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Synthesis and Fastness Studies of New Nitro Substituted - 6 - Phenacitamido Quniophthalone

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Abstract
Substituted quinoline yellow dyes have been assessed to possess potentialities as dyes for wool and silk with good fastness properties. Their application as anti-malarial, antiseptic, trypanocidal agents and as photosensitizes have been mentioned in the literature. The new dyes have been synthesized by condensing 6-phenacitamido quinaldine with phthalic anhydride 3-Nitro Phthalic anhydride and 4-Nitro-Phthalic anhydride in presence of anhydrous zinc chloride. The synthesized derivatives were characterized by elemental analysis and spectral data (IR and ^{1}H NMR).

Keywords: 6-Phenacitamido quniophthalone dyes

Introduction
Quinoline yellow [1] is one of the important groups of compound used as dye stuff. In earlier time the yellow dyes was extracted from ground root of the turmeric or Indian saffron plant. Turmeric was the only yellow dye that did not require mordant to fix is a wool, cotton and silk but it is very sensitive to soap and alkali.

A large number of compound containing quinoline nucleus have been synthesized with in dude. Some potential antimalarial [2-5]. Trypanocidal agents [6] and compounds with strong antiseptic activity[7]. The dyes derived from quinoline [8] have proved useful for photosensitization. Quinoline yellow among a few others has been considered inoffensive and has been used as artificial color in food stuff's [9-10] and also in certain pharmaceutical preparation [11].

A number of quinolyl dyes having good strength and fastness properties for nylon and wool have been reported [12].

In the present investigation the new quinoline yellow dyes have been synthesized by adaptation of the method suggested by Philips and Goss [13]. Substituted quinaldine and Phthalic anhydride in equivalent amounts were heated in presence of anhydrous zinc chloride in an oil bath for several hours.

Experimental
Synthesis of Quinophthalone - I
A mixture of quinaldine (4g., 0.027 mole) powdered Phthalic anhydride (4.5g., 0.030 mole) and anhydrous zinc chloride (1g., 0.007 mole) was heated in an oil bath at 180 ^{0}C for four hours. The dark red colored viscous liquid was cooled and washed with rectified sprit when yellow solid dye separated out. The crude product on re-crystallization from glacial acetic acid gave deep yellow crystalline solid m.p. 240 ^{0}C, yield 80%.

Synthesis of 6-Phenacitamido quinophthalone-II
A mixture of 6-phenacitamido quinaldine (5.6g., 0.019 mole) powdered Phthalic anhydride (2.6g., 0.017 mole) and anhydrous zinc chloride (1g., 0.007 mole) was heated in an oil bath at 180 ^{0}C for four hours. The dark red colored viscous liquid was cooled and washed with rectified sprit when yellow solid dye separated out. The crude product on re-crystallization from glacial acetic acid gave bright yellow crystalline solid m.p. 192 ^{0}C, yield 51.72%.

Synthesis of 3-Nitro-6-Phenacitamido quinophthalone-III
A mixture of 6-Phenacitamido quinaldine (5.6g. 0.019 mole) powdered 3-Nitro Phthalic anhydride (3.6g., 0.018 mole) and anhydrous zinc chloride (1g., 0.007 mole) was heated in an oil bath at 180 ^{0}C for four hours. The dark red colored viscous liquid was cooled and washed

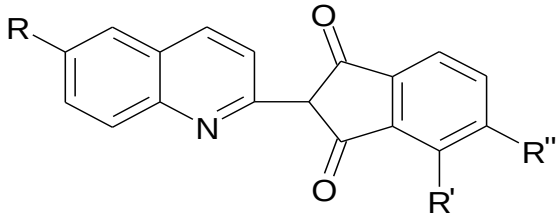
with rectified sprit when yellow solid dye separated out. The crude product on re-crystallization from glacial acetic acid gave yellow crystalline solid m.p. 181 ^{0}C, yield 52.1%.

Synthesis of 4-Nitro-6-Phenacitamido quinophthalone-IV

A mixture of 6-Phenacitamido quinaldine (5.6g., 0.019 mole) powdered 4-Nitro Phthalic anhydride (3.6g., 0.018 mole) and anhydrous zinc chloride (1g., 0.007 mole) was heated in an oil bath at 180 ^{0}C for four hours. The dark red colored viscous liquid was cooled and washed with rectified sprit when dark yellow solid dye separated out. The crude product on re-crystallization from glacial acetic acid gave yellow crystalline solid m.p. 188 ^{0}C, yield 61.5%.

Table 1: Physical Characterization data of synthesized compound I to IV

Compound	M.F.	M.W.	Dye	Yield	M.P.	Analysis	
			R R' R"	%	^{0}C	N	
						Found	Cal.
I	$C_{18}H_{11}O_2N$	273.29	- - -	80	240	4.80	5.12
II	$C_{26}H_{18}O_3N_2$	406.44	$NHCOCH_2C_6H_5$ H H	51.7	192	6.87	6.89
III	$C_{26}H_{17}O_5N_3$	451.44	$NHCOCH_2C_6H_5$ NO_2 H	52.1	181	9.26	9.31
IV	$C_{26}H_{17}O_5N_3$	451.44	$NHCOCH_2C_6H_5$ H NO_2	61.5	188	9.29	9.31



Spectral Interpretation

Compound - II

IR (kBr) : $\bar{\upsilon}$ cm^{-1} : 1665 (C=O),1642 (C=O), 1617 (C=C) 1452 (C=C), 1355 (Tertiary Nitrogen), 710 (Aromatic ring), ^{1}H NMR (500MHz, CDCl3): (ppm): 14.16 (S,^{1}H NH enaminone form), 8.62 (d, ^{1}H, ^{3}J = 9Hz = C-H), 8.05 (d ^{1}H ^{3}J = 9Hz = C-H), 7.64 - 7.74 (m 4H. Ar). 7.5 - 7.6 (m 3H Ar), 7.41 (d ^{1}H, ^{3}J = 8Hz, ^{4}J = ^{1}Hz Ar)

Compound - III

IR (kBr) : $\bar{\upsilon}$ cm^{-1} : 1665 (C=O),1642 (C=O), 1617 (C=C) 1452 (C=C), 1355 (Tertiary Nitrogen), 710 (Aromatic ring), 1555 (N=O), 1332 (N-O) ^{1}H NMR (500MHz, CDCl3): (ppm): 14.16 (S, ^{1}H NH enaminone form), 8.62 (d, ^{1}H, ^{3}J = 9Hz = C-H), 8.05 (d ^{1}H ^{3}J = 9Hz = C-H), 7.64 - 7.74 (m 4H. Ar). 7.5 - 7.6 (m 3H Ar), 7.41 (d ^{1}H, ^{3}J = 8Hz, ^{4}J = ^{1}Hz Ar)

Compound - IV

IR (kBr) : $\bar{\upsilon}$ cm^{-1} : 1665 (C=O),1642 (C=O), 1617 (C=C) 1452 (C=C), 1355 (Tertiary Nitrogen), 710 (Aromatic ring), 1555 (N=O), 1332 (N-O) ^{1}H NMR (500MHz, CDCl3): (ppm): 14.16 (S, ^{1}H NH enaminone form), 8.62 (d, ^{1}H, ^{3}J = 9Hz = C-H), 8.05 (d ^{1}H ^{3}J = 9Hz = C-H), 7.64 - 7.74 (m 4H. Ar). 7.5 - 7.6 (m 3H Ar), 7.41 (d ^{1}H, ^{3}J = 8Hz, ^{4}J = ^{1}Hz Ar)

Result and Discussion

New Quinophthalone yellow dyes have been prepared and characterized by elemental and spectral analysis. The % Yield of the synthesized dyes was 51.7 to 80%. Their dyeing performance one silk wool and nylon fabric was assessed. All the synthesized dyes almost produce variety of different yellow shades with poor to fairly good light fastness, good to excellent wash fastness and the percentage dye bath exhaustion 63.16 to 73.45%.

Dyeing and fastness properties

Two percent solution of the sodium salt of the dyes (II, III, and IV) was prepared by dissolving the requisite quantity of the salt in hot bath. A little acetic acid was added and PH-value of the solution was brought between 5 and 5.5. The fabric swatches were placed in the solution and temperature rose slowly to 100 ^{0}C in 40 minutes. An aqueous solution of formic acid (1g per liter) was then added and the fabric was left undisturbed for one hour. The swatches were then removed, washed well with soap water and then dried. The percentage exhaustion of 2% dyeing on silk fabric ranges from 66.35 to 72.45% for wool ranges from 63.16 to 70.82% and for nylon ranges from 64.32 to 68.12%. The percentage fixation of 2% dyeing on silk, fabric ranges from 84.01 to 91.94% for wool ranges from 84.15 to 90.15% and for nylon from 82.66 to 89.55%. The above procedure was used for dyeing pure silk, wool and nylon.

Table 2

Dye No.	λ max	Exhaustion %			Fixation %		
		S	W	N	S	W	N
I- Deep Yellow	434	72.45	63.16	67.42	87.25	84.15	86.72
II- Yellow	428	70.20	68.35	64.32	84.01	83.05	89.56
III- Yellow	428	69.26	70.25	69.12	86.40	87.15	84.88
IV- Dark Yellow	434	73.45	64.15	68.40	88.25	85.12	85.72

All the dyes have show good light fastness in silk, wool and nylon fabrics.

Conclusion

The new 3-Nitro & 4-Nitro substituted 6-Phenacitamido quinophthalone have been synthesized and characterized. The help of elemental analysis and IR, NMR, the fastness of the dyes give better results on wool, silk and nylon.

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