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Cleaner Production in the Process of p-amino azo benzene 4-sulfonic acid

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***Abstract*—The common indicators of economic welfare, such as national product and income have reflected the growth of the industry as a major indicator for the development of the nation. But the other angle of industrialization has been the serious damage to the surrounding environment so there is keen need of "Cleaner Production" to sustain against waste from industries and pollution. In this paper Cleaner Production(CP) methodology for a dye intermediate P-amino azo benzene 4-sulfonic acid(PAAB4SA) is described. The literature review on cleaner production tools is described. The paper discussed reactor design and simulation of it in the production of PAAB4SA. This modified design is cost saving and it also gives cleaner and easy operation. The paper describe COD reduction options for the case study of dye intermediate sulfotobias acid. Different methods are evaluated and compared to give cost effective method of COD reduction for the process. Experiments were performed for Sulfotobias acid with Advance oxidation process(AOP) of fenton reagent and adsorption using low cost adsorbent neem leaves powder. The detail process and experimental data are discussed in the paper.**

Index Terms— ***Advance Oxidation Process(AOP),Cleaner Production, COD reduction, Fenton Process, Dye Intermediate, Reactor design, Reactor Simulation.***

INTRODUCTION

THIS paper is about how one can implement on cleaner production tools in industry. For the dye intermediate p-amino azo benzene 4-sulfonic acid equipment modification is suggested and we have proposed the design of reactor and simulation of the same. This implemented design of reactor gives better heat transfer in reactor. This will reduce ice consumption as raw material in the reactor. The dye intermediate, p-amino azo benzene 4-sulfonic acid has some solubility in water so ice should not be used to control reaction temperature and this will unnecessary increase handling problems and also increasing quantity of waste effluent alongwith some product loss in it.

Also the case study of sulfo tobias acid is presented for COD reduction in waste water. COD in waste water can be reduced by many methods like flocculation, oxidation, adsorption by activated carbon, electrolysis etc. Here in this paper COD is reduced by fenton's advance oxidation process(AOP) and adsorption using low cost adsorbent of neem leaves powder. Experiments were carried out to study the operating conditions like pH, H_2O_2/Fe^{+2} ratio and reaction time of the advance oxidation process using H_2O_2/Fe^{+2}. Results of the experiments are also presented. For the same effluent trials are also taken with low cost adsorbent neem leaves powder. The detail procedure and results of the experiments are discussed.

Equipment modification

Existing Equipment design

P-amino azo benzene 4-sulfonic acid is used as one important dye intermediate for the production of azo dyes. It is manufactured at Industrial Chemical Works in GIDC Naroda. Manufacturing of p-amino azo benzene 4-sulfonic acid is 4 step process.

First stage is manufacturing of coupling component, benzene methyl aniline sodium sulfonate. It is produced from the reaction of aniline with formaldehyde and sodium bisulfate. The reaction is exothermic and temperature is maintained at 80°C for 3 hour. Water is used to provide cooling and to maintain temperature. But it is not sufficient so ice was also used to quench the reaction temperature. But ice should not be used to control temperature in the reactor. It has several disadvantages. As product has a little solubility in water, it is waste of product in water by adding more amount of water. Also it unnecessary increase volume of liquid effluent generated from the process. So for cleaner production in this stage they need to stop addition of ice in the reactor. It seems that the reactor which is existing there is giving poor heat transfer coefficient. So, there is need to modify the reactor design for this stage. We need to find heat transfer area required for this stage of reaction and can compare it to heat transfer area provided by the reactor to know whether proper heat transfer is taking place otherwise we can also insert cooling coils in the reactor to achieve good heat transfer.

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Existing reactor is having following parameters as in table (1). The design which is proposed is as given in table (2). In the proposed design, jacket used is spiral baffled jacket. It has many advantages over conventional existing reactor jacket.Heat transfer coefficient is improved by this design. Spiral baffles also work as stiffning ring thus shell thickness is reduced in large reactor having long diameter and height.

Table 1
Existing reactor data

Material of construction	MS	
outer diameter of shell	1828.8	mm
height of shell	2209.8	mm
thickness of shell	6	mm
type of agitator	anchor type	
Liquid height in shell	2159	mm
rotational speed of agitator	30	rpm
Jacket outer diameter	1981.2	mm
Area available	12.68962623	m^2

III Proposed equipment design

Determine Size of reactor

To Determine the size of Reactor

$$\text{Working Volume of Reactor} = \frac{\text{Mass of Raw Materials Charged}}{\text{Density of Liquid mixture charged}}$$

$$\frac{h}{D_i} = 1$$,

Let type of bottom head = Torispherical
Inside volume of Torispherical Head,

$$V = 0.084672\, D_i^3 + \frac{\pi}{4} D_i^2 * S_F$$

$$V_{working} = \frac{\pi}{4} D_i^2 * h + 0.084672\, D_i^3 + \frac{\pi}{4} D_i^2 * S_F$$

Consider provision of 20% extra space for vapor liquid disengagement, then actual height of shell of reactor H = 2.189005619m

Determine convective film coefficient

For flat blade disc turbine, for Re > 400

$$\frac{h_i D_a}{k} = 0.74 Re^{0.67} Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14}$$

Where, $$Re = \frac{n D_a^2 \rho_{mix}}{\mu_{mix}}$$

$$Pr = \frac{\mu_{mix} C_{p\,mix}}{k_{mix}}$$

Determine jacket side heat transfer coefficient

Use spiral baffled jacket for heat transfer
For Re > 10000

$$\frac{h_o D_e}{k} = 0.027\, Re^{0.8} Pr^{0.33} \left[\left(\frac{\mu}{\mu_w}\right)^{0.14} \left(1 + 3.5 \frac{D_e}{D_j}\right)\right]$$

Where, D_e *= Equivalent diameter for cross section, m*
D_e *= 4W*
W = Width of jacket,m
W = 50 mm
p = pitch of baffle spiral, m
p = 150 mm
m' = effective mass flow rate through spiral, kg/s

m' = 0.6 $\cdot$ m
m = actual mass flow rate through spiral jacket, kg/s
D_j = mean diameter of jacket, m

$$D_j = \frac{D_{jo} + D_{ji}}{2}$$

D_{ji} *= Jacket inside diameter = shell outside diameter*
D_{jo} *= Jacket outside diameter =* $D_{ji} + 2W$

Determine overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i\, h_{id}} + \frac{d_o}{d_i\, h_i}$$

$$\text{Fouling Coefficients} = h_{od} = h_{id} = 5000\ W/(m^2 \cdot {}^\circ C)$$

Determine heat transfer area

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$$\Delta T_{ln} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln\left(\frac{T_1 - t_2}{T_2 - t_1}\right)}$$

$$A_{req} = \frac{Q_t}{U_o \, \Delta T_{ln}}$$

$$A_{avai} = \pi \, d_o L'$$

Where, L' = length of cylinder that must be covered by jacket

Mechanical design of reactor

Thickness of shell can be determine based on internal design pressure and external design pressure.

$$t_s = \frac{PD_i}{2fJ - P} + CA$$

Same way thickness of top head, bottom head, jacket shell and jacket head has been found and the results of which are given in table (2). Jacket provided in this vessel is spiral baffled jacket so internal stiffning ring is provided in the reactor shell thus thickness of shell can be considerably reduced. The detail design will be presented.

Results and discussion

The design of reactor which has been proposed is in table (2).

Mass of Raw Materials Charged	4302	kg
Working Volume of Reactor	5.3775	m^3
Inside Diameter of Reactor(D_i)	1.824	m
Height of liquid in shell	1.824	m
Actual height of shell of reactor(H)	2.189	m
tip velocity, V	200	m/min
Diameter of agitator (D_a)	0.608	m
Convective heat transfer coefficient(h_i)	405365	$w/m^2 °C$
Inside Diameter of Reactor(D_i)	1.824	m
Jacket ouside diameter(D_{jo})	1.89	m
Jacket side heat transfer coefficient(h_o)	1443.61	$w/m^2 °C$
Outside Diameter of Reactor(Do)	1.840	m
Overall heat transfer coefficient(U)	784.00	$w/m^2 °C$
Area required(A_{req})	1.862	m^2
Area available(A_{avai})	10.540	m^2
Type of Agitator	45° pitch blade turbine	

Proposed design gives better heat transfer coefficient. Area available for heat transfer is also greater than area required by the reactor. Also proposed design have advantages of spiral baffeled jacket. The detailed cost estimation will be presented for this design. The detailed drawing of proposed design will also be presented in Autocad. The simulation of this reactor will be done in Aspen hysis and will be presented.

COD reduction

Using Fenton's advance oxidation Process(AOP)

The mechanism of fenton reaction has been known for a long time. This oxidation method is based on the use of amixture of hydrogen peroxide and iron salts(Fe+2) which produce hydroxyl radical(●OH) at acidic pH in ambient condition. Both H2O2 and Fe+2 can react with ●OH and therefore both can inhibit the oxidation reactions if either of them is not in optimal dosage. Many authors suggested Fe+2 to H2O2 mass ratio to be optimal at 1 to 10, but it must be optimized for particular waste water to minimize scavenging effects. Photo-fenton process(H2O2/Fe+2/UV) is also one of the advance oxidation process involve the hydroxyl radical ●OH formation in the reaction mixture through photolysis of hydrogen peroxide(H2O2/UV) and fenton reaction (H2O2/Fe+2). The H2O2/UV or fenton process alone was successful in removing COD from waste water. The peroxide dose is important in order to obtain better degradation efficiency, while the iron concentration is important for the reaction kinetics.

The experiments were performed with different quantity of hydrogen peroxide and the set-up is as shown in fig. (1)

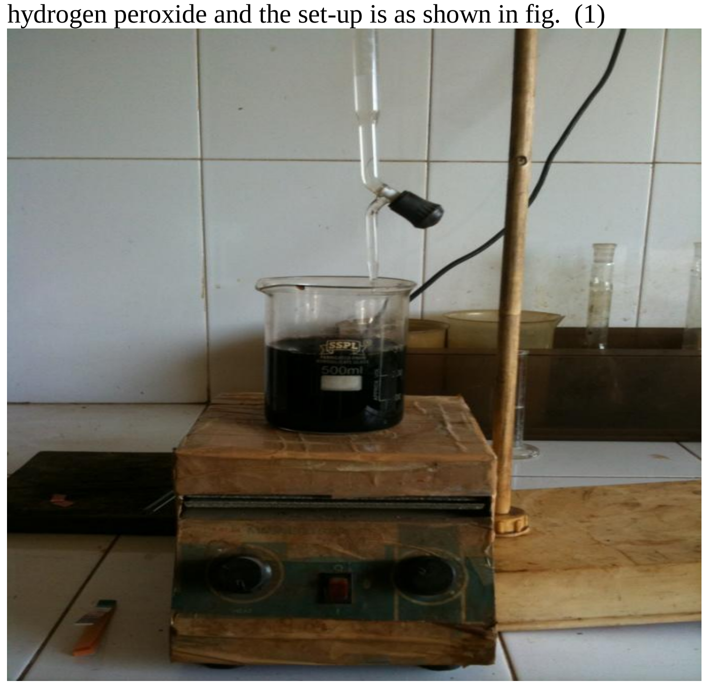

Fig. (1) Experimental set-up of fenton's process

Results and discussion

Results of the fenton's advance oxidation process for COD reduction has been given in table (3). It shows that optimum ratio of hydrogen peroxide to iron sulfate should be kept at 8.

Table 2
Variation of quantity of hydrogen peroxide in fenton's process

		Experiment 1	Experiment 2	Experiment 3
Volume of Sample	ml	250	250	250
Qty of 50% H_2O_2	ml	12.5	10	5
Qty of $FeSO_4.7H_2O$	gm	1.25	1.25	1.25
Time	hr	2	2	1
pH		2	2	2
Initial COD	PPM	5250	6000	3581
Final COD	**PPM**	**3800**	**2925**	**2412**
Difference in COD	**PPM**	**1450**	**3075**	**1169**
% reduction of COD	%	27.61904762	51.25	32.64451271
Ratio of $H_2O_2/FeSO_4.7H_2O$		10	8	4
Flow rate of H2O2	ml/se	0.104166667	0.166666667	0.083333333

The graph of % COD reduction is as shown in figure (2). Which depict that cod can be reduced to 2925 ppm with hydrogen peroxide quantity of 10ml, which is the optimum value from these experiments.

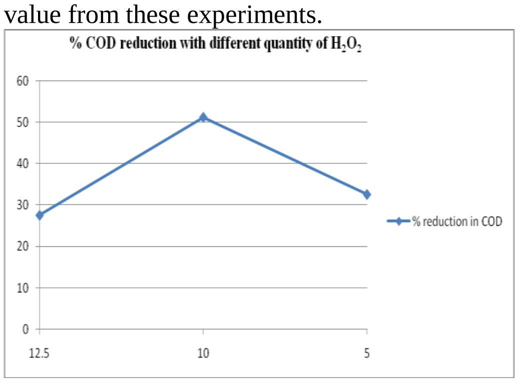

Fig. (2) Graph of H2O2 quantity vs % COD reduction

Conclusion

The detail of this design will be provided. Simulation of this proposed design will also be presented. The cost estimation of proposed design has also been done and pay back period is identified. The proposed design has advantages of spiral baffled jacket. It helps to improve heat transfer coefficient and also it help to reduce thickness of shell as baffles will work as external stiffning ring.

COD can be reduced successfully with fenton's advance oxidation process only we need to optimize various process parameters for different type of waste water.

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