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Study of the Performance of Batch Reactive Distillation Column

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Abstract

Batch reactive distillation was studied in packed bed column. Esterification of methanol with acetic acid to produce methyl acetate and water with homogenous sulfuric acid as a catalyst was considered. This system was chosen because the reaction is reversible and the boiling point of reactant and products are different.

The reaction was carried out with and without distillation column and shows that the reactive distillation is more efficient from the conventional process (reactor and then separation). The conversion of acetic acid and concentration of methyl acetate increase by (30.43% and 75.14%) respectively at the best condition (reflux ratio 2, feed mole ratio 2 and batch time 90 minute).

The influence of various parameters, such as batch time, reflux ratio, and feed mole ratio (methanol to acetic acid) on the performance of the batch reactive distillation column was studied, through the effect of the concentration of product and conversion of reactant.

The results obtained for the non-ideal packed bed reactive batch distillation column show that the conversion of acetic acid is 90% at the best condition reflux ratio 2, feed mole ratio 2, and batch time 90 minute

Introduction

In recent years, increasing attention has been directed towards reactive distillation process as alternative to conventional processes (reactor and then separation). This has led to the development of a variety of techniques for reactive multistage column; however the problem of design and synthesis of batch reactive distillation processes have not yet been addressed [1].

Most of the distillation synthesis studies to date have been concerned with multi component non-ideal mixtures. The main advantages of this process relative to the conventional alternatives are the possibility of carrying equilibrium–limited chemical reaction to completion, and the simultaneous separation of the reaction products in only one unit .This reduces or eliminates reactor and recycle costs. This advantages of reactive distillation, and flexibility of a batch process can be combined in batch reactive distillation device, and the effect of operating parameter on yield and selectivity is studied [2].

The aim of the present work is to study the efficiency and the characteristics of the batch reactive distillation process and distinguished between it and the conventional reactor – distillation process. The present work provides a process for the production high purity methyl acetate from methanol and acetic acid, to study the effect of major variables (such as batch time, reflux ratio, and feed mole ratio) on the conversion of acetic acid and the concentration of methyl acetate in the product.

Experimental Work

A schematic sketch of the apparatus is illustrated in figure (1). It is comprised of all items, flask with two necks, one for the thermometer to measure the temperature at the still (vapor temperature) and the other connected to 3cm diameter and 57cm length distillation

column. Packed with packing to a height of 30cm. The distillation column connected at the top with a thermometer to measure the top distillation temperature. The raising vapor from the distillation column was condensate by a condenser.
The condense accumulates in the receiver which has two necks one for product through valve (1) and the other return back to the distillation column as reflux with fixed ratio (R) through valve (2).
Dosing pump was used to control the quantity of the reflux to the distillation column. Heat was supplied to the still by electrical heater and was controlled on the temperature of the component in the still to desired value by using a variac.

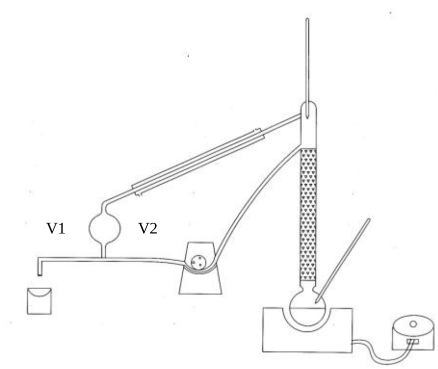

Fig. 1: Sketch of the experimental equipment

Selection of Liquid- Liquid System

The reaction of methyl acetate with acetic acid in the presence of sulfuric acid as homogenous catalyst to produced water and methyl acetate is:

$$CH_3COOH + CH_3OH \overset{H_2SO_4}{\Longleftrightarrow} CH_3COOCH_3 + H_2O$$

Specification of Variables

The variables considered for the present work are:

a- Reflux ratio (0, 1, 2, and 3).
b- Feed mole ratio (1, 1.5, 2, and 2.5) of methanol to acetic acid.
c- Batch time (40, 60, 90, and 120 minute)

These variables are studied and the results are compared with previous work.

Results and Discussion

Variable Effect on Product Concentration

-Effect of Batch Time

Figure (2) shows the relation between concentration of methyl acetate against time at variable feed mole ratio and constant reflux ratio (2). Form this figure it clear that the concentration of methyl acetate increase sharply up to (20) minute then it begin to increase slightly to value of (60) minute after that it remain constant, for a period of time depend on feed mole ratio and then decrease. At a low mole ratio along time is required for the concentration to be reduced because of the low mole ratio consist of high percent of acetic acid, methanol return to the still through the reflux and react with residual acetic acid to produce methyl acetate. For a high mole ratio, a short time is required for the concentration to be reduced. That mean a low percent of acetic acid which can be consumed rapidly and then concentration of methanol in the distillate will be increase to reduce the concentration of methyl acetate.

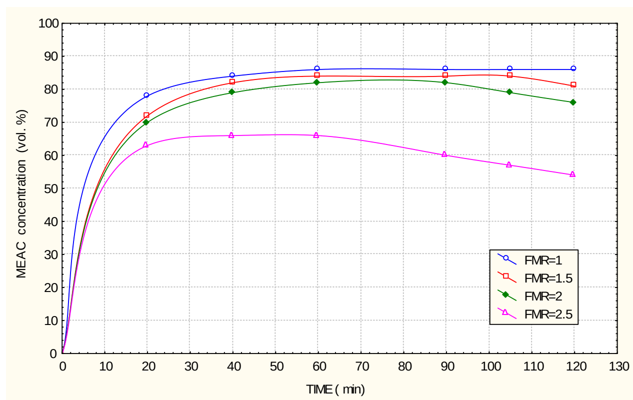

Fig. 2: Concentration of methyl acetate vs. time at reflux ratio=2 and different feed mole ratio

-Effect of Reflux Ratio

Figure (3) shows the relation of methyl acetate concentration against reflux ratio for a batch time 90 minute and different feed mole ratio. This figure shows that the maximum concentration is at reflux ratio equal to (2) for all feed mole ratio except the value of feed mole ratio (2.5) where in this feed mole ratio the concentration increase directly mainly in straight line as it be explained previously. From this figure it can be deduced that the best reflux ratio is (2).

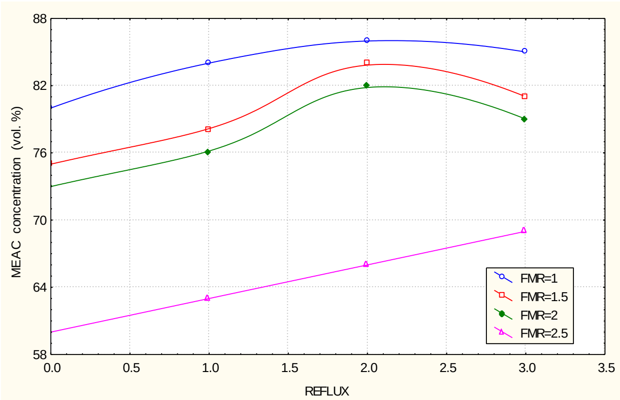

Fig. 3: concentration of methyl acetate vs. reflux ratio at 90 minute and different feed mole ratio.

-Effect of Feed Mole Ratio

Figure (4) show the concentration of methyl acetate against feed mole ratio at variable reflux ratio and constant time. The figures represented that if the mole ratio increase the concentration will decrease, because of if the percent of methanol increase in feed, it will vaporize more than methyl acetate, causes decrease of methyl acetate concentration.

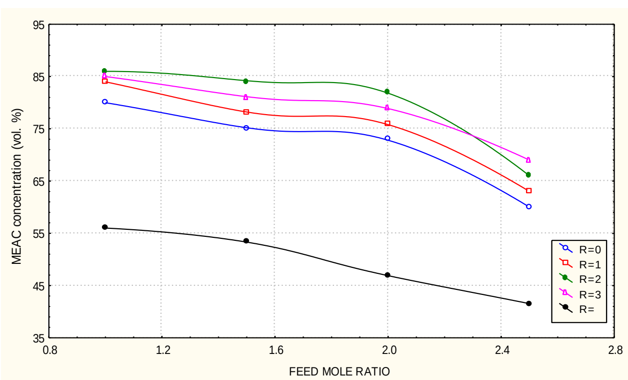

Fig.4: concentration of methyl acetate V.s. feed mole ratio at 90 minute and different reflux ratio

Variable Effect on Conversion of Acetic Acid

- Effect of batch Time

Figure (5) shows the conversion of acetic acid against batch time for variable feed mole ratio and constant reflux ratio 2. this figure shows that the conversion of acetic acid increase slightly up to (60) minute then increase sharply up to the (120) minute as an over all the conversion of acetic acid increase with time due to the increase of consumption of acetic acid with time which cause a high conversion value.

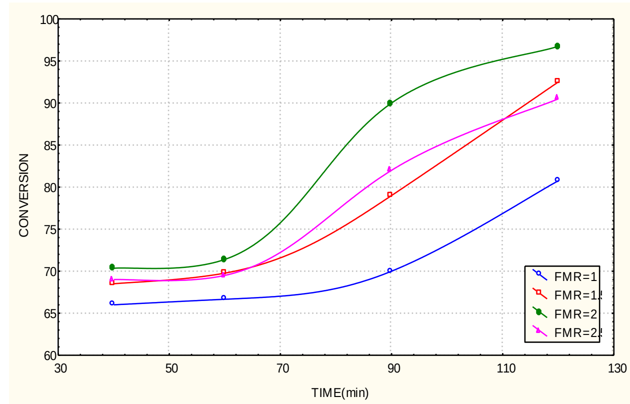

Fig.5: conversion of acetic acid V.s. time at R=2 and different feed mole ratio.

- Effect of reflux ratio

Figure (6) shows the conversion against reflux ratio at constant batch time 90 minute and different feed mole ratio. From this figure the conversion of acetic acid increase with reflux ratio up to reflux rate (2) and then decrease for all value of feed mole ratio, except for feed mole ratio (2.5) which shows a maximum value of conversion at reflux ratio equal to(1) this is in agreement with (Kai [6], Agreda [7], and Doherty [8]). This is because of at increase reflux ratio, the distillate is rich in methyl acetate and when it return to the still, the reversible reaction is occurred and more acetic acid is produced which causes a decrease in the conversion of acetic acid.

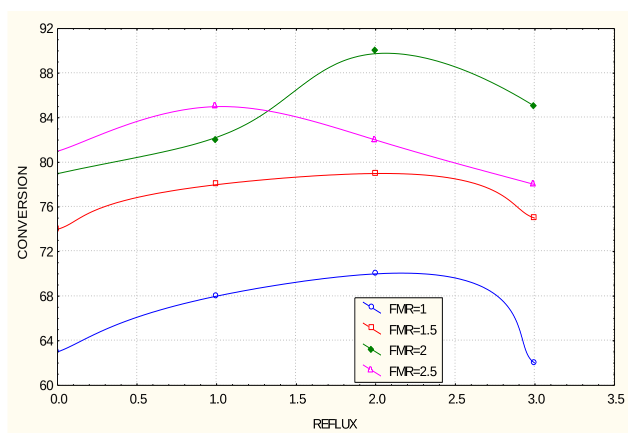

Fig. 6: conversion of acetic acid V.s. reflux at 90 minute and different feed mole ratio

- Effect of feed mole ratio

Figure (7) shows the conversion against feed mole ratio at batch time 90 minute and different reflux ratio. In the case of (0, 1) reflux ratio the conversion will increase when the mole ratio increase, because of the continues of with draw the desired product, the reaction is always to foreword and cause consumption of the limited component (acetic acid), and increase it is conversion. If reflux ratio is increase to (2, 3) the conversion will increase with mole ratio up to (2) after that the conversion will decrease with increase of the feed mole ratio. That is because of low percent of methanol in feed, not all acid will react, so the conversion is low. As the percent of methanol in the feed increase to a limited amount the conversion increase, then it will decrease because of reversible reaction will occur.

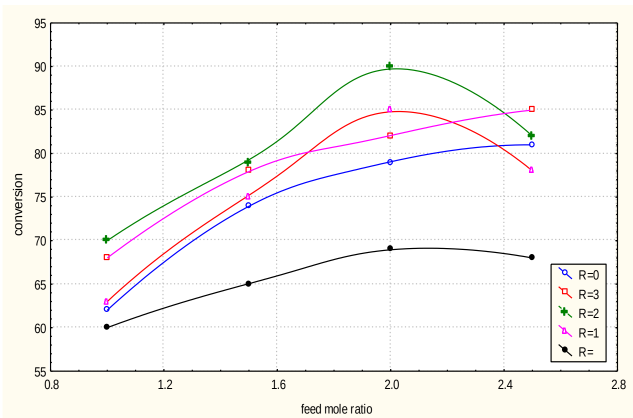

Fig.7: conversion of acetic acid VS. feed mole ratio at 90 minute and different reflux ratio.

CONCLUSIONS

From the present work the following conclusions are made:

1- The concentration of methyl acetate increase with increasing the reflux ratio to maximum value of reflux ratio equal (2) then decrease.

2- The concentration of methyl acetate increase with decrease feed mole ratio.

3- The concentration of methyl acetate increase with time to a limit value depending on the feed mole ratio.

4- The higher concentration of methyl acetate in thee product was obtained at reflux ratio 2, feed mole ratio 1, and for batch time 60 minute.

5- The conversion of acetic acid increase with feed mole ratio and reflux ratio to maximum value equal 2 then decrease.

6- The conversion of acetic acid increase with time.

7- The reactive distillation is more efficient than the conventional process at the best condition mentioned above for reactive distillation the conversion of acetic acid increase by 30.43% than that of conventional process, and the concentration of methyl acetate in the product increase by 75.14% than the conventional process.

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دراسة اداءعمود التقطير التفاعلي

الخلاصة

تم دراسة التقطير التفاعلي ذو الدفعات في عمود ذو حشوات, والنظام المستخدم هو تفاعل الميثانول مع حامض الخليك لانتاج خلات المثيل والماء بوجود حامض الكبريتيك كعامل مساعد, وتم اختيار هذا التفاعل لان التفاعل عكسي ودرجات الغليان للمواد المتفاعله والناتجه متباعده.

تم العمل بهذا النظام بوجود او عدم وجود عمود التقطير وتبين ان التقطير التفاعلي اكفأ من الطريقه العاديه (التفاعل لوحده والتقطير لوحده). حيث ان نسبه التحول لحامض الخليك والتركيز لخلات المثيل تزيد بمقدار (% 30.43 و %75.14) على التوالي عند افضل الظروف(عند راجع 2 ونسبه المواد المتفاعله 2 وزمن 90 دقيقه).

تم دراسه تاثير العديد من المتغيرات منها نسبه الراجع ونسب المواد المتفاعله وتاثير الزمن على اداء برج التقطير ذو الدفعات المصاحب للتفاعل الكيمياوي خلال جريان السائل والبخار داخل البرج وتاثيرها على تركيز المواد الناتجه وتحول المواد المتفاعله.

النتائج التي تم الحصول عليها للخلائط الغير مثاليه لجهاز التقطير ذو الدفعات المصاحب للتفاعل الكيمياوي كانت نسبه التحول لحامض الخليك تساوي 90%عندما تكون نسبه الراجع 2 ونسبه المواد المتفاعله 2 وزمن 90 دقيقه.