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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.

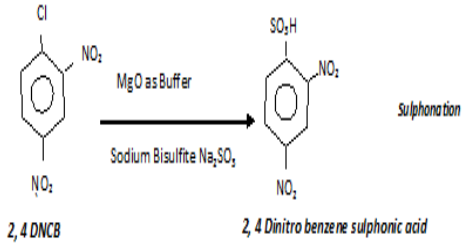
Key words – meta phenylene diamine 4 Sulphonic acid,, Oleum 23%, Oleum65%

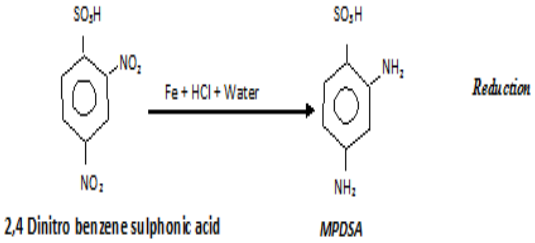
1. Introduction

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 intermediat acid which is a by product of the process. are derived from two principal sources, coal tar and petroleum.Intermediates may be conveniently divided into primary intermediates (primaries) and dye intermediates. Large amounts of inorganic materials are consumed in both intermediates and dyes manufacture. [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]

2. Manufacturing Process in plant.





Process Step:-

1) **Sulphonation Process:**

Take 1000 kg 2,4 DNCB can make partial sulfonation reaction with 625kg sodium by sulphite in presence of 150 kg magnesium oxide in Under stirring reaction vessel. P^H maintaining at 7 to 7.5 and Temperature Maintain at 8 hour in 52°C.

After cooling through, adding 1700 kg salt, cooling. Filter, centrifugal, Dehydration, get 1200kg 2,4 dinitrobenzene sulphonic acid(Nitro mass)

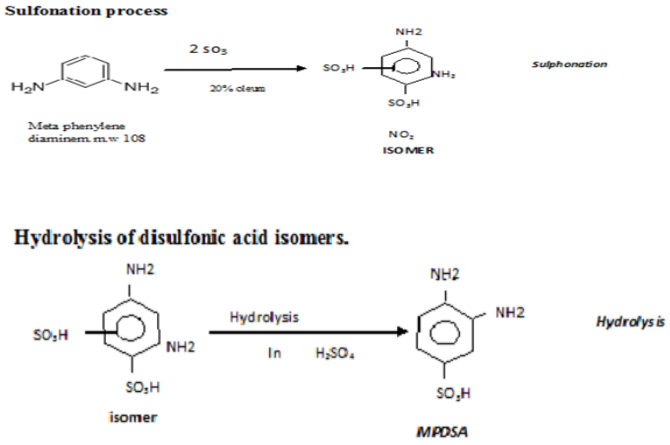
2) Reduction of nitro mass:

Take 1200kg 2, 4-dinitrobenzenesulfonic acid into the reduction vessel which contains 1100kg scrap iron and

hydrochloric acid. After boiling, maintain it 99°C ,whenever this lot is clear we checking by spot paper ,the spot is o.k. then going for a filter press then reactions with HCL salt ,then centrifuges it dry it and packing .t It is saleable MPDSA. Centrifuge or nutch water collect at collection tank, we get 600 kg MPDSA. Second step filtration in after liquor take for second batch.

Take out from filter press is iron oxide 600 kgs. It is packed in HDPE bags and store in factory then after dispatch at NEPL other site. [3]

3. 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 hydrolysed 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]

4. 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= 336 gm
2. 98% H_2SO_4 =590 ml
3. 25% Oleum=199 ml
4. 60% Oleum=380 ml
5. common solt =20 gm

STEP:-

1) Take a 1000 ml capacity of 3 neck flask (Round bottom flask) having stirrer with connected with electric motor.
2) Adding
   - 590ml of 98% sulfuric acid and
   - 199 ml of 25% Oleum in round bottom flask. In under Stirring condition.
3) After 23% Oleum addition, gradually add 336gm 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 380ml of 60% Oleum over a period of 1 ½ hour.

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

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

Hydrolysis of disulfonic acid isomer

6) Now charge 88 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 600 ml water and 2300 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 300 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

RESULT:

Weight the centrifuge wet cakes :-(Total cake mass) = **910 gm**

Moisture =40% = **364 gm**

Dry mass **= 546gm**

UN reacted MPD=**10%** = **91 gm**

MPDSA REAL = **455 gm**.

- **Calculation of yield :-**
  - Initially weight taken of m-phenylene diamine **336gm**
  - According to that moles of m-phenylene diamine **3.1 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 **455gm**
  - Molecular weight of MPDSA is **188gm/mol**
  - So, total mole of MPDSA product is **2.42** moles.

Moles of MPD reacted to produce

$$\text{MPDSA \%Yield} = \frac{\text{(moles of MPDSA)}}{\text{Total moles of MPD initially}} = \frac{2.42}{3.11} * 100$$

= **77 % yield**

Comparison of exp results with plant data

1) Yield of MPDSA:

Process	**Yield**
Plant	60%
Experiment	77%

5. Conclusion

In company get product of MPDSA yield is 60%. And by Performing this experiment by raw material changes like meta phenylene diamine instead of 2,4 dinitrichlorobenzene get yield is 77%,when some changes like maintaining process ,we will also increasing yield.

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