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DYEING BEHAVIOR AND PERFORMANCE OF PREPARED BENZIMIDAZOLE AND CHROMENE DISPERSE DYES AS APPLIED ON POLYESTER FIBRE, AND ITS BLENDS WITH WOOL.

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Abstract

Azo disperse dyes Ia-f - Va-f were synthesized from corresponding amines, where IR and visible spectra were evaluated. The prepared dyes were used for dyeing polyester, polyester woolen type and wool fibres. A range of bright and leveled shade have been obtained on application of these disperse dyes. The K/S values for various dyed substrate are in the following order, Wool > polyester/wool > polyester.

Introduction

Dyes based on heterocyclic Compounds as azo Components tend mostly to produce brilliant shade high tinctorial strength and hue ranging from yellow to dark brown (1).

Disperse dyes have been applied to a large number and variety of fibres, such as: cellulose acetate, triacetate, polyamide, and polyester fibres. Disperse dyes have therefore achieved an importance which is probably greater than the suggested weights used. These were the first type to be applied directly to fibre from an insoluble suspension. Their preparation for dyeing included a process adopting particle size reduction, and often dyeing in the presence of dispersing agent. The mechanism of dyeing with them would assume initially their passing from suspension through solution onto the fibre to which they become attached by non-polar forces. (2).

In recent years, the introductions of man-made and synthetic fibres have accelerated the development of new application methods, therefore assisting the dyer to utilize such more complex dyes.

Experimental

Synthesis of dyestuffs:

1) Synthesis of 5-aminobenzimidazole derivatives (A, B&C) (3-6) (General procedure).

A suspension of 5-nitrobezimidazole derivative (0.048 mol) in acetic acid (300 ml, 20%) was stirred at 60C^o while iron powder (20gm) was added gradually. After 2 hrs. the solution was cooled, diluted with water and the formed precipitate was filtered off, the product (5-aminobenzimidazole, A; 5-amino-2-methylbenzimidazole, B and 5-amino-2-trifluoromethylbenzimidazole, C) was extracted from the precipitate by acetone. After evaporation of acetone the product was crystallized from ethanol.

(Compound A, yield, 80%, m.p., 113 c^o; Compound B, yield, 88%, m.p., 229-231 c^o and Compound C, yield, 68%, m.p., 185-189 c^o)

2) Synthesis of 2-amino-3-cyano-7-hydroxy-4-phenyl (4-chlorophenyl)-4H -Chromene D&E (7-8) (General procedure).

Triethylamine (0.5ml) was added to a solution of benzaldehyde (10 mmol), malonoitrile (10 mmol) and resorcinol (10 mmol) in ethanol (30 ml). The reaction mixture was refluxed for 0.5hr. The precipitate that formed (2-amino– 3-cyano-7-hydroxy -4 -phenyl-4H - Chromene , D and 2-amino-3-cyano-7-hydroxy-4-(4-chlorophenyl)-4H-Chromene, E) was filtered off, and crystallized from ethanol .

(Compound D, yield, 63%, m.p., 209-211 c^o& Compound E, yield, 61%, m.p., 154-156 c^o).

3) Synthesis of Dyes I, II, III,IV and V:

The synthesized amino compound (A, B, C, D or E) was diazotized in the usual way by dissolving (0.1mole) of the appropriate amine in hydrochloric acid (10ml), cooling to 0-5 c^o, then adding an equivalent amount of cold sodium nitrite solution with stirring. The cold diazonium salt solution was used for coupling with phenols, amines and compounds containing active methylene group.

The structures and physical data of the synthesized disperse dyes are shown in tables (1 and 2).

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II- Materials for dyeing.

Polyester, Polyester woolen type denir 1.4 and wool produced locally at (Misr Rayon Co. Kafr El Dawer and El Mehalla El Kubra), Egypt.

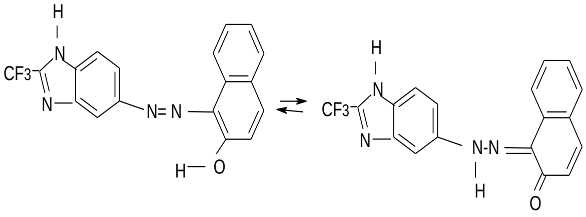
III. Instrumentation and measurements.

1- Melting Point apparatus	2- Infrared spectra
3- Spectrophotometer	4- Elemental analyses

Results and Discussions

The infrared spectra of parent compounds is basically retained for the reaction products. Some spectral changes are expected to appear as being associated with the formation of new species at the expense of vanished amino group, confirming the appearance of some new characteristic bands for example. 1456 – 1543 corresponding to- N= N –azo group.

The infrared spectra of dyes I,II, and V which contain the hydroxyl group in ortho position from the azo group show distinct bands in the hydroxyl stretching region. Generally, a series of weak broad bands can be detected in the range 3100-3420 cm^{-1} which may be ascribed to a chelate hydrogen- bonded system and the IIIc dye show a band for CO at 1627.2, this indicate that the dye may be in the hydrazone form.



These dyes Ib , IIa,IIb IIIa,IIIb and IVc are not containing hydroxyl group in ortho position to azo group therefor IR, specta show azo group (-N=N-) bands at 1460 – 1509 and also show (NH) band at 3327 – 3419 cm^{-1} this suggests that these dyes are predominately in azo form, as opposed to the hydrazo form in the solid state. IR,spectra of dyes Ic, IIc and IIIc are showing a broad band at 3479, 3423cm^{-1} assigned to (OH/NH) .

The infrared spectra of compounds Ie, IIe , IIIe, IVe show a new band at 3400 - 3500 cm-1, respectively, which can be assigned to stretching vibration of OH group . and this indicates that compound would favour the hydrazone form also, the IR spectra of these compounds show absorption band characteristic of --N=N-- stretching at 1557, 1543. Thus, these compounds exist in solid state as a mixture of azo - hydrazo form, but for the compound IIe the infrared show the disappearance of – N = N- azo group, this indicate that this compound may be found in the hydrazo form**.**

3.2. Assessment of prepared derivatives as disperse dyestuffs .

Table 3 :- UV – visible spectra of prepared dyes in organic solvents .

Dye No	**Colour in Crystal**	**Colour in organic solvents**							
		Ethyl alcohol		**D M F**		**CC l $_4$**	**Dichloro ethylene**	**Tetra chloro ethylene**	**acetone**
		colour	**λ max**	**Colour**	**λ max**	**Colour**	**Colour**	**colour**	**Colour**
I a	Redish Brown	deep orange	421	deep Brown	452	yellow	yellow	orange	orange
b	Dark Brown	Greenish yellow	436	Greenish yellow	461	yellow	yellow	**G** yellow	**G** yellow
c	yellowish Brown	Greenish orange	474	Green	498	Pale green	Pale green	Brownish orange	Pale green
d	orange	Light yellow	429	yellow	442	Pale yellow	Pale yellow	yellow	yellow
e	Brown	Light yellow	490	yellow	515	Pale yellow	Pale yellow	Pale yellow	Pale yellow
f	Brownish yellow	Pale Brown	414	Brown	452	Pale yellow	Pale Brown	Pale orange	pale yellow
II a	Dark Green	greenish yellow	440	Greenish yellow	480	Pale yellow	Pale yellow	**G** yellow	yellow
b	Greenish gray	Pale yellow	444	Greenish yellow	482	Pale Brown	Pale Brown	Pale yellow	**G** yellow
c	Greenish orange	G.yellow	485	G.yellow	511	G.yellow	G. yellow	**G** yellow	**G** yellow
d	orange	Pale yellow	419	yellow	489	Pale yellow	Pale yellow	yellow	yellow
e	orange greenish	Brownish orange	498	Brownish orange	520	Pale orange	Pale orange	Brownish orange	Brownish orange
f	Brownish yellow	yellowish Brown	421	Brown	452	yellowish Brown	Pale yellow	yellowish Brown	yellowish Brown
III a	Redish Brown	deep orange	474	deep orange	498	Pale yellow	yellow	orange	orange
b	Brownish green	Greenish yellow	461	G yellow	489	Pale yellow	Brownish yellow	Brownish yellow	Greenish yellow
c	Dark Grey	G yellow	481	Brownish yellow	509	–	Greenish yellow	Greenish yellow	G yellow
d	orange	deep yellow	456	deep yellow	482	Pale yellow	deep yellow	deep yellow	Pale yellow
e	orange	Pale yellow	508	yellow	534	–	yellow	yellow	Pale yellow
f	Brown	Brownish yellow	452	Brownish yellow	472	–	Brownish yellow	Brownish yellow	Brownish yellow
IV a	deep Brown	Brownish orange	505	Redish orange	535	Pale yellow	Pale pink	Pale yellow	Redish orange
b	deep orange	Brownish orange	479	Brownish orange	533	Pale pink	Pale Brown	Pale pink	Redish orange
c	deep yellow	deep orange	437	deep orange	474	Pale pink	deep orange	deep orange	orange
d	deep yellow	deep yellow	479	deep yellow	530	Pale yellow	Light yellow	Light yellow	orange
e	yellowish orange	deep yellow	479	orange	533	–	yellow	yellow	yellow
f	Redish Brown	Redish Brown	515	Redish Brown	534	–	Brownish orange	Redish Brown	Brownish orange
g	Redish Brown	Redish Brown	518	Redish Brown	535	Brown	Brownish orange	Redish Brown	Brownish orange

Table 3 Cont:- UV – visible spectra of prepared dyes in organic solvents .

Dye No	**Colour in Crystal**	**Colour in organic solvents**							
		Ethyl alcohol		**D M F**		**CC l$_4$**	**Dichloro ethylene**	**Tetra chloro ethylene**	**acetone**
		colour	**λ max**	**Colour**	**λ max**	**Colour**	**Colour**	**colour**	**Colour**
h	deep yellow	deep yellow	484	deep yellow	535	yellow	yellow	yellow	orange
i	yellowish orange	yellowish orange	488	orange	535	yellowish orange	yellow	yellow	orange
V a	Dark Brown	Brown	503	Brown	505	Orangish Brown	Brown	Brown	Brownish orange
b	Brown	Brown	498	Brown	504	Orangish Brown	Brown	Brown	Brownish orange
c	Brown	Brown	495	Brown	502	Orangish Brown	Brown	Brown	Brownish orange
d	yellowish orange	yellowish orange	486	orange	530	yellowish orange	yellow	yellow	orange
e	deep yellow	deep yellow	480	deep yellow	530	yellow	yellow	yellow	Orange
f	Brown	Brown	488	Brown	498	Orangish Brown	Brown	Brown	Brownish orange

The visible spectra of the azo compounds under investigation show one band within the wavelength range 421 – 535, this band would be characteristic of – N = N – system .

The tautomeric structures of some dyes show a charge transfer from electron donating group to azo group. This electronic migration for dyes to from hydrazo form . (9).

The tatuomeric structures of the investigated dyes (Ic, IIc, IIIc) show a charge transfer from oxygen atom of the hydroxyl group to the azo group. This electronic migration for dyes to form hydrazo form. (10)

From the obtained results it is clear that:-

The introduction of some substituents on the dye molecule causes some shifts in the position as well as the intensity of the absorption band for example;-

i) The absorption spectrum of dye Ia show band at λ max. 421nm. introduction of acetyl group (electron withdrawing group) in dye I_f causes a decrease in λ max. From 421 n m to 414.0 nm. (Blue shift) .

The presence of any substituent group is important. Either to enhance or inhibit this electronic migration thus causing a bathochromic or a hypsochromic shiftsThis can be discussed as follow.

ii) Bathochromic shift may be occurred due to increase conjugation in the parent compounds e.g dye No IIIe.

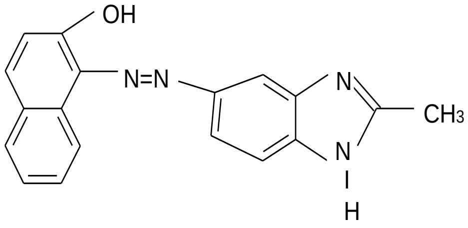
ii) The visible spectra of I_b, II_b and III_b, show an absorption band for dye II_b at 444 nm. and absorption band for dye III_b at 461nm. This means that a bathochromic shift occurred for dye IIb and IIIb as compared with dye Ib and this is attributed to the increased conjugation in the parent compound.

iii) It was noticed that the increase in the bathochromic shift was due to increase in polarity of solvent and dipole moment of the organic solvent e.g ethanol and carbon tetrachloride.

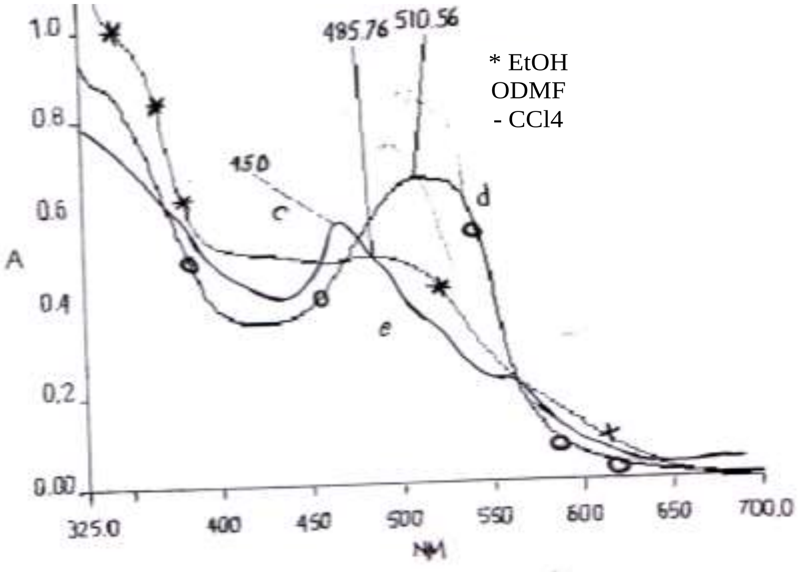
iv) The visible spectra of dyesIV show that :-

The introduction of electron withdonating group in the coupling component causes a bathochromic effect for dyes IVe and IVf .

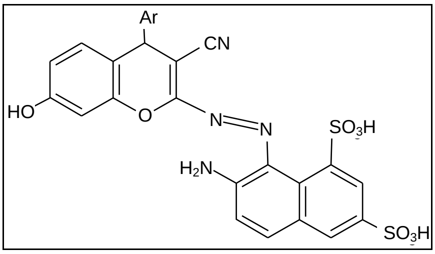
v) The effect of hydroxyl or nitro groups in ortho position is more than its effect of hydroxyl or nitro groups in para position e.g dye Ib and Ic and IVh and IVi respectively.



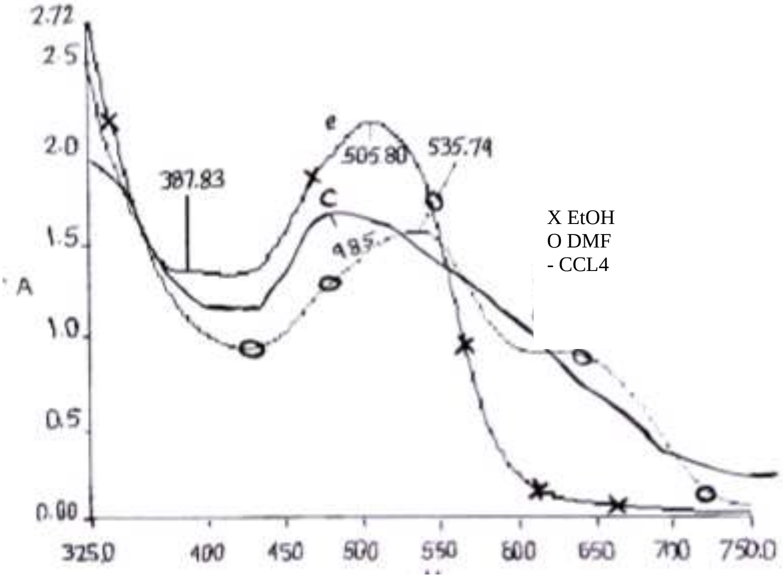
IIc dye



Absorption spectra of dye IIc
Fig., (1)



IVa dye



Absorption spectra of dye Iva
Fig., (2)

3.3. Application of the Prepared disperses dyes.

It was observed visual colour with different shades varied from yellow. Orange to dark brown for disperses dyes under investigation. The coupling component seemed to affect noticeably the depth and colour shade (11).

A range of bright and leveled shade have been obtained on application of these dyes to polyester, polyester woolen type and wool fibre (12).

3.3.2 Assessment of colour measurement.

In the present study, the colour strength expressed as K/S values was measured by applying the Kubelka- Munk equation as follow:

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-Ro)^2}{2Ro}$$

The K/M function has been tested for several prepared dyes on various substrates including polyester, polyester/ wool and wool. As we have seen from tables (4-7) the fibre with increased strength of colour shows low reflection R% value but higher K/S value indicating darker shade on dyed fibre (13).

Table(4): - Colour measurement of the dyed polyester fabric samples .

Dye No.	Visual colour on PET	Measured colour on PET . fibre .						
		L*	a*	b*	C*	H	R	K/S
I a	Pale brown	51.7	24.2	24.7	34.6	46.0	18	2.3
b	Brown	50.6	16.5	28.9	32.5	33.9	20	2.5
c	Orange	62.3	24.6	16.5	28.0	79.2	18	2.3
d	Deep yellow	50.9	15.6	19.3	24.8	51.1	18	2.4
e	light yellow	64.9	3.1	13.6	13.9	47.0	22	1.3
f	Yellowish orange	61.7	24.2	24.7	34.6	46.0	18	2.9
II a	Deep yellow	64.9	3.2	15.9	15.9	60.2	30	1.9
b	Orangish Brown	56.7	17.1	27.2	32.3	58.1	31	3.7
c	Deep orange	50.1	17.8	18.7	14.8	35.6	18	2.1
d	Yellowish orange	50.1	12.0	14.0	15.6	38.9	16	2.8
e	Yellow	63.1	26.2	26.7	35.6	41.2	35	1.6
f	Yellow	50.9	15.6	19.3	24.8	51.1	18	2.4
III a	Orange	50.9	30.39	20.1	35.9	32.1	13	3.3
b	Yellowish Brown	62.9	19.1	28.1	32.1	58.1	37	3.6
c	Deep orange	50.1	17.8	18.7	14.8	35.6	18	2.3
d	Yellowish orange	50.1	12.0	14.0	15.6	38.9	16	2.8
e	Yellow	61.7	24.2	24.7	34.6	46.0	18	1.9
f	Deep Orange	50.9	30.39	20.1	35.9	32.1	13	3.3
Iv a	Pale Brown	56.72	17.01	27.13	32.02	57.91	11	3.7
b	Pale orange	49.98	5.4	9.0	10.5	59.2	15	2.8
c	Deep yellow	69.82	14.7	19.3	24.8	52.1	18	2.5
d	Dark Yellow	64.9	3.09	13.6	13.88	77.3	30	1.6
e	yellow	65.8	3.1	13.5	13.80	77.1	35	1.9
f	Brown	44.5	10.5	4.76	11.53	24.4	9	3.3

Table(5): Colour measurement of the dyed wool fabric samples .

Dye No.	**Visual colour on Wool**	**Measured colour on wool . fibre**						
		L*	**a***	**b***	**C***	**H**	**R**	**K/S**
III a	Brown	52.7	32.2	27.1	31.9	57.9	20	4.3
b	Deep Brown	44.55	10.5	4.76	11.6	24.4	10	6.3
c	Pale Brown	49.86	7.5	9.6	12.4	34.2	28	3.13
d	Deep yellow	62.6	36.8	43.7	57.1	49.9	22	3.9
e	Pale Orange	50.9	30.5	20.3	35.9	32.1	28	3.8
f	Pale Orange	50.6	16.5	28.9	32.5	33.9	20	4.5

Table (6) : Colour measurement on Polyester/ wool:

Dye No	**Visual colour P.E.T/ wool**	**Measured Colour on PET/ wool fibre**						
		L*	**a***	**b***	**C***	**H**	**R**	**k/S**
I_f	Deep Yellow	61.7	24.2	24.7	34.6	46.0	18	3.1
II_f	Deep yellow	50.9	15.6	19.3	24.8	51.1	18	2.9
III_f	Deep Orange	50.9	30.39	20.1	35.9	32.1	13	3.8
IV_d	Deep yellow	69.82	14.7	19.3	24.8	52.1	18	2.9
IV_e	Yellow	65.8	3.1	13.5	13.8	77.1	35	1.9

Table (7) : Colour measurement on wool fibre:

Dye No	**Visual colour on Wool**	**Measured Colour on Wool fibre**						
		L*	**a***	**b***	**C***	**H**	**R**	**k/S**
I_f	Orange	50.6	16.5	28.9	32.5	33.9	24	4.3
II_f	Orange	50.9	30.39	20.1	35.9	32.1	28	3.8
III_f	Pale Brown	56.7	16.98	27.1	31.9	57.9	18	4.5
IV_d	Yellow	63.9	38.95	46.23	60.4	49.8	22	3.9
IV_e	Yellow	62.6	36.8	43.75	57.1	49.9	22	3.9

From the above results the K/S values for various dyed substrate are in the following order. Wool > polyester/wool > polyester.

Conclusion

In this research we could prepared disperse dyes from benzimidazole and chromene applied to dye polyester , polyester woolen type and wool.A range of bright and leveled shade have been obtained on application of these disperse dyes. From the above results the K/S values for various dyed substrate are in the following order, Wool > polyester/wool > polyester .

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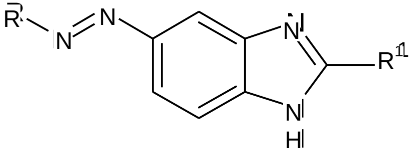
Table 1: physical data of disperse dyes derived from 5-aminobenzimidazole derivatives I- III.



I - III

I, R^1 =H; II, R^1 =CH_3; III, R^1 = CF_3.

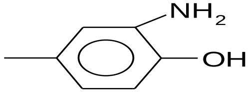
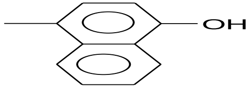
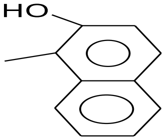
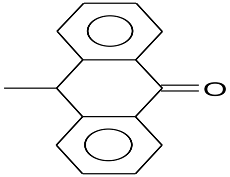
Dye No	R	**Yield %**	**m.p°C**	**M.formula (M.W.t)**	**Elemental analysis (cal/ found) C H N**	**IR(υ,cm^{-1})**	**Colour of solid**
Ia		74	> 300	$C_{13}H_{11}N_5O$ (253)	61.66 4.35 27.67 61.60 4.31 27.62	IR:3416.3(OH/NH_2/NH)broad;2928.3(CH); 1595.3 (C=N),1395.7(C-N) 1460 (N=N). .	Redish Brown
Ib		90	>300	$C_{17}H_{12}N_4O$ (288)	70.83 4.16 19.44 70.80 4.10 19.40	IR: 3417.3(OH,NH)broad;2930.2(CH) 1383.1 (C- N), 1457.4 (N=N) .	Dark Brown
Ic		76	210 decom.	$C_{17}H_{12}N_4O$ (288)	70.83 4.16 19.44 70.70 4.08 19.42	IR: 3585.1(OH), 3396.1(NH)3000.6(CH), 1587.6 (C=N)1374.4 (C-N),1456.4 (N=N)	yellowish Brown
Id		94	> 300	$C_{21}H_{14}$ N_4O (338)	74.56 4.14 16.57 74.50 4.10 16.50	IR: 3469.4 (OH), 3417.3 (NH), 2926.4 (CH) , 1638.7(C=O), 1460.3 (N = N) .	Brownish orange

Table 1:(Cont.).

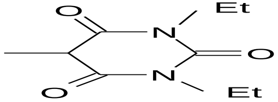
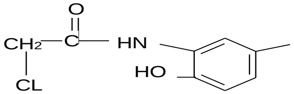
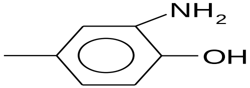
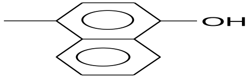
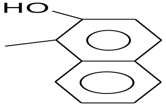
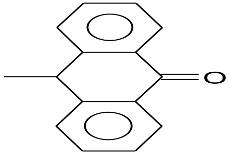
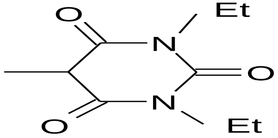
Ie		80	292+4	$C_{15}H_{16}N_6O_3$ (328)	54.88 4.88 25.60 54.80 4.80 25.60	IR : 3511.8(OH) , 3321.8, 3239.8 (NH) , 1630 (C= 0) , 1380.2(C- N), 1557.7 (C=N) , (N=N)	Brown
If		78	220 decom	$C_{15}H_{12}N_5O_2Cl$ (329.5)	54.63 3.64 1.24 54.55 3.60 1.20	IR: 3482.9(OH, NH) broad;2932.2868.2 (CH.aliph) ;1671.5(C=0);:1498.9(-N=N-); :868.1(C- CL)	Brownish gray
IIa		90	> 300	$C_{14}H_{13}N_5O$ (267)	62.92 4.87 26.21 62.89 4.82 26.19	IR : 3419.2 (OH, NH_2, NH)broad, 2932.2 (CH), 1509.51 (N=N), 1374.4 (C- N) .	Dark Brown
IIb		94	> 300	$C_{18}H_{14}N_4O$ (302)	71.52 4.64 18.54 71.50 4.60 18.50	IR: 3327.6 (OH, NH) broad , 3055.6 (CH) , 1586.6(C= N), 1371.6 (C-N) , 1517.2 (N= N).).	Brownish gray
IIc		96	> 300	$C_{18}H_{14}N_4O$ (302)	71.52 4.64 18.54 71.50 4.60 18.49	IR: 3479(OH) , 3399(NH) , 3050.8 (CH) , 1599.2 (C= N) , 1378.3 (C- N) 1466.1 (N=N) .	Brownish orange
IId		88	280 Decomp.	$C_{22}H_{16}N_4O$ (352)	75.0 4.55 15.90 75.0 4.49 15.85	IR: 3405.7(OH/NH)broad,1652.2(C=O) 1596.3 (C= N) , 1382.2 (C-N) 1466.1 (N= N).).	Orangeish Brown
IIe		92	280 Decomp	$C_{16}H_{18}N_6O_3$ (342)	56.14 5.26 24.56 56.10 5.20 24 52	IR : 3400.0 (OH) / (NH) ,3082.6(CH), 2942.8, 28675 (CH) , 1763.2, 1695. (2 C= O) , (C- N) 1387.0.	orange Brownish

Table 1:(Cont.).

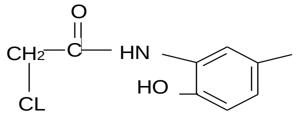
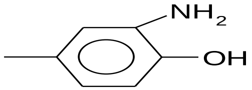
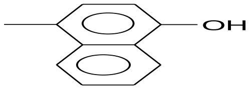
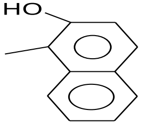
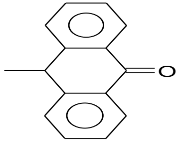
IIf		96	> 300	$C_{16}H_{14}N_5O_2cl$ (343.5)	55.90 4.10 20.38 55.88 4.10 20.35	IR:3420.2 (OH, NH) broad; 2940,2752.8 CH. aliph.);1604.0(C=0); 1490.2 (-N=N-); 765.8(C-CL).	Brownish yellow
IIIa		70	230	$C_{14}H_{10}N_5OF_3$ (321)	52.34 3.12 21.80 52.28 3.10 21.60	IR : 3469.4 (OH) . 3417.3 (NH_2). 3247.6 (NH) , 2928.3 (CH) , 1624.3(C=N) .	Redish Brown
IIIb		79	> 300	$C_{18}H_{11}N_4OF_3$ (356)	60.67 3.10 15.73 60.59 3.10 15.69	IR : 3235.0 (OH/NH) , 1633.9 (C=N) , 1384.1 .(C-N) ..	Brownish green
IIIc		78	> 300	$C_{18}H_{11}N_4OF_3$ (356)	60.67 3.10 15.73 60.57 3.10 15.70	IR : 3404.8 (OH), 3247.6(NH) , 1627.2 (C=N) , CO, 1385.1 ((C-N) ..	Dark Grey
IIId		90	216 Decom.	$C_{22}H_{13}N_4OF_3$ (406)	65.03 3.22 13.70 65.00 3.1813.70	IR : 3250.4 (NH) , 1635.8 (CO)1635 (C=N) , 1461.3(N=N) , 1400 (C-N) .	orange

Table 1:(Cont.).

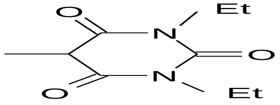
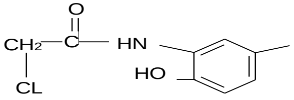
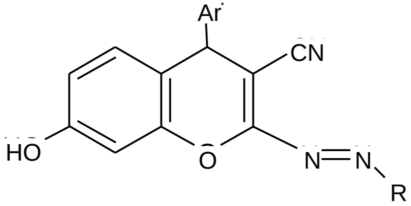
IIIe		80	284	$C_{16}H_{15}N_6O_3F_3$ (396)	48.48 3.79 21.20 48.40 3.70 21.18	IR 3435.6 (OH, NH) broad, 2931.2(CH) , 1658 (C=O) ,1543.2 (C=N) (N=N) , 1385.1 (C-N) .	Brownish orange
IIIf		80	> 300	$C_{16}H_{11}N_5O_2CLF_3$ (397.5)	48.30 2.7 17.61 48.20 2.7 17.58	IR :3502.2(OH, NH) broad, 24932.2, 2870.4(CH. aliph):1672.5(C=0);1498.9(-N=N-); 866.1 C-CL	Dark Brown

Table 2 : physical data of disperse dyes derived from 2-amino- 3-cyano -7-hydroxy -4-aryl -4H-chromene.



IV & V
IV, Ar=phenyl & V,Ar =4-chlorophenyl.

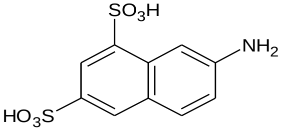
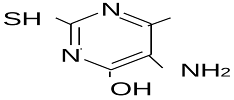
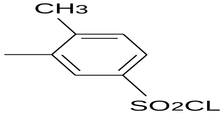
Dye No	R	yield	m.p^{o}C	M.formula (M.W.t)	Elemental analysis (cal/ found) C H N	IR(υ,cm^{-1})	Colour of solid
IVa		90	248	$C_{26}H_{19}N_4O_8S_2$ (579)	53.88 3.28 9.67 53.82 3.25 9.63	IR : 3400.9 (OH) ; 3252.4 (NH_2); 2219.4 (C ≡ N) : 1450.6 (- N = N-).	Deep Brown
IVb		82	299	$C_{20}H_{15}N_6O_3S$ (419)	57.279 3.58 20.05 57.220 3.49 19.90	IR : 3405.7(OH); 3309.3 (OH) ; 3080.7 (NH2) ; 2191.4 (C≡N) ; 1453.5 (- N= N).	Deep orange
IVc		78	240 decom.	$C_{23}H_{17}N_3O_4S$ Cl (466.5)	59.16 3.64 9.00 59.10 3.57 8.96	IR : 3420.2(OH); 3063.3 (CH Arom.); 2194.3 (≡N); 1490.9(-N= N-) .	Deep Brown

Table 2:(Cont.).

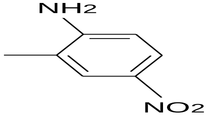
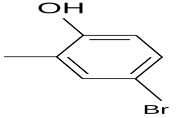
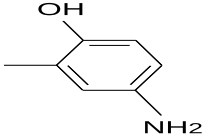
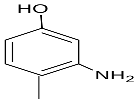
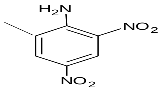
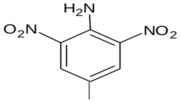
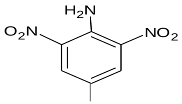
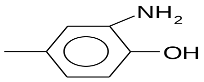
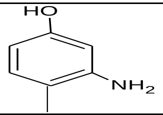
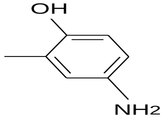
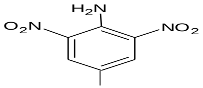
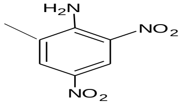
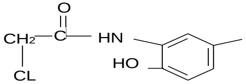
IVd		84	200 decom.	$C_{22}H_{16}N_5O_4$ (414)	63.77 3.86 16.91 63.70 3.80 16.83	IR: 3427.9 (OH) : 3153 (NH_2) ; 2207.8(C ≡ N); 1598.2 , 1540.3 NO_2 , 1467 (-N= N-).	Deep Yellow
IVe		53	270	$C_{22}H_{15}N_3O_3Br$ (449)	58.80 3.34 9.35 58.73 3.28 9.30	IR: 3424.1 , 3331.5 (2 OH) , 2184.6 (C ≡ N) ; 1403.4 (-N = N-).	Brownish yellow
IVf		92	235	$C_{22}H_{17}N_4O_3$ (385)	68.57 4.42 14.55 68.49 4.40 14.48	IR: 3423.1 , 3334.4 (2OH) , 3223.4 (NH2) ; 2188.5 (C ≡ N) ; 1402.4 (-N= N-)	Reddish brown
IVg		86	210	$C_{22}H_{17}N_4O_3$ (385)	68.57 4.42 14.55 68.53 4.40 14.53		Reddish brown
IVh		89	180 Decomp.	$C_{22}H_{15}N_6O_6$ (459)	57.52 3.27 18.30 57.51 3.25 18.29		Deep Yellow
IVi		90	175 Decomp.	$C_{22}H_{15}N_6O_6$ (459)	57.57 3.27 18.30 57.51 3.23 18.28		Deep Yellow

Table 2: (cont.)

IVi		$C_{22}H_{15}N_6O_6$ (459)	175 Decomp.	90	57.57 3.27 18.30 57.51 3.23 18.28	Yellowish Orange
V a		$C_{22}H_{16}N_4O_3Cl$ (419.5)	190 Decomp.	82	62.93 3.81 13.35 62.91 3.81 13.33	Dark Brown
V b		$C_{22}H_{16}N_4O_3Cl$ (419.5)	170 Decomp.	80	62.92 3.81 13.35 62.91 3.80 13.32	Brown
V c		$C_{22}H_{16}N_4O_3Cl$ (419.5)	185 Decomp.	84	62.93 3.81 13.35 62.92 3.80 13.32	Brown
V d		$C_{22}H_{14}N_6O_6Cl$ (493.5)	195 Decomp.	90	53.49 2.84 17.00 53.48 2.83 17.00	Dark Orange
V e		$C_{22}H_{14}N_6O_6Cl$ (493.5)	180 Decomp.	90	53.50 2.84 17.02 53.48 2.82 17.01	Yellowish Brown
V f		$C_{24}H_{18}N_4O_4Cl_2$ (496)	150 Decomp.	78	58.06 3.63 11.29 58.03 3.62 11.26	Brown