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Društvo za procesnu tehniku
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pri SMEITS-u



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PREDGOVOR

Naučno-stručni odbor 33. Procesinga, posle obavljenih recenzija, prihvatio je za izlaganje i u program uvrstio ukupno 23 rada. Elektronski Zbornik radova (sa CIP i ISBN brojem) sadrži radove u celini, i slobodan je za preuzimanje na sajtu skupa. Pojedinačni radovi su objavljeni na sajtu www.izdanja.smeits.rs.

Apstrakti prihvaćenih radova objavljeni su u štampanom dvojezičnom zborniku zajedno sa programom Procesinga '20.

Jedan broj radova je izabran za objavljivanje u drugom broju časopisa „Procesna tehnika“ (M53) koji izlazi krajem 2020 godine.

Programski koncept Kongresa o procesnoj industriji – Procesinga obuhvata bazne oblasti procesne tehnike:

Procesne tehnologije (naftna, hemijska i petrohemijska industrija, crna i obojena metalurgija, industrija nemetalnih minerala, industrija plastičnih materijala, industrija građevinskih materijala, industrija vatrostalnih i termoizolacionih materijala, industrija papira i celuloze, farmaceutska industrija, prehrambena industrija, proizvodnja alkoholnih i bezalkoholnih pića, proizvodnja stočne hrane, proizvodnja veštačkih đubriva i agrohemijska, duvanska industrija, tekstilna industrija, gumarska industrija);

Projektovanje, izgradnja, eksploatacija i održavanje procesnih postrojenja (projektovanje procesnih postrojenja, izgradnja procesnih postrojenja, puštanje u rad, eksploatacija i održavanje procesnih postrojenja, ispitivanje funkcionalnosti i bezbednosti, sistemi automatskog upravljanja i kontrole procesa, informacione tehnologije u projektovanju i upravljanju procesnim postrojenjima);

Osnovne i pomoćne operacije, aparati i mašine u procesnoj industriji (toplotne, difuzione, mehaničke, hidromehaničke, biohemijske i hemijske operacije, pomoćne operacije i opremu, cevovodi i armature);

Merenja i upravljanje u procesnoj industriji (osnovne merne veličine u procesnoj industriji; zakonska regulativa i standardizacija iz oblasti merenja i ispitivanja, merenja u cilju kontrole, vođenja i automatskog upravljanja procesom, merni sistemi, organizacija složenih sistema merenja sa akvizicijom podataka).

Razvoj novih tehnologija kao i potrebe povećanja energetske efikasnosti, uštede energije, korišćenja obnovljivih izvora energije, tretmana svih vrsta otpadnih materija, uvođenja koncepta cirkularne ekonomije i zaštite životne sredine u procesnoj industriji, nametnulo je da su ove teme poslednjih godina sve više uključene i u program Procesinga.

Program Procesinga '20 obuhvatio je 11 tematskih oblasti: 1. Procesne tehnologije; 2. Projektovanje, izgradnja, eksploatacija i održavanje procesnih postrojenja; 3. Osnovne i pomoćne operacije, aparati i mašine u procesnoj industriji; 4. Energetska efikasnost u industriji; 5. Inženjerstvo životne sredine i održivi razvoj u procesnoj industriji; 6. Procesi i postrojenja u pripremi i prečišćavanju vode u procesnoj industriji; 7. Sušenje i sušare; 8. Gasna tehnika; 9. Modelovanje i optimizacija procesnih i termoenergetskih postrojenja; 10. Merenja i upravljanje u procesnoj industriji; 11. Tehnička regulativa, standardizacija i sistem kvaliteta.

Procesing '20 organizovalo je Društvo za procesnu tehniku pri SMEITS-u, a u Naučno-stručnom i Organizacionom odboru prisutni su predstavnici mašinskih i tehnoloških fakulteta iz Srbije. Međunarodni karakter kongresa karakteriše učešće u Naučno-stručnom odboru predstavnika drugih zemalja, kao i jedan broj prijavljenih radova iz inostranstva.

U Beogradu
oktobra 2020.

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POBOLJŠANJE PROCESA BOJENJA IZ PERSPEKTIVE ARILAZO PIRIDONSKIH BOJA

TOWARDS ENHANCED DYEING PROCESS: ARYLAZO PYRIDONE DYES

Aleksandra MAŠULOVIĆ*¹, Julijana TADIĆ¹, Luka MATOVIĆ¹, Jelena LAĐAREVIĆ²,
Aleksandra IVANOVSKA², Mirjana KOSTIĆ², Dušan MIJIN²

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Kao grupa disperznih azo boja, azo piridonske boje nerijetko se koriste u svrhe bojenja tekstila. Najčešće, sintetska vlakna, boje se u kiseloj sredini, pri čemu povećanje pH vrijednosti može dovesti do narušavanja strukture boje, i lošijeg obojenja tkanine. Shodno ovome, potrebe tekstilne industrije zahvtaju nove boje za bojenje kako sintetskih tako i prirodnih vlakana. Novosintetisane disperzne azo boje zadovoljavaju ove potrebe dajući intenzivna obojenja pri čemu je struktura stabilna i u alkalnoj sredini. Arhitektura boja počiva na tri prstena od kojih dva, piridinium- i fenil-, sadrže elektron akceptorske grupe. Tkanine, kako sintetske, tako i prirodne, obojene ovim bojama imaju bolju UV zaštitu. Boje su okarakterisane tradicionalnim analitičkim metodama, nakon čega su obojene tkanine različitog hemijskog sastava. Obojenje je ispitano CIElab testom, a faktor zaštite od UV zračenja UPF-om testom. Na osnovu dobijenih rezultata može se zaključiti da se sintetisane boje uspješno mogu primjenjivati za bojenje vune na pH 8.5, kao i da njihovom primjenom tekstil dobija dvostruko veću zaštitu od UV zračenja.

Ključne reči: Azo boje; tekstil; UPF; UV

Azo pyridone dyes as a group of synthetic disperse azo dyes are commonly used in textile fiber dyeing. Traditionally, synthetic fibers are tinted in an acid environment, whereas their stability has been ruptured by base addition. Therefore demands of the textile industry have not been fully satisfied and the need for new dyes has aroused bearing the necessity for natural fiber dyeing. New disperse azo dyes have been designed satisfying nowadays industrial demands yielding tinctorially strong, alkali stable dyes, exhibiting good all-around properties. The invention begins on the architecture of two novel dyes bearing acceptors on the different scaffolds of the structure, pyridinium and phenyl core of the pyridone moiety. These dyes ameliorate the UV protection of natural, as well as synthetic fibers. The dyes are characterized by traditional analytical methods, prior to characterizing their ability for dyeing fibers of different chemical composition. Based on the obtained results, it can be concluded that the studied dyes can be successfully used for dyeing wool at pH 8.5. They also contributed to an increase in wool UV protection factor for about two times.

The tintorial strength has been established by CIElab and UV protection factor by UPF.

Key words: Azo dyes; textile; UV protection

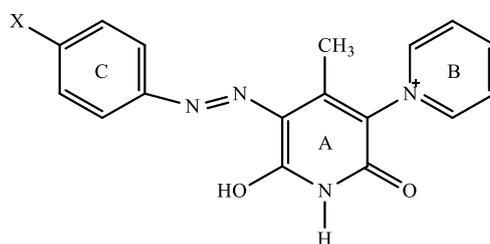
1 Azo dyes from a textile perspective

When it comes to industrial colorants azo dyes are considered to be the most important class due to their wide application from non-optical materials (NLO) [1], dye-sensitized solar cells (DSSC) [2], to cosmetics, medicines [3] and textile fiber dyeing [4-7]. Azo pyridone dyes have been studied extensively from a tautomeric point of view throughout years. It has been shown that pyridone based azo dyes adopt hydrazone tautomeric form when found in neutral and acidic medium, whereas deprotonated azo anion is to be found in alkaline conditions. These claims are supported by

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a broad range of queried UV-Vis, X-ray and NMR spectra, as well as quantum chemistry calculations [4]. This appears as an interesting fact from a structural point of view, but it represents a certain obstacle when it comes to the dyeing process, namely because of the presence of the alkali-unstable hydroxyl groups, wherein the result is that disperse dyes can only be used for dyeing under acidic conditions and the color is unstable and fades quickly under basic conditions. Most of the disperse azo dyes are water insoluble and used for synthetic fiber dyeing such as polyester, polyamides and acrylics [5]. Post modification of the molecule represents a challenge seeking the usage of complex synthetic routes and economic excesses. Textile industries are in the demand of the more soluble, cleaner and more stable products. Hence, the design and development of such azo dyes still represent an active research area [4-7].

In this work two arylazo pyridone dyes (Figure 1.) are synthesized in order to acquire water soluble dyes which can be used for dyeing natural fibers in a basic environment. The introduction of the pyridinium scaffold in the pyridone (B) backbone is in aspiration to increase the solubility of resulting dyes and facilitate processes of purification and characterization.



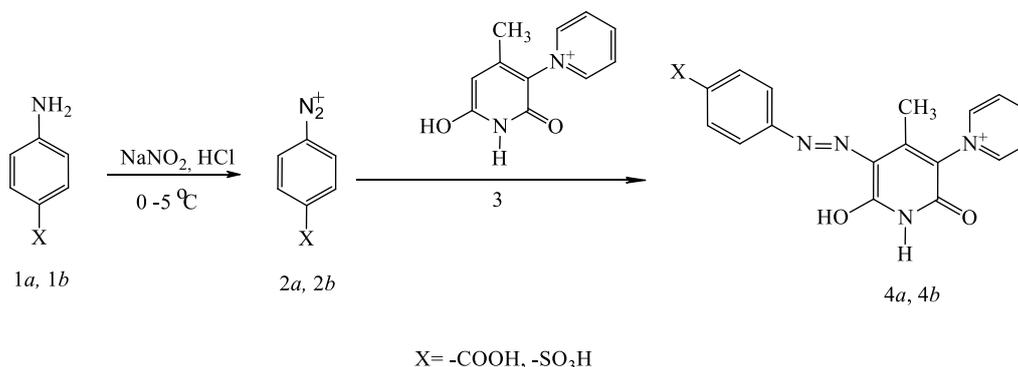
X = -COOH (a), -SO₃H (b)

Figure 1. The structure of 5-(4-substituted phenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridones

2 Experimental part

2.1 Synthesis of azo dyes

Azo dyes are synthesized by a common procedure of diazo coupling previously found in literature [8]. Equimolar amounts of diazonium salt (2a, 2b) obtained from substituted anilines (1a, 1b) were added to previously prepared pyridone (3). Diazonium salts dissolved hydrochloric acid were stirred with sodium nitrite for an hour at the temperature of -5°C to obtain diazonium salt. The entire synthetic route at -5°C was performed as depicted on Scheme 1. Dyes are further purified whereas orange (4a) and yellow (4b) powders are obtained.



X = -COOH, -SO₃H

Scheme 1. Synthetic route of 5-(4-substituted-phenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridones

2.1.1 Dyes characterization

1. 5-(4-carboxy-phenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone **4a**: Orange powder; t.t. 332-334 °C; ATR-FTIR (v/cm⁻¹): 3350 (N-H hydrazone), 3122 (N-H pyridone), 1665, 1628, 1603 (C=O);

2. *5-(4-carboxy-phenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone 4b*: Yellow powder; t.t. 325-331 °C; ATR-FTIR (ν/cm^{-1}): 3337 (N–H hydrazone), 3177 (OH), 3112 (N–H pyridone), 1698, 1653 (C=O), 1407 (SO₃H);

2.2 Dyeing procedure and characterization of dyed fabrics

The Multifiber Adjacent Fabric TV Style 42 comprising wide bands of: diacetate (CA), bleached cotton (CO), polyamide (Nylon 6.6) (PA), polyester (PES), acrylic (PAN) and wool (WO) was used as a substrate for dyeing. In order to assess the behavior of dyes marked as a and b (Figure 1) towards each component of the multifiber substrate, the dyeing of the samples was performed under pH 8.5, at 80 °C for 90 min under constant shaking. The dyebaths were prepared by dissolving the corresponding amount of each dye (3% o.w.f., i.e. on the weight of fiber) in distilled water. After dyeing, the samples were thoroughly washed with warm distilled water and dried at room temperature.

In order to determine the colorimetric properties of each component of the dyed multifiber sample, the fabrics' color coordinates (L , a^* , b^*) were measured in the CIELab color space. In order to determine the colorimetric properties of each component of the dyed multifiber sample, the colorimetric measurements (under illuminant D65 using the 10° standard observer) were performed using SF300 (Datacolor, USA) reflectance spectrophotometer with ultra-small area view (USAV). The UV protection factor (UPF) of dyed fabrics was derived from the measurement of transmittance of ultraviolet radiation through fabrics (SRPS EN 13758-1). UPF value will be derived from the measurement of transmittance of ultraviolet radiation through fabrics (according to ATCC 183-2004 standard).

3 Characterized dyes and dyed fabric access

3.1 UV-Vis characterization

UV-Vis spectra of *4a* and *4b* were recorded in methanol. Acid-base equilibrium was discussed in order to detect tautomeric forms, as it is stated that hydrazone (Figure 2a) tautomer is favored in a neutral and acid environment, whereas azo anionic (Figure 2b) form appears to be dominant in basic conditions.

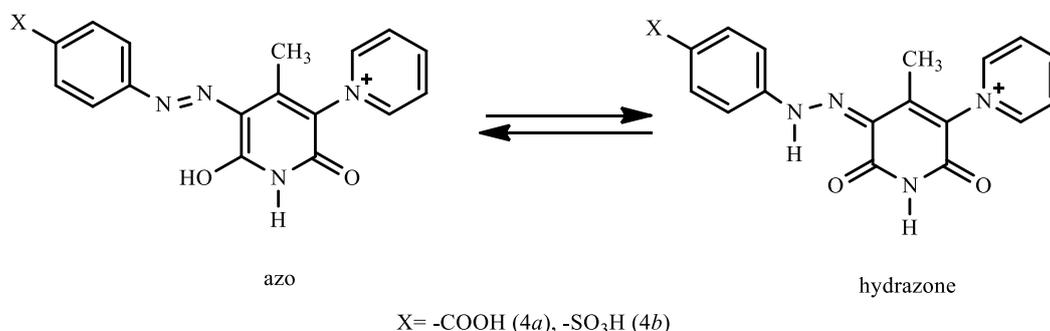


Figure 2. Azo-hydrazone tautomerism of the investigated dyes.

Absorption spectra in Figure 3. as well as ATR-FTIR show the presence of only one tautomeric form.

As seen from the spectra, the shift of the absorption maxima is negligible. Compound *4a* in methanol has absorption maxima at 427 nm, whereas when acid or base are added maxima shifts hipsochromically to 422 and 421 respectively. There is a slight shoulder present when the base is added, but overall we can observe that compound *4a* is not pH sensitive. Moreover, the presence of azo anionic form in this compound is insignificant.

UV-Vis spectra in methanol are recorded for compound *4b* and depicted in Figure 4.

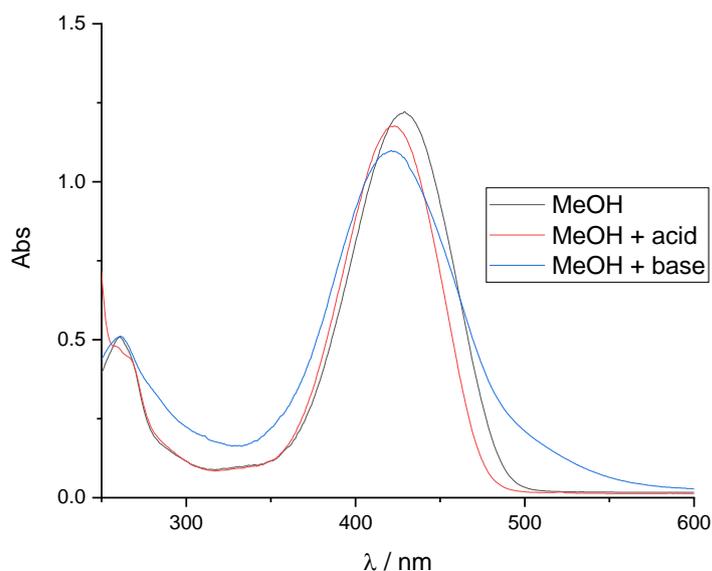


Figure 3. UV-Vis spectra of 5-(4-carboxyphenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone

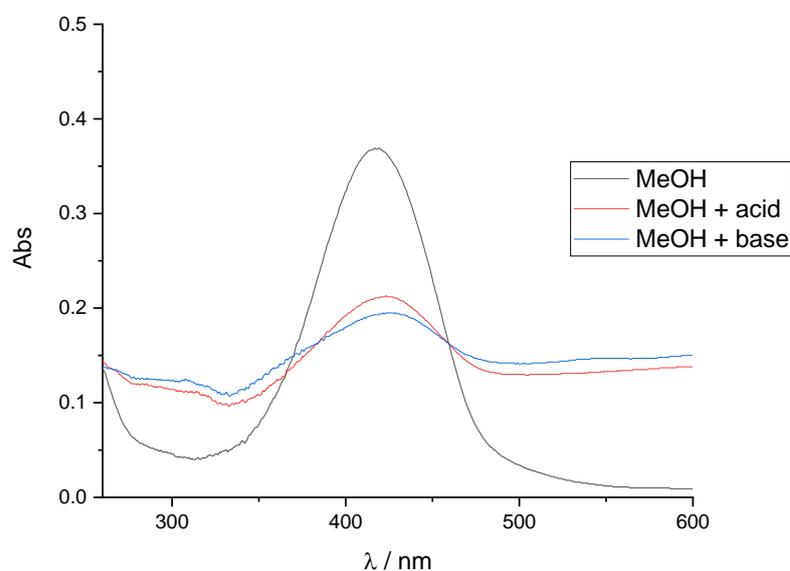


Figure 4. UV-Vis spectra of 5-(4-sulfoxyphenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone

The absorption maxima of dye *b* in methanol appears at 425 nm, while when acid or base were added the shift of the maxima is hypochromic to 423 nm and 418 nm respectively. When compared to spectra of the *4a*, we can state that *4b* is pH insensitive as well. Moreover, *4b* dominantly exists in hydrazone tautomeric form as concluded from Figure 4.

3.2 Characterization of dyed fabrics

According to the results presented in Figure 5, it is evident that both studied dyes are selective for wool, i.e. they can be successfully utilized for dyeing wool fabric at pH 8.5. The fabrics' CIELab color coordinates are listed in Table 1.

Wool fabric dyed with dye *4a* has almost the same lightness (*L*) but more red (higher value of parameter *a**) and more yellow (higher value of parameter *b**) shade than wool fabric dyed with dye *4b*. It has to be emphasized that dyed wool fabrics have about two times higher UPF value than undyed ones. UPF values for wool are 2.32, while when dyed with dyes *4a* and *4b* values are 4.11 respectively.

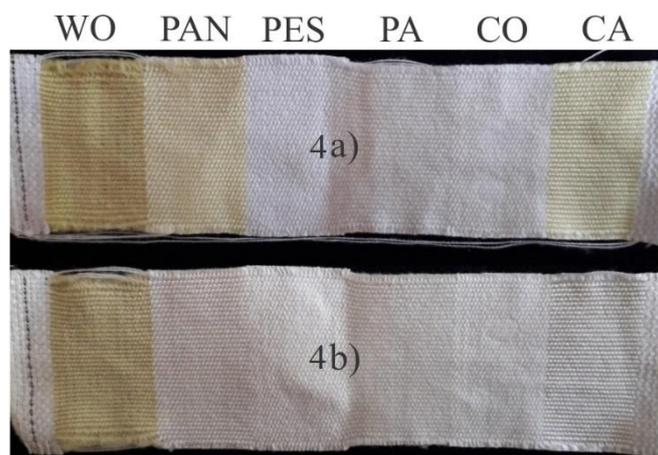


Figure 5. Multifiber adjacent fabric dyed with dye: 4a a and 4b

Table 1 CIELab color coordinates of wool fabric

	<i>L</i>	<i>a</i> *	<i>b</i> *
Wool dyed with dye a	85.43	-3.6	50
Wool dyed with dye b	86.55	-5.7	38.7

4 Conclusion

We can state that 5-(4-sulfoxyphenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone and 5-(4-carboxyphenyl)-6-hydroxy-4-methyl-3-pyridinium-2-pyridone are stable in alkaline environment based on UV-Vis spectra if dyes and their tautomer forms barely transform due to the pH adjusting. Moreover, the synthesized dyes probably exist in only one – hydrazone tautomeric form. Based on the obtained results, it can be concluded that the studied dyes can be successfully used for dyeing wool at pH 8.5. They also contributed to an increase in wool UV protection factor for about two times.

5 Acknowledgement

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