



Impact of Chitosan-poly(amidoamine) Dendreimer Hybrid Treatment on Dyeing and Color Fastness Properties of Wool Yarn with Madder Natural Dye

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ABSTRACT

In recent years, chitosan and its different derivatives have been extensively used in surface modification and functional finishing of textiles. In this study, wool yarns used in handmade carpet were treated with different concentrations of chitosan-poly (amidoamine) dendrimer hybrid (Ch-PAMAM) and characterized by scanning electron microscope (SEM), and their dyeing properties were investigated through dyeing with madder as natural dye. SEM images showed that Ch-PAMAM was almost evenly grafted on the wool surface which resulted in significant improvement in dyeing characteristics with madder. Indeed, treated wool exhibited lower optimal dyeing temperature and dyeing time and required lower amount of madder dye to achieve a desired color strength, as compared to raw wool. Moreover, due to its "bio-mordant" role, Ch-PAMAM was replaced with metal mordant and acid auxiliary in wool dyeing with natural dye. In addition, rub and light fastness of dyed treated wool was improved to some extent. The results of this study confirmed that Ch-PAMAM can be effectively used as alternative bio-mordant in eco-friendly dyeing of wool with madder dye. Prog. Color Colorants Coat. 12 (2019), 241-250© Institute for Color Science and Technology.

1. Introduction

Natural dyes are well-known for coloration of food substrates, leather, wood as well as natural fibers like wool, silk, cotton and flax since ancient times [1, 2]. Natural dyes and pigments can be extracted from various parts of plants including roots, bark, leaves, flowers, and fruit, and provide a wide range of shades [3, 4]. The dyeing of textiles with natural dyes has become an important matter because of increasing environmental awareness and prohibition of the use of some hazardous synthetic dyes [5, 6]. However, the application of natural dyes on textiles has been mainly limited to craftsman, small scale exporters, dyers and printers [7-9]. Lately, the use of natural dyes for dyeing and printing of textiles have started to overcome environmental pollution

produced by the synthetic dyes [1, 3, 7]. Generally, natural dyes create very uncommon, eye-catching, soothing and soft shades as compared to synthetic dyes [1].

For successful commercial use of natural dyes, the appropriate and standardized dyeing techniques are required to obtain the proper quality of dyed textile materials. Although natural dyes are suggested as more sustainable and cleaner sources than synthetic dyes, their negative environmental impacts due to content of residual toxic metal ions, generally used as mordant to enhance dye up-take and fixation, in wastewater are well understood. In fact, variety of metallic salt are generally used as mordants with the aim to increase the affinity of natural dyes towards textile fibers, enhance dye up-take,

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extend color shades, and enhance color fastness properties [10]. However, with the increase of ecological and economical restrictions imposed on textile industry, it is required to find environmentally favorable alternatives in pre-treatment and mordanting of textiles [11, 12].

To reduce above mentioned drawbacks and improvement of natural dye absorption on wool fibers with deep color shades, pre-treatment of wool fibers with compounds containing cationic groups and various bio-mordants have been investigated [13]. However, the usage of hazardous chemical for pretreatment of textiles produce considerable amount of polluted effluent which needs to be treated before discharge into the environment. Hence, to make the textile dyeing and/or finishing more environmental friendly, the pretreatment material should generally be nontoxic, biodegradable, sustainable, durable against repeated washing, compatible with human skin and also have no detrimental effect on the resultant color hue. For this purpose, chitosan and its derivatives are extensively investigated as the best alternatives [14, 15].

Chitosan is a derivative of chitin which obtained from crustaceans like crab and shrimp shell wastes. It has many useful chemical and physical properties such as biodegradability, non-toxicity, antibacterial activity, antioxidant property, etc. [15]. Dendrimers consist of well-defined size, shape, molecular weight and monodispersity. This unique structure creates the best places for the host molecules between the branches [16]. Literature survey shows that dendrimer modified chitosan materials have been extensively used in drug delivery systems, elimination of dyes from colored effluents, extraction of dyes, dyeing and finishing of textiles, etc [17]. It has been reported that pretreatment with chitosan-dendrimer hybrid has significantly improved chemical and natural dye uptake, dyeing properties and also imparted antibacterial properties to wool [17-19], cotton [20, 21], nylon [22] substrates. Moreover, the difference of dyeing performance between damaged and undamaged wool fibers can be decreased through the increasing of dyeing rate and dyeing ability of fibers [17].

To the best out of knowledge, no reports are available in the published literatures concerning the study of dyeing properties of wool pretreated with chitosan-polyamidoamine hybrid (Ch-PAMAM) [23]. Therefore, in this study, the wool yarns were pretreated with Ch-PAMAM, dyed with madder as a source of

common natural dye, and investigated for dyeing characteristics and color fastness properties.

2. Experimental

2.1. Materials and apparatus

Commercial scoured 100% wool yarn (20Tex/4 fold) was supplied by Azar Barf Iran Co. Chitosan (degree of deacetylation (DD): 85%, M_w : 1000 kDa) was provided by Chitotech Co., Iran. Madder was purchased from Yazd province, Iran. Alum (potassium aluminium sulphate) as a metal mordant was supplied by Merck Co. All other chemicals used in this study were of analytical grade.

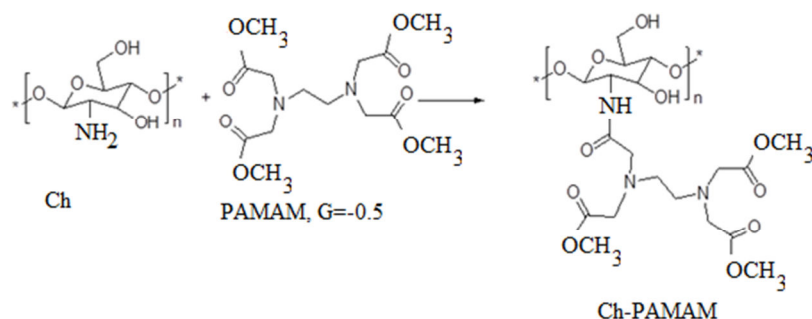
Wool dyeing was performed in acidic media of (pH =5; glacial acetic acid, Merck) using laboratory HT dyeing machine. UV-Visible absorption spectra were measured using a Spectronic Helios Alpha. The color parameters of dyed samples were measured by a surface reflectance Color-Eye XTH Spectrophotometer, X-Rite Inc., using D65 illumination and 10° standard observer.

2.2. Preparation of PAMAM and CS-PAMAM hybrid

PAMAM dendrimer was prepared by the divergent synthesis method starting from ethylenediamine. Briefly, a solution of ethylenediamine (EA) in methanol was added to methyl acrylate (four molar excess). The mixture was stirred for 5 days at 40 °C. Then, the mixture was evaporated to give PAMAM ($G=0.5$) [23]. Chitosan was dispersed in methanol, PAMAM ($G=0.5$) was then added to the prepared suspension and the mixture stirred at 40 °C for 3 days. Then, the mixture was filtered, and dried at room temperature to give Ch-PAMAM hybrid. Preparation of Ch-PAMAM hybrid is illustrated in Scheme 1.

2.3. Pretreatment of wool yarns

Wool yarns were scoured using a solution containing 5 g/L nonionic detergent, 3% Na_2CO_3 at 60 °C for 30 min, liquor ratio (L.R) 40:1, then dried in room temperature. A fine powder of Ch-PAMAM (5-20 % owf (on weight of fiber)) was dissolved in acetic acid solution (pH=4-5). The scoured wool yarns were then immersed for 6 h at 65 °C. The samples were then dried at 80 °C for 5 min, and cured in an oven at 120 °C for 3 min. Then, the samples were dyed with madder natural dye, and fastness properties of treated wool yarns were evaluated.



Scheme 1: Preparation method of Ch-PAMAM hybrid [23] .

2.4. Pre-mordanting of wool yarns

Prior to dyeing, scoured wool yarns were pre-mordanted with alum (5% owf) at boil for 60 min, L.R 40:1, thoroughly rinsed and dried at room temperature and used in dyeing with madder dye.

2.5. Dyeing method

Raw, alum mordanted and Ch-PAMAM grafted wool yarns were dyed with madder dye (70, 100, 125 and 150 % owf), dyeing temperature (60, 80 and 100 °C), dyeing time (15, 30, 45, 60 and 120 min), and dyeing pH values (4 and 7) using L.R 40:1. Dyeing was performed in a laboratory dyeing machine. The samples were wetted for 5 min in the dye bath at 30 °C before the addition of dye, then the dye was added, the temperature was raised to dyeing temperature, and dyeing performed as per dyeing graph shown in Figure 1. As dyeing completed, the samples were removed from dye bath, rinsed with hot and cold water, and dried at ambient temperature.

The absorbance of the dye solution was measured at λ_{\max} of each dye prior and after dyeing tests. Then, the percentage of dye bath exhaustion (E %) was calculated using Eq. 1.

$$E \% = \frac{(A_0 - A_1)}{A_0} \times 100 \quad (1)$$

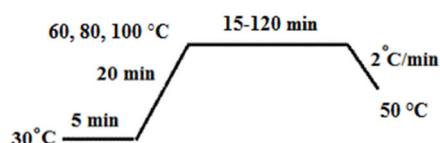


Figure 1: Graph of dyeing wool yarns with madder dye.

Where, A_0 and A_1 were the absorbance of the dye solution before and after the dyeing process, respectively.

Colorimetric parameters of dyed samples such as color strength (K/S), lightness (L^*), redness-greenness (a^*), yellowness-blueness (b^*), chroma (C^*), and hue angle (h°) were calculated from reflectance data. For each sample, at least four individual measurements were performed, averaged and reported. The reproducibility of the experimental data, calculated as relative standard deviation, was in acceptable range of ≤ 4 %.

2.6. Fastness properties

Wash fastness was measured by the standard ISO 105 C06 C2S:1994 (E) method. The washing was conducted for 30 min at 60 °C, rinsed with cold water, air dried, and analyzed. Light fastness test ISO 105 B02:1988 (E) was evaluated with the xenon arc lamp using blue reference samples.

3. Results and Discussion

In our previous studies, we have reported that chitosan-poly(propylene imine) dendreimer hybrid was very effective in surface modification of wool, cotton and nylon textiles which resulted in appreciable improvement in dyeing rate and dye uptake of synthetic and natural dyes [17-22]. Since dyeing properties and color fastness of Ch-PAMAM modified wool with madder natural dye have not been investigated so far, therefore, it has been investigated in this research.

3.1. SEM images

SEM was used to study surface morphology of wool yarns (Figure 2). It is evident that the morphologies of

raw and treated wool are different. In the case of raw wool, the scales are clearly distinct and the wool surface is almost clean with no impurities or foreign materials. But, the surface of Ch-PAMAM treated wool is smoother and the presence of foreign particles distributed on wool surface is evident confirming the successful grafting of Ch-PAMAM on the surface of wool fibers.

3.2. Dyeing and related parameters

3.2.1. Effect of Ch-PAMAM concentration on dye uptake

The effect of Ch-PAMAM concentration on dye uptake of wool yarns is shown in Figure 3. It is well observed that the dye uptake of treated wool is markedly higher, especially with higher concentrations of Ch-PAMAM,

as compared to untreated wool. Further, the dye uptake steadily increased with the increase of Ch-PAMAM concentration and reached the maximum at around 15 % owf Ch-PAMAM. This behavior is likely due to the increase in the number of functional groups like free amino, amide, hydroxyl, and carbonyl groups on the surface of treated wool which are able to interact frequently and more easily with madder dye molecules via formation of hydrogen bonds and/or electrostatic forces, thereby giving rise to sequentially increased dyeing property of the grafted wool [18, 24]. However, a decrease in dye uptake after 15% owf Ch-PAMAM may be attributed to saturation of wool fibers and overcrowding of dye molecules. As a result, Ch-PAMAM 15% owf was selected as the appropriate concentration for treatment of wool yarns, and used for further investigation of dyeing properties.

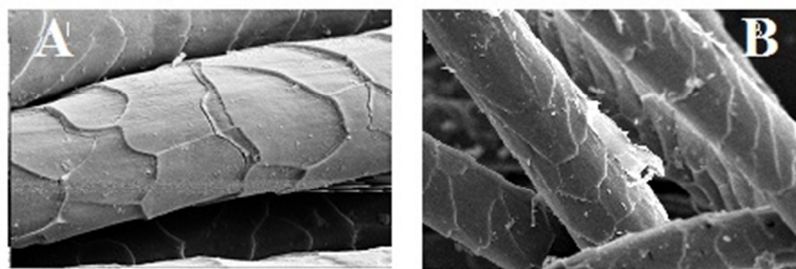


Figure 2: SEM images (20 KX) of (A) raw wool, and (B) Ch-PAMAM (15 % owf) treated wool.

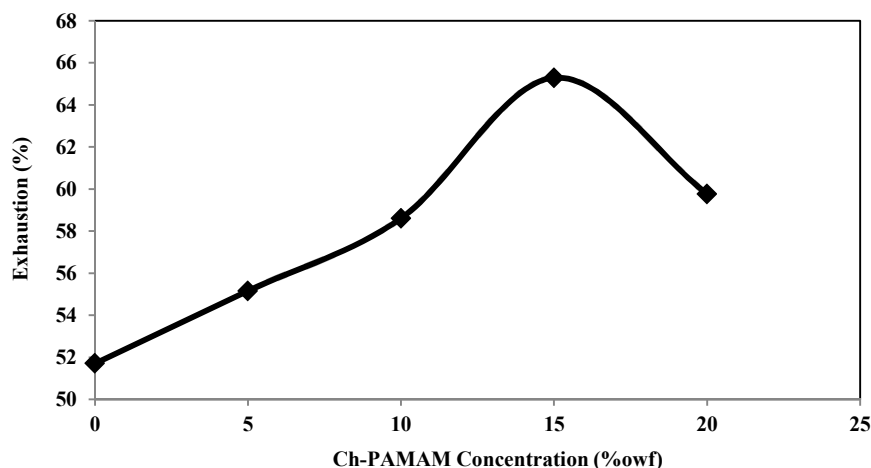


Figure 3: Effect of Ch-PAMAM concentration on madder dye uptake by wool yarns (pH: 4, T: 100 °C, time:60 min, L.R: 40.1 and C_{Madder} : 100 % owf).

3.2.2. Effect of initial dye concentration on dye uptake

The effect of initial concentrations of madder dye (70-150 % owf) on dye uptake of raw and Ch-PAMAM treated wool yarns are shown in Figure 4. For raw wool, percent dye exhaustion steadily increased with almost higher rate with the increase in initial dye concentration presumably due to the enhancement in the driving force at higher initial dye concentrations [24, 25]. However, treated wool exhibited relatively higher dye uptake at lower dye concentrations, so, the rate of increase in dye exhaustion as a function of initial dye concentration was comparatively lower than that of raw wool. Modified wool almost reached equilibrium using 125 % owf dye, while raw wool still required further amount of dye to get saturated. Interestingly, treated wool dyed with 70 % owf dye demonstrated similar dye exhaustion to that of raw wool dyed with 125 % owf dye. These behaviors are related to different morphologies and surface characteristics of raw and treated wool which play key role in dye/fiber interactions. In fact, large number of functional groups on surface of treated wool facilitate interactions with dye molecules in dye bath and consequently enhance dye uptake. A decrease in dyeing saturation concentration as well as the enhancement in the amount of dye uptake is an important outcome since the usage of dye and chemicals can be reduced which is important from environmental and economical points of view [17, 18].

3.2.3. Effect of dyeing pH on dye uptake

Dyeing pH is a key factor which has significant effect on dye uptake and the stability of dye molecules in the solution [8, 10]. Generally, it is recommended to dye wool fibers with natural dyes at weak acidic media ($\text{pH} \approx 4$), below the isoelectric point of wool ($\text{pH} = 4.2$) [8, 10, 18, 19] since in this condition relatively high dye exhaustion is obtained. Figure 5, demonstrates percent dye exhaustion as a function of two dyeing pH values. It is observed that dye uptake of treated wool was slightly higher at both dyeing pH values. Also, dye exhaustion in neutral medium ($\text{pH} = 7$) is relatively higher than weak acidic medium ($\text{pH} = 4$) for both raw and treated wool. Considering the isoelectric point of wool ($\text{pH} = 4.2$) and chemical structures, types and ratio of madder colorants, this behavior can be explained by the type of interactive forces between madder dye and wool polymer chains [18]. In fact, alizarin and purpurin as two main colorants of madder dye are less soluble/insoluble in water, and their solubility in water decrease with decreasing of pH. These colorants are mainly absorbed by wool via hydrogen bonding rather than ionic forces. Compared to neutral condition, in acidic medium due to protonation of wool, the possibility of hydrogen bonding of madder dye molecules with wool polymer chains significantly decreases. As a result, above the isoelectric point of wool ($\text{pH} = 7$), madder dye uptake is relatively higher. Therefore, $\text{pH} = 7$ (acid free) medium was selected for the rest of dyeing tests.

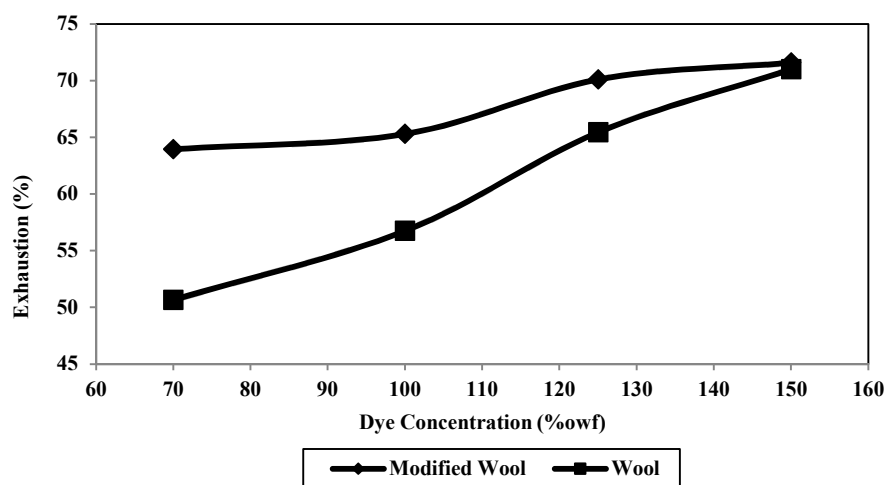


Figure 4: Variation of dye exhaustion as a function of dye concentration in dye bath ($\text{pH} = 4$, T : 100°C , t : 60 min, L.R: 40.1 and $C_{\text{Ch-PAMAM}}$: 15 % owf).

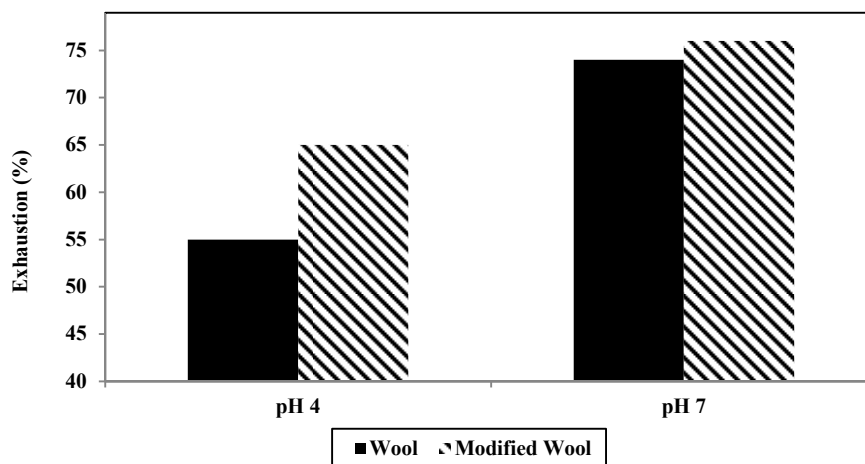


Figure 5: Effect of dyeing pH on madder dye exhaustion by wool yarns (T: 100 °C, t: 60 min, L.R: 40:1, $C_{\text{Ch-PAMAM}}$: 15% owf and C_{Madder} : 100 % owf).

3.2.4. Effects of dyeing temperature and time on dye uptake

Temperature has several important effects in process of textile fibers. Increasing temperature raises thermal energy which loosens fiber structure and facilitates the breaking down of dye agglomerates and diffusion of dye molecules within the fiber structure, increases the uniformity and levelness of dyeing, but causes hydrolytic degradation of wool chemical structure, especially at highly elevated temperatures, i.e., boiling temperature [18, 19]. The effect of dyeing temperature (60, 80 and 100 °C) on dye exhaustion of raw and treated wool yarns is shown in Figure 6. Temperature has similar effect on dye uptake trend on raw and treated wool. Meanwhile, treated wool has higher dye

exhaustion than raw wool. This can be due to more interaction between dye absorbing places and dye molecules, enhanced solubility and movement, and easier penetration and diffusion of dye molecules within wool fiber structure. It is also found that treated wool yarns dyed at 60 and 80 °C possess similar dye exhaustion compared to raw wool dyed at 80 and 100 °C, respectively. This finding confirms that the optimum dyeing temperature of treated wool has decreased at least 20 °C, thereby less energy is needed for dyeing. Moreover, the likely hydrolytic degradation of macromolecular chains of wool fibers generally occur at boil can be reduced by decreasing of dyeing temperature [18, 19].

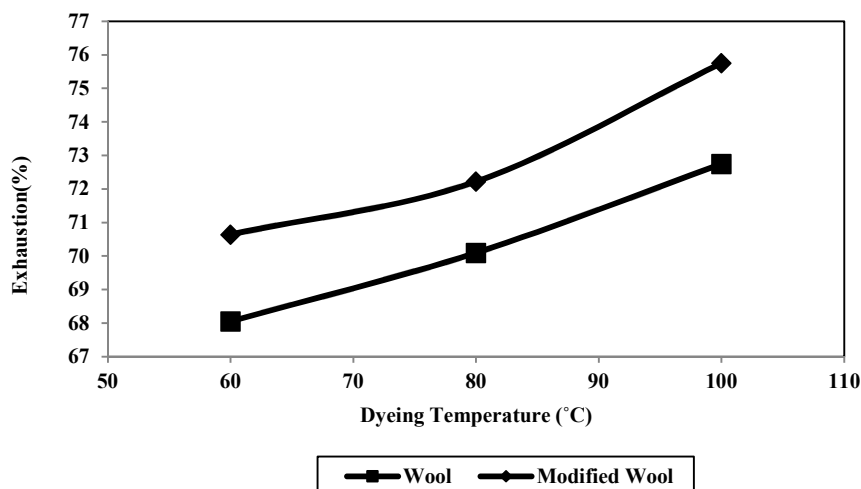


Figure 6: Variation of madder dye exhaustion by raw and Ch-PAMAM (15 % owf) treated wool as a function of dyeing temperature (pH= 7, time: 60 min, L.R: 40:1 and C_{Madder} : 100% owf).

Dye exhaustion behavior of untreated and Ch-PAMAM treated wool as a function of dyeing time is shown in Figure 7. It is clear that, at any given time, treated wool showed higher dye exhaustion particularly at early stages of dyeing compared to raw wool. In addition, treated wool reached equilibrium with 60 min, thereafter no further dye uptake occurred by prolonging dyeing time. In the case of raw wool, however, dye exhaustion steadily increased as a function of time and equilibrium was not realized within the studied time range (0-120 min). Furthermore, it is observed that treated wool yarn dyed for 30 min possesses similar dye exhaustion to that of raw wool dyed for 60 min.

As discussed earlier, the differences in the surface properties of wool fibers are responsible for different dyeing behaviors. In fact, large number of functional groups on treated wool, as vacant dye absorbing sites, are easily accessible to dye molecules and thus dye molecules are easily adsorbed with much faster rate compared to raw wool and get saturated within short range of time (≈ 60 min) [18, 19]. However, besides the limited number of functional groups in the surface of untreated wool fibers, the performance of these groups as dye absorbing places is strongly influenced by the hydrophobic nature of wool, as a result, the dye exhaustion is generally slow and time consuming process [8]. This explains that why equilibrium was not

reached within the investigated time range (120 min). Therefore, it is concluded that a decrease in time of dyeing and equilibrium as well as enhanced dye uptake can be realized by Ch-PAMAM treatment of wool yarns which is worthwhile finding in terms of time, energy, and chemical saving in dyeing with madder natural dye.

Referring to Figure 5, the modified wool attained equilibrium at around 60 min while the untreated wool still required higher amount of time (~ 120 min) for saturation in the measured time range. In the analysis of a dyeing system including particular dye and fiber, a reduction in dyeing saturation time along with the improvement in the amount of $E\%$ is very significance since perceptible amount of time, energy, and chemicals could be saved in both economical and environmental points of view. This aim was achieved in this study by a reduction in the necessary amount of dye for a preferred depth of color on the substrate, and a decrease in the dyeing time and energy.

3.3. Color Fastness

Color fastness of dyed samples were measured according to standard methods, and the changes in color and the degree of staining on wool and cotton were investigated visually with grey scale, and the results are summarized in Table 1.

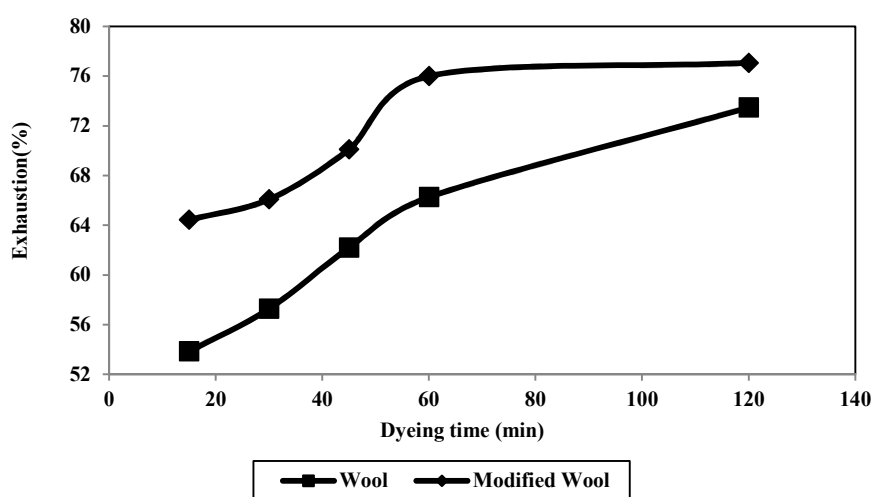


Figure 7: Variation of dye uptake with dyeing time for raw and Ch-PAMAM (15 % owf) treated wool (pH= 7, T: 100 °C, L.R: 40:1 and C_{Madder} : 100 % owf).

Table 1: Color fastness of wool yarns dyed with 100 % owf madder dye at 1/1 standard depth (CC: Color change, SC: Staining on cotton and SW: Staining on wool).

Sample	Wash fastness			Light fastness	Rub fastness	
	CC	SC	SW		Dry	Wet
Wool	4-5	3	3	6	3-4	3
Wool-Ch-PAMAM	4-5	4	4	6-7	4-5	4
Wool-Al	4-5	4	4	6	3-4	2-3

It is apparent from data that the wash fastness of all dyed wool samples is satisfactory, and treatment with Ch-PAMAM resulted in 1 grade improvement in the change of shade and staining of adjacent fabrics compared to dyed raw wool. Both dry and wet rub fastness of Ch-PAMAM treated wool showed 1 grade improvement compared to raw or alum mordanted wool. Slightly improved wash and rub fastness can be related to enhanced interactions of numerous functional groups of Ch-PAMAM treated wool with those of madder dye molecules so that hold dye molecules more tightly and avoid detachment or movement of dye molecules [18, 19]. Light fastness of madder dye with anthraquinone-based chemical structure on raw wool has also good rating (6) which was slightly improved by treatment with Ch-PAMAM. This might be due to intrinsic characteristics of dendrimer structures generally used to improve the optical stability of dye materials [26, 27]. Overall, color fastness results showed that Ch-PAMAM pretreatment not only had no adverse effect on fastness of color against wash, rub and light, but also slightly improved color strength and color fastness.

3.4. Colorimetric properties

CIE $L^*a^*b^*$ values of untreated, Ch-PAMAM treated, and alum mordanted wool yarns dyed with madder dye are presented in Table 2. In fact, dyeing with madder

produced red brick color hue with slight variations in color coordinates but no changes in color hue depending on the type of wool yarn used. Ch-PAMAM treated yarns exhibited darker shades (lower L^* value) which was in agreement with higher color strength (higher K/S value) compared to untreated wool. The variations in colorimetric data (L^* , a^* and b^* values) can be attributed to the new chemical interaction formed between various functional groups of dye/Ch-PAMAM-wool system.

Metal mordants play important role in dyeing of wool yarn with natural dyes because they have substantivity for both dye and fiber and form insoluble complex between the dye and the wool fiber. A comparison of color data revealed that K/S values of raw wool yarns were enhanced by incorporation of alum mordant. Further, Ch-PAMAM treated wool exhibited K/S values higher than that of alum mordanted wool confirming that treated wool could be successfully dyed with same or higher color depth without using alum mordant. In other words, metal mordant could be eliminated and replaced with Ch-PAMAM as “bio-mordant” in wool dyeing process with madder natural dye. This finding is worthwhile in terms of environmentally friendlier dyeing of wool with natural dyes having lowered load of discharged wastewater and associated human health risks.

Table 2: Colorimetric data of different wool yarns dyed with madder dye.

Sample	L^*	a^*	b^*	C^*	h°	K/S
Wool	30.34	29.14	18.39	34.46	32.26	23.05
Wool-Ch-PAMAM	29.36	31.49	22.48	38.69	35.53	25.75
Alum mordanted Wool	31.88	36.56	27.60	45.81	37.05	24.01

4. Conclusions

The results of this study revealed that Chitosan-Poly(amidoamine) hybrid (Ch-PAMAM) can be used as a biocompatible agent to modify surface characteristics of wool yarns. SEM images confirmed almost even distribution of Ch-PAMAM on the surface of treated wool yarns. Due to introduction of numerous polar functional groups on wool surface, dyeing characteristics was appreciably improved. Similar color depth for treated wool dyed at 80 °C, compared to raw wool dyed at 100 °C revealed that optimal dyeing temperature has moved towards lower temperatures. It was also noticed that the amount of dye needed for a desired color depth as well as dyeing time could be significantly decreased when treated wool yarns are used. In addition, as an alternative bio-mordant, Ch-PAMAM can be replaced with alum metal mordant in wool dyeing with madder natural dye, thus makes dyeing environmentally friendlier than conventional

metal mordant dyeing. Colorimetric data and color fastness tests confirmed that wool treatment has slightly improved light and rub fastness of madder dye on wool. Therefore, it was concluded that wool treatment with Ch-PAMAM can greatly assist to cleaner and cost effective production of naturally dyed wool.

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