



## Waste minimization in manufacturing of Reactive dye

\* J. R. Vadher

Lecturer, Chemical Engineering Department, Govt. Polytechnic, Gandhinagar

### ABSTRACT

There is no denying the fact that dyestuff industry is the major growth driver all around especially in the textile and chemical industries in the country. Reactive dyes are important in dyeing textiles because they are unequal in their ability to confer bright wet fast shades on cotton fabric. India is one of the major supplier of the dyes and dye intermediates from the Asian region and can meet the requirements of the world at large provided adequate incentives and facilities are made available. The dye industry achieved growth rate of 15% during last year. India's share in the world market is estimated at 5-6%. The Share of Gujarat in all Indian export is about 60%. The major markets for Indian dyestuff are the European country, USA, South Korea, Hong Kong. Manufacturer are suffering due to competition in market by the other country like china, so to with stand with them it is necessary to optimize the manufacturing process either Production cost, Raw material cost or Energy cost which is the ultimate way to stand in competitive market. Fiber reactive dyes are commonly employed for textile dyeing; the use of these dyes can introduce high costs and environmental concerns. For example, their fixation levels can be as low as 50% and high salt levels are typically needed to achieve desired shades. Thus, a mechanism for increasing fixation and exhaustion efficiencies in an economical way would enhance the value of these dyes to the textile industry. With these points in mind, studied waste minimization in Manufacturing of Reactive Dye. Specifically, the reactivity and affinity of Reactive Blue (Blue RGB) dye.

**Keywords : Dye, Reactive dye, Fixation, Affinity, Adsorption,**

### 1.0 Introduction

The field of synthetic dye chemistry was now exploding as the increased textile production of the late 19th century industrial revolution increased the demand for inexpensive dyes. Soon Germany would become the leader in the synthetic dye industry as chemists educated by the likes of Liebig and Wohler dominated the synthesis of "coal-tar" dyes. These chemists discovered dozens of new dyes each year which were processed at and shipped from large-scale factories. These improvements in the making of dyes quickly eliminated the production of natural dyes due to their expense and inefficiency as compared to the new synthetic colors.

The synthetic dye industry today is vast and contains many groups of dyeing processes and dyes. From the synthesis of biological stains used in the preparation of microscope slides to the production of acetate rayon dyes and nylon dyes used in the preparation of commercial textiles, the industry continues to develop new processes and dyes to serve the needs and wants of humanity. One area of early synthetic dye chemistry though, azo dyes, remains one of the largest and most important to the industry. The birth of azo dyes came in 1858, the same year Perkin started his factory for the production of mauve, although their value was not appreciated until Bottiger produced congo red, the first direct cotton dye, in 1884. Johann Peter Griess had made the original discovery that a diazo compound could be derived from the reaction of nitrous acid with aromatic amines. Upon experimentation, he further concluded that this diazo compound could couple to another aromatic amine resulting in the formation of a dye. This area of chemistry has been greatly expanded and refined and now includes trisazo, tetrakisazo and polyazo dyes. The arenediazonium ion, containing the  $-N=N-$  chromophore, serves as a weak electrophile which may perform an electrophilic aromatic substitution on an aromatic ring to produce a vast and diverse array of different dyes. Upon referral to the above discussion of the chemistry behind the colors, one can see how these dyes with their great amounts of conjugated  $\Pi$  bonds serve as excellent dyes.

### 2.0 Experimental work

Manufacturing Process for manufacturing of Reactive blue 250 involves five step processes. The raw material requires for that processes are H-Acid, Vinyl Sulphon, Ortho Anicidine Vinyl Sulphon, Hydrochloric acid, sodium nitrite and Sodium Hydroxide. The processes involve are diazotization and coupling.

#### 2.1 First Diazotization:-

Vinyl Sulfone (Acetanilide based) charged to an M.S.R.L. reaction vessel along with water and ice to maintain temperature between 0 to 5 °C. Then Hydrochloric Acid was added followed by Sodium Nitrite powder gradually till diazotization completed, which can be confirmed by starch iodide paper. Starch iodine paper will convert in to purple color if diazotization not complete (if excess nitrite will present in reaction mass). If all nitrite will consumed than there will not any change on iodine paper. Any excess nitrite will be removed by adding Sulfamic Acid just before coupling.

#### 2.2 Prepare Clear Solution of H-Acid:-

H-Acid (1 Hydroxy 8 amino 3, 6 di sulphonic acid) was charged to a M.S.R.L. reaction vessel along with Caustic lye and maintained at Temperature at 15 – 20 °C and pH at 6.5 to 6.8 stir it till clear brown solution appeared.

#### 2.3 First Coupling:-

Prepared clear slurry of H-Acid was charged in to the diazotized vinyl sulfone, and stirred for 6 to 8 hrs keeping the temperature between 0 to 5 °C by adding of ice

#### 2.4 Second Diazotization:-

As mention in first coupling, Vinyl Sulfone (Ortho Anicidine Based) charged to a M.S.R.L. reaction vessel along with water and ice to maintain temperature between 0 to 5 °C. Then Hydrochloric Acid was added followed by Sodium Nitrite powder gradually till diazotization completed, any excess nitrite was removed by adding Sulfamic Acid just before coupling. Keep temperature between 0 to 5 °C throughout the diazotization reaction.

**2.5 Second Coupling:-**

Charge diazo of O.A.V.S. to the first coupling mass and stir for 3 hours keeping temperature 0 to 5 °C pH of the coupling mass was raised by addition of sodium bicarbonate and maintained temperature at 0 to 5 °C by addition of ice. Properties of dye (Strength, tone etc.) was checked before starting spray drying, if any correction needed apply. At last transferred batch to spray dryer.

**2.6 Spray Drying:-**

The standardized dye liquid of reactive Blue 250 is transferred to the spray drying holding tank and spray dry. After drying in to spray dryer the crude dye was packed in plastic bags to avoid contamination from moisture and dust.

As per stoichiometric calculation raw material to be charge for reaction.

**Table: 2.1 Material Balance data sheet as per stoichiometric**

Material In (in Gms.)		Material Out (in Gms.)	
H-Acid (80%)	39.05	Dye + Salt	133.48
V.S (96%)	28.67		
NaNO <sub>2</sub> (100%)	13.52	H <sub>2</sub> O	52.67
OAVS (95%)	32.06	Co <sub>2</sub>	17.24
NaOH (48%)	16.32		
Sodium Bi carbonates (98%)	32.91		
HCl (35%)	40.86		
	203.39		203.39

For Experimental purpose material was charged as per table and than finished product was compared for various parameter with standard test dye sample

**Table: 2.2 Material Balance data sheet For Experiment**

Material In (in Gms.)		Material Out (in Gms.)	
H-Acid (80%)	39.05	Dye + Salt	126.00
V.S (96%)	28.67		
NaNO <sub>2</sub> (100%)	13.52	H <sub>2</sub> O	52.67
OAVS (96%)	31.73	CO <sub>2</sub>	17.24
NaOH (48%)	16.32	Diff in Material	0.84
Sodium Bi carbonates (98%)	26.60		
HCl (35%)	40.86		
	196.75		196.75

**3.0 Result and Discussion :**

After performing the experiment the crude dye is tested in the liquid as well as in powder form with and without dying on cotton. The test method for prepared dye is tested for its strength in liquid form by its simadzu value tested by UV 1601 Spectrophotometer with standard sample.

After drying into oven the dye in powder form is used for dying on cotton of 2% shade (2 Gms of dye per 100 Gms of cotton). Before dying, the same powder is also checked for strength as per above method. The dye powder is diluted in CPTLAB solution Maker 2.5 instrument, which itself dilutes dye as per standard method, So that solution prepared is perfect and up to mark.

**BLUE RGB LAB EXPERIMENT**

**Dvine Report**

Name of : Greatg Macbeth  
 Instrument : Spectrophotometer  
 STANDARD: Lab Batch Standard.

BATCH : 1

FORMULA : CIEL\*a\*b                      Obs. : 10                      Tolerance : 1

ILLUM:	PASS/FAIL	DE	DL*	Da*	Db*
D65	Fail	3.85	-3.81 Darker	0.45 Less Green	0.21 Less Blue
TL84	Fail	3.98	-3.88 Darker	0.90 Less Green	0.13 Less Blue
A	Fail	3.82	-3.78 Darker	0.51 Less Green	0.24 Less Blue

Strength : Min                      Strength Value : 1.31                      Res. DE : 0.49

Difference at equal Strength:( formula : CIEL\*a\*b)                      Obs : 10                      Illum : D65

DE*	DL*	Da*	Db*	DC*	DH*
0.49	-0.09	-0.14	-0.46	0.48	0.09

## Relative Strength Analysis Report

Name of Instrument : AGS- NOVASCAN  
Spectrophotometer : CE 7000

Color Space : CIE Based on User Wavelength  
Illuminant : D65 Obs. Degree : 10

ID Name	WL nm	K/S	Strength in %	Strength in Parts
Blue RGB	560	0.394	100	100
Exp 1	560	0.391	99.211	100.795

## Color difference on strength Adjustment to 100%

Batch Name	dE	dL	da	db	dC	dH
EXP 1	4.854	0.365	-4.26	-1.06	3.025	3.254

## Color difference as it Exists

Batch Name	dE	dL	da	db	dC	dH
EXP 1	4.92	0.462	-4.325	-0.952	3.011	3.251

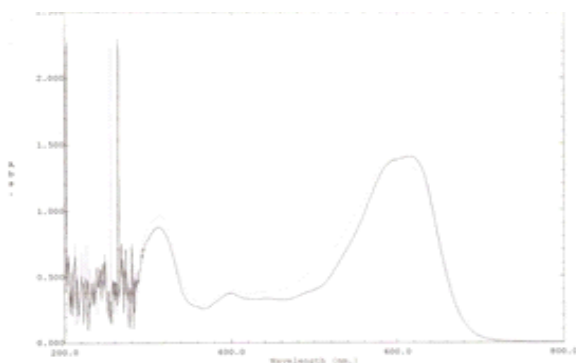


Figure: 3.1 Nova scan report of experiment

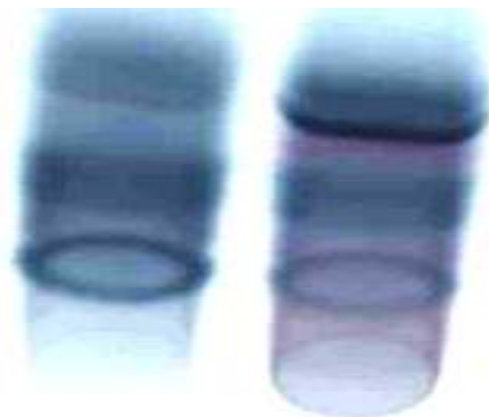


Figure : 3.2 TLC report at 2 % of shade

Nova Scan report was carried out with UV 1601 spectrophotometer. The comparison of both samples in graphical nature (absorption vs. wavelength) was plotted.

The visible spectrum of the blue dye was ranging from 300nm to 750 nm. In this range absorption of light rays was plotted against wavelength of standard sample and experimental batch. Comparison of both is shown in the above figure.

If absorption of sample was low that means its strength was low. For better results absorption of experimental sample must be higher or equal than standard sample.

After checking result of above experiment dyeing report was not good compared to standard sample, here permissible tolerance limit for DE was set to 1.00 and result obtained for this was more than 3.00, colour tone was not as per standard. Here colour tone was diverting to reddish side. Dye of experiment was 10 % weak compared to standard. It was found by checking TLC report that first coupling was not proper because red dye was separated on it. As per stoichiometry, quantity of Bi-Carbonate required for neutralization of second coupling mass was 32.91 gms., but in experiment I consumption of Bi-carbonate was 26.60 gms., so that Acid formed during second coupling was less. Following are the possible reason for incomplete reaction

1. Purity of raw material.
2. Insufficient reaction time.
3. Temperature and pH of the reaction

#### 4.0 Conclusion :

Following modification were made during next experiment.

1. Time given for coupling reaction was increased.
2. NaOH was added drop by drop to solution of H-Acid to prepare clear brown solution of Sodium salt of H-Acid of pH 6.5 – 6.8.

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