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# Synthesis of Some Disperse Dyes Based on Pyridine-2,6-dione: Part 1. Synthesis, Applications and Antibacterial Activities

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**Abstract**

The aim of this survey is, novel dispersion dyes based on pyridones are synthesized. These disperse dyes were made by reacting methyl propionylacetate with ethyl cyanoacetate and ethyl amine. Utilizing elements analysis, FTIR, mass, UV, and NMR spectroscopy, their structures were determined. The spectrum features and fastness properties of the dyes were assessed after application to polyester fibers. These disperse dyes' antimicrobial properties were also assessed

***Keywords***: Arylhydrazopyridone; dispersion dyes; antimicrobial

## 1. Introduction

Since the different societies have been interested in the dyeing process from previous ages, and since the dyes and their attractive colors affect human behavior and therefore society as a whole, researchers and scientists have been interested in preparing industrial dyes with multiple colors for their application in different fields. Synthetic dyes contributed greatly to natural dyes, because they had multiple and varied colors. And with the emergence of synthetic fabrics, it was necessary to have dispersed dyes that were applied to synthetic fabrics, the most important of which is polyester fabrics. Dyes that contain the azo group and the nucleus of these dyes are heterogeneous rings that have received great attention due to their importance in many fields [1, 2], the most important of which is textile dyeing. In addition to dyeing textiles, it has importance in biological and medical studies. As some of these synthetic dyes showed activity against microorganisms as well as viruses, and they also helped in inducing some diseases. Solar cells are among the most important fields affected by synthetic dyes when applied to them [3-5]. Pyridine-2,6-dione (pyridone) is one of the most important pigments that belong to the azo group and contains a heterocyclic ring pyridones are a class of organic compounds that have been extensively studied for their applications as dyes [6-8].Pyridones have been used as dyes in a variety of applications, including textiles and paper [9]. They are particularly useful in the textile industry, where they are used to dye natural and synthetic fibers [10, 11]. Pyridones are known for their excellent color fastness, which means that they do not fade or wash out easily. This makes them ideal for use in clothing and other textiles that are exposed to sunlight and washing [12, 13].

In addition to their use as dyes, pyridones have also been studied for their potential applications in other areas. For example, they had been investigated as influential medication for the remedy of cancer and other diseases. Pyridones have also been studied for their antibacterial and antifungal properties, which could make them useful in the development of new antibiotics.

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## 2. Martial and methods

All melting points were calculated without correction using an electrothermal digital melting point equipment. At Assiut University, the infrared spectra were captured using a KBr pellet approach on a Pye Unicam SP3-100 spectrophotometer and a Shimadzu IR-470 infrared spectrophotometer. At Cairo University, deuterated dimethysulfoxide (DMSO-d6) $^1$H-NMR spectra were optained on a Varian 300 MHZ with tetramethylsilane (TMS) as the internal reference, and the chemical shifts are represented in ppm. At Assiut University, mass spectra were conducted using an HP model MS-5988. At Al-Azhar University in Cairo, microanalyses for C, H, N, and halogen were conducted using a Vario El Elementar analyzer.

### *2.1. General procedure*

Methyl propionylacetate (0.01 mol), was refluxed with ethyl cyanoacetate (0.01mol) and ethyl amin (0.015 mol) for 6 hours to form compound **4**.

### *2.1.1 The general process for the synthesis of azo pyridones dispersion dyes*

First, prepare the diazanium salt. 10 ml of water were used to dissolve 1 gram of sodium nitrite, and the formed solution was cooled between 0 and 5 °C, and then added to a solution of arylamine hydrochloride cooled at a temperature of 0 to 5 °C also with stirring for an hour. Secondly, a solution of aryldiazanium salt is added to a solution of compound **4** (0.01 mole dissolved in 20 ml of ethyl alcohol in addition to 2 grams of sodium acetate). For 60 minutes, the ingredients were mixed at room temperature. The precipitates were filtered and then recrystallized using ethanol **6a-c** (Scheme 2).

**1,4-diethyl-5-(2-(naphthalen-1-yl)hydrazineylidene)-2,6-dioxo-1,2,5,6-tetrahydropyridine-3-carbonitrile (6a).**

Yield (75.2%), m.p. 198-200 ºC. IR spectrum showed absorption bands at υ = 3435 cm$^{-1}$(-NH), 3072(CH-aromatic), 2926(CH-aliphatic), 2206(C≡N) and 1668(C=O), 1592 cm$^{-1}$ (C=C). While the $^1$H-NMR indicated signals at 1.14 (t, 3H, *J* = 7.18 Hz, $CH_3$), 1.21 (t, 3H, *J* = 7.78 Hz, $CH_3$), 2.93 (q, 2H, *J* = 7.77 Hz, $CH_2$), 3.88 (q, 2H, *J* = 7.78 Hz, $CH_2$), 7.56 (m, 7H, *J* = 7.19 Hz, Ar-H), 12.8 (s, 1H, NH). MS (*m/z*) 346.37 ($M^+$, 54%), 142.18 (46%). Anal. Calcd. for $C_{20}H_{18}N_4O_2$ (346): C 69.30; H 5.24, N 16.17. Found: C 69.62; H 5.37; N 16.41.

**1,4-diethyl-2,6-dioxo-5-(2-(pyridin-3-yl)hydrazineylidene)-1,2,5,6-tetrahydropyridine-3-carbonitrile (6b).**

Yield (69.7%), m.p. 152-154 ºC. IR spectrum showed absorption bands at υ = 3367.16 cm$^{-1}$(-NH), 2963.3(CH-Ar), 2930(CH-aliph), 2224(C≡N), 1604(C=N) and 1678(C=O), 1510 cm$^{-1}$ (C=C). While the $^1$H-NMR indicated signals at 1.16 (t, 3H, *J* = 7.17 Hz, $CH_3$), 1.25 (t, 3H, *J* = 7.78 Hz, $CH_3$), 2.94 (q, 2H, *J* = 7.77 Hz, $CH_2$), , 3.89 (q, 2H, *J* = 7.78 Hz, $CH_2$), 7.17 (d, 1H, *J* = 7.18 Hz, Ar-H), 7.62 (d, 1H, *J* = 7.19 Hz, Ar-H), 7.91 (d, 1H, *J* = 7.18 Hz, Ar-H), 8.17 (d, 1H, *J* = 7.18 Hz, Ar-H),11.82 (s, 1H, NH). MS (*m/z*) 298.23 ($M^+$, 54%), 94 (100%), 182 (80%). Anal. Calcd. for $C_{15}H_{15}N_5O_2$ (297): C 60.60; H 5.09, N 23.56. Found: C 60.84; H 5.21; N 23.75.

**5-(2-(3-acetylphenyl)hydrazineylidene)-1,4-diethyl-2,6-dioxo-1,2,5,6-tetrahydropyridine-3-carbonitrile (6c).**

Yield (78.3%), m.p. 172-174 ºC. IR spectrum showed absorption bands at υ = 3436.26 cm$^{-1}$(-NH), 2979.91(CH-aromatic), 2936.25(CH-aliphatic), 2224(C≡N) and 1671(C=O), 1519 cm$^{-1}$ (C=C). While the $^1$H-NMR indicated signals at 1.22 (t, 3H, *J* = 7.18 Hz, $CH_3$), 1.28 (t, 3H, *J* = 7.78 Hz, $CH_3$), 2.91 (q, 2H, *J* = 7.78 Hz, $CH_2$), 3.34 (s, 3H, *J* = 7.18 Hz, $CH_3$), 3.91 (q, 2H, *J* = 7.78 Hz, $CH_2$), 67.48-7.52 (m, 1H, *J* = 7.18 Hz, Ar-H), 7.62 (dd, 1H, *J* = 7.18 Hz, Ar-H), 7.91 (dd, 1H, *J* = 7.19 Hz, Ar-H), 8.46 (s, 1H, Ar-H),12.82 (s, 1H, NH). MS (*m/z*) 339.23 ($M^+$, 12%), 94 (100%), Anal. Calcd. for $C_{18}H_{18}N_4O_3$ (338): C 63.89; H 5.36, N 16.65.56. Found: C 64.15; H 5.45; N 16.73.

### 2.2 Dyeing process

Fabric scoured and bleached polyester fabric was gotten from local market.

#### 2.2.1. Conventional *dyeing*

The dye (1%, 2%, and 3% weight of cloth) was used to make the dye baths, which had a final liquor ration of 50:1 (w: w). Using a 2% dispersion agent and 2% carrier in a pH range of 4.5–5.0, 100 °C, and a 60-minute dyeing process, fabrics were dyed. The fabrics were cleanly rinsed in water before being immersed in a solution containing 2 g/L sodium hydroxide and 2 g/L sodium hydrosulphite for 30 minutes at a temperature of 50 °C. They were then carefully washed and allowed to air dry.

#### *2.2.2. Color measurements*

For assessing the colorimetric characteristics of the colored polyester fabrics, a reflectance spectrophotometer was used. The UltraScan PRO D65 UV/VIS spectrophotometer was used to evaluate the yield of dye on the colored fabric utilizing light reflection technology.

The Kubelka-Munk Equation (1) was used to determine the dyes' color strengths, which are represented by the symbol K/S [14].
K/S is calculated as

$$K/S = [(1-R)^2 / 2R] - [(1-R_o)^2 / 2R_o]. \quad (1)$$

Where $R_o$ is the fraction of the reflectance of the undyed fabric, K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance of dyed samples.

### *2.3. Fastness properties*

#### *2.3.1. Color fastness to washing*

The ISO 105-C02:1989 procedure was used to achieve the color fastness to washing [15]. A sample of the dyed fabric was sandwiched between two bleached pieces, one made of cotton fabric and the other of wool fabric, and fastened with sutures. It was then immersed in an aqueous solution containing 5 g/L of nonionic detergents at a liquor ratio of 1:50 for 30 minutes at 60 °C, rinsed thoroughly with a manual squeeze, and left to dry. To evaluate the color fastness to washing, the gray scale was used.

#### *2.3.2. Color fastness to perspiration*

Two artful sweat solutions were created using the ISO 105-E04:1989 test methodology, one in an acidic medium and the other in a basic one. Where the acidic perspiration solution contains sodium chloride (5 g), sodium dihydrogen orthophosphate dihydrate (2.2 g), and L-histidine monohydro-chloride monohydrate (0.5 g) in one liter of pure water. After adding 0.1 N NaOH to the solution, the pH was adjusted to 5.5. The basic perspiration solution contained one liter of distilled water along with L-histidine monohydro-chloride monohydrate (0.5 g), sodium chloride (5 g), and sodium dihydrogen orthophosphate dihydrate (2.2 g). After adding 0.1 N NaOH to the solution, the pH was changed to 8.0. The following procedures were followed in order to conduct the fastness test: A sample of dyed cloth (5 x 4 cm) was stitched between two separate slices of plain designs. For 15–30 minutes, the samples were immersed in both solutions while being agitated and compressed to ensure complete wetting. The examined sample was held between two plates of plastic or glass and subjected to a load of 4-5 kg. The color fastness to perspiration was then evaluated using a gray scale change approach after these plates had been exposed to a temperature of 37 °C in a vertical position for four hours.

### *2.4. . Antimicrobial activities test*

The Agar-well diffusion technique [16, 17] was used to assess the antibacterial effects of new dispersion arylhydrazopyridone dyes against six distinct microbe cultures. The test included pure cultures of *Candida albicans* (Yeast), *Aspergillus flavus*, *Bacillus cereus*, *Staphylococcus aureus* (Gram positive bacteria), *Micrococcus luteus*, and *Pseudomonas aeruginosa* (Gram negative bacteria). Each bacterial strain was inoculated in an aliquot of 0.1 mL and spread on nutrient agar (NA), while the yeast was spread in an aliquot of 0.1 mL on potato dextrose agar (PDA). Each of the dispersion dyes under investigation had an ultimate concentration of 100 mg/mL., was given to the infected plates in a volume of 100 L. The sterile cork borer created 4 mm wells with the dispersed colours inside of them. The NA plates underwent a 37 $^oC$ incubation period.

## *3.* Result and discussion

### 3.1. Synthesis and Features.

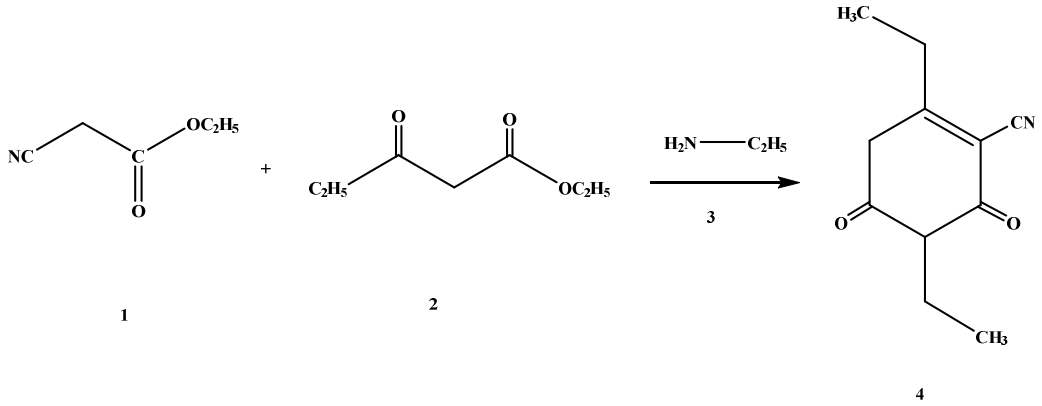
The present investigation deals with the synthesis of some new azo disperse dyes containing a pyridones moiety starting from methyl propionylacetate. Methyl propionylacetate was reacted with ethyl cyanoacetate and ethyl amin (scheme 1) to obtained 2,5-diethyl-4,6-dioxocyclohex-1-ene-1-carbonitrile **4** which then reacted with aryldiazanium salt of arylamine to produce new disperse dyes consist of 2,6-dioxo-1,2,5,6-tetrahydropyridine-3-carbonitrile derivatives (6 a-c ) (scheme 2). Structure of compounds **6 a-c** are assured by elemental analysis and spectral data, In IR spectra is found of new band for NH group after 3400 $cm^{-1}$, in $^1$H-NMR spectra showed the presence of exchangeable signal after δH 11 ppm for NH.
In addition of IR spectrum, $^1$H-MNR enhance chemical structure of new synthesized dye where it shows presence of (1H for –NH) at signals 12.8, 11.82 and 12.82 for compound **6a**, **6b** and **6c** respectively. Aromatic protons also exist in $^1$H-NMR spectrum at signals 7.56 for **6a**, (7.17 and 7.62) for **6b** and compound **6c** showed signals at (7.91, 8.46) for aromatic protons.
$^1$H-NMR showed the presence of ethyl groups in the new synthetic dyes, as it showed the presence of a signal for ($CH_2$) at 2.93, 3.88 and signals for ($CH_3$) at 1.14, 1.21 for compound **6a**, while **6b** signals for ($CH_2$) found at 2.94, 3.89 and signals for ($CH_3$) at 1.16, 1.25 and finally signals for ($CH_2$) of **6c** at 2.91, 3.34 in addition to signals at 1.22, 1.28 for ($CH_3$).
The congruence of the calculated elemental analysis with the found elemental analysis enhanced the chemical structure of the new synthetic dyes.

### 3.2. Fastness and Dyeing Characteristics

### 3.2 Property of Fastness

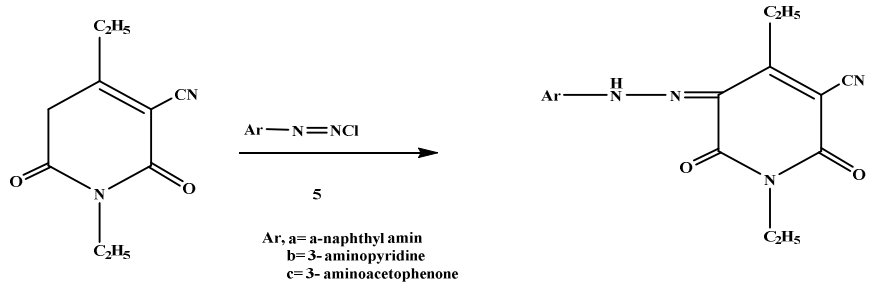
The results obtained and recorded in Table 1 indicate that with an increase in the concentration of the dye in the dyeing bath, the intensity of the color on the fabric increases accordingly. Filling more tissue gaps with the dye, this will increase the intensity of the color. And by looking at the L values, we find that

the dyes which applied to the fabric sheds in the direction of darkness by increasing the depth of the



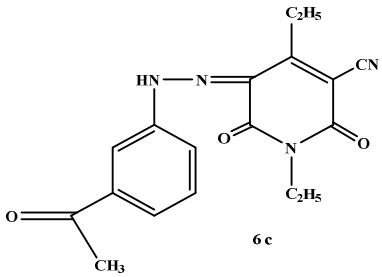

1 2 4

Scheme 1




6 a-c


6 a 6b




6 c

Scheme 2

**Table 1**
Colour strength measurements of synthesized dyes on the substrate

| Dye No. | Shade % | K/S | *L* | *a* | *b* | *c* | *h* |
|---|---|---|---|---|---|---|---|
| **6a** | 1% | 2.14 | 68.16 | 24.67 | 28.37 | 37.59 | 48.99 |
| | 2% | 2.60 | 63.88 | 26.23 | 26.61 | 37.37 | 45.42 |
| | 3% | 3.63 | 57.67 | 26.33 | 25.55 | 36.68 | 44.14 |
| **6b** | 1% | 0.52 | 81.75 | - 0.84 | 29.03 | 29.05 | 91.65 |
| | 2% | 0.53 | 80.64 | - 2.53 | 33.38 | 33.48 | 94.33 |
| | 3% | 0.61 | 80.24 | - 4.05 | 33.04 | 33.29 | 96.99 |
| **6c** | 1% | 0.26 | 82.82 | - 3.81 | 24.33 | 24.62 | 98.89 |
| | 2% | 0.36 | 81.61 | -3.22 | 28.49 | 28.67 | 96.46 |
| | 3% | 0.68 | 80.91 | - 4.37 | 38.11 | 38.36 | 96.55 |

**Table 2**
Fastness characteristics of synthesized dyes.(Where: Alt = Alteration, SC = Staining on cotton, SW = Staining on wool)

| Dye No | Shade % | Washing fastness | | | Perspiration fastness | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Acidic | | | Alkaline | | |
| | | Alt | SC | SW | Alt | SC | SW | Alt | SC | SW |
| **6a** | 1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| | 2 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| | 3 | 5 | 5 | 4-5 | 5 | 4-5 | 4-5 | 5 | 4-5 | 4-5 |
| **6b** | 1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| | 2 | 5 | 5 | 5 | 5 | 5 | 4-5 | 5 | 5 | 5 |
| | 3 | 5 | 5 | 4-5 | 5 | 4-5 | 4-5 | 5 | 4-5 | 4-5 |
| **6c** | 1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| | 2 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| | 3 | 5 | 5 | 4-5 | 5 | 4-5 | 4-5 | 5 | 5 | 4-5 |

**Table 3**
Results of antimicrobial properties of the synthetic disperse dyes

| Dye No. | $G^-$( inhibition zone in mm) | | $G^+$( inhibition zone in mm) | | Fungal strain | |
|---|---|---|---|---|---|---|
| | Pseudomonas aeruginosa (cont.42) | Bacillus cereus (cont.18) | Micrococcus luteus (cont.24) | Staphylococcus Aureus (cont.34) | *Aspergillus flavus* (cont.24) | *Candida albicans* (cont.22) |
| **6a** | 24 | 10 | 10 | 18 | 0 | 14 |
| **6b** | 29 | 10 | 0 | 20 | 0 | 18 |
| **6c** | 21 | 8 | 10 | 19 | 0 | 12 |

shade applied to the fabric, and this behavior includes dye **6a** , **6b** and **6c**.when looking at the values of (a, b, and c) for the dyes **6a**, **6b**, and **6c**, we find that the dye **6a** has positive (a) values, which indicates that the dye is in the direction of the red color, and the b values indicate that the color is positive, indicating that the color is in the direction of the yellow color.

(h) The hue is in the orange color range, ranging from 44 to 48 for shades 1%, 2% and 3%

Dye **6b** showed negative values for (a), meaning that the dye goes in the direction of the green color, and since the values do not exceed 1, the dye is in the brightness zone. The positive (b) values indicate that the dye is in the direction of the yellow color, and this is confirmed by the (h) values for the different concentrations of the dye bath, as the values are close to 90, meaning that the color is in the yellow color region. As in the case of dye **6b**, dye **6c** has almost the same characteristics, as the values of (a) are negative and do not exceed 1, meaning that the dye is in the direction of the green color and in the brightness region, and the positive values of (b) indicate that the dye is in the direction of the yellow color, and the values of (h) Close to 90 indicates that the dye is in the yellow color region.

#### *3.2.1. Washing fastness*

It is clear from the data recorded in Table No. 2 that the newly synthesized dyes have a fastness between excellent and very good against washing, and the difference is very small at shade 3% g. Perhaps this is due to the strength of the dye penetration into the gaps of the polyester fabric, which increased the bonding of the dye to the fabric

#### *3.2.2. Perspiration fastness*

As is the case in the stability of our new synthetic dye against washing, these dyes have excellent fastness against perspiration with shades of 1% and 2%, and very good fastness at a shade of 3% against perspiration, whether the medium is acidic or alkaline, which suggests that our new synthetic dyes bind to the fabric Strong bonding and it penetrates well into the gaps of the polyester fabric.

### *3.3 Antimicrobial Activity*

The newly synthesized arylhydrazopyridone dyes (6 a-c) were evaluated in vitro for antibacterial and antifungal activities against two different strains of Gram-negative (Bacillus cereus, Pseudomonas aeruginosa) and Gram-positive (Micrococcus luteus, Staphylococcus Aureus) bacteria and two fungal strains (Candida albicans and Aspergillus flavus) by the agar diffusion technique. To create a solution with a concentration of 1000 g mL-1, the investigated substances were dissolved in DMSO. Nutrient agar was used to maintain the bacteria cultures. There were no inhibitory zones in DMSO. Different bacteria were culture tested on the agar media. The diameter of the inhibitory zone (mm) was determined following 24 hours of bacterial incubation at 30 °C (Table 1). The findings acquired, which are given in Table 1, demonstrated that the synthesized dyes produced significant and encouraging outcomes.

## 4. Conclusion

In this research, three new dispersed dyes were synthesized, and then their structures had proven. Then different applications were made on these new synthetic dyes. They were applied as a dye for polyester fabric, and the effect of changing the concentration of the dyeing bath was studied. These dyes showed excellent stability against washing and perspiration, especially with the lower concentration of dyeing bath. When studying the biological activity of these new synthetic dyes, it was found that they have good biological activity

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