ICS-Texicon Limited, Kennetside Park, New Burg, Berkshire, England. The colour strength expressed as K/S values was assessed by applying the 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- Colour fastness:

Different fastness properties of dyed samples were tested according to ISO standard methods. The specific tests were: ISO-X12(1987), colour fastness to rubbing; ISO 105-C02 (1989), colour fastness to washing; and ISO 105-E04, colour fastness to perspiration. The dyed samples were subjected to tests, for fastness to light by AATCC test method 16-1993

2.3.2- Determination of wettability:

The effect of enzymatic treatment on the degree of absorbency of wool/nylon blend can be observed by testing the wettability of wool/nylon blended fabric as the different concentrations of brewers' yeast suspension .

This test was carried out by putting two drops of water on the blended fabric surface and calculate the time that will be passed until the fabric absorb the water.

3- Results and Discussion:

The use of biotechnology in the processing of fibers and textiles is rapidly gaining wider recognition because of their non-toxic and eco-friendly characteristics. The best established application of biotechnology in textile industry is the use of enzymes,[9] especially with using natural dyes .Most of the enzymes used are derived from fungal sources such as Amylase, Protease and Lipase [10].

A great many studies have been carried out on the application of enzymes on natural fibers, Recently, extensive researches on modifying synthetic fibers using biotechnology are performed. The chemical composition of the fibers influences their susceptibility to enzymes. Enzymatic hydrolysis of synthetic fibers improves some undesired properties such as hydrophobicity, less wearing comfort, low dyeability, difficulties in finishing, build-up of electrostatic charge, the tendency to pilling and insufficient washability.

Very few research papers have been reported on different properties of enzymatically hydrolyzed polyamides.[9] Miscibility of polymers strongly depends on polymer–polymer interactions such as hydrogen bonding, ion–dipole and dipole–dipole interactions . Nylon 6 is known to be hygroscopic, due to the presence of H-bonds. [11,12]. An integral part of macromolecular polyamide chains are amino groups, so bending with wool fibers is suitable . Therefore , polyamide fiber is chemically similar to wool and can be dyed with the same dyes as wool , but morphological difference over molecular structure of fibers cause the difference in the speeds of dyeing, which makes nuancing "shade-to-shade "difficult.[13]

Blends of synthetic fibers with natural fibers offer the most valuable possibilities for combining desirable physical properties , because the two components are so dissimilar. Different fibers can be blended in textile structures to obtain the desirable properties of each of the fibers in the blend.[14]

3.1- Factors affect the enzymatic treatment of wool/nylon blended:

3.1.1 Effect of Brewer's Yeast Suspension Conc. :

Wool/Nylon blended fabrics were pretreated by using brewer's yeast suspension at different conc. (25-50-75-100 ml/l) under fixed conditions of time, temp, and pH . After enzymatic treatment , dyeing process with camphor extract was carried out .

The colour strengths of the dyed blend were measured and the results are plotted in fig. (1)

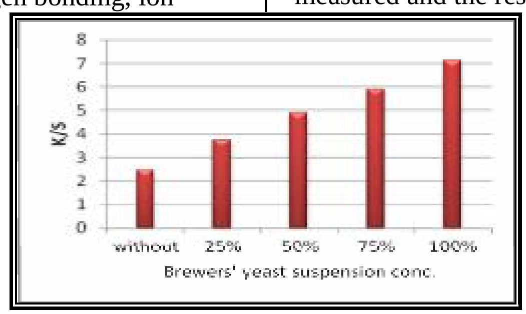


Fig.(1):Effect of enzyme concentration of the bio-treatment on the colour strength (K/S) of dyed wool/ nylon blend .

Bio-treatment condition:

Dyeing condition:

X enzymes conc.,pH8,60min.,temp.80^{o}c

50% champor extract,PH 7, 60min,temp.10

Figure (1) indicates that the K/S was increased according to increasing of enzymes conc. on wool/nylon blended fabric until reaches its maximum value at the higher conc. of enzymes . this result is logic one because the enzymes that found in the brewer's yeast increasing dye up take on the two fabrics . The enzymes which be found in brewer's yeast suspension can be showed in (Table1). since the rate and magnitude of hydrolytic reaction between enzyme and the impurities depends on great extent on enzyme concentration and The action of enzymes consists of increasing dye absorption and also seems to produce a better diffusion of the dye into the fabric . [15]

Table (1) :The composition brewer's yeast suspension (100 ml)

Enzymes	Content (U.S.P)
Protease	350
Lipase	28
Amylase	350

Some researchers were investigated the changes induced on nylon 6,6 fiber by enzymatic system using different proteolytic enzymes and the results showed higher dyebath exhaustion on the enzyme treated samples compared to raw material .[16]

Treated nylon 6,6 with protease enzymes clearly suggest that the substrate has undergone severe chemical hydrolysis especially at amide (–CONH–) functional groups. and color fastness of nylon 6 fiber Fig. 2 shows the schematic hydrolysis

of nylon 6,6 after protease treatment . Protease enzymes can attack preferentially the amorphous or less-ordered (paracrystalline) regions rather than the crystalline or moreordered regions because of the enzymes capability of migration into these less-ordered regions compared to the more-ordered ones, hence, it accelerates the thermal degradation of nylon 6,6 samples. [9]

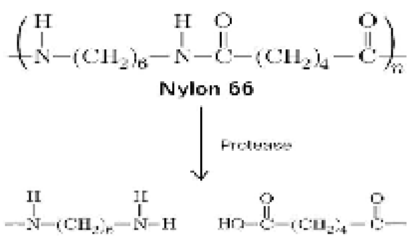


Fig. 2. Biohydrolysis of nylon 6,6 fiber after treatment with protease enzymes

Lipase enzyme can change dyeability, moisture absorption, surface properties ,and the results were confirmed that the darkness of the samples increased with an increase in the enzyme percentage in the solution. The results of moisture regain showed that treatment of nylon fabrics with lipolytic enzymes caused to increase the moisture absorbency.[17]

There have been relatively few papers studying the effect of protease on dyeing properties of wool with natural dyes.Proteases are one of the major enzymes which have received much interest for their use in wool processing . Protease enzyme also tends to improve natural dyes absorption on wool fibers. An increase in the enzyme concentration resulted in an increase in the dyebath exhaustion. This confirms that protease catalyse degradation of epicuticle produce the fiber with more amine terminal groups, and as a consequence, improve susceptibility of dyes attraction and allowing the dye molecules to penetrate more easily into the fiber cortex.[18] Also the lipase enzymes have been used in to remove lipids from the outer layer of the wool fiber .

3.1.2 Effect pH of Enzymatic treatment :

It is important to control pH to suit enzyme , in order to get the most efficient enzyme performance .

Different wool/nylon blended samples were treated in various treat-baths under constant conditions i.e. conc. of brewers 'yeast suspension, temp., and time whereas the pH level of treatment was varied from one bath to another. The colour strengths of the dyed samples were measured and the received data are plotted in Figure 3

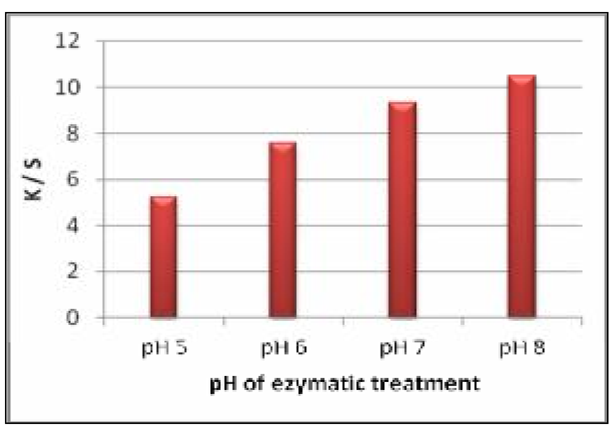


Fig.(3):Effect of pH of pretreatment on the colour strength (K/S) of dyed wool/nylon blend .

Figure 2 indicates that the colour strength obtained was increased as the pH increases up to 8 . Enzyme has an optimum pH which the high activity of enzymes were achieved . pH 8 is the optimum of brewers'yeast suspension which contain many enzymes as previous studies .[15,19,20]

3.1.2 Effect of Enzymatic treatment temp. :

Temperature of the reaction medium between enzymes and fiber is important because the activity of enzyme is very sensitive to temperature .

To determine the most suitable pretreatment temp. , wool/nylon blend samples were bio-treated at various degrees of temperature and then dyed . The results show in figure 4.

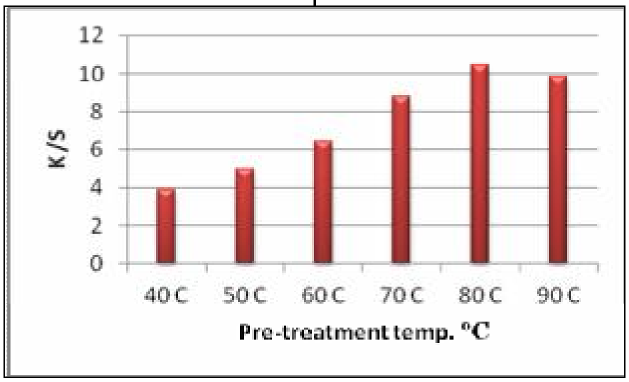


Fig.(4):Effect of pretreatment temp. on the colour strength (K/S) of dyed wool/nylon blend .

It is obvious, from Figure 4 that the rate of enzymatic treatment increases gradually by raising the temperature . this result means that at 80^o C the activity of the applied enzymes suspension reach its maximum which more removing the impurities from two fibers .

3.1.3 Effect of Enzymatic treatment time :

The wool/nylon were pretreated with brewer's yeast suspension at 80^o C by using a constant conc. of enzymes at pH8 for different durations (30-60-90-120-min.) after which the knitted blend was dyed .

The results are found in fig. 5 which illustrate the relation between time of treatment and attained color strength .

Figure 4 illustrates the effect of bio-treatment time on the K/S of wool/nylon blend which it can be observed that by lasting time of treatment the K/S is increased .The chosen time was 60 min .because after this time slight increase in K/S was achieved .

this means that increasing of the time of pretreatment , the reactivity of the enzymes was increased .

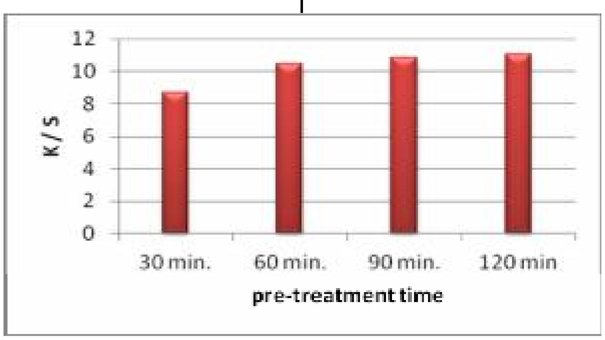


Fig.(5):Effect of pretreatment time on the colour strength (K/S) of dyed wool/nylon blend.

3.1.4 Wettability of wool/nylon blend :

The surface morphology of wool and nylon plays an important role in dyeing processing[21] .Enzymatic treated samples are hydrophilic, independent of the sample composition (wool/nylon blend), while untreated wool/nylon blend are hydrophobic , the wettability test can be indicated this results .

Table (2) illustrates the alteration in the wettability as a function of enzymatic treatment using different concentrations of brewer's yeast suspension .

Conc. of brewer's yeast suspension (%)	Time of disappearance of water drop on cotton/wool knitted blend
zero	340 sec.
25	230 sec.
50	140 sec.
75	90 sec.
100	50 sec.

These results show that the wettability of wool/nylon blend increases gradually as the conc. of the used enzymes increase which may be attributed to the modification of morphological structure of two fiber .

3.2- Factors affect dyeing of wool/nylon blended with camphor plant:

Wool/nylon blend can be dyed by using new natural dye which extracted from the bark of camphor tree for after enzymatic treatment . Camphor tree (*Eucalyptus citriodora*) belongs to *Lauraceae* family.The stems and bark on young branches of Camphor-Tree are bright green, tinged with red when young, maturing into a dark grey-brown. The major parts of natural dyes are anthraquinone, anthocyanin and flavonoid dyes or polyphenolic compounds, most of which have yellow, red, brown, and olive shades. Flavoniods present in most of the natural dye obtained from fruits and vegetable are found to be important in producing colour in textile dyeing [20,22] .

3.2.1-Effect of dye concentration:

Fig.6 shows the effect of dye concentration on the colour strength obtained for the dyed wool/nylon fabrics. The pretreated wool/nylon blend were dyed by using extract of camphor plant with different concentrations (25,50,75,100 %) under fixed dyeing condition .
As shown, the K/S value increases as the concentration of the natural dye increased . The results of color measurements showed that the more concentration of enzyme used, the darker the color of dyed sample is .
This confirms that protease catalyses the hydrolysis occurred in the amorphous regions of the polyamide chains producing more free functional groups on the surface which in turn, improve the susceptibility of reactive and acid dyes attraction. Further hydrolysis occurs with any increase in the enzyme concentration .[9]
Also the fatty layer which decrease the affinity of natural dyes to wool fiber can be removed by using enzymatic treatment .

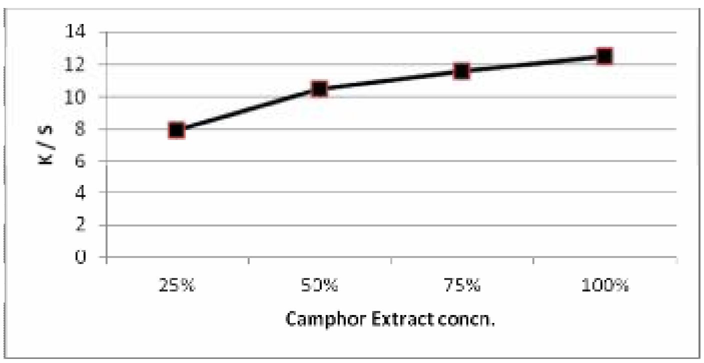


Fig.(6):Effect of champor extraction conc. on the colour strength (K/S) of pretreated wool/nylon blend .

3.2.2Effect of pH values:

The relation between the uptake of natural dye on wool/nylon blend was studied and the results are plotted in fig.7
Fig. 7 shows that the pH values of the dye bath have considerable effect on the dyeability of wool/nylon blend fabric with camphor dyes. The effect of dye bath pH can be attributed to the pre-enzymatic treatment , correlation between dye structure, the fibres used and dye stability.
In the presence of water, and depending on the pH within the fibre, the amino and carboxylic acid groups will be ionised to a greater or lesser extent. that accompanied a decrease in pH of application can be attributed to a corresponding increase in ion-ion
attractive interaction operating between the anionic sulphonate groups of the dye and the cationic protonated amino end groups of the nylon.The ability of the amino groups in nylon to protonate at low pH effectively reduces the concentration of nucleophilic free amine and increases the affinity of anionic dyes for (cationic) nylon.[23] This indicates that the electrostatic interactions between the positively charged amino groups in nylon and wool with the anionic groups.[24]

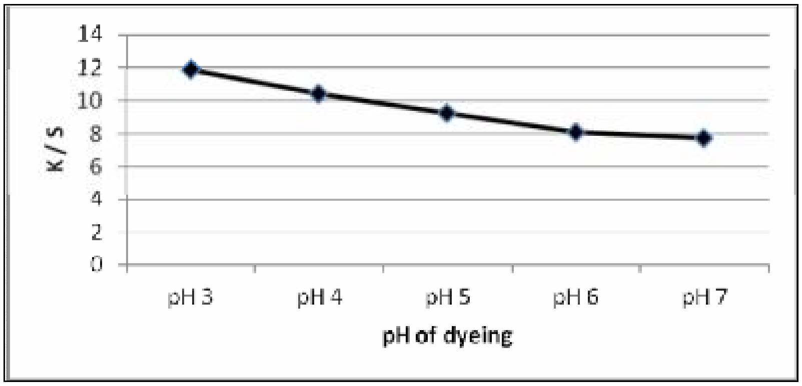


Fig.(7):Effect of pH of dyeing bath on the colour strength (K/S) of pretreated wool/ nylon blend .

Increasing the acidity of the dyebath is accompanied with higher dyeability of wool/nylon fabric towards natural dyes . Since the camphor dye used is a water soluble dye containing carboxyl groups, it would interact ionically with the protonated terminal amino groups of wool and nylon fibres at acidic pH via ion exchange reaction. The weak carboxylate anion of the dye that may be present at low pH replaces that of acid due to its higher affinity. The anion of the dye has a complex character, and when it is bound on fibre,further kinds of interactions take place together with ionic forces.[25]

3.2.3Effect of salt concentration:

Neutral electrolytes such as sodium chloride and sodium sulphate play a great role in dyeing textile fibers . Pretreated blend were dyed in presence different concentrations of NaCl (without,1, 3,5,7,9) .

Fig. 7 shows the effect of the presence salt in the dyeing bath .

In case of protein fibres and polyamide the electrolytes are considered as leveling agents owing to the unsimilarity of charges on both fibres and dyes. Dyeing of proteinic fibres with high affinity anionic dyes necessitates the use of salt in the dye bath for obtaining level dyeing.

Fig.8 shows the effect of salt concentration on the colour strength for the dyed wool/nylon fabrics. Expectedly, the colour strength was better in the absence than in the presence of salt[25], whereas it plays as leveling agent in case of dyeing wool fiber specially at low pH medium .

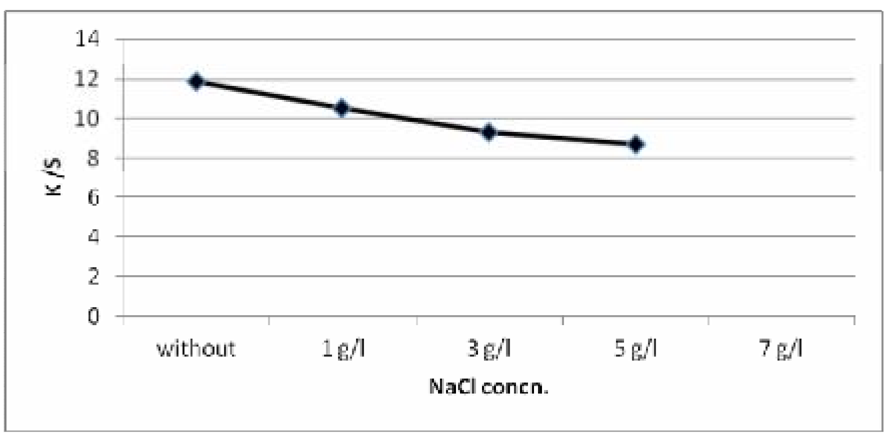


Fig.(8):Effect of salt concentration in the dyeing bath on the colour strength (K/S) of pretreated wool/nylon blend .

3.2.4.Effect of dyeing temp. :

The effect of temperature on the dyeability of wool/nylon blend fabrics with Camphor natural dye was conducted at different temperatures (40, 60, 80, and 100 °C). As shown in Fig. 9, it is clear that the colour strength increases with the dyeing temperature .

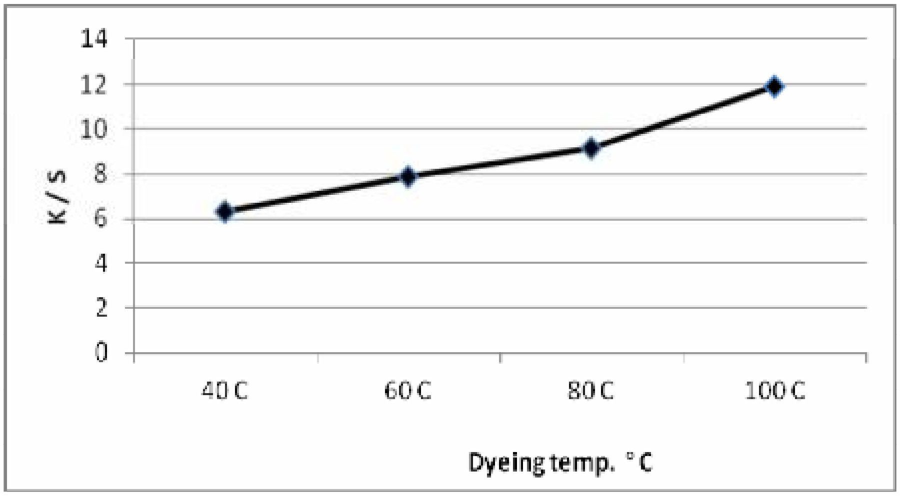


Fig.(9):Effect of dyeing temperature on the colour strength (K/S) of pretreated wool/nylon blend .

It is obvious, from Figure 9 that the rate of dyeing increases gradually by raising the temperature. Maximum dye fixation is achieved at boiling .

This efficiency can be attributed to the higher kinetic energy of the dye molecules and their consequent greater migration power within the substrates. In addition, a higher extent of fiber swelling will have contributed to increased dye exhaustion. As maximum fixation efficiency was obtained around 100 ^{o}C, this temperature was used in subsequent experiments .[26]

3.2.5.Effect of dyeing time :

Fig. 10 shows that the colour strength obtained depend on the dyeing time. The K/S value was increased as the time increases up to 60 min and then it decreases. From 60 min, the effect of dyeing time can be attributed to the thermal stability of camphor dye and its escape from the fibre. The result indicates that 60 min is a suitable time for dyeing with camphor natural dyes

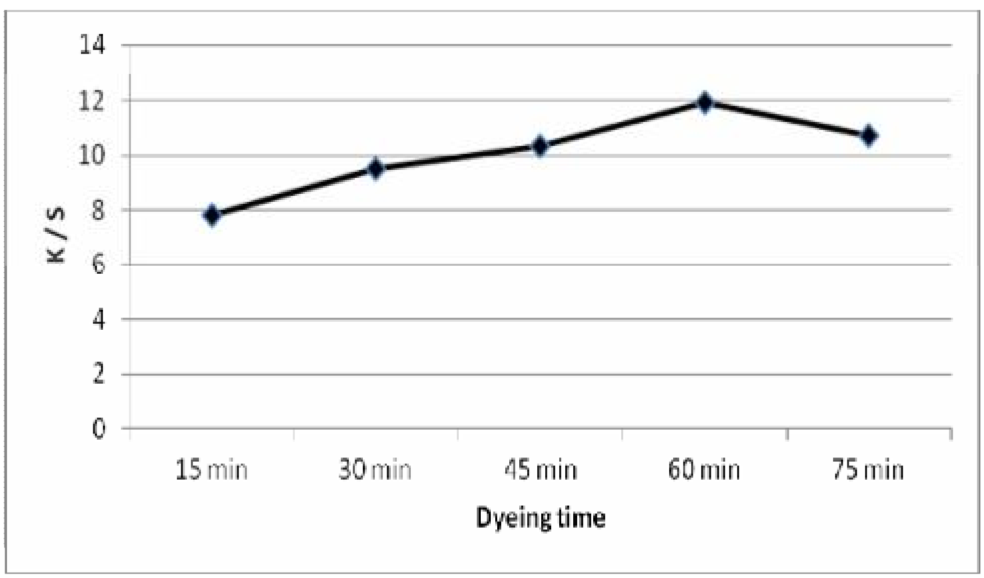


Fig.(10):Effect of dyeing time on the colour strength (K/S) of pretreated wool/nylon blend .

3.4- Fastness properties :

Wool/nylon fabrics dyed with camphor natural dyes and mordanted with four different metal salts (alum ,ferric ,ferrous ,copper) were subjected to fastness tests to washing, perspiration and light .The results of these tests are polluted in table 3

***Table 3*:**Colour fastness properties of wool/nylon blended fabrics dyed with camphor plant

Light	Perspiration						Washing			Samples
	Alkali			Acid			Staining		Alt.	
	Staining		Alt.	Staining		Alt.				
	**	*		**	*		**	*		
4-5	4	3-4	4	3-4	4	3	3-4	4	3	Treated dyed wool/nylon blend
6	4-5	4	4-5	4	5	4-5	4-5	4-5	3-4	Post Modanted sample with alum
5-6	4-5	4-5	5	5	4-5	4-5	4	5	4-5	Post Mordanted sample with ferric sulphate
6	4-5	5	4-5	5	4-5	5	5	4-5	5	Post Mordanted sample with ferrous sulphate
6-7	5	4-5	5	4-5	5	4	4-5	4-5	4	Post Modanted sample with copper

* staining on cotton **staining on wool

From the table, it may be concluded that the fastness properties were good to excellent specially after post-mordanting with mordants . A metallic mordant,that enable them to form a stable, coordination complex with a metal ion in situ within the wool and nylon fibre, this being accompanied by a dramatic improvement in both the fastness of the dyeing to light and wet agencies, as well as a marked change in the colour of the dyeing .[27]

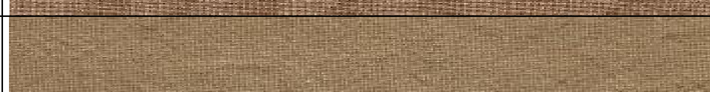
4- Conclusion:

This investigation was focused on using one-bath for dyeing wool/nylon blend by using new natural dye (camphor extract), after successful bio-treatment by using brewer's yeast suspension which contain three enzymes (lipase , amylase , protease) .

The rate of diffusion of camphor dye increased into two fibers and the wettability was improved.

Using brewer's yeast suspension and natural camphor dye minimize pollution at the same time improve the dyeability of both wool and nylon fibres in the blended fabrics. Post-mordanting was improved the fastness properties and the results ranged from good to excellent .

Table (4) : The colored samples of wool/nylon blend dyed with camphor plant and fixed with different mordants

Different Mordant	Colored sample
Champor plant without treatment	
Champor plant after treatment	
Champor plant with Alum	
Champor plant with copper	
Champor plant with ferric	
Champor plant with ferrous	

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