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Concentration of Hydrogen Peroxide by Batch Distillation Column

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Abstract

An investigation was conducted to study the concentration of hydrogen peroxide by vacuum distillation.The effect of the process variables (such as vacuum pressure, reflux ratio, time of distillation, and packing height of the column used in the distillation process) on the concentration of hydrogen peroxide were investigated. During the third stage of distillation (95 wt.%) concentration was obtained.

Box-Wilson central composite rotatable design is used to design the experimental work for the mentioned variables. It was found that the concentration of hydrogen peroxide increases with:Increasing vacuum pressure, decreasing reflux ratio, increasing the time of distillation and increasing the packing height.

The second order polynomial regression analysis of the objective response (concentration of hydrogen peroxide). with respect to the four variable, using statistical program gave the following equations:

$$Y_1 = 54.87 - 0.27\,P - 81.45\,R + 16.36\,t + 0.69\,H + 55.67\,R^2 - 0.0035\,H^2$$
$$Y_2 = 104.04 - 0.44\,P - 140.62\,R + 19.8\,t + 0.211\,H + 0.0018\,P^2 + 105.25\,R^2 - 3.33\,t^2$$
$$Y_3 = 83.79 - 0.18\,P - 19.04\,R + 13.14\,t - 0.094\,H - 0.0047\,P^2 - 26.78\,R^2 - 2.78\,t^2$$

Keywords: hydrogen peroxide, batch distillation.

Introduction

Hydrogen peroxide is a major industrial chemical material which is rapidly increasing in scale of manufacturing and uses[1].

Its scale of manufacture and use have increased markedly since about 1925 when electrolytic processes were introduced to the United States and industrial bleach applications were developed[2].

Hydrogen peroxide can be prepared electrochemically by the oxidation of sulfuric acid or the partial reduction of O_2. And by catalytic process, such as the anthraquinone process[3].

Hydrogen peroxide solutions are strong oxidizing agents. In some cases, hydrogen peroxide also acts are a reducing agent but only toward those materials that are themselves strong oxidizing agents, e.g, in acid solutions hydrogen peroxide reduces potassium permanganate to manganese sulfate. Hydrogen peroxide above (52 wt.%) is classified as "Corrosive material". Actually hydrogen peroxide is extremely weak acid[2].

Commercial products of hydrogen peroxide (10 to 70 wt% H_2O_2), usually contain impurities such as metallic (Iron, Aluminum) and inorganic impurities (NO_3, PO_4, SO_4)[4].

A number of the purification techniques are used to remove metallic and inorganic impurities such as distillation, crystallization, adsorption, filtration and reverse osmosis. Depending on the degree of purity of hydrogen peroxide addition of more stages of these methods are required[5].

Experimental Work

Apparatus

Hydrogen peroxide was concentrated by using a vacuum distillation apparatus. The vacuum distillation unit consists of a distillation apparatus and vacuum unit Fig. (1).

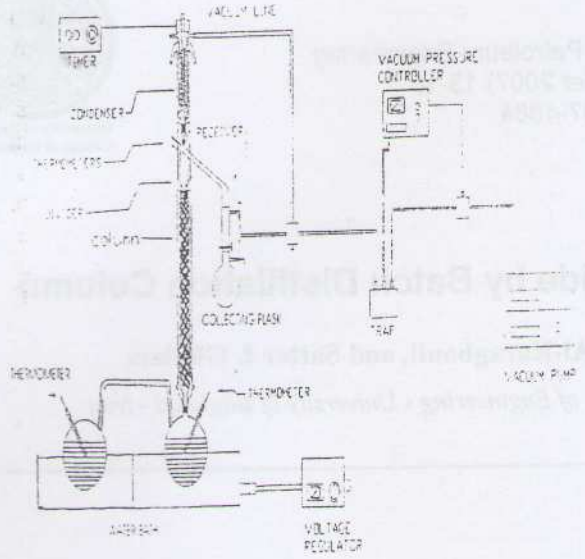


Fig. 1 Vacuum distillation unit

1- Water Bath: The water is heated by means of electronical immersion coil placed inside the bath. Electrical power to the immersion coil is regulated and provided by adjustments of the voltage regulator. The water bath is capable of holding two still pot flasks of (250 ml) volume each. The still pot has a 2-necks round bottom Pyrex flask. One neck is used for thermometer while the other one used for the connection with the other flask, which has three necks. The thermometer is immersed to about (2 cm) above the flask bottom.

2- The packed column: The experiments in the present work were carried out using a (2 cm) I.D glass column filled with 5 mm glass Rashing rings. The column is designed for a batch rectification process. It has two branches for placing the thermometer for measuring the temperature. One of these branches contains a thermometer which measures the temperature of the entering vapor to the base of the column and the other is used to measure the temperature of leaving vapor from the column.

3- Divider: Reflux divider is placed between the column and condenser. Divider is used to give an adjustable ratio between the takeoff water distillate and the reflux. The height of the divider is (20 cm) with an outer diameter of (3.5 cm). The divider has two branches for placing thermometer. One of these branches is used to measure the temperature of the vapor while the other branch is used to measure the temperature of the liquid condensate. The distillate liquid is collected in the round flask which is connected with tube to transfer the liquid condensate to the controlling device which adjusts reflux ratio.

4- Condenser : The divider was connected to the helical-tube condenser which is (30 cm) height and (3.5 cm) outer diameter. The condenser has two nozzles for cooling water flow. The top of the condenser was connected to the vacuum line.

5- Receiver : The receiver with volume of (150 ml) is connected to the vacuum line through two solenoid values to control the flow of the condensate.
The bottom of receiver was connected to a round flask with volume of 250 ml.

6- Vacuum Unit :
The vacuum lines from the condenser and the receiver were joined by means of -T- shape valve. The resulting connection is called main vacuum line. Two traps are connected to the main vacuum line. These traps are cooled by liquid nitrogen to protect the vacuum pump, from entraining vapor and liquid. One of the two outlet lines from the traps was connected to one-way valve, and the other was connected to the vacuum pressure controller.
The vacuum pressure controller was used to adjust the pressure drop in the distillation system by comparing the vacuum pressure set values and vacuum pressure values in the trap indicated by controller sensor.
The controller sends its signal to the one-way valve to open or close the vacuum line depending on the vacuum pressure in the trap.
The one-way valve is connected to rotary vacuum pump which produce a vacuum pressure in the system. All parts of the equipment are made of glass.

Experimental Procedure

The vacuum distillation experiments were carried out by using a vacuum distillation apparatus as shown in Fig. (1).

Before each experimental all connections and joints in the apparatus were lubricated by silicon grease in order to obtain the desired vacuum pressure. The feed for each distillation experiment was 100 ml of hydrogen peroxide solution with defined concentration (25, 50, 75 wt.% H_2O_2). This quantity was charged into the still pot then evaporation takes place, followed by distillation experiment where proceeds for a certain time and defined vacuum during the operation.

Samples from the desired product, residue and the condensate water are taken for analysis of hydrogen peroxide concentration.

After each experiment all parts of the vacuum distillation apparatus are washed with demineralized water in order to remove any possible contaminants accumulated in the column, condenser, still pot.

The washing procedure was an operation of atmospheric distillation using (250 ml) of demineralized water to wash the contaminants and accumulate them at the bottom of the still pot where they can be removed.

These procedures are the same as for three stages to get a highly concentrated hydrogen peroxide (90-95 wt.%).

The higher concentration hydrogen peroxide obtained was collected in the still pot number two. While the concentration of residue in the still pot number one increased which must be drawn off to remove impurities. The water distillate is collected in the receiver and collection flask.

After the distillation process is preformed the vacuum system is switched off and the required hydrogen peroxide is separated from the distillation system and stored in refrigerator.

Results and Discussion

Analysis of Results

The response of experiments represented by the concentration of hydrogen peroxide which is fitted to the results, was used to determine the operating conditions that give the concentration of hydrogen peroxide for each stage of the vacuum distillation.

Mathematical Models

A second order polynomial which has the general form for four variables, was employed in the range of the independent variable, as represented by the following equation :

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_5X_1X_2 + B_6X_1X_3 + B_7X_1X_4 + B_8X_2X_3 + B_9X_2X_4 + B_{10}X_3X_4 + B_{11}X_1^2 + B_{12}X_2^2 + B_{13}X_3^2 + B_{14}X_4^2 \quad (1)$$

Effect of Vacuum Pressure :

The effect of vacuum pressure on the concentration of hydrogen peroxide was studied in order to determine the pressure at which distillation process is best performed.

Figure (2) show that for all reflux ratios studied the concentration of hydrogen peroxide increases as the vacuum pressure increases. Figure (2) shows that at R=0.3 the concentration of hydrogen peroxide appears to have the highest value but it is not desired, since the amount of hydrogen peroxide lost with water distilled is maximum. Therefore the best reflux ratio for first stage distillation was found to be (0.5-0.6) this range of reflux ratio given the desired concentration of hydrogen peroxide and minimum loss.

The concentration of hydrogen peroxide in the water distilled is increased when the vacuum pressure increased and the reflux ratio decreased. Therefore the best condition for concentration of hydrogen peroxide was found to be in the vacuum pressure range (80-60 mmHg) for first stage, the (60-40 mmHg) for second stage and (30-20 mmHg) for third stage.

The vacuum pressure influences the concentration of hydrogen peroxide in different ways, mainly the operation at low pressure is normally used to avoid the formation of explosive vapor composition (76 wt.% H_2O_2 and above) and leaving behind the organic impurities.

Similarly the relation between hydrogen peroxide concentration and vacuum pressure was plotted for given times of distillation as shown in Figure (3). The highest values of hydrogen peroxide concentration were obtained at longer times of distillation, but it was observed experimentally that the best time was about (1.5 hr) for the experiment conditions used. It was observed that the stability of hydrogen peroxide solution produced at longer time of distillation (2.5 hr) was not high because the organic impurities evaporated along with hydrogen peroxide product and the concentration of impurities in the residue possibly reaches hazardous level.

Therefore the optimum vacuum pressure is essential to achieve the desired concentration of product with highest purity and consequently more stable with minimum loss.

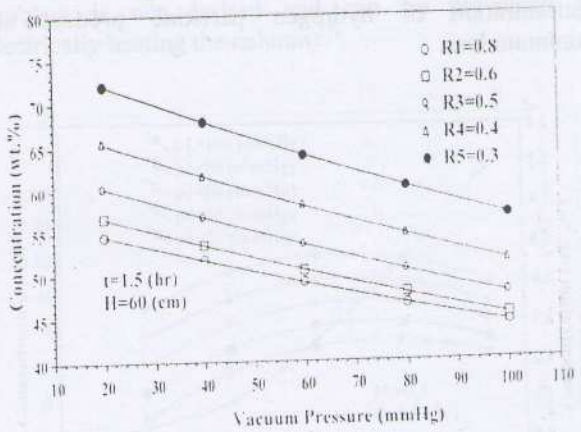


Fig. (2) : Hydrogen peroxide concentration vs. vacuum pressure at various reflux ratios

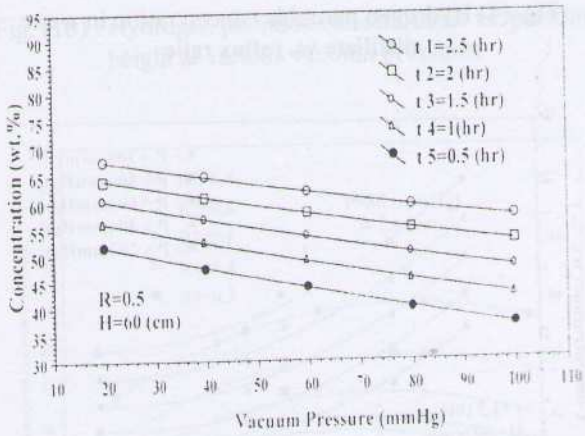


Fig. (3) : Hydrogen peroxide concentration vs. vacuum pressure at various times

Effect of Reflux Ratio

The effect of reflux ratio on the concentration of hydrogen peroxide at a given (vacuum pressure, time of distillation), is shown in Figures (5) and (6).

Examining these Figures it can be seen that the hydrogen peroxide concentration increases with decreasing the reflux ratio. For example at R=0.3 the concentration of hydrogen peroxide was the highest, but it is not desired for first and second stages of vacuum distillation, because the concentration of hydrogen peroxide lost in the distillate water is maximum.

Therefore the optimum reflux ratio in first stage is (0.5-0.6) and in second stage (0.4-0.5). But in the third stage optimum reflux ratio is (0.4-0.3) for limited the concentration of hydrogen peroxide in distillate water using packing height of (80 cm). Referring to Figure (4), which clearly illustrates that the losses of hydrogen peroxide in the water distillate increase when the reflux ratio decrease.

The best reflux ratio is the ratio which gives the desired concentration of hydrogen peroxide product and minimum loss.

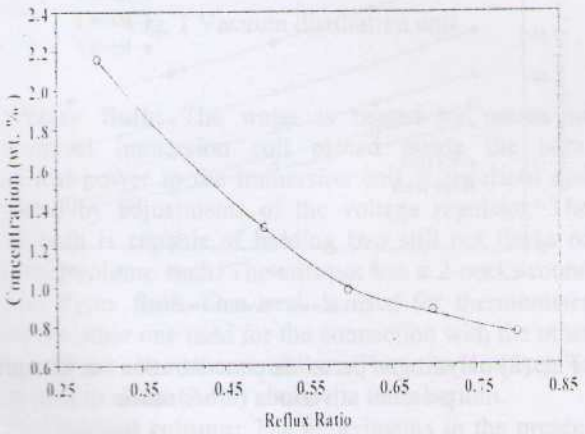


Fig. (4) Hydrogen peroxide concentration in water distillate vs. reflux ratio

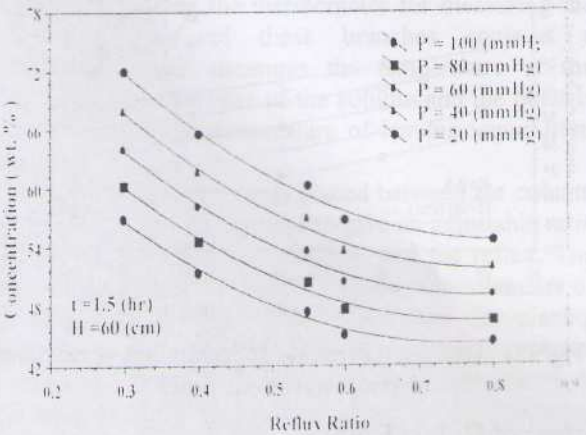


Fig. (5) : Hydrogen peroxide concentration vs. reflux ratio at various vacuum pressures

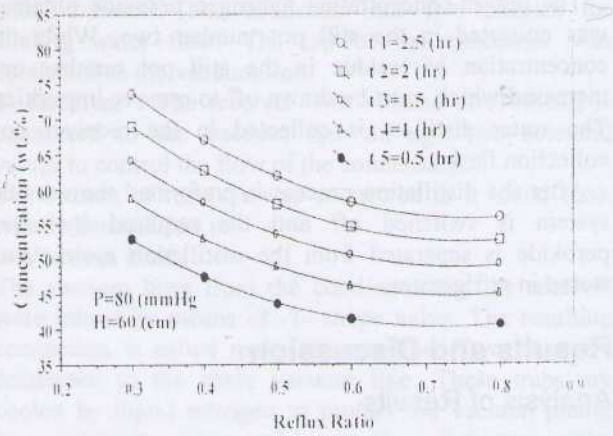


Fig. (6) : Hydrogen peroxide concentration vs. reflux ratio at various times

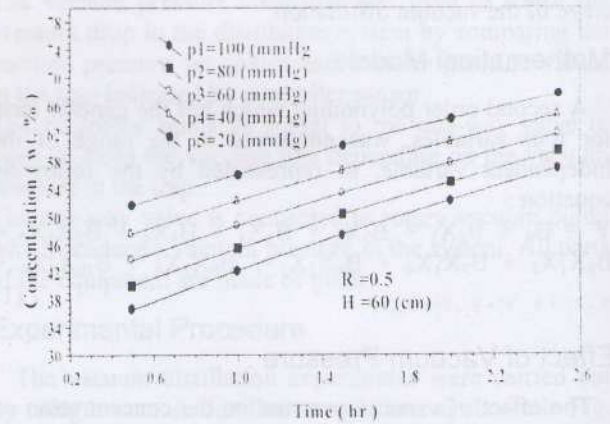


Fig. (7) : Hydrogen peroxide concentration vs. time at various vacuum pressures

Effect of Time of Distillation

The best time of distillation is an important factor affecting the concentration of hydrogen peroxide, which must be known to avoid the rise of concentration of impurities in the hydrogen peroxide product to hazardous level.

Since the process of distillation is batch wise, therefore it is essential to determine the rate of evaporation so that the time required should be just sufficient to evaporate about 80-90% of the original feed.

Examining Figures (7) and (8) it can be seen that the concentration of hydrogen peroxide increases with increasing the time of distillation.

It is clear from those Figures that when time of distillation is equal (2.5 hr) the concentration of hydrogen peroxide is highest. But the product was not stable, because impurities evaporated along with the product and the amount of residue was very little (3-5 ml) out of (100 ml) is originally fed. But when time was (1.5 hr) the

amount of residue (10-15 ml) and the product was stable therefore the best distillation time which leads to a desired concentration of hydrogen peroxide is greatly dependent on the specific conditions of the experiment (i.e., amount of hydrogen peroxide, vacuum pressure, temperature).

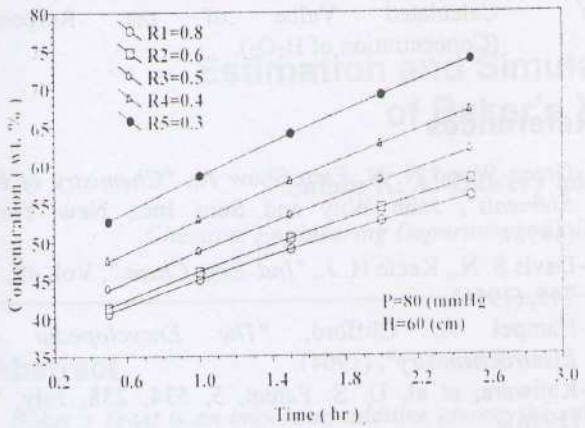


Fig. (8) : Hydrogen peroxide concentration vs. time at various reflux ratios

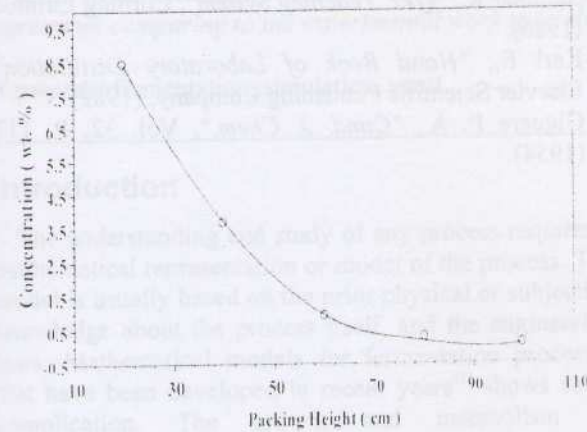


Fig. (9) : Hydrogen peroxide concentration in water distillate vs. packing height

Effect of Packing Height

The effect of packing height is clearly observed by concentration hydrogen peroxide in water distillate.

Examining Figure (9), it can be seen that increasing packing height will decrease the concentration of hydrogen peroxide in the water distillate.

Figures (10) and (11) show that increasing of packing height will increase the concentration of hydrogen peroxide product (The efficiency of distillation column depends upon its ability to transfer heat and material between the liquid and vapor streams. Since, in the case of packed column, this transfer takes place on the surface area of distillation column packing,, then it follows that high surface areas result in high heat and material transfer. Maximum efficiency is obtained by using long columns of high surface area)[6].

These figures show also that for first and second stage the best packing height is (60 cm) because this height gives the desired concentration of product and minimum loss of hydrogen peroxide in water distillate. In the third stage however, the packing height used was (80 cm). Since the feed to this stage is relatively high (75 wt.%) and to avoid excessive losses of hydrogen peroxide in the water distillate and to achieve the desired concentration of product (> 90 wt.%).

When packing height of (1 m) was used a high concentration product of hydrogen peroxide was also obtained, but a problem of flooding was observed. (this problem is not desired and can be prevented by electrically heating the column)[7].

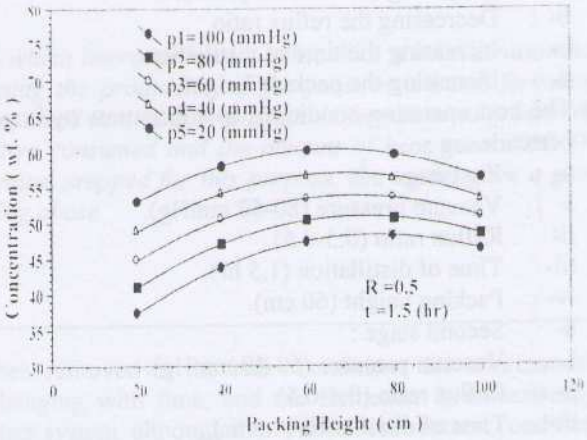


Fig. (10) : Hydrogen peroxide concentration vs. packing height at various vacuum pressures

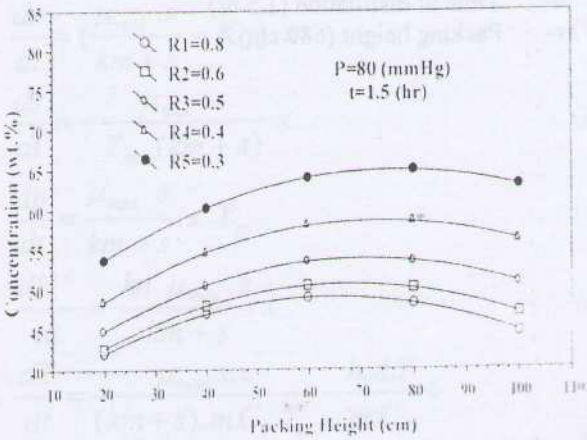


Fig. (11) : Hydrogen peroxide concentration vs. packing height at various reflux ratios

Conclusions

The following conclusions could be drawn from this investigation :

1-The second order polynomial regression analysis of the objective function i.e., the concentration of hydrogen peroxide and four variables (i.e., vacuum pressure, reflux ratio, time of distillation, and packing height), for each stages of distillation were found.

2-It is necessary to concentrate hydrogen peroxide by using vacuum distillation in two or three stages because :

a- Decrease of the boiling point of hydrogen peroxide leads to decrease the rate of decomposition of hydrogen peroxide.

b-Obtaining hydrogen peroxide with high purity and good stability.

3-The concentration of hydrogen peroxide increases with:

a- Increasing the vacuum pressure.
b- Decreasing the reflux ratio.
c- Increasing the time of distillation.
d- Increasing the packing height.

4-The best operating conditions to concentrate hydrogen peroxide are :

a- First stage :
i- Vacuum pressure (80-60 mmHg).
ii- Reflux ratio (0.5-0.6).
iii- Time of distillation (1.5 hr).
iv- Packing height (60 cm).
b- Second stage :
i- Vacuum pressure (60-40 mmHg).
ii- Reflux ratio (0.4-0.5).
iii- Time of distillation (1.5 hr).
iv- Packing height (60 cm).
c- Third stage :
i- Vacuum pressure (30-20 mmHg).
ii- Reflux ratio (0.3-0.4).
iii- Time of distillation (1.5 hr).
iv- Packing height (680 cm).

Nomenclature

H	Packing Height.	m
P	Vacuum Pressure.	mmHg
R	Reflux Ratio.	
t	Time.	hr
Y	Calculated Value of the Response (Concentration of H_2O_2).	

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