

# Synthesis of Disperse Azo Dyes by using Various Combinations of Base & Coupling Agents

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**ABSTRACT:** Synthetic dyes are ingredients often derived from petrochemical derivatives and used as colouring agents for many different products in market. Common types of synthetic dyes are known as azo dyes. Synthetic dyes are widely used worldwide due to their excellent properties which satisfy need of textile industry. The textile sector consumes around 80% of total production of synthetic dyes due to high demand. In this work we synthesized disperse azo dyes by using diazotization and coupling reaction. The bases used are 4-Nitroaniline, 2- Aminophenol, 4-Chloroaniline, 3-Chloroaniline and 2, 4-Dihydroxybenzophenone as coupling agent. In this work we made various combinations of base and coupling agent to produce azo dyes. The impact on various proportion of base and coupling agents shows the variation in colour and colour strength. An attempt has been made here to expose the possibility of synthesizing disperse azo dyes by employing diazotization and coupling reaction with various bases and coupling agent in their various molar proportion. The yield of synthesized dyes was calculated to confirm which dyes get produced in larger quantity with better utilization of chemicals.

**KEYWORDS:** Base & Coupling agent, Colour yield, Disperse Azo dyes, Synthetic Dyes.

## I. INTRODUCTION

Synthetic dyes are manufactured from organic molecules. Before synthetic dyes were discovered in 1856, dyestuffs were manufactured from natural products such as flowers, roots, vegetables, insects, minerals and woods. Batches of natural dye were never exactly alike in hue and intensity, whereas synthetic dyestuffs can be manufactured consistently. The use of computers and computer colour matching (CCM) system produces colour that is identical from batch to batch. William Henry Perkin, an eighteen-year-old English chemist, was searching for a cure for malaria, synthetic quinine, and accidentally discovered the first synthetic dye. He found that the oxidation of aniline could colour silk. From a coal tar derivative, he made a reddish-purple dye. The brilliant purple was called mauve. This discovery resulted in additional research with coal tar derivatives and other organic compounds and an entire new industry of synthetic dyes was born [1, 2]. In the twenty-first century, synthetic dyes are less expensive, have better colourfastness, and completely dominate the industry as compared with natural dyes. Thousands of distinctly different synthetic dyes are manufactured in the world.

## II. RELATED WORK

Prajapati et al [3] studied on Formation of some novel disperse azo dyes synthesis by the coupling component of diazonium salt with 1-(4-Nacetyl amino) 2-methyl phenyl-2-chloro ethanone to give the corresponding various azo disperse dyes (D13- D18). These dyes were applied to polyester and nylon fabric and their fastness properties were evaluated. Dyes were characterized by IR, elemental analysis, UV-Visible spectral studies. Dixit et al [4] studied on novel mordant and disperse azo dyes were prepared by the coupling of various diazo solutions of aromatic amines with 2,4- dihydroxybenzophenone. The resultant dyes were characterized by elemental analysis as well as IR and NMR spectral studies. Mariam et al [5] studied on new 1,2,4- trizole colorants were obtained, in high yields, by coupling 3-ethylthio-5-cynomethyl-4-phenyl-1,2,4 with diazotized aniline derivatives 2,4 and 6. Vithal et al [6] studied on the synthesis of some novel heterocyclic azo disperse dyes which are derived from 5-acetyl-2-amino-4-methylthiazole (1) using various Nalkyl derivatives of aniline (2a-e) and their dyeing performance disperse dyes have been assessed on polyester and nylon fabrics. The spectral properties of these dyes were measured. Patel et al [7] studied on some novel azo disperse dyes which synthesis by the coupling component of diazonium salt with 1-(4-N-acetyl amino) 2-methyl phenyl 2- chloroethanone. Thus, a series of azo disperse dyes has been prepared. After synthesis compounds were characterized by chemical as well as instrumental methods like Melting point, elemental analysis and UV-Visible spectral studies. Yusuf Lams et al [8] studied on synthesize disperse dyes in the derivative of 2- amino-4-chloro-5-

formylthiazole by conventional diazotization and couplings with pyridone and resorcinol. The dyes were characterized by visible absorption spectroscopy, IR spectral studies, and <sup>1</sup>H and <sup>13</sup>C NMR. The pyridine and resorcinol substituted dyes exhausted well with good depth on 100% polyester fabrics with a shade of brown and purple colours, respectively. In this work we have synthesised Disperse Azo dyes with various combinations of Base & Coupling agents.

### III. MATERIALS & METHODS

The following chemicals have been used in this study,

S No	Name of chemical	Function
1	2,4-Dihydroxybenzophenone	Coupling agent
2	4-Nitroaniline	Base
3	2-Aminophenol	Base
4	4-Chloroaniline	Base
5	3-Chloroaniline	Base
6	Acetic acid	Protecting agent for dyes
7	CHT Dispergator SMS	Dispersing agent
8	Lixatron DFT	Levelling agent

Table 1. Chemicals used for this research

#### 3.1 Diazotization Process:

The process of conversion of primary aromatic amines into its diazonium salt is called diazotization. Diazonium salts are important synthetic intermediates that can undergo coupling reaction to form azo dyes and electrophilic substitution reactions to introduce functional group. The reaction temperature should up to 0-5°C. The mixture is kept for 30min with continuous stirring.

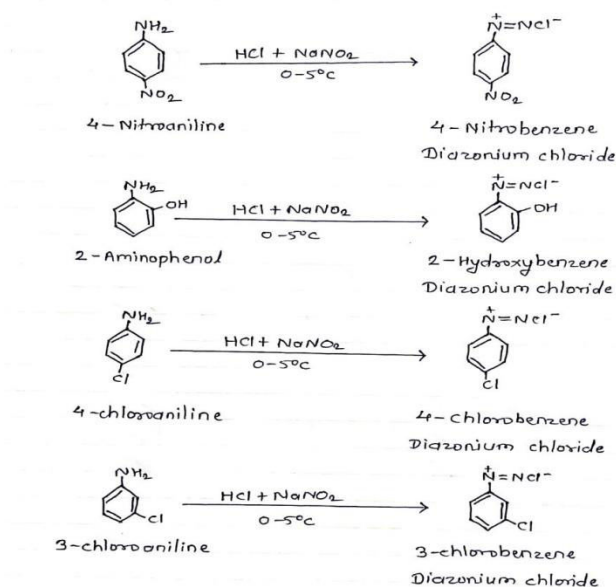


Figure 1. Diazotization Process sequence

#### 3.2 Coupling Process

An azo coupling is an organic reaction between a diazonium compound and another aromatic compound that produces an azo compound. This reaction favours to proceed at lower temperature of 0-5°C. This can be done by pouring diazonium salt solution in coupling agent solution over 30min and mixture is kept for 2hrs with continuous stirring. Then dried and filtered.

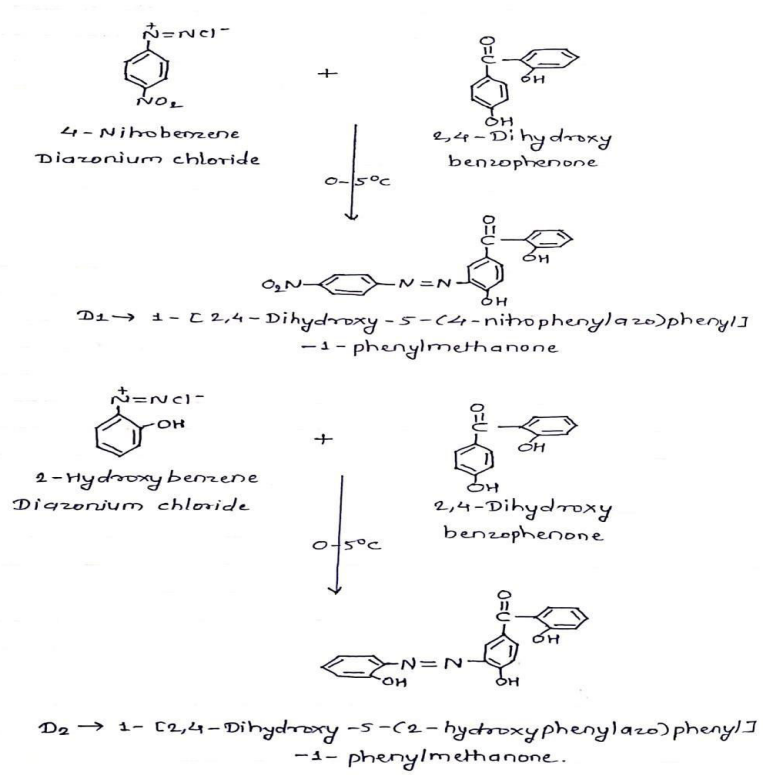


Figure 2. Coupling process sequence

### 3.3 Proportion of base and coupling agent

Proportion of base and coupling agent as follows;

S No	Base: coupling agent
1	1:1
2	1:1
3	1:3
4	2:1
5	2:3
6	3:1
7	3:2

Table 2. Proportion of base and coupling agent

**Base:** 4-Nitroaniline, 2-Aminophenol, 4-Chloroaniline, 3-chloroaniline

**Coupling agent:** 2, 4-Dihydroxybenzophenone

## IV. RESULTS AND DISCUSSION

We have made an attempt to synthesize disperse azo dyes by employing diazotization and coupling reaction. For this work 2, 4-Dihydroxybenzophenone has been used as coupling agent while 4-Nitroaniline, 2-Aminophenol, 4-Chloroaniline, 3-Chloroaniline as bases. Initially 1:1 proportion of base and coupling agent has been selected to synthesize disperse azo dyes.

### 4.1 Synthesize of Disperse azo dyes

In the present work we have synthesize following 4 disperse azo dyes employing 1:1 molar proportion base and coupling agent.

D1 - 4-Nitroaniline + 2, 4-Dihydroxybenzophenone

Dye Name- 1-[2, 4-Dihydroxy-5-(4-nitrophenylazo) phenyl]-1-phenylmethanone

D2 - 2-Aminophenol + 2, 4-Dihydroxybenzophenone

Dye Name- 1-[2,4-Dihydroxy-5-(2-hydroxyphenylazo)phenyl]-1-phenylmethanone

D3 - 4-Chloroaniline + 2, 4-Dihydroxybenzophenone

Dye Name- 1-[2,4-Dihydroxy-5-(4-chlorophenylazo)phenyl]-1-phenylmethanone

D4 - 3-Chloroaniline + 2, 4-Dihydroxybenzophenone

Dye Name- 1-[2,4-Dihydroxy-5-(3-chlorophenylazo)phenyl]-1-phenylmethanone

Properties of these dyes are shown below table;

Sr. No.	Dye	Chemical formula	Molecular weight
1	D1	$C_{19}H_{13}O_5N_3$	363
2	D2	$C_{19}H_{14}O_4N_2$	334
3	D3	$C_{19}H_{13}O_3N_2Cl$	352.5
4	D4	$C_{19}H_{13}O_3N_2Cl$	352.5

Table 3. Properties of Dyes

#### 4.2 Synthesis of disperse azo dyes using various molar proportions of base and coupling agent

To observe the effect of base and coupling agent, we have synthesized 24 disperse azo dyes using various molar proportion of bases and coupling agent. Molar proportion of bases and coupling agent are shown in below table.

Sr. No.	Dye No.	Combination	Proportion
1	Dye 1	D1	1:1
2	Dye 2	D1	1:2
3	Dye 3	D1	1:3
4	Dye 4	D1	2:1
5	Dye 5	D1	2:3
6	Dye 6	D1	3:1
7	Dye 7	D1	3:2
8	Dye 8	D2	1:1
9	Dye 9	D2	1:2
10	Dye 10	D2	1:3
11	Dye 11	D2	2:1
12	Dye 12	D2	2:3
13	Dye 13	D2	3:1
14	Dye 14	D2	3:2
15	Dye 15	D3	1:1
16	Dye 16	D3	1:2
17	Dye 17	D3	1:3
18	Dye 18	D3	2:1
19	Dye 19	D3	2:3
20	Dye 20	D3	3:1
21	Dye 21	D3	3:2
22	Dye 22	D4	1:1
23	Dye 23	D4	1:2
24	Dye 24	D4	1:3
25	Dye 25	D4	2:1
26	Dye 26	D4	2:3
27	Dye 27	D4	3:1
28	Dye 28	D4	3:2

Table 4. molar proportions of base and coupling agent

#### 4.3 Percentage yield of the dyes

Percentage yield of dyes is an important criterion of synthesizing dye. We are estimated percent yield of 28 dyes prepared in lab. The results of percentage yield of dye synthesizing is shown in table no 5. The all prepared 28 dyes yield calculated by following formula,

$$\text{Yield (\%)} = (\text{weight of dye prepared} / \text{Weight of chemical used}) * 100$$

Sr. No.	Dye No.	Yield (%)
1	Dye 1	77.65
2	Dye 2	100
3	Dye 3	93.71
4	Dye 4	43.31
5	Dye 5	100
6	Dye 6	100
7	Dye 7	100
8	Dye 8	61.91
9	Dye 9	80.89
10	Dye 10	79.62
11	Dye 11	52.55
12	Dye 12	43.95
13	Dye 13	70.36
14	Dye 14	79.47
15	Dye 15	84.26
16	Dye 16	86.07
17	Dye 17	83.22
18	Dye 18	78.63
19	Dye 19	80.35
20	Dye 20	72.42
21	Dye 21	84.67
22	Dye 22	70.38
23	Dye 23	100
24	Dye 24	100
25	Dye 25	82.97
26	Dye 26	69.19
27	Dye 27	100
28	Dye 28	96.41

Table 5. Percentage yield of the dyes

Critical analysis of Table No 5, clear that dye 5, 6, 7, 23, 24 and 28 have 100% yield. From this table it has been observed that a combination of 4-Nitroaniline and 2, 4-Dihydroxybenzophenone in the molar proportion of 2:3, 3:1 and 3:2 give 100% yield. The combination of 3-Chloroaniline and 2, 4-Dihydroxybenzophenone at molar ratio proportions 1:2, 1:3 and 3:1 gives 100% yield. In all above combination 100% yield has been observed may be due to cent utilization of base and coupling agent. Further dye 12 gives which is synthesizing by using 2:1 molar proportion of 2-Aminophenol and 2, 4-Dihydroxybenzophenone give lowest yield this may be due to underutilization of base and coupling agent.

#### 4.4 Colour appearance of synthesized dyes

The colour of the various combinations shows tonal change in appearance of dye by visual method. The results obtain shown in below table.

Sr. No.	Dye No.	Appearance
1	D1	Yellowish red
2	D2	Light brown
3	D3	Light orange
4	D4	Light maroon

Table No 6. Colour appearance of dye

Tone of the dye is also important criteria of dyes synthesized from table no.6, it is clear that colour of the dye 28 synthesized dyes is majorly yellowish red, brown and light orange. This may be due to variation in azo group as well as their position which results in variation in amount of nitrogen may present the dye.

#### V. CONCLUSIONS

In the present study an attempt has been made to expose the possibility of synthesizing disperse azo dyes employing diazotization and coupling reaction with various bases and coupling agent in their various molar proportion. Following conclusions have been made,

1. A combination of 4-Nitroaniline and 2, 4-Dihydroxybenzophenone in molar proportion of 2:3, 3:1 and 3:2 give 100% yield.
2. A combination of 3-Chloroaniline and 2, 4-Dihydroxybenzophenone at molar proportion of 1:2, 1:3 and 3:2 give 100% yield.
3. A combination of 3-Chloroaniline and 2, 4-Dihydroxybenzophenone at molar proportion of 1:2, 1:3 and 3:2 give 100% yield.
4. The colours of synthesized dyes are majorly reddish yellow, light maroon, light orange and light brown.

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