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COLORING PROPERTIES OF WOOL FABRIC COLORED BY NEW DYESTUFFS - AZOMETHINES

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***Abstract*:** *The azomethines have broad applications in food and dyestuff industries, and in analytical chemistry, catalysis and also in the field of agrochemical. These have played an influential part in the improvement of modern coordination chemistry, but also they can also be found at key points in the development of inorganic biochemistry, catalysis and also in optical materials. The present paper describes coloring properties of wool fabric colored by new dyestuffs - azomethines, derivate of isatin. Synthesizing of dyestuffs can often have one to six chromogen, which can be defined as the photoactive components that contain colored or uncolored absorbent components. In addition of monoazo, diazo, poly-azo, anthraquinone, xanthan and similar systems, the azomethines or imines, also includes to the chromogen groups. Azomethines, such as, isatin-3-hydrazone, isatin-3-thiosemicarbazone and isatin-3-phenylhydrazone, were synthesized and their coloring performance on wool fabric assessed. The synthesized azomethines showed very good substantively for wool fibers with good coloring performance according to CIEL*a*b* system which characterized quantitative and qualitative coloring property. Dyestuff 3 or isatin-3-phenylhydrazone bound to woolen textiles to a greater extent and greater intensity (minimum value of L). Dyestuff 2 or isatin-3-thiosemicarbazone linked to the minimum amount for textiles (the largest value of L). Although it must be noted that it is a lighter shade (yellow color) as opposed to the dyestuff 3 (red color).*

Key words: *Azomethines, Dyestuff, Coloring, Wool, CIEL*a*b*.*

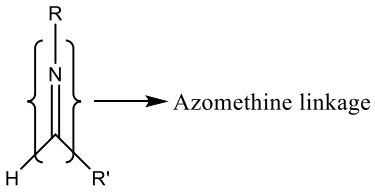
1. INTRODUCTION

Natural and synthetic dyestuffs are compounds of great interest since they play an important role in our everyday life. The broad variety of technical and industrial applications, which includes “classical” utilizations like dyeing of textiles and other consumer goods. Widely applied and interesting representatives amongst the large number of dyestuff categories are triphenylmethane, azo, anthraquinone, perylene, and indigoid dyestuffs [1].

The azomethines are the compounds having azomethine linkage (Fig. 1) and can be synthesized from an amino and a carbonyl compound. These are significant chelating ligands in co-

ordination chemistry to co-ordinate metals ions through azomethine nitrogen and have been considered broadly [2, 3].

As is known, azomethines (isatin derivatives) are reagents which are becoming increasingly important in the pharmaceutical, dyestuff and plastic industries as well as for liquid–crystal technology and mechanistic investigations of the drugs used in pharmacology, biochemistry and physiology [2-5]. Azomethines containing derivatives of salicylaldehyde and aliphatic amines have been studied for their dioxygen uptake and oxidative catalysis because of their similarities to the biological dioxygen carriers, as well as their catalysis properties for the insertion of oxygen into organic substrates [6, 7].



R = amino part
R' = aldehyde or ketonic part

Fig. 1: *General representation of the structure of an azomethines (Schiff bases)*

There are not many results in the world for azomethine application in dyeing processes of different textile materials. The aim of this work was tested synthetized azomethines as dyestuffs in treatment of wool textile fiber. It was necessary to find an adequate recipe for the the highest exhaustion of dyestuff from the baths and good color fastness on fabric.

2. EXPERIMENTAL

2.1. Materials

Equimolar amounts of isatin and amine component (hydrazine, phenylhydrazine and thiosemicarbazide) were dissolved in 95 % ethanol. The solution was heated under reflux for 1 hour. The products were filtered, washed with ethanol and dried in vacuum over $CaCl_2$ [8].

Their structure is shown in Fig. 2.

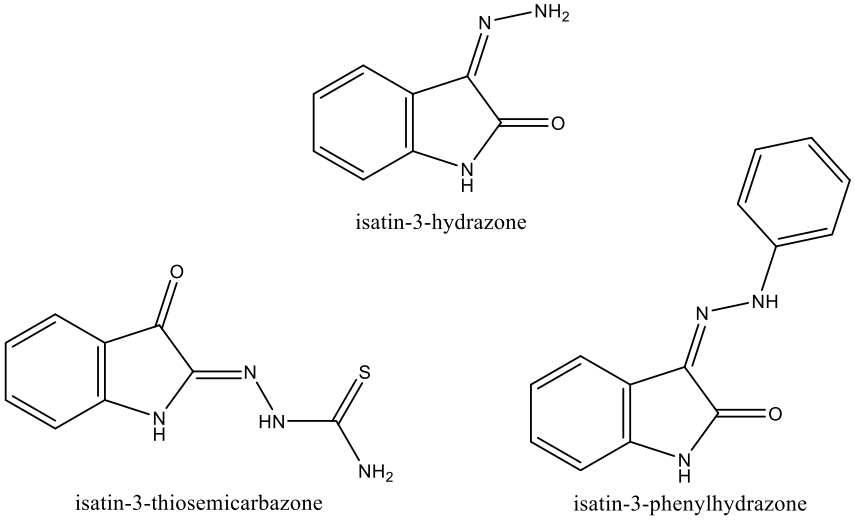


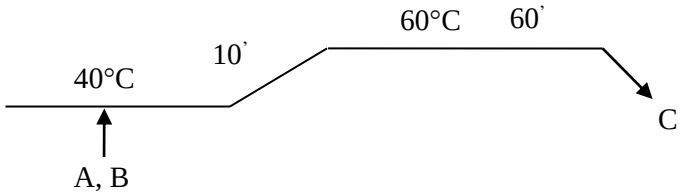
Fig. 2: *Structure of synthesized azomethines*

2.2. Dyeing procedure

The dyeing was carried out in a solution of ethanol/water (50/50 %) without additives, on 60°C for 60 min, in the presence of the new dyestuffs. The reason was the fact that the used active agents, azomethine, insoluble in water and soluble in an alcohol. Higher temperature and dispersant in pure aqueous solutions of azomethine did not give satisfactory results of wool fabrics dyeing.

Isatin-3-hydrazone (yellow powder) marked as dyestuff 1, isatin-3-thiosemicarbazone (orange powder) marked as dyestuff 2 and isatin-3-phenylhydrazone marked as dyestuff 3.

The wool fabric dyeing was performed in Linitest aparature for laboratory dyeing with temperature time regime like at scheme:



(A) - dyestuff (B) - textile (C) rinsing.

To avoid any subjective judgment when it comes to visual valuation of fiber dyeing, we used CIEL*a*b* control system of textile dyeing, considered reflection spectrophotometer, and specific computer software. In this work, we measured the reflectance (remission) of textile samples using reflection spectrophotometer Dye Eye 3000 (ICS – TEXICON) connected with personal computer and specific softer system. With this procedure, it is possible to obtain not only degree of reflection, but also Kubelka - Munk function (color strength) that express the reflection coefficient dependence on fiber color content.

3. RESULTS AND DISCUSSION

Commonly, the wool textile is colored with chromic, acid, reactive, metal-complex etc. dyestuffs. The azomethines are relatively small molecules with low water solubility and possess substantively for hydrophilic or hydrophobic fibers such as wool or polyester.

The figures from 3 to 5 show the reflection dependence on wavelength for colored samples of wool textile. Colored samples possess less reflection, i.e., the highest values for K/S (color strength), which indicates their darkness coloring compare to uncolored samples, what was expected.

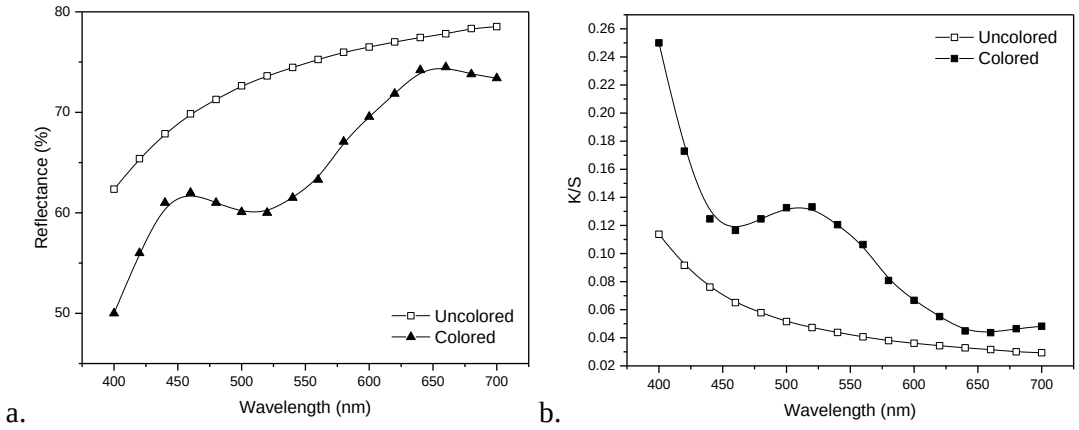
The remission on wavelength for wool (dyestuff 1) represented at Fig. 3a. It can be seen that colored textile absorbed at about 510 nm, which correspond to blue-green color of spectra, and reflects at about 650 nm, which is attributed to red (pink) color. This is actually the color of sample or color that can be seen on textile, visually.

The Fig. 3b shows the dyestuff depth indicator, i.e. the parameter K/S, dependence on wavelengths in distinct area, where the maximum of absorption can be seen. This parameter is in inverse proportion to remission of colored textile by the dyestuff. The parameter K/S increases with depth color enhancement.

The Fig. 4a and 4b represented the values of remission and K/S for samples colored by dyestuff 2. The Fig. 4a shows that the maximum of reflection is at 580-590 nm (yellow color of spectra, visible color), while the maximum absorption is at about 420 nm (blue, complementary color). The parameter, as indicator of intensity of depth dyeing, suggests that colored sample possess the higher absorption and less reflection than control, uncolored sample. Since the difference in

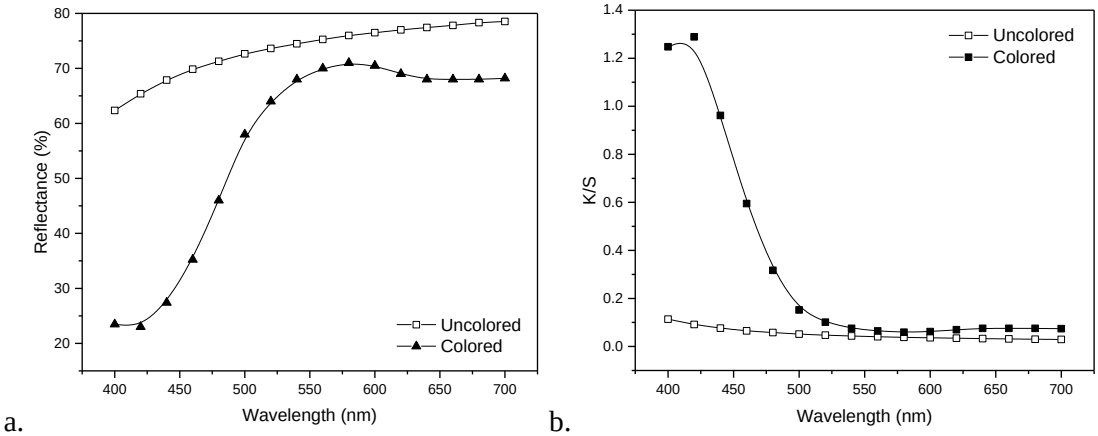
intensity of remission and K/S of colored and control sample is not such a large, it can be concluded that samples are colored in yellow and red (pink) tones by azomethine 1 and 2, respectively.

The Fig. 5a and 5b show the bigger difference in intensity of remission and color strength (K/S), which means stronger and deeper tones of sample colored by azomethine - dyestuff 3. The colored textile absorbs at 510-520 nm (blue-green color of spectra), and reflects at about 670 nm – red-pink color of spectra, which is color of sample.



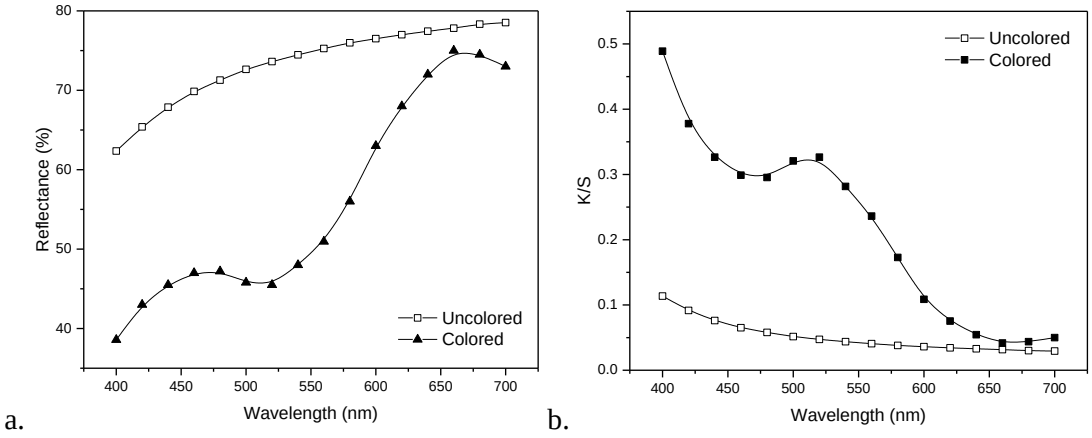
a. b.

Fig. 3: *Spectral reflectance and K/S diagram of wool textile colored by dyestuff 1*



a. b.

Fig. 4: *Spectral reflectance and K/S diagram of wool textile colored by dyestuff 2*



a. b.

Fig. 5: *Spectral reflectance and K/S diagram of wool textile colored by dyestuff 3*

Taking account, that parameter L is smaller while the sample is darker (at all samples); it is obviously that samples are colored by azomethine. It is similar with other parameters of CIEL*a*b* system speaking in favor of the existence of colored wool textile.

Parameter a* is negative at uncolored sample (indicate green), while positive is for sample colored by all the azomethines (indicate magenta). Parameter b* is positive at all colored samples (indicate yellow) and uncolored sample (light of source D65). Also, there are the difference between shades, i.e., hue of dyestuff (H*) and saturation (C*) of dyestuff on sample. h* describes the hue angle. It ranges of colored samples from about 68 to 75, i.e. it is known that, h=0° = red; and h=90° = yellow; but the uncolored sample indicates result around 92 (light of source D65).

According to the results in Table 1, colored woolen samples show higher saturation or "color purity" of non-colored sample woolen textiles, their value range from 36 to 76, while the uncolored sample has the value 12 (light of source D65).

Table 1: *Results of characteristic parameters of CIEL*a*b* system of colored wool fabric by some azomethines*

Sample	Light of source	L*	a*	b*	C*	h*
Uncolored	D65 10 Deg	81.36	-0.53	12.77	12.78	92.37
	A 10 Deg	82.14	2.83	12.83	13.14	77.54
	F2 10 Deg	81.85	-0.46	14.52	14.53	91.80
Dyestuff 1	D65 10 Deg	53.73	10.51	39.35	40.73	75.05
	A 10 Deg	56.88	16.40	42.12	45.20	68.73
	F2 10 Deg	55.34	6.53	43.75	44.24	81.52
Dyestuff 2	D65 10 Deg	62.51	28.57	71.28	76.79	68.16
	A 10 Deg	68.50	29.76	79.68	85.05	69.52
	F2 10 Deg	67.12	17.85	79.76	81.74	77.38
Dyestuff 3	D65 10 Deg	50.00	10.53	35.19	36.73	73.34
	A 10 Deg	52.98	15.82	38.09	41.24	67.44
	F2 10 Deg	51.52	6.66	39.18	39.74	80.35

5. CONCLUSIONS

The azomethines have broad applications in food and dyestuff industries, and in analytical chemistry, catalysis and also in the field of agrochemical. These have played an influential part in the improvement of modern coordination chemistry, but also they can also be found at key points in the development of inorganic biochemistry, catalysis and also in optical materials.

Azomethine derivatives, associated with the amino heterocyclic, can be used for dyeing of wool fabric. The synthesized azomethines showed very good substantively for wool fibers with good coloring performance according to CIEL*a*b* system which characterized quantitative and qualitative coloring property.

Dyestuff 3 or isatin-3-phenylhydrazone bound to woolen textiles to a greater extent and greater intensity (minimum value of L). Dyestuff 2 or isatin-3-thiosemicarbazone linked to the minimum amount for textiles (the largest value of L). Although it must be noted that it is a lighter shade (yellow color) as opposed to the dyestuff 3 (red color).

REFERENCES

[1] R. Rondao, J. S. de Melo, F. Schaberle and G. Voss, *"Excited state characterization of a polymeric indigo"* Phys. Chem. Chem. Phys., vol. 14, pp. 1778–1783, 2012.

[2] M. Sarigul, P. Deveci, M. Kose, U. Arslan, H. T. Dagi and M. Kurtoglu, *"New tridentate azo–azomethines and their copper(II) complexes: Synthesis, solvent effect on tautomerism, electrochemical and biological studies"*, J. Mol. Struct., vol. 1096, pp. 64–73, 2015.

[3] S. Eskikanbur, K. Sayin, M. Kose, H. Zengin, V. McKee and M. Kurtoglu, *"Synthesis of two new azo-azomethines; spectral characterization, crystal structures, computational and fluorescence studies"*, J. Mol. Struct., vol. 1094, pp. 183–194, 2015.

[4] N. Kurtoglu, *"Synthesis, characterization, chelation with transition metal ions, and antibacterial and antifungal studies of the 4-[(E)--phenyldiazenyl]-2-[(E)-(phenylimino)methyl]phenol dye"*, J. Serb. Chem. Soc., vol. 74, pp. 917–926, 2009.

[5] I. Anis, M. Aslam, N. Afza, A. Hussain, F. S. Ahmad, L. Iqbal, M. Lateef, M. T. Hussain and T. H. Bokhari, *"A one-pot synthesis, characterization and pharmacological investigations: schiff bases"*, IJPC, vol. 2, pp. 73-77, 2012.

[6] A. Jarrahpour, D. Khalili, E. De Clercq, C. Salmi and J. M. Brunel, *"Synthesis, Antibacterial, antifungal and antiviral activity evaluation of some new bis-Schiff bases of isatin and their derivatives"*, Molecules, vol. 12, pp. 1720-1730, 2007.

[7] M. Sarigul, A. Sari, M. Kose, V. McKee, M. Elmastas, I. Demirtas and M. Kurtoglu, *"New bio-active azo-azomethine based Cu(II) complexes"*, Inorg. Chim. Acta, vol. 444, pp. 166–175, 2016.

[8] H. J. Vashi and K. R. Desai, *"Syntheses and dyeing performance of azo disperse dyes based on schiffs base system"*, Indian J. Fibre Text., vol. 21, pp. 225-227, 1996.