Increasing Yield in the Manufacturing of MPDSA

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Abstract— Meta phenylenediamine 4 sulphonic acid (MPDSA) is one of the important products in the dyestuff sector. The present conventional manufacturing process for MPDSA being carried out at the industry is less efficient and gives an overall yield of about 60 %. But the use of alternative raw materials for the manufacture of MPDSA can give yield as high as upto 80%. In addition, this process also eliminates the use of metal catalysts which cause downstream problems. This method involves the use Metaphenylene Diamine as the raw material instead of 2, 4, dinitrochlorobenzene for the manufacture of MPDSA.

A lab scale experiment has been carried out and the overall yield has been found to be higher than that obtained from the conventional process. This paper describes this new manufacturing process and its possible economic benefits. Overall yield is calculated and compared with the conventional process.

Key words – Meta phenylene diamine 4 Sulphonic acid, Oleum 23%, Oleum65%

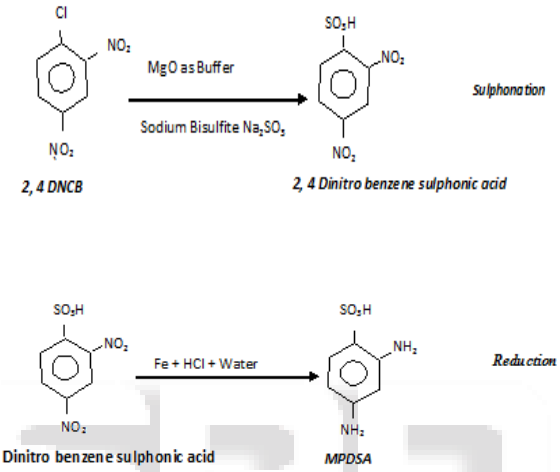
I. INTRODUCTION

Dye Intermediates: The precursors of dyes are called dye intermediates. They are obtained from simple raw materials, such as benzene and naphthalene, by a variety of chemical reactions.

Usually, the raw materials are cyclic aromatic compounds, but acyclic precursors are used to synthesize heterocyclic intermediate acid which is a by-product of the process. Are derived from two principal sources, coal tar and petroleum. [1]

The first synthetic dye, Mauveine, was discovered by Perkin in 1856. Hence the dyestuffs industry can rightly be described as mature. However, it remains a vibrant, challenging industry requiring a continuous stream of new products because of the quickly changing world in which we live. The early dyes industry saw the discovery of the principal dye chromo gens (the basic arrangement of atoms responsible for the color of a dye). Indeed apart from one or two notable exceptions, all the dye types used today were discovered in the nineteenth century. The introduction of the synthetic fibers, nylon, polyester, and polyacrylonitrile during the period 1930-1950. [2] sativa *L)*. Section 3 talks about the materials and methods proposed for calculating parameters for the quality of rice seeds (Oryza sativa *L)*.The proposed system and proposed algorithm for computing Rice seed (Oryza sativa *L)*with long seed as well as small seed being present in the sample is also discussed in the same section. Section 4 discusses the quantification for the quality of rice seeds based on image processing and analysis. Section 5 discusses results based on quality analysis. Section 6 provides the conclusion of the proposed process.

II. MANUFACTURING PROCESS IN PLANT



A. *Process Step:-*

1) Sulphonation Process:

1000 kg 2, 4 DNCB is partially sulfonated with 625 kg sodium bisulphite in presence of 150 kg magnesium oxide in reaction vessel under constant stirring condition.

PH is maintained at 7 to7.5 and temperature maintained at 52°C for 8 hour.

After Cooling, adding 1700 kg salt, filter, centrifugal, get 1200 kg 2, 4 dinitrobenzene sulphonic acid (Nitro mass).

2) Reduction of nitro mass:

- 1200 Kg 2, 4-dinitrobenzenesulfonic acid is taken into the reduction vessel which contains 1100 kg scrap iron and hydrochloric acid. After boiling, maintain temperature at 99° C
- After reaction completion, mass is sent to a filter press
- Finally, Centrifugal and drying gives 600 kg MPDSA.

III. YIELD IMPROVEMENT METHOD FROM LITERATURE.

(1) Use of Agitated Nutche Filter by Equipment changing.

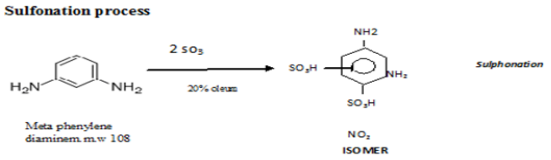
(2) Use of spray dryer with scrubber

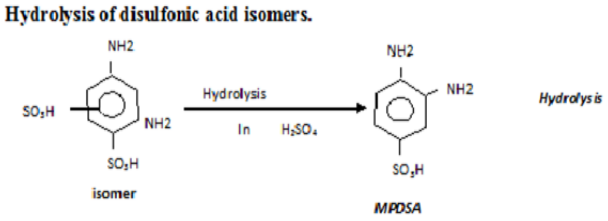
(3) Using Different catalyst instead of Fe (iron powder) in reduction process. Like Zno (Zinc oxide), platinum, palladium, Raney nickel, polysulfide (caustic+sulfer) etc. can be used. [11]

(4) Recycle of mother liquor for reduction process

(5) Raw material changes process modification

IV. IMPROVING YIELD BY PROCESS MODIFICATION





Principal:-Solid m phenylene diamine (MPD), about 98-99 %.is sulfonated with 20% Oleum to m-phenylene Diamine 4-sulfonic acid and partly to some 4:5 and 4:6 isomer which are subsequently hydrolyzed by boiling with weekend sulfuric acid to monosulfonic acid, thus resulting in the formation of the objective compound.[13]

The hydrolysis batch is finally drowned into an ice water mixture and the precipitated product is filtered thought nutch filter the wet product is further sucked in hydro extractor to raise the solid contents. The centrifuged cakes are finally evaluated for real content and the yield calculated.

The product is used or solid in form of wet cake**.** [4]

V. FIGURE: EXPERIMENTAL SETUP

Apparatus:

Thermometer, Borosilicate glass 1000ml sspl 3 neck flask, (round bottom flask.),Separation funnel, Magnetic stirrer, Beaker, Flask, funnel, Heater mantle.

Raw material:

1 Mpd= 70 gm

2. 98% H_2SO_4 =118 ml

3. 25% Oleum=40 ml

4. 60% Oleum=100 ml

5. Common solt =16 gm

STEP:-

1) Take a 1000 ml capacity of 3 neck flask (Round bottom flask) having stirrer with connected with electric motor.

2) Adding 118ml of 98% sulfuric acid and 40 ml of 25% Oleum in round bottom flask. In under Stirring condition.

3) After 23% Oleum addition, gradually add 70 gm real Dry m phenylene diamine

(M..Wt. 108.0) over a period of about 1 hour, maintain the temperature between 85°C -90 °C.

4) Then add to 100 of 60% Oleum over a period of 1 ½ hour.

Heat the Sulphonation mass to 156°C during about 4 hours. And maintain it for 3 hour. Now Sulphonation is positively complete.

5) Now cool the mass to 88°C by adding ice externally.

Hydrolysis of disulfonic acid isomer

6) Now charge 18 ml water drop by drop to the sulfonation mass for 1 hour.

7) Maintaining the temp between 85°C to 90°C

8) Now heat the mass gradually 135°C for 4 hour, and then cool the mass to 85 °C

9) Take a separate beaker and add 120 ml water and 480 gm of ice.

10) Then transfer the content from the sulfonater to beaker at that time temperature is 40°C to 50 °C

11) Cool the batch 30 °C to 2hour gradually and further 18°C in a 4 hour.

Filtration:-

12) Filter the batch and wash the cakes by 60 ml of as is 25% weight/vol brine. Centrifuge the wet cakes to further suck down the liquor. Weight the centrifuge wet cakes and record.

13) Check sample of centrifuge wet cake and determine its real content calculation and record yield.

VI. RESULTS

Weight the centrifuge wet cakes :- (Total cake mass)

= 185 gm

Moisture =40% *= 77.6 gm*

Dry mass *= 107.4gm*

UN reacted MPD=**8%** *= 14.8 gm*

MPDSA REAL *= 92.6 gm.*

- *Calculation of yield :-*
  - Initially weight taken of m-phenylene diamine *70 gm*

- According to that moles of m-phenylene diamine *0.6481 mole*
- After that, for step-2 disulfonic acid isomer is send for hydrolysis, and at final end product produce is Meta phenylene diamine sulfonic acid.
- Total weight of MPDSA product is *92.6gm*
- Molecular weight of MPDSA is *188gm/mol*
- So, total mole of MPDSA product is *0.4925* moles.

$$\text{MPDSA \%Yield} = \frac{\text{Moles of MPD reacted to produce (moles of MPDSA)}}{\text{Total moles of MPD initially}}$$

$$= \frac{0.4925}{0.6481} * 100$$

ANS= *75.99% yield*

Comparison of exp results with plant data

1) Yield of MPDSA:

Process	*Yield*
Plant	60%
Experiment	76%

VII. CONCLUSION

In company, we get yield of MPDSA as 60%. In this experiment, Meta phenylene diamine was used instead of 2, 4 dinitrochlorobenzene as raw material and the yield was found to be about 75%. The yield can be further increased to 85% by allowing Sulphonation to occur for 2 more hours.

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