Recovery of 4-Aminophenol from Aqueous Solution Using Different techniques

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ABSTRACT

4-Aminophenol belong to a class of organic substances of particular environmental interest. Due to their widespread discharge into environment and their toxicity to many living organisms, phenol and its amine derivates are presently found on most priority lists of hazardous substances.

Different techniques to separate phenolic compounds from aqueous solution has been developed which includes extraction, absorption, distillation , freeze crystallization ,etc. To separate a phenolic compound from effluent & to recover it is an essential need for the industry today. This report presents experimental for aqueous solution of 4-aminophenol with help of Distillation and freeze crystallization system which is utilized in finding % recovery of 4-aminophenol. Using freeze crystallization method we did not get any recovery of 4-aminophenol so freeze crystallization method don't work in recovery of 4-aminophenol from aqueous solution. then used distillation technique we get 80% recovery of p-aminophenol from aqueous solution. Based on experimental data, we proposed to design the batch distillation column.

Keyword: *4-Aminophenol, Distillation , Freeze crystallization,*

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1. INTRODUCTION

P-Aminophenol (PAP), an important intermediates in pharmaceutical, dyestuffs and organics synthesis, are widely used in the production paracetamol and acetaminophenol.[1,2] Phenolic contaminant are found in waste water of various industry such as coal conversion, pharma, petroleum refining, plastics, iron, textile and steel manufacturing as well as pulp and paper manufacturing. [3,4]phenolic compounds are under great concern for their toxicity and possible accumulation in the environment. Therefore, it is of great important to remove PAP from industrial wastewaters and environmental water effectively.

A number of treatment technology have been used to removed phenolic compound from aqueous solution. These include distillation, solvent extraction, membrane separation and Freeze crystallization.[5] In this paper, distillation and freeze crystallization process to used recovery of 4-aminophenol from aqueous solution.

2. EXPERIMENTAL WORK

To recovery of 4-aminophenol first we perform freeze crystallization process in Cascaded refrigeration apparatus. The details of the experiment carried out in this study as following.

2.1 EXPERIMENT NO :-1 FREEZE CRYSTALLIZATION

2.1.1 Experimental set up

Figure 1: Cascaded refrigeration apparatus

2.1.2 Experimental Procedure

Take aqueous solution sample in glass test jar up to the filling mark and start the equipment. Then solution is frozen up to -20`C and sent to the separator(filter).and ice and melt are separated out. After stage, the concentration of 4-aminophenol is measured by analyzing the sample.

2.1.3 Analysis Method

Take 25 ml filtrate, 5 ml HCL and approx water in 250 ml conical flask. Place the flask in the crushed ice ground the flask to facilitate cooling of the mass in the conical flask .Titrate the content flask against 0.1 M std. $NaNO_2$ solution using starch iodide paper as external indicator.
Observed blue spot on starch indicator paper for 3 min.

$$\% \text{ PAP} = \frac{\text{Titrate value x } 0.0109 \text{ x } 100}{\text{Sample volume (25 ml)}}$$

2.1.4 Result Analysis

From the results obtained after analysis,

1). Concentration of 4-aminophenol in ice = 1.69%
2). Concentration of 4-aminophenol in melt = 1.79 %

We conclude that emphasis should be applied to freeze crystallization technique but not recovery of 4-aminophenol in melt as well as ice. So we perform second experiment of simple distillation process in distillation column apparatus .

2.2 EXPERIMENT NO : 2 DISTILLATION

2.2.1 Experimental set up

Fig 2. distillation column apparatus

2.2.2 Experimental Procedure

Take 1500ml of aqueous solution into round bottom flask and then start heating. Cold water is used for circulation in condenser for condensation. Also put the thermometer on the round bottom flask for the measurement of temperature and Set the temperature of heater as 80^{o} C. The vapour will condense and we collect it as distilled. The temperature is around 170-180^{o} C , In this temp 70% water will be vaporized and it will collect in to the flask is called cut-1. The temperature was allowed to increase from 180-195^{o} C , In this temp 12% water and 4-aminophenol mixture will be vaporized and it will collect in to the flask is called cut-2 .Some part remains in the flask as residue. From the 1500ml feed we get, cut-1 1070 ml, cut-2 170 ml, Residue- 285 ml.

Fig -3 Sample of Aqueous solution

Fig - 4 Sample of cut-1

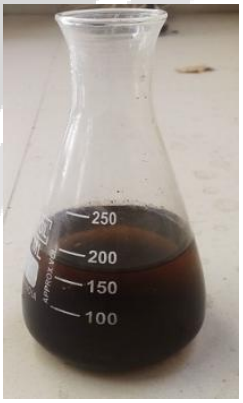

Fig -5 Sample of cut-2

2.2.3 RESULT

Comp.	Cut-1		Cut-2		Residue	
	%	Vol(ml)	%	Vol(ml)	%	Vol(ml)
PAP	0.1	1.02	4.95	8.415	0.39	1.11
Water	99.9	1068.9	95.05	161.5	10.59	30.18
Salt	-	-	-	-	89.02	253.7
Total		1070		170		285

3.CONCLUTION

We are Using freeze crystallization method we did not get any recovery of 4-aminophenol so freeze crystallization method don't work in recovery of 4-aminophenol from aqueous solution. then used distillation technique we get 80% recovery of p-aminophenol from aqueous solution .

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