Performance of packed bed reactive distillation column

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ABSTRACT – Lab-scale packed bed reactive distillation column was utilized to evaluate the performance of reactive distillation for the reaction between isopropyl alcohol and acetic acid. Two different packing materials and different bed heights were compared to obtain the optimum bed height and packing material for the chosen reaction. Glass Raschig rings and spherical shaped rubber were chosen as the packing materials. Experiments were conducted for the same batch time and desirable packing material and bed height was chosen according to the experiment results. It was indicated that the glass Raschig rings gave isopropyl acetate rich products.

Keywords: Reactive distillation; Isopropyl acetate; Esterification

INTRODUCTION

Reactive distillation is mainly performed for esterification reactions as those reactions are equilibrium limited reactions. Many studies were carried out on reactive distillation with esterification reactions giving reasons such as lower energy requirement and capital cost saving because the simultaneous separation which happens in the integrated reactive distillation column causes the time that was allowed for the backward reaction to occur to be less (Edreder, et al., 2015). As a result, the forward reaction was carried out and the conversion was enhanced in the integrated configuration when compared to that of the separate reactor distillation column design (Gonzalez, et al., 2017).

Esterification reaction between glycerol and acetic acid was studied in a pilot-scale reactive distillation column. In this study as well experimented data was used to validate the simulation of the pilot plant. For the esterification reaction, seepage catalytic packing internals (SCPI), filled with NKC-9 cation exchange resin was used (Li, et al., 2018). Acetic acid and methanol esterification reaction was studied to evaluate the performance of the reactive distillation column. Two different packing materials and catalyst were used in the experiment. Packing materials were Raschig rings and amberlyst IR 120 H+ and the catalyst was 1M sulphuric acid for both cases (Prapainainar, et al., 2014). A different study on methanol and acetic acid esterification reaction was performed by a group of researchers. The low reflux operation with longer batch time would lower the conversion and would not produce the distillate at the required purity (Edreder, et al., 2015). Most of the esterification reactions that researchers had a keen interest in were involved with acetic acid. It was found that the type of packing material had different impacts on the performance. Therefore, the packing materials that were utilized in the present work were glass Raschig rings and spherical shaped rubber.

METHODOLOGY

Process analysis

Components used here were isopropyl alcohol and acetic acid, and the reaction was reversible. The main product was isopropyl acetate and all the experiments were done in atmospheric pressure conditions. Concentration of acetic acid in distillate was determined by a simple titration with NaOH and concentration of ester was determined by a titration with dilute H_2SO_4. A special experimental setup was designed using a column, a condenser, a heating mantle and the other equipment in order to perform experiments, by changing parameters of packed bed column and reactants.

Experimental setup

A glass condenser was used as the packed bed column section. A base to hold the packing material was made by a thin metal net. The bottom and top parts of the column were connected to a three necks still pot and the condenser upper section. The condensation of vapor coming from the packed bed column was done by the condenser. A three neck quick fit still pot (19/25) was used as the reactor and three necks were used to connect the bottom of the packed bed column, to add chemicals into the pot and to hold the thermometer which measures the reaction temperature within the still pot. The still pot was placed within the heating mantle. The two simple titration setups were used to measure the conversion in the still pot, acetic acid accumulation in the distillate and also to measure ester concentration.

Experiments

The parameters that were supposed to change were feed mole ratio, packed bed height and packing material type. First, the experiments were done by changing the feed mole ratio in three steps, moles of acetic acid to moles of IPA - (1:1), (2:1) and (1:2). After that, experiments were done by changing packed bed height under the most productive feed molar ratio. Packed bed height was varied in two steps, full bed height – (15cm) and half bed height – (7.5cm). After that, experiments were done by varying the packing material type, Raschig rings and rubber pieces.

Acetic acid and IPA were allowed to react at 70°C. Samples were taken from the distillate per 20 minutes and titrated with 0.5M NaOH solution. After that, 20ml of heated 0.5M NaOH solution was added to the sample. Finally, the sample was titrated with 0.5M H_2SO_4 to measure the residue NaOH amount.

RESULTS AND DISCUSSION

Effect of feed mole ratio

As described in methodology, all the above experiments were done under three changing conditions.

1. Feed molar ratio
2. Packed bed height
3. Packed bed material

In this scenario the first experiment was done by varying the feed mole ratio. In this research, the two main reactants were acetic acid and isopropyl alcohol. The reactants were heated up and the produced vapour was condensed through the condenser lines. The product was titrated with a 0.1 M NaOH solution and then the product solution was titrated with 0.5 H_2SO_4 again. Finally, ester concentration was evaluated. Constants were bed height (15cm) and bed type (Raschig rings).

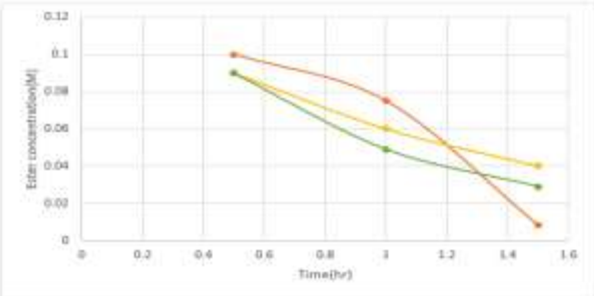
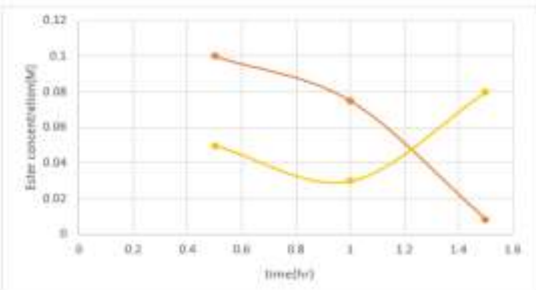


Figure 1. Optimum results were obtained at equal feed mole ratio.

Effect of bed height

From the above set of experiments, it was derived that the equal molar ratio was the optimum condition. Next task was to estimate the best bed height using the optimum molar ratio. Packed bed height was another important factor that affected to the final product concentration. The purpose of this experiment was estimating the optimum height. Experiment constants were the feed mole ratio and the bed type (Raschig rings) Same NaOH and H_2SO_4 solutions were used for the titration process.



From the above comparison, it seems that efficiency was higher when bed height increased. But in experiment 2.1(bed height 7.5 cm), the third value was not accurate. Ester concentration suddenly increased. Analyzing the above graph, it can be estimated that the highest packed bed height (15 cm) gave the optimum condition for the reactive distillation process.

Effect of packed bed type

The efficiency of synthesis of isopropyl acetate depended on the packing material of the bed. Throughout these experiments, Raschig rings were used. In experiment section 1, equal feed mole ratio was the optimum one. The second experiment gave the optimum bed height. The final goal was to find better packing material. For these experiments, optimum parameters that were obtained in previous experiments were used. Here two packing materials were compared while keeping the feed mole ratio (1:1) and bed height (15cm) at their optimum conditions.

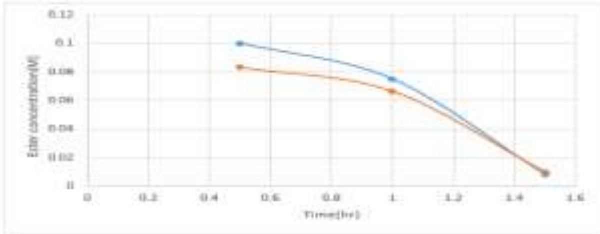


Figure 2. Optimum conditions were satisfied by the experiment which used Raschig rings as the packing material.

CONCLUSION

Performance of packed bed reactive distillation process which is a famous and efficient process in industry. Here the performance of a packed bed reactive distillation column was analyzed by varying major parameters. The first parameter was feed moles ratio. Here, experiments were done using equal molar ratio, using higher acid and IPA concentrations. From those experiments, equal molar ratio gave the optimum performance. Other experiments were done under equal molar ratio and changing the packed bed height. Highest height of the packed bed (15 cm) gave the optimum performance. Finally, the packing material was changed. Experiments were done by varying packing materials (Raschig rings and rubber) while keeping feed mole ratio and bed height at optimum conditions. Raschig rings gave the optimum performance. Finally, the performance of packed bed reactive distillation column was estimated under those three parameters.

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