Research Paper

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Optimization Process in manufacturing of Reactive dye

Engineering

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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 unequally 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 dying; 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 dye.

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 dying 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 Π 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 0C. 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 – 20^0 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 ^{0}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 ^{0}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 ^{0}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 0C pH of the coupling mass was raised by addition of sodium bicarbonate and maintained temperature at 0 to 5 0C 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.

3.0 Experimental Work:

Experimental work for this study is done at R& D department of Kiri Dyes and Chemical Ltd. At Vatva. Before taking any raw material for use it was tested in quality control laboratory of the industry. After Preparation of dye dying on cotton is done as per standard procedure. All testing of dyeand dyed cotton done with different instrument as per

Table 3.1 Material Balance

	Materials input	Materials output									
	H-Acid	V.S	$NaNO_2$	OAVS	NaOH	Na2CO3	HCl	Dye + Salt	H_2O	CO_2	ANY OTHE R
Existing Process	39.05	28.67	13.52	32.06	16.32	32.91	40.86	133.48	52.67	17.24	
Exp.-1	39.05	28.67	13.52	31.73	16.32	26.60	40.86	126.00	52.67	17.24	0.84
Exp.-2	39.05	28.67	13.52	31.73	9.72	27.00	40.85	120.00	46.34	24.19	
Exp.-3	38.27	28.67	13.78	32.06	10.62	33.00	40.85	126.00	45.79	24.99	
Exp.-4	37.49	28.67	13.78	32.06	10.34	33.00	40.85	125.00	45.86	24.85	
Exp.-5	37.49	28.67	13.52	32.06	8.73	32.91	28.12	131.92	32.35	17.24	
Exp.-6	374.93	286.69	135.16	320.64	87.32	329.09	281.23	1319.17	323.49	172.38	

4.0 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.

Table : 4.1 Result Data

EXP NO.	PRODUC	STRENGT	RESULT	DE	DL*	Da*	Db*
Existing Process	133.48	1		0.56	-0.46	0.33	-0.19
1	126	1.31	FAIL	3.85	-3.81	0.45	0.21
2	120	0.89	FAIL	1.6	1.4	-0.20	-0.74
3	126	1.02	OK	0.59	0.4	0.15	-0.4
4	125	1.04	OK	0.59	-0.56	0.16	-0.13
5	131.92	1.03	OK	0.55	-0.42	0.33	0.12
6	1319.17	1.01	OK	0.54	-0.38	0.31	-0.04

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.

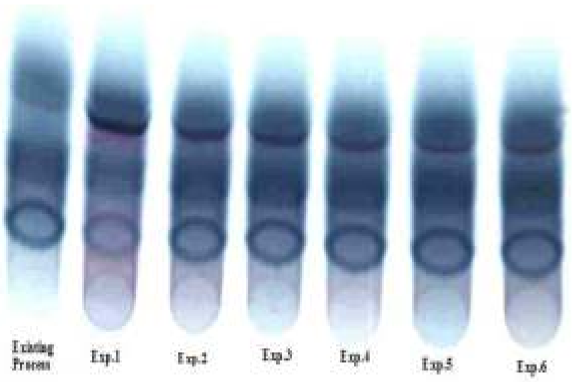

Figure 4.1: THIN LAYER CHROMATOGRAPHY REPORT OF 2% DYE SOLUTION

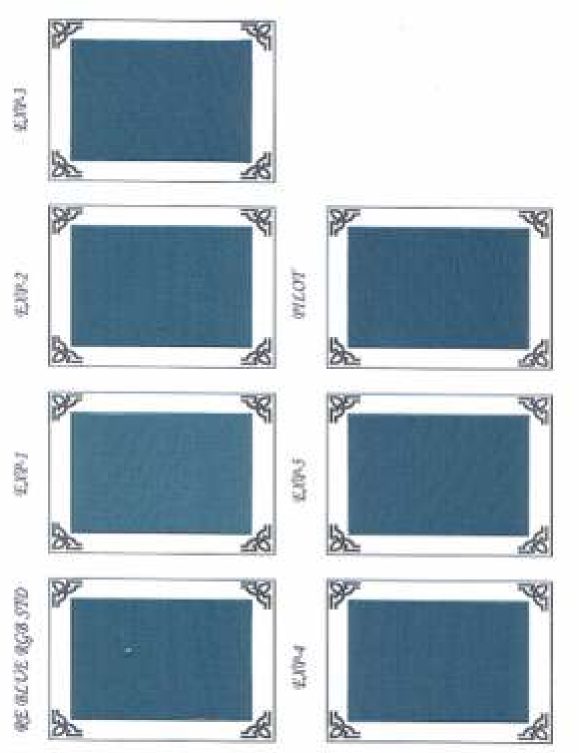

Figure 4.2 : Shade card for dying of 2% of concentration

After checking result of first experiment dying report was not good compared to standard sample, tone was not as per standard. Here colour tone was diverting to reddish side. After checking result of experiment II dying report was still not good compared to standard sample. Here tolerance limit for DE was 1.00 and result obtained for this was more than 1.00, colour tone was as per standard. By checking TLC report it was found that some red dye has remained untreated on TLC plate. To keep formation of red dye under control, quantity of H-Acid should be taken less compared to stoichometric requirement. After checking result of experiment III the dying report was ok, tone slightly differed as per standard. Strength value was 1.02 means exhaustion and fixation of dye was good as per standard sample. As checking TLC report, it was very near to standard sample. After performing experiment IV and checking result of it, the result showed excellent improvement as compared to standard sample, so we took another experiment V in which we carried out diazotization reaction by adding nitrite and HCl slowly and checking the completion of reaction by Congo and SI test. (Congo paper will convert from red to blue in presence of acid and Starch iodine paper change from colourless to violet in presence of nitrite). By this experiment it was found that in diazotization reaction of V.S. and O.A.V.S, for 100% consumption of sodium nitrite, requirement of HCl was less compared to stoichometric amount. Thus this excess acid required more Bicarbonate for neutralization. Finally this neutralized mass form more salt in the dye. The presence of excess salt in dye would reduce the strength, exhaustion and fixation property of dye.

5.0 Conclusion

After performing the experiments and checking the results, the quantity of HCl was reduced to 31% and quantity of NaOH was reduced to approximately 50 % as per stoichometric requirement. The quantity of acid and alkali charged was excess compared to stoichometric requirement, so this excess acid and alkali may formed more salt in the reaction mass. Ultimately, reducing the strength of dye.

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