Research Paper

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Cleaner Production in Dye Industries

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ABSTRACT

To reduce the manufacturing cost of dye process as well as to reduce salt formation during neutralization of dye. In preliminary studies, laboratory-scale preparations were conducted to further investigate the color strength relationships between the modified and commercial dyes. An optimized procedure was developed for the application of the dye. It is clear that high fixation levels and deep shades are obtained using the modified dye at lower cost and salt levels than commonly employed for the commercial dyes. 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. The dye industry achieved growth rate of 15% during last year. The Share of Gujarat in all Indian export is about 60%. To achieve the above mentioned thing Cleaner Production is applied. Cleaner Production means economic savings from reduced consumption of raw materials and energy and lower treatment costs as well as other benefits such as a better company image and better working conditions. Fiber reactive dyes are commonly employed for textile dying; the use of these dyes can introduce high costs and environmental concerns.

1.Introduction

Humanity, from antiquity to modernity, has carried a wish to use dyes as a way to symbolize or beautify. From the ancient Egyptian use of the madder and saffron plants to dye cloth for burial wrappings to the modern employment of dyes in the clothing industry, dyes have maintained their status as one of the most prevalent and widespread examples of applied chemical technology. Although some of the most ancient pigments were natural minerals, such as red lead oxide made by the Egyptians from the heating of lead with basic lead carbonate. A brief discussion of the chemistry behind the colors ensues and will proceed to an overview of the development of dye chemistry throughout the ages.

The visible spectrum of light consists of radiation of wavelengths between approximately 350 to 780 nm. Absorptions of light by a molecule for these wavelengths require the high energy of electronic transitions in a molecule. While sigma bonds are generally not affected by light with wavelengths in the visible region, electrons in bonds are more susceptible to promotion into a higher-energy orbital. Certain chromophores, or functional groups involved in absorption, such as conjugated bond systems, tend to have greater absorption in the visible region. The Woodward-Fieser rules summarize these tendencies and explain why dyes tend to be large conjugated molecules. With this very limited introduction to the chemistry of dyes, we are now ready to examine the rich history of dyes and dying. We now know that the dye from this plant is alizarin, which was synthesized in 1869 both by William Perkin and Heinrich Caro in independent laboratories. Until Perkin's breakthrough synthesis of the "coal-tar" dye mauve, which will be discussed in length below, most dyes came from natural sources. Some exceptions, though, are Runge's synthesis of a urine from phenol in 1834, and Laurent's successful conversion of phenol into picric acid in 1842. These natural dyes often utilized a mordant to improve color fastness. A mordant can simply be described as a "metallic salt with an affinity for both fibers and dyestuffs." Traditionally the focus of environment protection efforts has been upon setting up pollution control equipment to treat liquid effluents or gaseous emissions and bringing down the concentration of pollutants to within the stipulated limits. For most of the SME's this is just not feasible on account of poor profitability, space constraints etc.Realizing these drawbacks and looking into further aggravating problems as the pollutant assimilation capacity of the receiving bodies nearing exhaustion, the concerned agencies were forced to go backward in the industrial production processes and there emerged the concept of proactive approach of waste reduction at source in environment protection. In other words, Cleaner Production (CP) was realized the need of the day.

2. 0 MANUFACTURING PROCESS OF DYE:

First Diazotization:-

Pera nitro aniline(PNA) 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 Sulfuric Acid just before coupling.

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 – 200 C and pH at 6.5 to 6.8 stir it till clear brown solution appeared.

First Coupling: Prepared clear slurry of H-Acid was charged in to the diazotized PNA and stirred for 6 to 8 hrs keeping the temperature between 0 to 5^{0}C by adding of ice.

Second Diazotization:- As mention in first coupling, DASA (Diamine Sulfanilic acid) 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 0C throughout the diazotization reaction.

Second Coupling: Charge tetra azo of DASA. to the first coupling mass and stir for 8-10 hours keeping temperature 0 to 5^{0}C pH of the coupling mass was raised by addition of sodium hydroxyde and maintained temperature at 8 to 12 ^{0}C by addition of ice. Third Coupling:Prepare MPD(Meta Phenyl Diamine) solution with adding water and heat upto 500C. Then add MPD solution into second coupling mass. Stir for 2 hours and check pH. After completion of reaction heat the final mass upto 55^{0}C.

Filtration and Reverse Osmosis:-

After completion of third coupling reaction, reactant mass has some insoluble solid impurities like unreacted mass and polythene(due to solid material charging) which can be removed by filter press. Acid dye is soluble in the water so it comes in a liquid phase.

The concentration of acid black 210 is approximately 10% after the filtration and which is increased upto 28-30% by using reverse osmosis operation. In reverse osmosis operation, the light molecular weight substances is removed with a water stream and concentrated mass is go for spray drying.

Spray Drying:-
The standardized dye liquid of acid black 210 dye is transferred to the spray drying holding tank and spray dry. During spray drying the solid crude dye particle are collected at the bottom of the cyclone separator and they are removed at the bottom of cyclone separator with the help of rotary valve and through conveyor they are dumped into the blender. After blending in to ribbon blender the crude dye was packed in plastic bags to avoid contamination from moisture and dust.

Typical procedure for laboratory dying:
The following procedure is commonly used for a commercial reactive dye application today. This procedure is for a 2.0% exhaust dyeing on 100% nylon:

1. Fill dye container with water
2. Add cloth
3. Add pre-dissolved dye
4. Add 70 g/L common salt
5. Raise temperature 2 °C per minute to 105 °C
6. Run for 15 min
7. Add 10 g/L acetic acid
8. Run for 45 min
9. Drop bath
10. Rinse hot for 2 min and drop bath
11. Soap at 105 0C for 15 min and drop bath
12. Rinse cool and unload

2.1 Experimental Data:-
As described earlier experimental methodology is same for all this experiment. Material charged and Product recovered is listed here in the following table, As per Stoichiometric of the reaction the raw material required as per purity listed as shown in the following table: Each batch is taken of 100 Gms of Acid black 210 dye.

Table: 2.1 Material Balance data sheet

Material input(in Gms)					Material output(in Gms)		
	STD	EXP-1	EXP-2		STD	EXP-1	EXP-2
PNA	15.24	12.4	14	Dye + Salt	142.34	132.58	137.72
H-acid(80%)	44.05	44.05	44.05				
NaOH(48%)	36.81	32	36.81	H_2O	95.46	101.57	97.74
NaNO2	22.87	22.87	22.87	HCl	4.03		
HCl(35%)	80.63	80.63	75.53				
DASA(96%)	30.27	30.27	30.27				
MPD	11.93	11.93	11.93				
	241.8	234.15	235.46		241.8	234.15	235.46

3.0 Result Data Sheet:
EXPERIMENT 1:

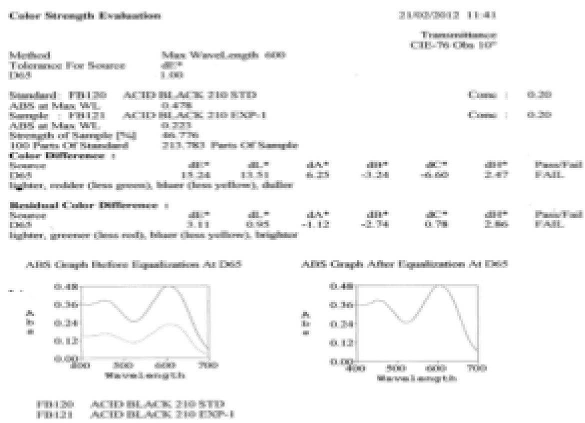

Figure 3.1Colour strength evaluation report

EXPERIMENT 2

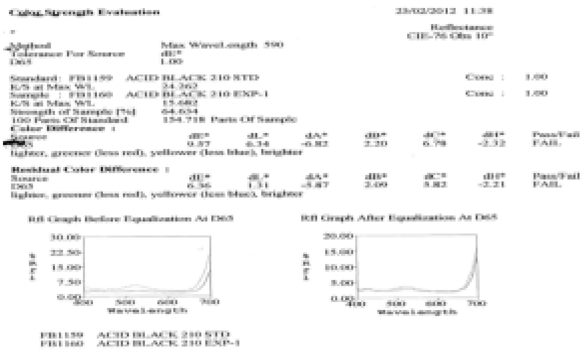

Figure 3.2: Colour strength evaluation report

After performing experiment and checking result of it, the result showed excellent improvement as compared to standard sample, so we took another experiment 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 PNA and DASA for 100% consumption of sodium nitrite, requirement of HCl was less compared to stoichiometric amount. Thus this excess acid required more caustic soda 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. The other raw material like PNA, DASA, MPD are act as colour donor group so if we reduced the quantity of them than we could not get the exact colour tone which we required.

4.0 CONCLUSION:
After verifying results, discussion and cost analysis of modified method we conclude that modified method is best both qualitatively and economically. Here quality of modified dye is good with less salt content. The production in modified method is reduced to 1.18% but on the other side quality of the dye is improved. Which is handsome amount. So, finally we conclude that modified method is definitely good in quality from the economic point of view. In modified method exhaustion and fixation properties were increased, So maximum quantity of dye is exhausted, hence colored waste generation in wash water is reduced; thus environmental point of view modified dye more environmental friendly.

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