The International Journal Of Engineering And Science (IJES)
||Volume||2 ||Issue|| 4 ||Pages|| 22-27 ||2013||
ISSN(e): 2319 – 1813 ISSN(p): 2319 – 1805

Liquid Waste Minimization in the Production of 4-Amino Di Phenylamine 2-Sulfonic Acid

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Abstract
minimizing waste is a sustainable and environment efficient process for any company to produce any product. In dyes and dye intermediates company production of liquid waste is a major issue. In production of 4-amino diphenyl amine 2-sulfonic acid liquid waste generated in large quantity is reduced by recycle of mother liquor which is generated during reduction process of $R\text{-}NO_2$ to $R\text{-}NH_2$. The inprocess recycle of acidic effluent will reduce the quantity of acidic effluent and reduce the load of ETP plant. Recycle process is associated during pH reducing after reduction process. This cleaner production process leads to the economic efficient product.

Keywords: *pH maintaining, waste minimizing, recycle, reduction*

Date Of Submission: 26 March 2013 — Date Of Publication: 15April 2013

I. INTRODUCTION OF WASTE MINIMIZATION

Traditionally, waste is viewed as an unnecessary element arising from the activities of any industry. In reality, waste is a misplaced resource, existing at a wrong place at a wrong time. Waste is also the inefficient use of utilities such as electricity, water, and fuel, which are often considered unavoidable overheads. The costs of these wastes are generally underestimated by managers. It is important to realize that the cost of waste is not only the cost of waste disposal, but also other costs such as:

- Disposal cost
- Inefficient energy use cost
- Purchase cost of wasted raw material
- Production cost for the waste material
- Management time spent on waste material
- Lost revenue for what could have been a product instead of waste
- Potential liabilities due to waste.

II. WHAT IS WASTE MINIMIZATION?

Waste minimization can be defined as "systematically reducing waste at source". It means:

- Prevention and/or reduction of waste generated
- Efficient use of raw materials and packaging
- Efficient use of fuel, electricity and water
- Improving the quality of waste generated to facilitate recycling and/or reduce hazard
- Encouraging re-use, recycling and recovery.

Waste minimization is also known by other terms such as waste reduction, pollution prevention, source reduction and cleaner technology. It makes use of managerial and/or technical interventions to make industrial operations inherently pollution free. It should be also clearly understood that waste minimization, however attractive, is not a panacea for all environmental problems and may have to be supported by conventional treatment/disposal solutions. Waste minimization is best practiced by reducing the generation of waste at the source itself. After exhausting the source reduction opportunities, attempts should be made to recycle the waste within the unit. Finally, modification or reformulation of products so as to manufacture it with least waste generation should be considered.

III. BENEFITS OF WASTE MINIMIZATION [16]

Reducing waste is good for reducing all three of the triple bottom lines of:

- environment
- financial (the project budget and your business)
- social/community impact

Environmental

- Conserve space in existing landfills and clean fills and reduce the need for future landfills and clean fills.
- Reduce pollution and energy consumption associated with the manufacture of new materials.
- For destructive demolition, reduce other impacts (e.g. noise, dust and traffic) on surrounding properties and streets.

Financial

- For construction, more efficient use of products means reduced costs of purchasing new materials.
- Improve work efficiencies through accurate detailed design, a focus on reducing rework, temporary works and mistakes.
- Reduce waste disposal costs.
- Win contracts for projects that specify waste reduction procedures.
- Improve productivity of staff.

Social

- Minimise the effect of hazardous or nuisance wastes on the community by sound management.
- Innovation and challenges (for example, the training and skills required for deconstruction) can help to attract and retain employees who are keen to develop skills.
- A high level of client satisfaction could enhance your company's image and encourage repeat business.
- Improve site safety through better waste management.
- For demolition, reduce risks from hazardous materials due to more careful dismantling techniques and correct removal and disposal (e.g. asbestos).

IV. CLASSIFICATION OF WASTE MINIMIZATION TECHNIQUES

The waste minimization is based on different techniques. These techniques are classified as hereunder.

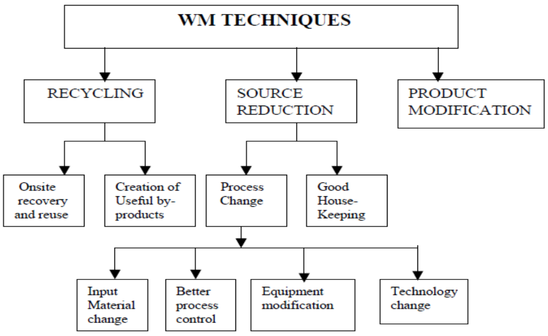


Fig. No. 1 flow diagrammed of waste minimization technique

4.1 Source Reduction

Under this category, four techniques of WM are briefly discussed below:

a) **Good Housekeeping-** Systems to prevent leakages & spillages through preventive maintenance schedules and routine equipment inspections. Also, well-written working instructions, supervision, awareness and regular training of workforce would facilitate good housekeeping.

b) **Process Change**: Under this head, four CP techniques are covered:

- Input Material Change - Substitution of input materials by eco-friendly (non-toxic or less toxic than existing and renewable) material preferably having longer service time.
- Better Process Control - Modifications of the working procedures, machine-operating instructions and process record keeping in order to run the processes at higher efficiency and with lower waste generation and emissions.

- Equipment Modification - Modification of existing production equipment and utilities, for instance, by the addition of measuring and controlling devices, in order to run the processes at higher efficiency and lower waste and emission generation rates.
- Technology Change - Replacement of the technology, processing sequence and/or synthesis route, in order to minimise waste and emission generation during production.

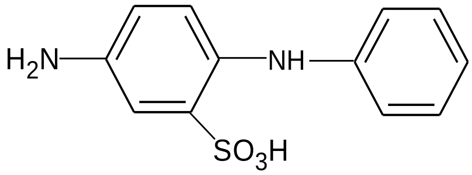
c**) Recycling**

- On-site Recovery and Reuse - Reuse of wasted materials in the same process or for another useful application within the industry.
- Production of Useful by-product - Modification of the waste generation process in order to transform the wasted material into a material that can be reused or recycled for another application within or outside the company.

d**) Product Modification**

Characteristics of the product can be modified to minimise the environmental impacts of its production or those of the product itself during or after its use (disposal).

V. BRIEF DESCRIPTION OF PRODUCT



4-ADAPSA / Neroolic Acid

4-Amino Diphenylamine -2- Sulfonic Acid (4-Adapsa) / (Nerolic Acid) / (Pados) that is used in dyestuffs and pigments. It is formulated from well-tested and certified ingredients that ensure the clients receive the best quality range. The main product of shree organo is 4-ADPSA(4-amino diphenylamine 2-sulphonic acid) for this product raw material are PNCB (pera nitro chloro benzene),aniline oil, soda ash, oleum, iron powder, HCL, salt Following are its specifications:

Product Name	4- ADAPSA
Chemical Name	4- Amino Di Phenylamine 2 – S. A.
Empirical Formula	$C_{12}H_{12}N_2O_3S$
Cas No.	91-30-5
Molecular Weight	264
Specification	Assay Dry : 90% Minimum Grey Colour
Use	Acid Orange 51 C. I.- 26550 Acid Black 26:2 C. I.- 26690 Acid Blue 59 C. I. - 50315 Acid Brown 213 C. I. - 20175 Acid Orange 3 C. I. - 10385

Table No. 5 specification of 4-adapsa

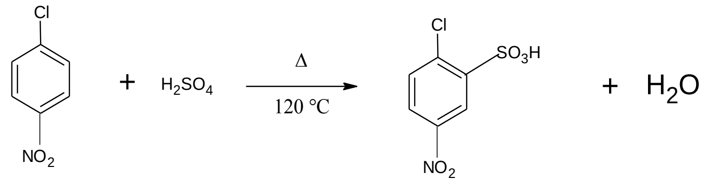
VI. BRIEF DESCRIPTION OF MANUFACTURING PROCESS FOR 4-ADPSA

There are three stages in the process

Stage-1 Sulfonation

Now in 3000 litre reactor take 650kg Oleum (65%) and 1500kg PNCB(Pera nitro chloro benzene). Heat this mixture at 85°C then add 1600 kg Oleum and heat it at 120°C for 5-6 hours. After reaction is over cool this sulfomass to 90°C.

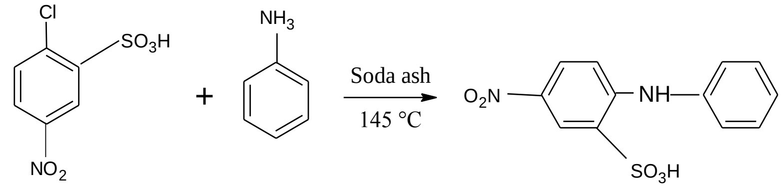
Take 9000 litre Water and sulfomass in 7-8 hours in another reactor and stir for 2 hours. Charge 1900 kg NaCL in step by step within two hours then total mass stirr for 12 to 16 hours (Exothermicity observed) & cool it up to 45°C. Filter in nutch filter up to 5-6 hrs then take the product in centrifuge for 1.5 hours. After centrifuge cake has 25% moisture. The cake is known as Pera Nitro Chloro Benzene Ortho Sulfonic Acid (PNCBOSA).



PNCB **PNCBOSA**

Stage-2 Condensation

In 6000 litre autoclave reactor take 2000 litre water and 100kg soda ash. Add 1000kg PNCBOSA and again add 400kg soda ash to this mixture and heat this mixture to 110°C then add 400kg aniline oil for 4 hours and reheat to 145°C for 12 hours and at that time maintain pressure up to 5-6 kg/cm².Cool this mixture to 125°C and release pressure to 0 kg/cm^2. Then stir for 2 hrs and cool up to temp 100°C. After that reaction mass send to isolation vessel which has 12000 litre capacity and made by Mild Steel Rubber Lined Brick.In isolation step stir the reaction mass for half an hour and charge HCl till pH 2 obtained and again stir for 2 hours and filter through nutch filter for 2 hours after that centrifuge the wet cack. After centrifuge wet cake is 20 to 22 % moisture of purity of aprox 70%. After this stage cake or product is 4-Nitro diphenyl Amine 2 sulphuric acid in yellowish colour.

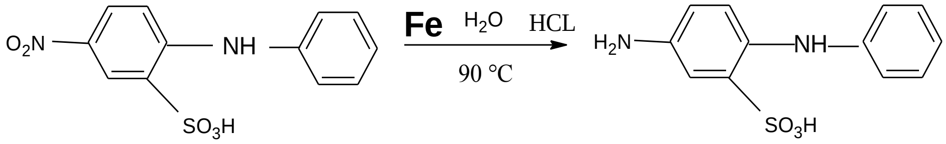


PNCBOSA **Aniline** **4 Nitro Di Phenyl Amine 2 Sulfonic Acid**

Stage-3 Reduction

In 12000 litre reactor take 4000 litre Water heat it up to 85°C. Add 120 kg HCl and 400 kg iron powder stir for 2 hour rise temp up to 90°C ± 2°C. Then add 1600 kg 4 nitro diphenyl amine 2 sulphonic acid for 8 to 10 hours and 250 kg iron powder and check reaction are complete or not through red colour via spot test & stir it for 2 hrs at 90°C ± 2 °C. Make volume 8000 litre with hot water. Maintain pH 8.5 to 9 by addition of soda ash.

Filter this reaction mass in filter press for 4 hours and collect filtrate mother liquor. Then wash cack in filter press with hot water. After that product has 7 to 8 % moisture. Acidify the filtrate by HCl up to pH 3. Stir for 2 hrs and filter in nutch filter for 6 to 7 hours. The final product is 4-Amino Di Phenyl Amine 2-Sulfonic Acid dull light bluish powder having purity of 90%.



4 Nitro Di Phenyl Amine 2 Sulfonic Acid **4 Amino Di Phenyl Amine 2 Sulfonic Acid**

(4-ADPSA)

VII. PROBLEMS DISCUSS:

During reduction process liquid waste generated is due to addition of water and hydrochloric acid in large quantity. The mother liquor is acidic in nature and having pH 3. So if we use this acidic effluent to reduced pH in place of HCl the quantity of final mother liquor will reduced. This mother liquor has 17% acidity. Treatment of large quantity of mother liquor is costly and difficult to dumping without treatment. If this effluent has 60% acidity then we can directly sold it.

VIII. EXPERIMENTAL PROCEDURE:

8.1 First setup:

Take 500 ml mother liquor which is coming after filter press which has pH 9. Now reduce its pH by concentrated hydrochloric acid from pH 9 to 3. After that filter it by vacuum filter and collect mother liquor. Now for first cycle use mother liquor of previous cycle to reduce pH then filter it. For second cycle use mother liquor of first cycle to reduce pH then filter it. Now for third cycle use mother liquor of second cycle to reduce pH then filter it. The flow diagrammed show below:

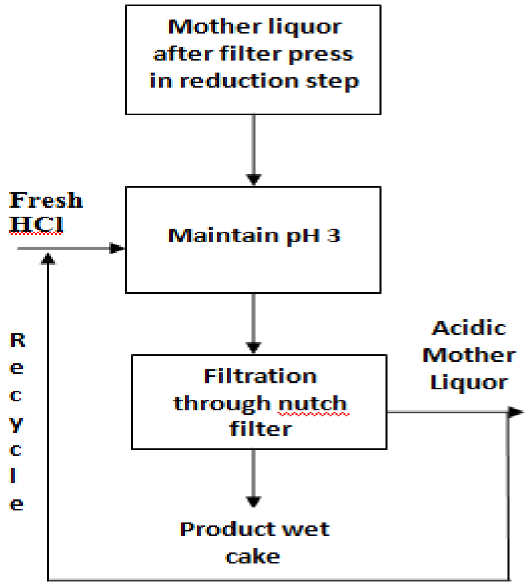


Fig No.2 flow chart of recycle process

8.2 Second setup:

Take 500 ml mother liquor which is coming after filter press which has pH 9. Now reduce its pH by concentrated hydrochloric acid from pH 9 to 3. After that filter it by vacuum filter and collect mother liquor. For first cycle use mother liquor of previous cycle to reduce pH then filter it. For second cycle use 300 ml mother liquor to reduce it pH upto 6 and after that use fresh HCl to reduced its pH upto 3. In every cycle we are using 50% mother liquor and remaining fresh HCl to reduce pH.

IX. RESULTS AND DISCUSSION:

By using complete recycle of Mother Liquor:

	Reaction mass(ml)	**M.L add Qty. (ml)**	**Fresh HCl add Qty. (ml)**	**Mother liquor (effluent) Qty. (ml)**	**Wet Cake Qty. (gm)**
Recycle 0	**500**	**----**	**12**	**300**	**300**
Recycle 1	**500**	**300**	**6**	**610**	**300**
Recycle 2	**500**	**610**	**5.5**	**980**	**240**
Recycle 3	**500**	**980**	**5**	**1310**	**200**

By Using 50% Mother liquor recycle:

	Reaction mass(ml)	M.L add Qty. (ml)	Fresh HCl add Qty. (ml)	Mother liquor (effluent) Qty. (ml)	Wet Cake Qty. (gm)
Recycle 0	**500**	**---**	**12**	**300**	**300**
Recycle 1	**500**	**300**	**6**	**605**	**298**
Recycle 2	**500**	**300**	**6**	**607**	**300**
Recycle 3	**500**	**300**	**6**	**615**	**305**

By using complete recycle of mother liquor to reduce pH from 9 to 3 the weight of wet cake is decrease continuously. By using 50% recycle of mother liquor to reduce pH the weight of wet cake remains same will not decrease much more. By using 50% recycle of mother liquor the pH will decrease from 9 to 6 and remaining pH will decrease by using fresh hydrochloric acid. The quantity of effluent is reducing 50% so the handling cost of effluent will reduce definitely.

X. CONCLUSION:

By recycling mother liquor the liquid waste generated will decrease. Quantity of effluent will decrease 50% so we conclude that 50% recycle of mother liquor will help to reduce mother liquor quantity and final material quantity will remain same.

REFERENCES:

[1] www.beeindia.in/energy_managers_auditors/.../guide.../4Ch13.pdf

[2] Identification of Hazardous Waste Streams, their characterization and waste reduction options in dyes and dyes Intermediate Sector, p-5-20

[3] Hazardous waste management in india by B.V babu and V Ramakrishna,p 1-8

[4] US Patent NO.: US 2012/0203031 A1, “sustainable chemical process for reduction of nitro compounds or nitros compounds containing sulphonic or carboxylic group into corresponding amino compounds with inherent recycle of all acidic streams generated in synthesis”

[5] http://www.branz.co.nz/waste minimization