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Synthesis and Characterization of Some Azo-heterocycles Incorporating pyrazolopyridine Moiety as Disperse Dyes

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IN THIS research paper, we used 3-aminopyrazolo[3,4-*b*]pyridine as key intermediate for the synthesis of some pyrazolopyridine azo compounds. The key starting fused heterocyclic 3-aminopyrazolo[3,4-*b*]pyridine is prepared by the reaction of 2-chloro-4,6-dimethylnicotinonitrile with hydrazine hydrate. The azo-coupling reaction of the pyrazolopyridinyl diazonium salt with a variety of coupling agents such as *β*-naphthylamine, *β*-naphthol, thiazole, and aniline derivatives afforded the corresponding arylazopyrazolo[3,4-*b*] pyridine derivatives in relatively high yield. The chemical structures were established using FT-IR, elemental analysis (C, H, N), and ^{1}H-NMR, ^{13}C-NMR. The prepared arylazopyrazolo[3,4-*b*] pyridine disperse dyes were applied to dye polyester garments to provide good color fastness properties.

Keywords: Aminopyrazolo[3,4-*b*]pyridine, Azo-coupling, Arylazopyrazolo[3,4-*b*]pyridine, Disperse dyes. Dyeing polyester.

Introduction

Nitrogen-containing heterocyclic compounds are a valuable class of materials because of their interesting biological characteristics as pharmacophores of extensive historical significance [1-5]. 1H-pyrazolo[3,4-*b*] pyridines especially comprise an attractive group of compounds due to their considerable and varied biological and pharmacological applications such as anti-malarial [6], anti-proliferative [7], anti-bacterial [8], kinase-inhibitor [9], cardiovascular [10], anti-viral [11], and anti-leishmanial medications [12]. Fused heterocycles such as pyrazolopyridines and pyrazolopyrimidines are well known to own a broad spectrum of biological activity. Specifically, pyrazolopyridines demonstrate anti-tubercular and anti-anxiety medical properties [13]. Furthermore, such fused heterocyclic compounds have been used as dyes for textile coloration [2, 5]. The biological activity received recently a very high attention from researcher due to the increased anti-microbial resistance developed by significant microorganisms [14]. In addition, emerging and reemerging microbial infections diseases still lead to global death and disability [15]. In view of the aforementioned findings and as continuation of our effort to discover new candidates that may be of importance in designing novel effective and selective anti-microbial material drugs, we report herein the preparation of some arylazo heterocycles incorporating pyrazolopyridine moiety starting from 3-aminopyrazolo [3,4-b] pyridine in order to investigate their spectral characteristics. Such water-insoluble and small molecular arylazopyrazolopyridine compounds can be utilized as disperse dyes able to potentially dye hydrophobic fabrics.

Experimental

Materials and methods

All melting points (uncorrected) are in degree centigrade and were determined on Gallenkamp electric melting point apparatus. Elemental analyses were carried out in the microanalytical unit, Faculty of Science, University of Mansoura IR spectra were recorded (KBr) with on a Mattson 5000 FTIR spectrometer. ^{1}H NMR spectra were measured on a Bruker WP 300 in deuterated DMSO as solvent, using TMS as an internal standard at 295°K. The color strength was tested using an UltraScan PRO Spectrophotometer (light source D65/10° observer). Solvents used in this study were obtained from Fluka and Aldrich. All reactions were monitored by thin layer chromatography (TLC) using Merck aluminum plates pre-coated with silica gel PF254; 20-20, 0.25

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mm, and detected by visualization of the plate under UV lamp (254 or 365 nm). Compounds were purified through recrystallization or using flash column chromatography which was performed on Scharlau silica gel, packed by the slurry method. Scoured and bleached 100% polyester fabrics were supplied by El-Mahalla El-Kobra Company, El-Mahalla, Egypt.

Dyeing procedure

The dyeing process was performed on polyester fibers via high temperature dyeing method by dispersing proper amount of dye (2% shade) in 1 mL DMF and then added dropwise with stirring to the dye-bath (liquor ratio 50:1) containing sodium lignin sulfonate as dispersing agent (ratio of dispersing agent to dye is 1:1). The pH of the dye-bath is kept at ~ 4.85 employing aqueous acetic acid and the wetted-out polyester samples are added. The dyeing procedure was accomplished by increasing the dye-bath temperature to 130°C for 180 minutes under pressure in an infra-red dyeing machine. After dyeing, the polyester samples are rinsed followed by surface reduction clearing in a solution composed of ((2 g NaOH + 2 g sodium hydrosulphite)/L, and soaped with 2% nonionic detergent) for 45 minutes at 80°C. Wash well in cold water and neutralize with 1 g/L acetic acid for 5 minutes at 40°C, followed by rinsing under tap water and dried at ambient temperature [16].

Color strength and fastness measurements

Fastness to wash, rubbing, perspiration and light were tested according to standard dyeing ISO methods [17-20]. The color strength of the dyed polyester samples expressed as K/S was assessed by the high reflectance method [21].

Synthetic approaches

Synthesis of 3- amino- 4,6- dimethyl- 1H-pyrazolo [3,4-b] pyridine 1

The key starting material 3- aminopyrazolo [3,4-b]pyridine 1 could be prepared by the reaction of 2-chloro-4,6-dimethylnicotinonitrile with hydrazine hydrate. Its versatility derives from the easy access of 2-chloro-4,6-dimethylnicotinonitrile from condensation reaction of acetyl acetone and cyanoacetamide [22-25]. m.p. 276-277°C, lit. m.p. 272-273°C. IR (ν/cm^{-1}): 3388, 32387, 3188 (NH_2, NH) and 1630 cm^{-1} (C=N). ^{1}H NMR (DMSO-d_6): δ/ppm 2.40 (s, 3H, CH_3), 2.50 (s, 3H, CH_3), 5.05 (s, 2H, NH_2), 6.60 (s, 1H, pyridine C-5), 11.65 (s, 1H, NH). ^{13}C NMR (DMSO-d_6): δ/ppm 18.27, 24.04, 104.16, 115.48, 142.21, 148.14, 152.85, 157.41.

Synthesis of 5- (2- (4,6- dimethyl -1H -pyrazolo [3,4-b]pyridin-3-yl) diazenyl)- N-substituted-4-phenylthiazol-2-amine 4

Sodium nitrite solution (0.69 g, 10 mmol) was added dropwise with stirring during 15 min. to an ice-cooled sample of compound 1 (1.62 g, 10 mmol) at 0-5°C, dissolved in concentrated HCl (3 ml) and water (2 ml). At the end of addition, the stirring was continued for further 15 min. To a cold solution of phenylthiazoleamine derivatives 3a-c (0.01 mol of each) in acetic acid (30 ml), the diazonium salt solution 2 (0.01 mol) was added dropwise with stirring. Stirring was continued for 2 hours. The products so formed were collected by filtration and washed with water followed by cold ethanol. The isolated compounds were re-crystallized from ethanol to give the corresponding heteroarylazopyrazolopyridine 4a-c.

Synthesis of 1-(4,6-Dimethyl-1H-pyrazolo[3,4-b]-pyridin-3-ylazo)-2-(2-aminonaphthalen-6-ylsulfonyl)ethanol 5

m.p. > 300°C. Yield = 82%. IR (ν/cm^{-1}): 3369 (NH_2), 3241 (NH), 1625 cm^{-1} (C=N), 1136 (–SO_2).^{1}H NMR (DMSO-d_6): δ/ppm = 2.55 (s, 3H, CH_3), 2.66 (s, 3H, CH_3), 3.46-3.51 (t, 2H, –CH_2OH), 3.68-3.71 (t, 2H, –SO_2CH_2–), 4.92 (1H, s, OH), 6.97 (s, 1H, pyridine C_5-H), 7.31-8.86 (m, 5H, Ar-H), 8.89 (s, 2H, NH_2), 13.65 (s,1H NH). Analysis: $C_{20}H_{20}N_6O_3S$ (424.48); Calcd. %: C, 56.59; H, 4.75; N, 19.80; S, 7.55; Found %: C, 56.32; H, 4.96; N, 19.64; S, 7.76

Synthesis of 3- (4,6- Dimethyl- 1H- pyrazolo [3,4-b]-pyridin-3-ylazo)-2-naphthol 6

m.p. > 300°C. Yield = 88%. IR (ν/cm^{-1}): 3538 cm^{-1} (OH), 3280 cm^{-1} (NH), 1612 cm^{-1} (C=N). ^{1}H NMR (DMSO-d_6): δ/ppm 2.55 (s, 3H, CH_3), 2.68 (s, 3H, CH_3), 6.97 (s, 1H, pyridine C_5-H), 6.72-8.57 (m, 6H, naphthyl-H), 13.62 (s, 1H, NH). Analysis: $C_{18}H_{15}N_5O$ (317.34); Calcd. %: C, 68.13; H, 4.76; N, 22.07; Found %: C, 67.33; H, 5.05; N, 22.48

Synthesis of N,N-Diethyl-4-(4,6-dimethyl-1H-pyrazolo[3,4-b]pyridin- 3-ylazo)aniline 7

m.p. > 300°C. Yield = 88%. IR (ν/cm^{-1}): 3130 cm^{-1} (NH), 2970, 2928, 2862, 1430, 1408, 1351 (C-H stretching and bending), 1601 cm^{-1} (C=N). ^{1}H NMR (DMSO-d_6): δ/ppm 1.14-1.21 (t, 6H, 2 × −CH_2CH_3), 2.54 (s, 3H, CH_3), 2.67 (s, 3H, CH_3), 3.35-3.60 (q, 4H, 2 × −CH_2CH_3), 6.97 (s, 1H, pyridine C_5-H), 6.79 (d, 2H, J 8.6, Ar-H), 7.80 (d, 2H, Ar-H), 13.65 (s,1H NH). Analysis: $C_{18}H_{22}N_6$ (322.41); Calcd. %: C, 67.06; H, 6.88; N, 26.07; Found %: C, 67.81; H, 7.02; N, 25.48

Synthesis of 3-(4,6-dimethyl-1H-pyrazolo[3,4-b] pyridin-3-ylazo)-2-naphylamine 8

m.p. > 300°C. Yield = 88%. IR (ν/cm^{-1}): 3395, 3315 (NH_2), 3144 (NH), 1601 cm^{-1} (C=N). ^{1}H NMR (DMSO-d_6): δ/ppm = 2.55 (s, 3H, CH_3), 2.68 (s, 3H, CH_3), 6.97 (s, 1H, pyridine C_5-H), 7.13-8.65 (m, 6H, naphthyl-H), 8.85 (s, 2H, NH_2), 13.62 (s, 1H, NH). Analysis: $C_{18}H_{16}N_6$ (316.36); Calcd. %: C, 68.34; H, 5.10; N, 26.56; Found %: C, 67.50; H, 4.91; N, 25.81

Synthesis of N- ethyl -N- benzyl -4-(4,6-dimethyl-1H-pyrazolo[3,4-b]pyridin-3-ylazo)aniline 9

m.p. > 300°C. Yield = 88%. IR (ν/cm^{-1}): 3112 (NH), 2967, 2928, 2871, 1425, 1393, 1352 (C-H stretching and bending), 1601 cm^{-1} (C=N). ^{1}H NMR (DMSO-d_6): δ/ppm 1.15-1.22 (t, 3H, $-CH_2CH_3$), 2.53 (s, 3H, CH_3), 2.67 (s, 3H, CH_3), 3.35-3.61 (q, 2H, $-CH_2CH_3$), 4.68 (s, 2H, $-CH_2Ph$), 6.97 (s, 1H, pyridine C_5-H), 6.81-7.80 (m, 9H, Ar-H), 13.70 (s, 1H, NH). Analysis: $C_{18}H_{16}N_6$ (316.36); Calcd. %: C, 71.85; H, 6.29; N, 21.86; Found %: C, 70.15; H, 5.96; N, 20.65

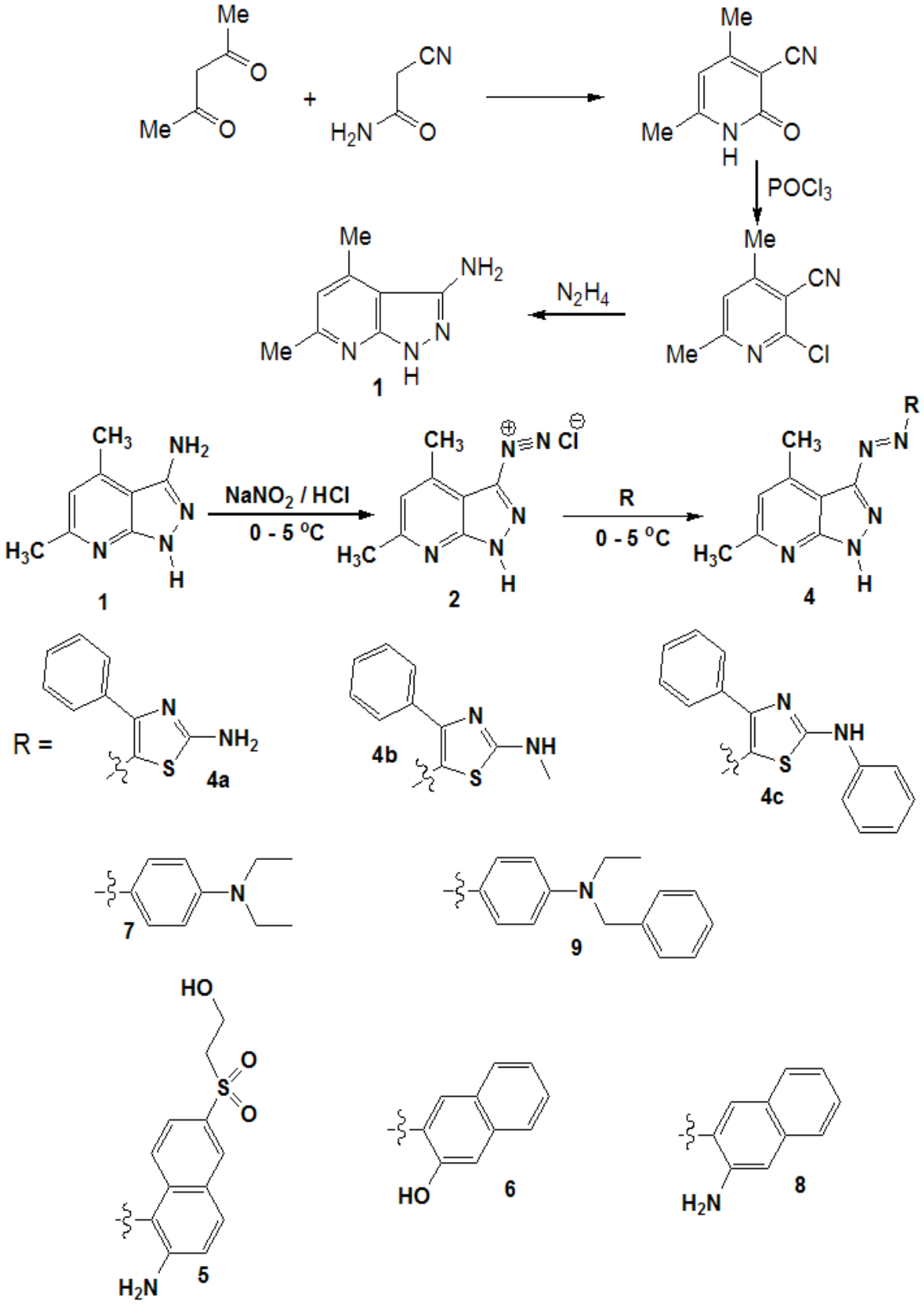
Results and Discussion

Chemistry of synthesis and characterization

The increasing importance of 3-aminopyrazolo [3,4-b]pyridine as an adaptable intermediate for the preparation of numerous fused heterocyclic rings as drugs for various pharmaceutical purposes led to the ongoing growth of novel uncomplicated procedures for their production [6-12]. In continuation of the preparation of nitrogen-containing heterocyclic structures, it was found that diazotized heterocyclic-containing amine can be considered as an excellent building unit for the production of the intended compound. Thus, azo-coupling of the pyrazolopyridinyl diazonium salt 2 with a variety of coupling agents including β-naphthylamine, β-naphthol, thiazole, and aniline derivatives afforded the corresponding arylazopyrazolo[3,4-b]pyridines 5-9. The chemical structure of compound 5 was demonstrated accordring to its analytical and spectral data. The FT-IR spectrum displayed stretching frequencies of two peaks at 3395, 3315 cm^{-1} assigned to NH_2 group, 3144 cm^{-1} for NH, 1601 cm^{-1} for C=N. The ^{1}H-NMR spectrum in deuterated DMSO displayed two singlet peaks at 2.55 and 2.86 ppm assigned to the two methyl protons, singlet peak at 6.97 ppm for pyridine C5-H proton and multiplet peak at 7.13-8.65 ppm for the aromatic ring protons. By considering the above data in addition to (C, H, N) elemental analysis, this reaction product could be formulated as the 4,6-dimethyl -1H- pyrazolo [3,4-b]pyridine derivative while azo coupling of the pyrazolopyridinyl diazonium salt 2 with another coupling agent such as N,N-diethylamine gave N,N- Diethyl -4-(4,6- dimethyl -1H-pyrazolo[3,4-b]pyridin-3-ylazo)aniline 7. The chemical structure of arylazopyrazolo[3,4-b]pyridine 7 is proved by both of analytical and spectral techniques. FT-IR demonstrated peak with one spike 3130 cm^{-1} (NH), 2970, 2928, 2862, 1430, 1408, 1351 (C-H stretching and bending), 1601 cm^{-1} (C=N). The ^{1}H-NMR spectrum in deuterated DMSO as a solvent showed two triplet peaks at 1.14,1.12 ppm and two quartet peaks at 3.35,3.6 ppm for two ethyl groups. The reaction of the pyrazolopyridinyl diazonium salt 2 with N-ethyl-N-benzylpyrazolo-pyridine affords N-ethyl -N- benzyl-4-(4,6-dimethyl-1H-pyrazolo[3,4-b]pyridin-3-ylazo) aniline 9 is assigned by its analytical and spectral data. ^{1}H-NMR in deuterated DMSO shows a triplet at 1.15-1.22 ppm for 3H, and 3.35-3.61 ppm for 2H of ($-CH_2CH_3$), and two singlet peaks at 2.53 and 2.67 ppm for two methyl groups on pyridine ring , a quartet peak for ethyl group at 3.35-3.61 ppm, singlet peak at 4.68 ppm for $-CH_2Ph$ the higher value of chemical shift due to withdrawal effect of phenyl ring), also aromatic protons appears at 6.97 ppm singlet 1H, of pyridine C_5-H, multiplet at 6.81-7.80 ppm for 9H, Ar-H, finally singlet at 13.70 ppm for 1H of (NH). The synthetic procedures to achieve the target pyrazolo[3,4-b]pyridine derivatives are described in Scheme 1.

Dyeing performance

Our azo dyes can be considered as disperse-type dyes able to potentially dye hydrophobic polyester fibers because they can be recognized as non-ionic small molecules with low solubility in water. They are applied to dye polyester fibers at 130˚C and 2% shade utilizing the high temperature pressure dyeing technique [16]. The color shades varied from orange-red to red. All dyestuffs 4a-c and 5-9 displayed good color strength and acceptable fastness properties (Table 1).



Scheme 1. Synthetic approach of pyrazolo[3,4-*b*]pyridine.

TABLE 1. Color strength and fastness properties of dyes 4a-c and 5-9 on polyester garments.

Dye No.	λ_{max} (nm)	K/S	Shade	Wash		Perspiration				Rubbing		Light**
						Acidic		Basic		Dry	Wet	
				Alt.*	St.*	Alt.*	St.*	Alt.*	St.*			
4a	481	3.60	Red	3-4	4	4	4	4	4	4	4	6
4b	477	4.29	Red	4	4-5	4	4	4	4-5	4	4	5-6
4c	470	4.83	Red	3-4	4	4	4	4	4	4	4	5-6
5	478	7.11	Orange-red	4	4-5	3-4	3	3-4	4	4	3-4	5-6
6	465	3.89	Orange-red	3-4	3-4	4	4	3-4	4	4	4	6-7
7	483	4.63	Orange-red	4	4	3-4	4-5	4	4	4	3-4	6
8	496	6.47	Orange-red	4	4	4	4	4	4	4	3-4	5-6
9	489	4.26	Orange-red	4	4	4	3-4	4	4	4	4	6-7

*Alt. = alteration in color; St. = staining on cotton.

Conclusion

In conclusion, we could show that a series of azo-pyrazolopyridine disperse dyestuffs have been synthesized. The key starting fused heterocyclic compound 3-aminopyrazolo[3,4-*b*] pyridine is prepared by the reaction of 2-chloro-3,6-dimethylnicotinonitrile with hydrazine hydrate. The azo-coupling reaction of the pyrazolopyridinyl diazonium salt afforded the corresponding arylazopyrazolo[3,4-*b*]pyridine derivatives in relatively high yield. The produced arylazopyrazolo[3,4-*b*] pyridine dyestuffs were applied on polyester samples via high temperature pressure dyeing technique to provide solid shades with acceptable color constancy and deepness. The shades occurred between orange-red and red. The color fastness properties displayed mostly good fastness against light, rubbing, perspiration and washing.

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تحضير وتوصيف بعض مركبات الازو الحلقية الغير متجانسة التي تحتوي علي وحدة بيرازولوبيريدين كصبغات منتشرة

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يهدف هذا البحث الي تحضير مركبات الاريل ازو بيرازولوبيريدين باستخدام مركبات -3امينو بيرازولوبيريدين ودراسة تطبيق هذه المركبات الناتجة كصبغات منتشرة علي اقمشة البولي استر. تم تحضير -3امينو بيرازولوبيريدين من خلال تفاعل -2كلورو-4،6-ديميثيل نيكوتينونتريل مع هيدرات الهيدرازين. يمكن الحصول علي مركبات الاريل ازو بيرازولوبيريدين عند اجراء تفاعل الأزو بين ملح ديازونيوم البيرازولوبيريدين مع مشتقات اقتران متنوعة مثل بيتا-نفثيل امين، بيتا-نفثول، ثيازول، ومشتقات الأنيلين. وتم اثبات التركيب الكيميائي للأصباغ التي تم تحضيرها من خلال التحليل العنصري (كربون، هيدروجين، نيتروجين)، الرنين النووي المغناطيسي لنظائر الكربون والهيدروجين، والاشعة تحت الحمراء. تم تطبيق ملونات الاريل ازو بيرازولوبيريدين المنتجة لصبغ اقمشة البوليستر لتعطي خصائص ثبات جيدة للغسيل، العرق، الاحتكاك والضوء.