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COMPARATIVE STUDY FOR ENHANCEMENT OF ESTERIFICATION OF ACETIC ACID WITH N-BUTANOL

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Abstract

In this paper the esterification reaction between acetic acid and n-butanol in the presence of Amberlyst 15 as a catalyst was studied by using ultrasonication technique and by water bath shaker.The experimental conversions obtained by using ultrasonication were observed which are higher than that obtained by using the general water bath shaker. The important point to be noted is that, the ultrasonication gives more yields in lesser time.The influence of severalprocess variables, such as process temperature, initial mole ratio of acetic acid over ethanol, and catalyst content, on the esterification was discussed.In most esterification reactions equilibrium is reached without conversion to a sufficient level .This method is used to get more conversion of acid in lesser time.

Keywords:*Acid, Alcohol, Catalyst, Esterification, Ultrasonication*

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1. INTRODUCTION

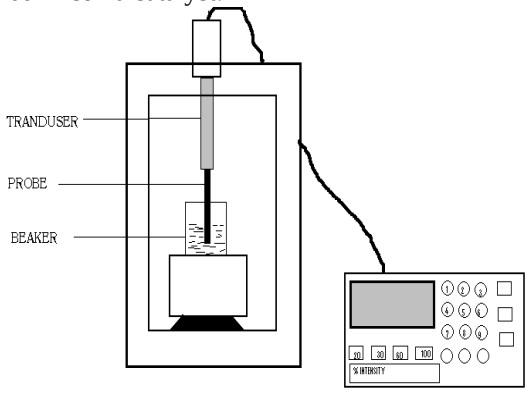
Esterification reactions are typical examples ofreactions controlled by thermodynamic equilibrium and are commonlycatalysed using liquid mineral acids. Commonly esters are prepared by direct esterification of carboxylic acids with alcohols in the presence of acid catalyst. Mineral liquid acids, such as sulfuric acid and p-toluenesulphonic acid are widely used in industrial esterification reactions, owing to their low price and high activity. However the drawbacks they suffered are obvious, such as their corrosive nature, the existence of side reactions, and the difficulty for the separation of the catalyst from the reaction mixture. They also cannot be reused and lead to low selectivity for desired products and produce a large amount of environmentally hazardous acid waste.

All these problems could be solved thoroughly with the solid acid catalysts, which could eliminate the corrosive nature and the side reactions to achieve higher purity of products. The substitution of traditionally homogenous Lewis and Bronstedacidcatalysts by heterogeneous ones, e.g. solid acid catalysts In addition, the solid acid catalyst can be easily reused bysolid decantation or filtration from the reaction mixture. A variety of materials such as clays, zeolites ,sulfated metal oxides , heteropolyacids, etc.have been used as solid acid catalysts for esterification reactions. Zirconia has been widely used as catalyst or catalyst support for acidic catalyst reactions, such as hydrocarbon isomerization, alkylation, and esterification.

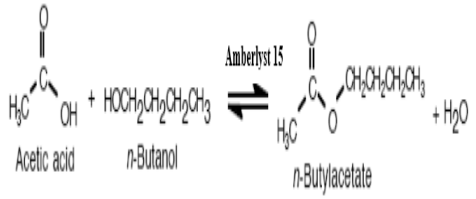
2. METHODOLOGY

The research is at the experimental work for solid catalyst. Esterification Reactions: The esterification reaction of acetic acid with n-butanol is performed by using simple water bath shaker (withoutultrasonication) and using ultrasonication technique. The sample is made up of of acetic acid and butanol, 10 ml of acetic acid (99.5% concentrated) and 12.52 ml of butanol is mixed with 0.2625 gm of solid acid catalyst Amberlyst 15 (Dry). The reaction is carried out in Water bath shaker with at different temperatures (70° C, 80° C, 90° C etc.).Also This sample is kept in B-03 Ultrasonic processor and stirred for 15 minutes by varying intensity (60% , 100%etc).3 ml sample is pipette out from the stirred sample and titrated by using 0.25NNaOH solution. Titrate it till pink colour is observed in the sample which shows formation of ester.

with the solid catalysts by using Conversion vs. time graphs were obtained. These will be compared with the results obtained by esterification reaction using the synthesised nano sized zirconia catalyst.



2.1 REACTION MECHANISM



3.RESULTS AND DISCUSSION

The results obtained for Esterification of Acetic acid and n-Butanolfor 1: 1 ratio using catalyst Amberlyst-15 are as follows:

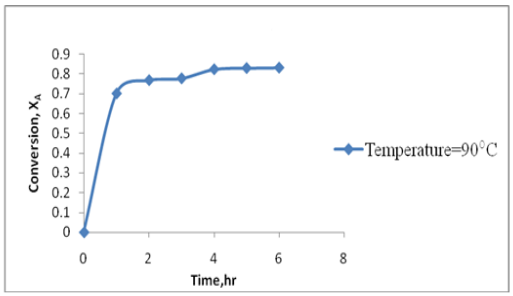


Figure 1: Experimental results for acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 without Ultrasonication.(T=90°C; Acetic acid: Butanol = 1:1; CC=5g/100ml Acetic acid)

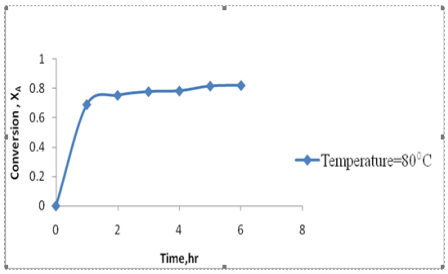
At 90°C, the conversion of acetic acid (XA) increases with time upto 4 hrs and then it becomes almost constant reaching the maximum conversion of 0.83 .Thus the above result

shows that the acetic acid conversion for esterification of acetic acid and n-butanol at 90°C is nearly 0.83 using solid acid catalyst Amberlyst 15 without Ultrasonication.

Figure 2: Experimental results for acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 without Ultrasonication. (T=80°C; Acetic acid: Butanol = 1:1; CC=5g/100ml Acetic acid)

At 80°C, the conversion of acetic acid (XA) increases with time upto 5 hrs and then it becomes almost constant reaching the maximum conversion of 0.81 .Thus the above result shows that the acetic acid conversion for esterification of acetic acid and n-butanol at 80°C is nearly 0.81 using solid acid catalyst Amberlyst 15 without Ultrasonication.

Figure 3: Experimental results for acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 without Ultrasonication.(T=70°C; Acetic acid)



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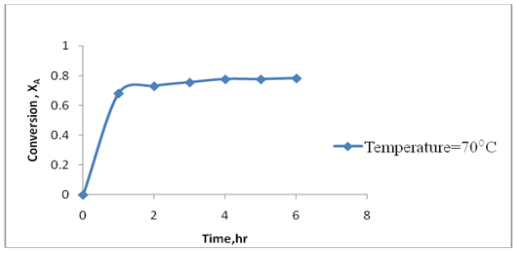

Fig-3: Experimental results for acetic acid conversion for Esterification reactionusing solid acid catalyst Amberlyst15 without Ultrasonication.(T=70°C; Acetic acid: Butanol = 1:1; CC=5g/100ml Acetic acid)

At 70°C, the conversion of acetic acid (XA) increases with time upto 4 hrs and then it becomes almost constant reaching the maximum conversion of 0.77 .Thus the above result shows that the acetic acid conversion for esterification of acetic acid and n-butanol at 70°C is nearly 0.77 using solid acid catalyst Amberlyst 15 without Ultrasonication.

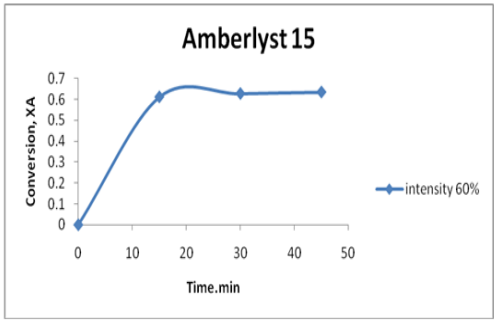

Fig-5: Experimental results for acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 with Ultrasonication.(Intensity 100%; Acetic acid: Butanol = 1:1; CC=5g/100ml Acetic acid)

From the results obtained from Ultrasonication it is observed that at the same reaction time higher conversion is obtained as compared to without ultrasonication

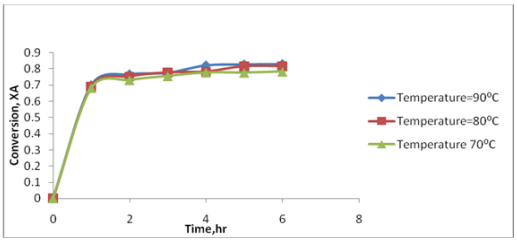

Fig-6: Effect of Temperature on acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 without Ultrasonication.(Temperature=90°C,80°C ,70°C;Acetic acid: Butanol = 1:1;CC=5g/100ml Acetic acid)

From the above figure it is observed that at higher temperature the conversion increases. Thus, increasing the reaction temperature we can increase the conversion.

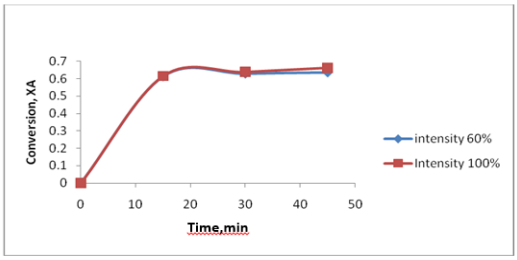

Fig-7: Effect of Temperature on acetic acid conversion for Esterification reaction using solid acid catalyst Amberlyst15 withUltrasonication. (Intensity=100% ,60%; Acetic acid: Butanol = 1:1;CC=5g/100ml Acetic acid)

At 100% intensity the conversion obtained is higher than at 60 % intensity. It shows that conversion increases with intensity for ultrasonication

4.CONCLUSIONS

The esterification reaction of acetic acid and n-butanol is carried out with and without ultrasonication using solid acid catalyst Amberlyst 15. The results are shown as conversion vs time. It is observed that the conversion increases with temperature and reaction time . Also Using Ultrasonication the conversion is increased. Amberlyst 15 is found to be effective catalyst for the esterification reaction along with the advantage of reusability.

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