



Application of Derivative Spectrophotometry to Determine the Relation Between Color Intensity and Dye Concentration of Madder

S. Ghanean and M. Ghanbar Afjeh*

Department of Textile Engineering, Amirkabir University of Technology. P. O. Box: 15875-4413, Tehran, Iran.

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ABSTRACT

The Derivative Spectrophotometry usually improves resolution bands, eliminates the influence of background and provides more defined fingerprints than original spectra, hence enhancing the detectability of minor spectral features. In this work, different madder types were collected and applied for dyeing woolen yarns used in Persian carpet in conventional pre-mordanting and dyeing process with different amounts of the dye. The second derivative of the Kubelka-Munk function of dyed samples has been calculated and implemented for eliciting a function that relates the color intensity of madder to dye concentration used in dyeing of Persian woolen carpets. It was found that for all selected madders, an exponential function could be appropriately adjusted to control this relation. Thus the concentration of madder used in an unknown woolen yarn could be approximately determined with the fitted function. Prog. Color Colorants Coat. 9 (2016), 183-194 © Institute for Color Science and Technology.

1. Introduction

Derivative Spectrophotometry (DS) is a simple non-destructive powerful analytical technique of great utility for extracting both qualitative and quantitative information from spectra. It consists of calculating and plotting one of the mathematical derivatives of spectral curves with respect to wavelength. DS has been widely used to enhance the signal and resolve the overlapped peak-signals due to its advantages in differentiating closely adjacent peaks, and identifying weak peaks obscured by sharp peaks. When DS is implemented, the structure of the original spectral curve is magnified and the detectability of minor spectral features is enhanced.

Thus, the information content of a spectrum is presented in a potentially more useful form, offering a convenient solution to a number of analytical problems, such as resolution of multi-component systems, elimination of sample turbidity, matrix background and improvement of spectral details. Derivative methods were first applied to ultraviolet/visible and infrared spectroscopy in 1953 and have been widely used as a tool for quantitative and qualitative analysis, characterization and quality control in various fields such as pharmaceutical, inorganic, organic, biological, food, environmental, agricultural and etc. [1-7]. Pharmaceutical [8-12] and

*Corresponding author: ghanbar@aut.ac.ir

inorganic [13, 14] analyses are two areas where DS has been more used.

The well-known Kubelka-Munk function in its derivative form provides a gainful indicative tool for characterization and quantitative analysis of pigment mixtures due to its ability in enhancement of sensitivity and specificity in mixture investigations [15, 16].

A variety of natural dyes such as Madder, Indigo, Cochineal, Weld, Walnut shells, Onion skins, Pomegranate shells, Henna and etc. are used on woolen yarn to prepare a broad range of colors for Persian carpet. Different metallic salts known as mordants are used by traditional dyers which not only improve the fastness of woolen yarns, but also extend the resultant color gamut. Among diverse conventional natural dyes used for dyeing Persian carpet, Madder is widely used for centuries due to its accessibility in Iran, acceptable general fastness properties and besides; it provides extensive range of various reds, oranges, browns, etc [17-19]. Belonging to the group of mordant dyes, madder is extracted from dried roots of *Rubiatinctorum* L., and has been used since antiquity for dyeing textiles. It is also known as a traditional herbal medicine used for treatment of kidney and bladder stones [20, 21]. The structure of the dye constituents is based on anthraquinone or similar skeleton. So far, up to 36 anthraquinones have been detected in madder, the most important one being alizarin (1,2-dihydroxy-anthraquinone) followed by purpurin (1,2,4-trihydroxy-anthraquinone) which yields orange or yellow dye. Other anthraquinone components are ruberythric acid, lucidin, rubiadin, xanthopurpurin, psedopurpurin, quinizarin and etc [22-25].

The color strength of a dye is a measure of its ability to impart color to other materials. This property is characterised by the absorption in the visible region of the spectrum and can be expressed as a color strength value. The relationship between reflectance value and dyestuff concentration has a complicated structure and this relationship must be known in order to solve the problems related to color measurement methods. This relationship is also very significant being a basis for calculation of dye formulation where one or more combination of several dyes is involved [26, 27]. For a known wavelength when the reflectance values are plotted against dye concentration, a markedly nonlinear relationship will be obtained. The most useful function which linearly relates reflection to color concentration is the Kubelka-Munk function as

illustrated in Equation 1.

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (1)$$

Where K and S are the absorption and scattering coefficients of the film and R is the reflectance of the light with a given wavelength by a sample of infinite thickness.

A valuable feature of Kubelka-Munk theory is that the absorption and scattering coefficients of a colored material can be built up from the individual absorption and scattering coefficients of the individual pigments or dyes (Equation 2).

$$\frac{K}{S} = c_1 \left(\frac{K_1}{S_s} \right) + c_2 \left(\frac{K_2}{S_s} \right) + c_3 \left(\frac{K_3}{S_s} \right) + \dots + \frac{K_s}{S_s} \quad (2)$$

Where c is concentrations of colorants, subscript s identifies the substrate and subscripts 1, 2, 3 ... identify the individual colorants [28].

In this paper, the second derivative of the Kubelka-Munk equation has been implemented to determine the functions which relate the color intensity to madder concentration in woolen yarns used in Persian carpet.

2. Experimental

2.1. Madder

5 different madders were collected including: 1) Mediterranean powdered madder with no clear information about the sample's age, 2) Three years old Madder root from Bafq, Yazd, Iran, 3) Three years old Madder root from Khur and Biabanak, Isfahan, Iran, 4) Four years old Madder root from Nain, Isfahan, Iran (the last three samples were grinded into powder by electrical grinder and then sifted), and 5) Final specimen was gathered from a Madder root silo in Ardakan, Yazd, Iran, where is known for its best quality madder in the whole country. These roots were finely powdered in the local traditional gristmill.

2.2. Dyeing

3ply woolen yarns with 98.03 S t.p.m and 0.22 metric number was selected in the form of hank from Tabriz, East Azerbaijan province of Iran and were scoured with 3 g/lit non-ionic detergent at 50-60 °C for 20 minutes. Prior to the dyeing process, woolen yarns were pre-

mordanted with 2% (o.m.f) Alum, $AlK(SO_4)_2$. Formic acid was used for adjusting the pH at 5 and the L:G was 40:1. Mordanting was started at 40 °C and then the temperature gradually was raised to the boiling point over 45 minutes. The samples were treated at the boiling temperature for 75 minutes and after rinsing, they were subjected to dyeing. For dyeing, different amounts of Madder (i.e. 5%, 10%, 20%, 40%, 60%, 80% and 100% (o.m.f)) were used for each selected type. The pH of dyebaths was kept at 5.5 by adding acetic acid and L:G was the same as the mordanting step. The dyeing process was started at 40 °C and then the dyebath temperature was raised to the boil over 30 minutes. Finally, dyeing was carried out at boiling point for an hour and samples were thoroughly rinsed.

2.3. Spectrophotometry

All dyed yarns were wound over a 2cm×4cm cards. A portable ColorEyeXTH spectrophotometer from GretagMacBeth Company was used for the reflectance

measurements of samples. The specular component of reflectance was excluded. Samples were measured from 360 to 750 nm at 10 nm intervals. In order to minimize the sample presentation effects, the reflectance of each sample was measured at four different rotational positions and the average was thought to be the true reflectance. Finally, the spectral range of measured spectra was narrowed to 400 to 700 nm for further mathematical operations.

3. Results and discussions

The spectral reflectance of woolen yarns dyed with 40% of different selected madders is plotted in Figure 1. As the plot shows, the spectral reflectance of different madder types is qualitatively very similar, and they haven't any distinguishable peak and differ only in altitude. Using the well-known Kubelka-Munk function as illustrated in Equation 1, the K/S values were calculated from measured spectral reflectance of dyed samples and plotted in Figure 2.

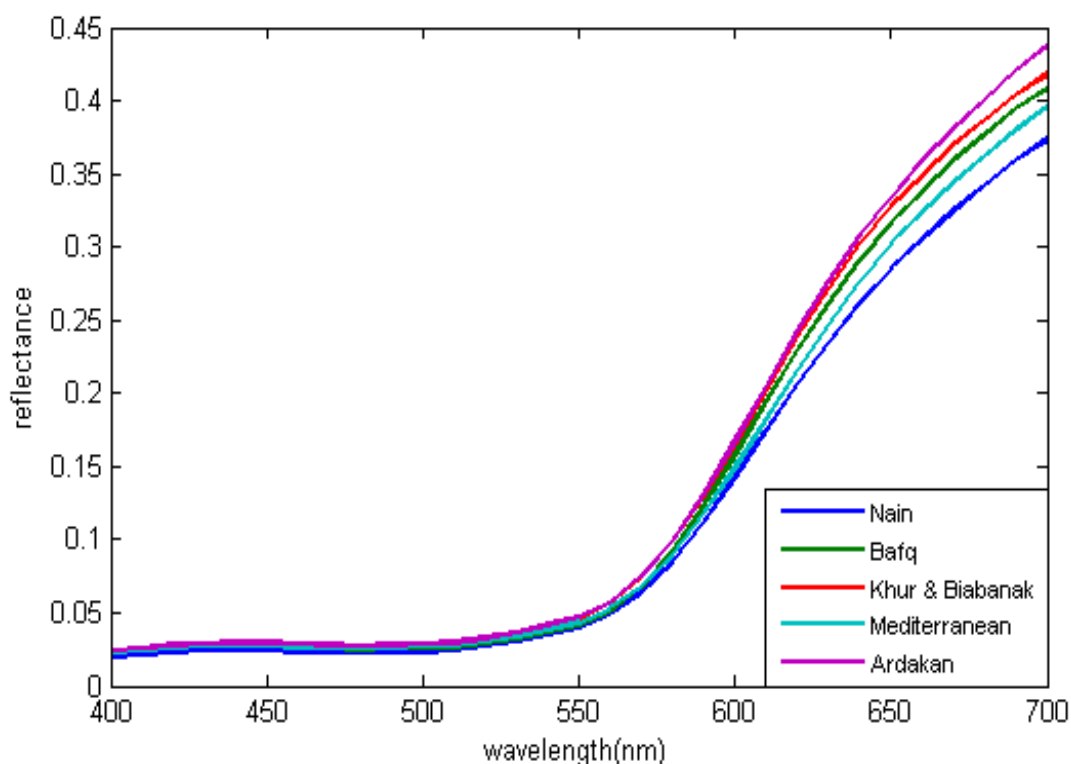


Figure 1: Spectral reflectance of Tabriz woolen yarn dyed with 40% (o.m.f.) of different madders.

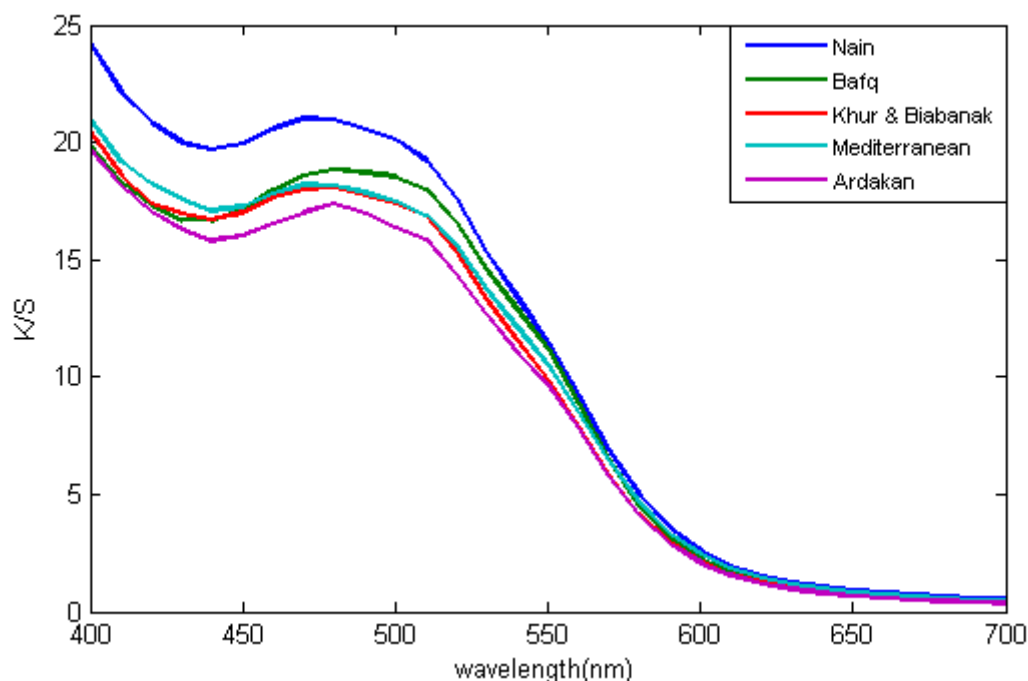
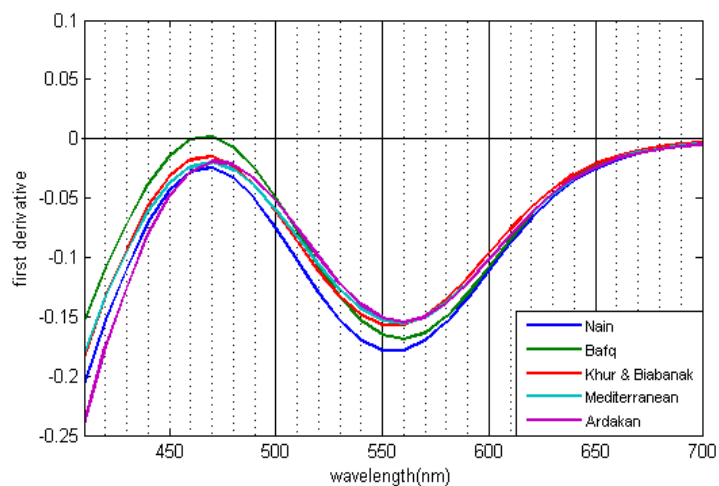


Figure 2: $\frac{K}{S}$ of Tabriz woolen yarn dyed with 40% o.m.f. of different madders.

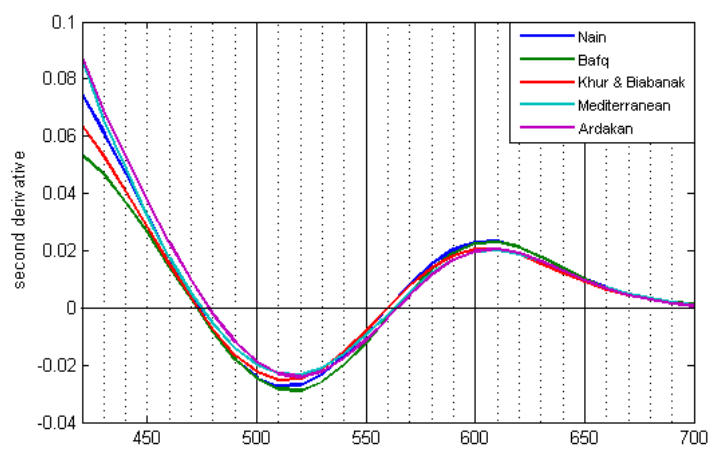
Although the smoothness of the K/S curves is less than the reflectance curves but they still do not have sharp peaks. To increase the sensitivity of the curves, the first, second and third derivatives of obtained K/S curves with respect to wavelength were calculated, smoothed in order to eliminate fluctuations and then plotted respectively in Figure 3 (a-c). According to Figure 1, each reflectance curve which is the characteristic of that sample does not provide any conclusive data by itself, because it presents only broad bands instead of define peak. As Figure 3 shows, the derivatives greatly enhances minor convexities and concavities of the original curve (K/S) and gives a much narrower band width which provides improved resolution of subtle bands. Comparison of the zero order curve with the second derivative of K/S curve shows that the areas of the original curve that have the greatest change in slope correspond with the minima and maxima in the second derivative. In other words, a key feature of the second derivative spectrum is its ability to resolve zero order curve with no maxima and

minima into a second derivative curve with distinct maxima and minima. Considering the second derivative curves of different madders in Figure 3, it is obvious that same significant minimum and maximum peaks are resulted for all cases. As the plot shows, although madders are different in age and place of cultivation, distinct minimum and maximum peaks at 510 nm and 605 nm are identified respectively for all samples.

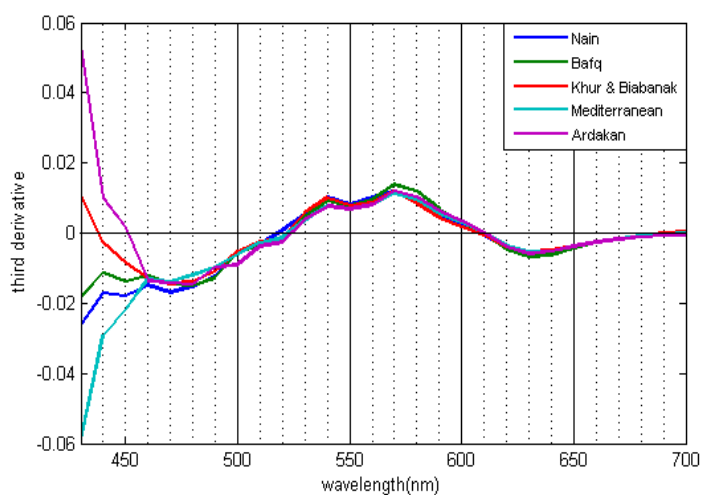
In Figure 4 second derivative of the Kubelka-Munk function for Bafq madder at different concentrations is plotted. The band with distinctive maximum peak at 605 nm was selected for measurement of intensity. The whole peak heights of the second derivative for every concentration of Bafq madder were measured to find the function which fits the data. It can be seen that curves at different madder concentrations i.e. 100%, 80%, 60%, 40%, 20%, 10% and 5% (o.m.f) represent 100%, 89.9%, 86.4%, 69.5%, 54.2%, 25.4%, and 15.2% of peak intensity measured at the characteristic maximum, respectively.



(a)



(b)



(c)

Figure 3: Derivatives of K/S for 40% o.m.f. of different madders on woolen yarns; a: first order, b: second order, c: third order.

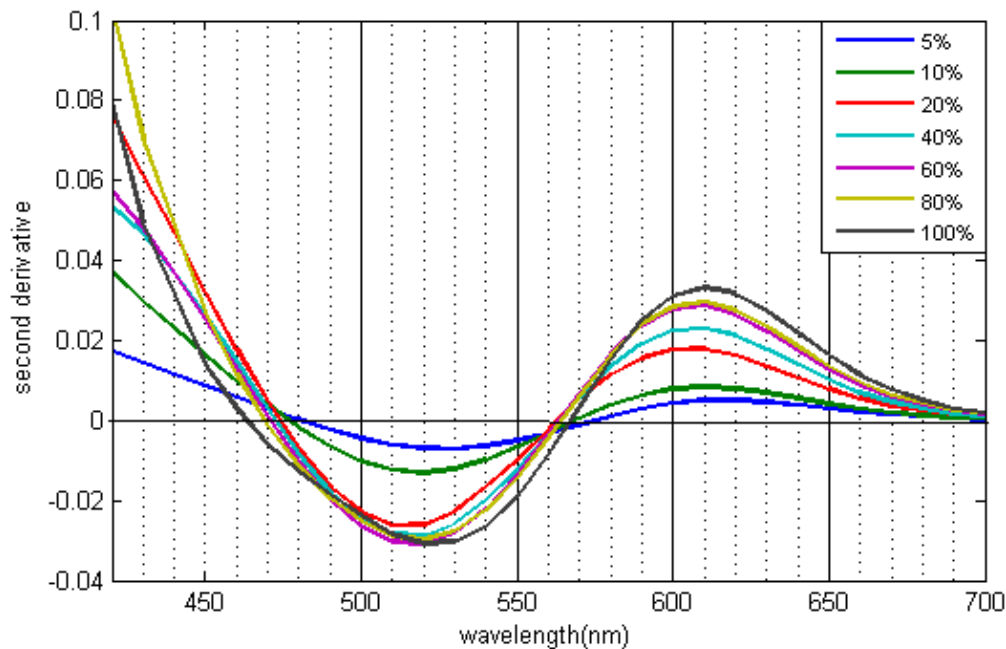


Figure 4: Second derivative of the Kubelka-Munk function for Bafq madder at different dye concentrations.

In order to obtain the mathematical function which provides the best estimation of the concentration of madder for a given intensity of dye, linear, logarithmic, exponential and power functions were tested. The adjustment of the function that rules the relation between the Bafq madder mass percentage and the relative peak percentage is demonstrated in Figure 5. It is found that the best fitness between the madder concentration and peak intensity is an exponential function as shown in Equation 3.

$$y = Ae^{Bx} \quad (3)$$

where y is the mass percentage of dye on mass of sample and x is the color intensity percentage for each concentration relating to highest concentration. Parameters A and B are 3.2539 and 0.0348, respectively.

In the case of Ardakan madder, the derivative curves at different concentrations such as 100%, 80%, 60%, 40%, 20%, 10% and 5% (o.m.f) represent 100%, 91.4%, 77.6%, 63.8%, 41.4%, 25.9%, and 13.8% of peak intensity measured at the 605 nm, respectively. The adjustment of the function that rules the relation between the Ardakan madder mass percentage and the relative peak percentage is demonstrated in Figure 6.

It is found out that the curves with the best fit were

again exponentials defined in Equation 2 and parameters A and B are 4.0273 and 0.0336, respectively.

This process was repeated for the other three selected madders to obtain their second derivative functions for adjusting the best relation between the madder's mass and peak intensity. The results again confirm the previous findings; the same exponential function with slight changes in A and B parameters is adjusted to indicate the relation between the dye concentration and color intensity. The values of these parameters as well as the correlation coefficient of curve fitting of regression, R^2 , for each madder are shown in Table 1. As data in Table 1 shows, all madder species have nearly equal coloring power no matter where the plants are from or how old they are. According to Equation 3, x and A are inversely related to each other. Comparing the A parameter of all madders shows that Nain and Ardakan madders have the least and the most values respectively; which means Nain has the most color intensity while Ardakan has the least one. This result is also reconfirmed by the K/S curves plotted in Figure 2.

Another feature of this method is the estimation of madder concentration on unknown woolen yarn without calculating K/S of the substrate.

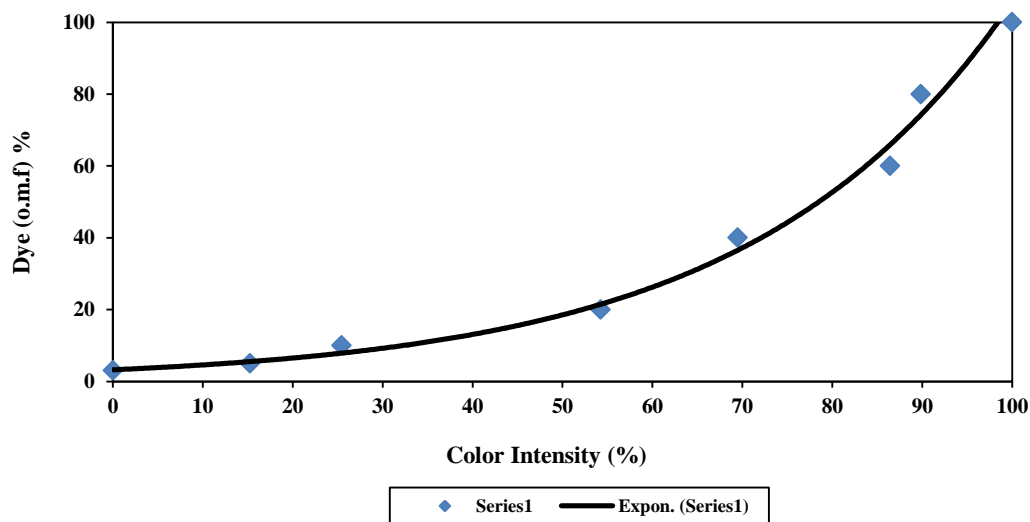


Figure 5: Curve fitting between dye concentration (o.m.f)% and peak intensity% for Bafq madder.

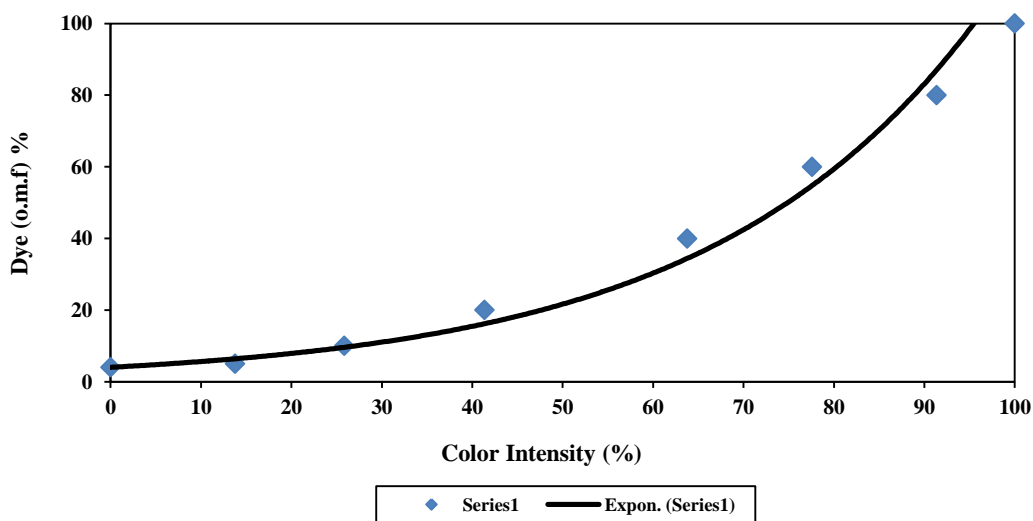


Figure 6: Curve fitting between dye concentration (o.m.f)% and peak intensity% for Ardakan madder.

Table1: Parameters of adjusted exponential function which relates color intensity of different madders to their mass percentage ($y=Ae^{Bx}$).

Madder Type	R ²	Parameters Values
Bafq	0.991	A=3.2539 B=0.0348
Nain	0.993	A=2.3738 B=0.0365
Khur&Biabanak	0.989	A=3.3420 B=0.0335
Ardakan	0.984	A=4.0273 B=0.0336
Mediterranean	0.975	A=3.3379 B=0.0347

For better clarity, the colorimetric specification of 35 woolen samples dyed with different amounts of various madders under D65 illuminant and 1964

standard observer were calculated and reported in Table 2. Resulted data in Table 2 have reasonable agreement with the previous findings.

Table2: Colorimetric Specification of woolen samples dyed with different amounts of various madders under D65 illuminant and 1964 standard observer.

Madder type	Dye Perc.	L*	a*	b*	c*	h°
Ard.	5%	53.5524	28.9191	18.4229	34.2887	32.4992
Ard.	10%	45.2461	31.8042	21.0502	38.1395	33.4993
Ard.	20%	39.2675	33.0153	22.8113	40.1294	34.6418
Ard.	40%	33.2433	31.5713	24.4632	39.9398	37.7705
Ard.	60%	30.8851	31.2411	24.0479	39.4247	37.5874
Ard.	80%	28.2133	30.0542	22.3537	37.4559	36.6412
Ard.	100%	27.4509	30.1527	22.4068	37.5666	36.6164
Med.	5%	50.4093	32.0613	20.3414	37.9697	32.3932
Med.	10%	44.4117	34.5087	22.7092	41.3105	33.3478
Med.	20%	37.9545	33.9276	25.6964	42.5604	37.1400
Med.	40%	33.1238	31.3751	24.8362	40.0155	38.3647
Med.	60%	28.5374	30.2621	21.9031	37.3569	35.8963
Med.	80%	25.6705	28.4663	19.1458	34.3058	33.9239
Med.	100%	24.2412	25.9289	16.5545	30.7630	32.5564
Khu.	5%	53.6732	29.5182	18.7099	34.9483	32.3683
Khu.	10%	40.9162	35.5151	26.1886	44.1267	36.4048
Khu.	20%	47.0337	33.6990	22.3610	40.4430	33.5664
Khu.	40%	34.4413	33.0556	26.7447	42.5200	38.9757

Table2: Continued.

Madder type	Dye Perc.	L*	a*	b*	c*	h°
Khu.	60%	31.5826	31.5007	25.9123	40.7890	39.4405
Khu.	80%	29.8223	30.8397	24.9792	39.6869	39.0064
Khu.	100%	30.2888	30.5900	24.7754	39.3646	39.0046
Baf.	5%	50.8815	31.0346	16.4431	35.1215	27.9161
Baf.	10%	45.4024	33.9245	20.4412	39.6070	31.0710
Baf.	20%	36.9563	34.8548	25.0738	42.9366	35.7304
Baf.	40%	33.4502	33.4286	25.2621	41.9004	37.0784
Baf.	60%	30.3346	32.0689	25.3982	40.9083	38.3788
Baf.	80%	29.1676	30.7563	24.5586	39.3584	38.6070
Baf.	100%	26.9382	29.1777	22.8888	37.0842	38.1128
Nai.	5%	50.6349	30.6579	19.6794	36.4306	32.6966
Nai.	10%	42.1509	32.8863	22.8803	40.0627	34.8278
Nai.	20%	37.4979	33.5344	25.6460	42.2169	37.4076
Nai.	40%	32.0832	31.4012	25.6493	40.5453	39.2429
Nai.	60%	30.6822	30.5935	25.3618	39.7390	39.6585
Nai.	80%	28.1906	28.7341	24.1514	37.5359	40.0475
Nai.	100%	26.8359	26.7270	21.7980	34.4889	39.2000

Finally the proposed method of evaluating color intensity in this study was compared to the well-known classic method of calculating the unit absorption coefficient. In textile, when only one dye is present, the relation between the Kubelka-Munk function and dye concentration in each wavelength will be illustrated as Equation 4.

$$\frac{K}{S} = \left[\frac{k}{s} \right]_{\text{sub}} + c \left[\frac{k}{s} \right]_{\text{unit}} \quad (4)$$

For different known concentrations of dye, while $\frac{K}{S}$ is calculated from the reflectance of dyed samples and $\left[\frac{k}{s} \right]_{\text{sub}}$ is obtained from reflectance of undyed substrate,

the term $\left[\frac{k}{s}\right]_{unit}$, the unit absorption coefficient, could be calculated by the least square method. Unlike their reflectance, $\left[\frac{k}{s}\right]_{unit}$ is not dependent on concentration. In other words, it doesn't belong to any specific concentration but indicates the spectral absorption characteristic of dye in all concentrations. Therefore, plotting $\left[\frac{k}{s}\right]_{unit}$ against wavelength could be considered as the color intensity of one dye on a specified substrate [29]. In this study, by measuring the reflectance of Tabriz woolen yarn as substrate, $\left[\frac{k}{s}\right]_{sub}$ was calculated from the Equation 1. Then for all 5 types of madder, the term $\left[\frac{k}{s}\right]_{unit}$ was obtained by least square method and finally plotted against the wavelength in Figure 7. As curves in Figure 7 show, changes of $\left[\frac{k}{s}\right]_{unit}$ against wavelength for all madders on Tabriz woolen yarn are nearly the same which confirms that same as previous findings from the

proposed method in this study, all selected madders have nearly same color strength.

4. Conclusion

For five different kinds of madder, the second derivative of the Kubelka-Munk function was applied at different dye concentrations in order to find the relationship between the madder concentration and the signature curve that this natural dye reflected in its derivative. It was shown that an exponential function fit the mass percentage and color intensity of all selected madder types with feasible accuracy. As an appropriate method to evaluate the color strength of dye, this relation will be able to estimate the approximate madder concentration in the unknown analyzed sample of woolen yarn dyed with madder and also could be strongly useful in analysis of woolen samples dyed with mixture of madder and other natural dyes used in Persian Carpet.

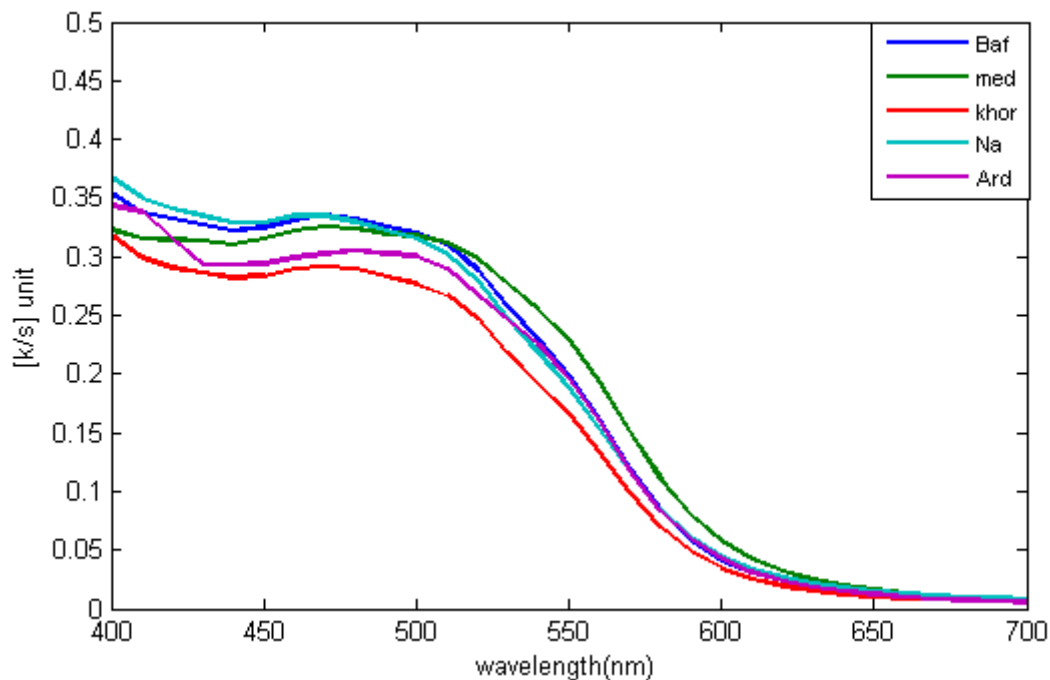


Figure 7: $\left[\frac{k}{s}\right]_{unit}$ of different madders on Tabriz woolen yarn against wavelength.

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