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Synthesis and Characterization of Novel Azo Disperse Dyes Containing a-amino Phosphonate and Their Dyeing Performance on Polyester Fabric

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THREE novel azo disperse dyes were designed, synthesized, and characterized by utilizing of α-aminophosphonates chromophores. The target compounds were prepared by employing of benzidine as amine and different heterocyclic formyl as aldehyde, and triphenylphosphite $P(OC_6H_5)_3$ that gives a-aminophosphonate in one position and free amino group in the other position, which was then coupled with β–naphthol to give the target products azo disperse dyes. The prepared dyes were structurally confirmed by IR, ^{1}HNMR and mass spectral data and applied to polyester fabric as azo disperse dyes and all fastness properties for the dyed samples were measured.

Keywords: α-aminophosphonates, Azo disperses dyes, Benzidine and polyester fabric.

Introduction

Polyester fabrics are the most demanded fabrics in the textile industry and disperse dyes are most important class of dyes for dyeing such fabrics because of its brilliance color, wide color range, stable fastness properties, environmental and economic reasons as well[1].

Organophosphorus compounds are important substrates and exhibit a wide range of biological activities. Organophosphorus intermediates are involved in many areas of agricultural, medicinal chemistry, and industrial applications due to their biological and physical properties[2-3]. α-Functionalized phosphonic acids are important intermediates for synthetic intermediates and preparation of many compounds as well [4-6].

Among α-functional phosphonic acids, α-aminophosphonic acids, and α-amino phosphonate are supreme important compounds which provides a diversity of important and preferable properties. A considerable number of α-amino phosphonate derivatives are known to be bioactive, they display an antiviral, antibacterial, antifungal, antimicrobial and antitumor activity [7-16].

In addition, the Organophosphorus compounds are very important precursors for preparation of fused heterocyclic compounds, Organophosphorus precursors owing great biological and pharmacological activities [17-20], which could be used as intermediates in the dyestuff industry [1, 21-23].

We also focused on whether bifunctional group like α-amino phosphonate located on the spacer of the azo dye moiety, would stimulate and expected to exhibit high acceptable and good biological resistance, in order to get colored sterile and/or biological resisting textile which could be applied in various fields of life applications.

In current study we used a one–pot Kabachnik–Fields reaction of different aryl substituted pyrazolaldehydes, benzidine, triphenylphosphite, by using Lewis acid catalyst such as copper(II) triflate $Cu(OSO_2CF_3)_2$ in dichloromethane which facilitate the condensation reaction at room temperature. A reaction of diphenyl [benzidine]1-aminoheterocyclicphosphonate coupled with β

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-naphthol was done to prepare 2-Naphthol-azo–diphenyl [benzidine] amino heterocyclic phosphonate compounds. The prepared compounds as disperse dyes were used for the application of dyeing polyester fabric with complete testing of fastness properties.

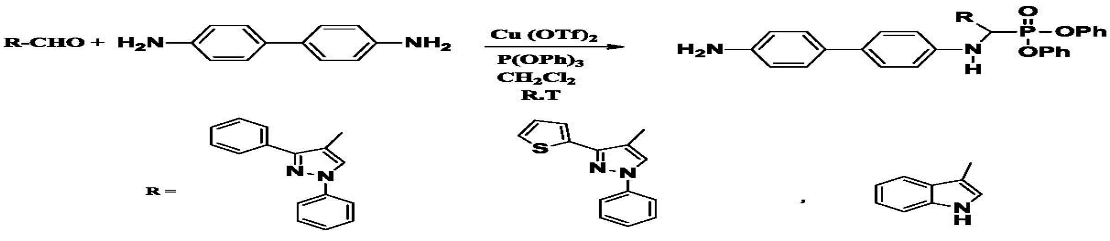
Results and Discussion

Synthesis

The Synthesis of novel azo disperses dyes containing α-aminophosphonates were achieved with high yields. While the synthetic routes of the target compounds were carried out by employing of benzidine as amine and aryl substituted pyrazolaldehydes, triphenylphosphite through copper (II) triflate $Cu(OSO_2CF_3)_2$ as lewis acid catalyst in dry dichloromethane at room temperature using one–pot Kabachnik–Fields reaction to afford α-aminophosphonate in one position and free amino group as shown in Scheme 1.

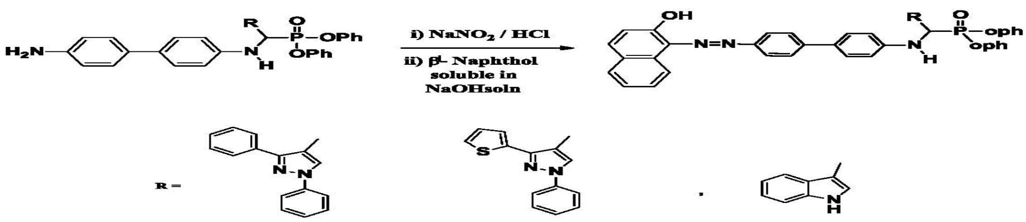
The free amino group was coupled with β–naphthol to produce different azo disperse dyes as shown in Scheme 2. The chemical structures, molecular formula, and the physical properties of synthesized dyes (D1-3) are shown in Table 1, and the prepared dyes were subjected for application of dyeing polyester fabric, and the fastness properties for the prepared dyes were also measured.

In addition, dyed polyester fabric colorimetric parameters were assessed by colorimetric measurements using reflectance spectrophotometer (Gretag Macbeth CE 7000a) (Table 2).



2a 2b 2c

Scheme 1



D1 D2 D3

Scheme 2

TABLE 1. Physical data for azo disperse dyes (D1-3).

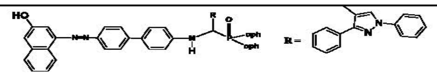
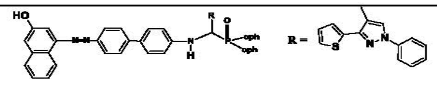
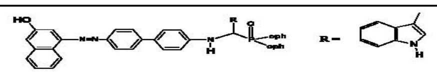
Compound	Molecular Structure	Mol. formula	Color (shade 2%)	Yield (%)
D1		$C_{44}H_{34}N_5O_4P$	Burgundy	95%
D2		$C_{48}H_{36}N_5O_4SP$	Rubine	95%
D3		$C_{43}H_{33}N_4O_4P$	Medium Pink	90%

TABLE 2. Color assessment of the dyes.

Dye	Absorption [λ_{max}(nm)]	K/S	L*	a*	b*
D1	505	30.03	60.15	43.12	36.06
D2	502	26.17	44.33	19.22	24.13
D3	385	18.33	45.14	22.45	19.03

K/S values given by the reflectance spectrometer are calculated at λ_{max} and are directly correlated with the dye concentration on the dye substrate according to the Kubelka–Munk equation [29]: K/S = (1-R) 2/2R, where K =absorbance coefficient, S = scattering coefficient, R = reflectance ratio.

Fastness tests for the prepared dyes (D1-3)

The prepared set of dyes (D1-3) was applied on dyeing of polyester fabric.

Fastness properties were measured according to ISO 105-X12(1987), ISO 105-co4 (1989), ISO105-EO4 (1989), ISO 105-BO2 (1988) corresponding to color fastness to rubbing, sublimation, washing, acid and alkali perspiration, respectively, for the fastness assessments, a rating scale of 1 (poor) to 5 (excellent) was used.

Table 3 shows that, all the dyed polyester fabric using D1-3 dyes exhibit good to very good washing fastness. On the other hand light fastness tests were carried out (According to the blue scale) and affected in the coupling component by the substituents. Mostly, better light fastness (good, 4-5 of the blue wool standard scale 1-8) 4 h irradiation to the dyed samples using a XENOTEST 1200 apparatus at a relative air humidity of 65% and 50˚C with duration 4 hours and the dyed samples showed very good light fastness. WhileTable 4 showed fairly good wet and very good dry rubbing fastness, the sublimation fastness for the dyed polyester fabric exhibit fairly good to poor sublimation resistance.

Fastness tests for acid and alkali perspiration were also performed and results are recorded in Table 5, it is clear from the results as it seen in Table 5 that all dyed polyester fabric are of fairly good to very good resistance acid and alkali perspiration properties.

TABLE 3. Washing and light fastness results for azo disperse compounds (D1-3).

Dye	Color (shade 2%)	Washing fastness		Light fastness
		ALT.	ST.	
D1	Purgundy	4	4	6
D2	Rubin	4	4-5	6
D3	Medium pink	4	4-5	6

ALT. = Alteration, ST. = staining on cotton

TABLE 4. Rubbing and sublimation fastness for azo disperse compounds (D1-3).

Dye	Color (shade 2%)	Rubbing fastness		sublimation fastness	
		Dry	wet	ALT.	ST.
D1	Purgundy	4-5	3-4	3-4	4
D2	Rubin	4-5	4	4	3-4
D3	Medium pink	4	4-5	4-5	4

ALT. = Alteration, ST. = Staining on cotton.

TABLE 5. Acidic and alkaline perspiration for azo disperse dyes (D1-3).

Dye	Color (shade 2%)	Acidic perspiration		Alkaline perspiration	
		ALT.	ST.	ALT.	ST.
D1	Burgundy	4-5	3-4	3-4	4
D2	Rubine	4	3-4	4	3-4
D3	Medium pink	4	4	3-4	4-5

ALT. = Alteration ST. = staining on cotton.

Conclusion

In this work, we have synthesized three novel azo disperse dyes containing α-aminophosphonates, and β–naphthol as coupling compound to give the target azo disperse dyes. The prepared dyes were used as disperse dyes for polyester fabric dyeing. These dyes gave a wide range of colors varying from burgundy, rubine, and medium pink shades on the fabric. The light, sublimation, washing, rubbing, and perspiration fastness of all patterns dyed with the prepared azo disperse dyes show good to very good fastness properties. In addition, the dyed samples showed that the dyes have good affinity with moderate to very good brightness and color depth to polyester fabric.

Experimental

All ^{1}HNMR experiments (solvent DMSO) were carried out with a 400 MHz Bruker Avance DRX-400 spectrometer at Okayama University, Japan. Chemical shifts are reported in part per million (ppm) relative to the respective solvent or tetramethylsilane (TMS). Melting points were recorded on Stuart scientific melting point apparatus and are uncorrected.

The mass spectroscopy and the microanalysis were performed in microanalysis laboratory at Cairo University. All reactions were followed by thin layer chromatography (TLC) on kiesel gel F254 precoated plates (Merck). Anhydrous THF, MeOH and CH_2Cl_2 were obtained from Sigma-Aldrich.

Synthesis of α-aminophosphonates 2a–2c

Aryl substituted pyrazolaldehydes (1.2 mmol), benzidine (1 mmol) and triphenylphosphite were dissolved in well dried anhyd. CH_2Cl_2 (5 ml). The lewis acid, copper (II) triflate (10 mol %) was added in one portion. The mixture was stirred at RT, until TLC analysis showed the complete consumption of benzidine. Then $CH_2Cl_2$was evaporated and the solid precipitated was filtered, dried, and crystallized from methanol as shown in Scheme 1.

Diphenyl(((4'-amino-[1,1'-biphenyl]-4-yl) amino)(1,3-diphenyl-1H-pyrazol-4-yl)methyl) phosphonate

$C_{40}H_{33}N_4O_3P$, Yield (95%); mp>300°C. ^{1}HNMR (DMSO , 400 MHz) : δ = 5.08 (s, 1H, CHP) , 5.29 (s, 1H, NH), 5.99 (d, *J* = 6.0 Hz, NH_2), 7.02 –7.68 (m, CH arom) ,8.28(s, CH pyrazole). The mass spectra show the molecular ion peak at m/e = 650 (M^+ + 2, 9.5 %) , the ion Peak at m/e = 77 (M^+ -$C_{34}H_{28}N_4O_3P$, 98.4 %) , the ion peak at m/e = 91 (M^+ – $C_{34}H_{28}N_3O_3P$, 23.8 %)

Diphenyl(((4'-amino-[1,1'-biphenyl]-4-yl) amino)(1-phenyl-3-(thiophen-2-yl)-1H-pyrazol-4-yl)methyl)phosphonate,

$C_{38}H_{31}N_4O_3PS$, Yield (85%); mp>300°C. ^{1}HNMR (DMSO , 400 MHz) : δ = 5.40 (s, 1H, CHP) , 5.80 (s, 1H, NH), 6.05 (d, *J* = 6.0 Hz, NH_2), 6.55–6.60 (m, 4H arom), 7.33–7.80 (m, CH arom)8.33(s, CHpyrazole). The mass spectra show the molecular ion peak at m/e = 655 (M^+ + 1, 9.4 %), the ion Peak at m/e = 77 (M^+ -$C_{34}H_{28}N_4O_3P$, 98.4 %) , the ion peak at m/e = 97 (M^+ – $C_{34}H_{28}N_3O_3P$, 33.8 %)

Diphenyl(((4'-amino-[1,1'-biphenyl]-4-yl) amino)(1H-indol-3-yl)methyl)phosphonate, $C_{33}H_{28}N_3O_3P$, Yield (85%); mp280–282°C. ^{1}HNMR

(DMSO , 400 MHz) : δ = 4.73 (s, 1H, CHP), 5.30 (s, 1H, NH), 6.22 (d, *J* = 6.0 Hz, NH_2), 6.40–6.58 (m, 4H arom), 7.20–7.58 (m, CH arom)9.66(s, NH).The mass spectra show the molecular ion peak at m/e = 546 (M^+ + 1, 9.6 %).

General procedure for the synthesis of diphenyl 1 [(benzidine) 1- azo β – Naphthole] aminoheterocyclephosphonate

A well-stirred solution of diphenyl [benzidine] 1-amino heterocylicphosphonate (2a-2c) (1.02 mmol) in 2N HCl (1.5 mL) was cooled in ice salt bath and diazotized with 1N $NaNO_2$ solution (1mmol; in 2 mL water). The mixture was then tested for complete diazotization using starch iodide paper, which gives a weak blue test. If the mixture does not give the test, more sodium nitrite was added dropwise until a positive test is obtained and the color is stable for few minutes. If, on the other hand, strong test for nitrite is obtained, a few drops of a dilute solution of the base hydrochloride are added until the nitrite test is nearly negative. The above cold diazonium solution was added slowly to a well stirred solution toβ – Naphthole (1mmol) in ethanol (20 mL) containing sodium acetate (2.5mmol) and the mixture was cooled in an ice salt bath. After the addition of the diazonium salt solution the reaction was tested for coupling reaction. A drop of the reaction mixture was placed on a filter paper and the colorless ring surrounding the spot dye was treated with a drop of an alkaline solution of a reactive coupler, such as sodium salt of 3-hydroxy-2-naphthanilide. If un-reacted diazonium salt is present, a dye is formed. The presence of un-reacted coupler can be determined in a similar manner using a diazonium salt solution to test the colorless ring. After the coupling reaction is complete, the reaction mixture was stirred for 50 minutes at room temperature. The crude product was filtered, dried and Purified by silica gel column chromatography (Ethyl acetate: Hexane 3:1) to give the dye.

Diphenyl((1,3-diphenyl-1H-pyrazol-4-yl)((4'-((2-hydroxynaphthalen-1-yl)diazenyl)-[1,1'-biphenyl]-4-yl)amino)methyl)phosphonate D1

$C_{50}H_{38}N_5O_4P$,Yield (95%); m.p =275–277°C. IR (KBr) cm^{-1}: OH at 3716 cm^{-1}, 3429 (NH), 1594 (N = N), 1227 (P = O), and 1027 (–P– O–C).^{1}HNMR (DMSO, 400 MHz): δ = 4.9 (s, 1H, CHP), 8.6 (s, 1H, NH), 7.1 – 7.29 (m, 5H arom), 7.29 –7.9 (m, CH arom), 9.9(s, 1H, OH).

Diphenyl(((4'-((2-hydroxynaphthalen-1-yl)diazenyl)-[1,1'-biphenyl]-4-yl)amino)(1-phenyl-3-(thiophen-2-yl)-1H-pyrazol-4-yl)methyl)phosphonate

D2: $C_{48}H_{35}N_5O_4SP$, Yield (95%);m.p =325 –327 °C. ^{1}HNMR (DMSO, 400 MHz): δ = 4.1 (s, 1H, CHP), 8.9 (s, 1H, NH), 6.55 – 6.88 (m, 2H arom), 7.29 –7.9 (m, CH arom), 10.4(s, 1H, OH). The mass spectra show the molecular ion peak at m/e = 809 (M^+, 42.9 %), the ion Peak at m/e = 808 (M^+ -1, 35.7 %), the ion peak at m/e= 810 (M^+ +1, 21.4%)

Diphenyl(((4'-((2-hydroxynaphthalen-1-yl)diazenyl)-[1,1'-biphenyl]-4-yl)amino)(1H-indol-3-yl)methyl)phosphonate

D3:$C_{43}H_{33}N_4O_4P$,Yield (95%); m.p=240–241°C.The mass spectra show the molecular ion peak at m/e = 700 (M^+, 0.3 %), the base ion Peak at m/e = 322 (M^+ -$C_{21}H_{19}N_2O_3P$, 100 %), the ion peak at m/e = 77 (62.4 %)

Substrate

Commercially available scoured, woven polyester fabric with weight of (122 g m^{-2}) was used for the experiment assess the dyeing behavior of the compounds under investigation.

Dyeing of polyester fabric

Azoic dyes (D1, D2, and D3) were applied on dyeing polyester 100% fabric that was performed by the following procedure:

- The required amount of the dye (2% shade) was suspended in methanol and was added dropwise to a stirred pollution of Dispersogen P (100 / L) [dispersing agent of Hoechst].
- A sample of polyester fabric (100%) was immersed in a bath of 50°C for 5 min . with a liquor ratio 1: 20.
- 2 g/L Eganal RAP (Levelling agent of Hoechst) and 4 g/L Hostatex Lo – ET (carrier of Hoechst) were added to the bath with stirring for 10 min. The PH is adjusted to 4-5 with the addition of acetic acid.
- The thoroughly dispersed dye solutions were added to the bath and the temperature is raised at 98 °C within 60 min. Total dyeing time is then being 90 min.
- Afterwards it is cooled to 60 °C and is supplied to washing, dyed samples rinsed and dried.

Color assessment

The colorimetric parameters (Table 2) of the dyed polyester fibers were determined on a reflectance spectrophotometer (Gretag-Macbeth CE 7000a), equipped with a D65/10° source and barium sulphate as standard blank, UV excluded, specular component included and three repeated measurements average settings. The following CIELAB coordinates are measured, lightness (L*), chroma (C*), hue angle from 0º to 360º (H), (a*) value represents the degree of redness (positive) and greenness (negative) and (b*) represents the degree of yellowness (positive) and blueness (negative). A reflectance spectrophotometer (Gretag Macbeth CE 7000a) was used for the colorimetric measurements on the dyed samples.

Color measurements

Fastness tests for the prepared dyes (D1-3)

The prepared set of dyes (D1-3) was applied on dyeing of polyester fabric: The dyed washed samples were tested by standard ISO methods. The specific tests were ISO 105-X12(1987), ISO 105-co4 (1989), ISO105-EO4 (1989), ISO 105-BO2 (1988) corresponding to color fastness to sublimation, rubbing, washing, perspiration, respectively, and light which was measured using samples 10x10 cm of dyed fabrics mounted on a white chart paper and irradiated using a XENOTEST 1200 apparatus at a relative air humidity of 65% and 50˚C with duration 4 hours.

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تحضير وتوصيف صبغات ازو مشتتة جديدة تحتوي علي مجموعة الفا امينوفوسفات وتطبيقاتها في صباغة الياف البولي استر

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يهدف هذ البحث الي تحضير صبغات ازو مشتتة جديدة مبنية علي مركبات البنزيدين والحاملة لمجموعة الفا امينو فوسفات التي تتميز بنشاطها البيولوجى المتنوع وتطبيق هذه الصبغات في صباغة الياف البولي استر.

فى هذا البحث تم تحضير وتوصيف عدة صبغات ازو مشتتة جديدة باستخدام مركبات البنزيدين المحتوي علي مجموعات امين طرفية وتفاعلها مع الدهيدات حلقية ومجموعة ثلاثي فينيل فوسفات لتحضير مركبات تحتوي علي مجموعة الفامينوفوسفات ومجموعة امين طرفية والتي تقترن مع مركبات البيتا نافثول لانتاج صبغات ازو مشتتة جديدة.

تم توصيف كل الصبغات الجديدة باستخدام اجهزة (IR, [1]HNMR, Mass spectra)

تم تطبيق الصبغات المحضرة في صباغة الياف البولي استر كما تم قياس خواص الثبات للالياف المصبوغة وقد اعطت نتائج جيدة.