Hydrolysis of ethyl acetate using low cost heterogeneous catalysts

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ABSTRACT

The hydrolysis of ethyl acetate is an important reaction in process engineering applications. In industrial scale the reaction is carried out under presence of homogeneous acid catalyst which leads to several environmental problems including generation of acidic waste, corrosion of equipment, wastage of catalyst and difficulties in catalyst separation. The objective of the research is to study the suitability of carbon based heterogeneous catalyst for the hydrolysis reaction of ethyl acetate. As the carbon based heterogeneous catalysts have several advantages over homogeneous acid catalysts including low cost of preparation, slower deactivation, abundance, cost effectiveness, higher stability, large number of active sites and minimal interference with the final products. According to the local availability, physical characteristics and practical applicability, saw dust, paddy husk and activated carbon are selected for the catalysis. These substances are pretreated using concentrated hydrochloric by wet impregnation method. Hydrolysis reaction experiments were done under constant temperature and catalyst loading. The percentage conversion of ethyl acetate was analysed in each sample using acid base titration method. The activated carbon catalyst showed better heterogeneous catalyst characteristics for hydrolysis of ethyl acetate out of the selected carbon based substances.

Keywords: ethyl acetate; hydrolysis; heterogeneous catalyst; wet impregnation

INTRODUCTION

Hydrolysis of esters plays a major role in industry to manufacture weak carbonic acids and alcohols for commercial usage in wide range of applications. Without the presence of a catalyst, the rate of hydrolysis of ester is slow and it is not commercially viable for large scale chemical manufacturing. Although homogeneous liquid acid catalysts are extensively used in process industry to increase the product yield, they create undesirable problems.

Performance of hydrolysis of ethyl acetate is studied under presence of carbon based solid catalysts. The chemical reaction can be represented as follows,

$$CH_3COOC_2H_5 + H_2O \rightleftharpoons CH_3COOH + C_2H_5OH$$

Homogeneous acid and base solutions have been used as catalysts in above reaction to achieve comparatively high conversion of ethyl acetate for long period of time in process industry. Utilization of base solution as the catalyst is quite rare as the product contaminated with metal ion exists in the base. Hence most of applications are based on acid catalysis of hydrolysis of esters.

Heterogeneous catalysts have several advantages over homogeneous catalysts including reusability, easiness of separation from the esters layers and no generation of wastewater during the purification stage. Several kinds of

heterogeneous catalysts have been reported in industry such as alkali metal catalysts, alkali and alkaline earth oxide, zeolites based catalysts, zirconia-alumina, amberlyst-15 and anion exchange resin (Yilin Ning, 2017).

Unfortunately, high cost of preparation, sensitivity to water, rapid deactivation, poor stability, rigorous reaction conditions and low acidic site are major obstacles in the application of these catalysts. Hence in recent years, there is a great interest shown in the generation of carbon-based catalysts for ester hydrolysis (J.Ahmada,2018).

This type of catalysts is still not fully explored. Carbon-based catalysts have several advantages over other metal-based catalysts; they have higher surface area, more stability under acidic and basic conditions, low preparation cost, high catalytic performance and low pollution in catalysing ester hydrolysis (Yilin Ning, 2017).

Heterogeneous solid catalysts cause widespread concerns for broad applicability of raw materials and green economy. (Siddiki, 2016) It makes the usage of low-cost materials such as paddy husk and saw dust for ester hydrolysis actualization to overcome the high cost obstacle. Activated carbon which is used in waste water treatment processes as an adsorbent also exhibit physical and chemical characteristics for preparation of supported catalyst.

METHODOLOGY

The performance of the reaction was determined without presence of catalyst. A sample of 200 ml of distilled water and 20 ml of ethyl acetate was reacted in a stopped bottle under 40$_0$C.Samples were pipetted out and kept in the ice bath in different time periods. Likewise, several samples were taken. They were titrated against a standard 0.001MNaOH solution using phenolphthalein indicator. After taking burette values, conversion of each case was calculated and a graph of time vs. conversion was drawn.

Next step was to investigate the system in the presence of catalysts. Paddy husk, saw dust and activated carbon were selected as catalysts. Using a sieve analysis, the particles in the rangex-y was separated. Then wet impregnation of H+ was done for each sample. A weighted amount of catalysts was taken and treated with concentrated HCl. For this a magnetic stirrer was employed and the agitation was carried out for 5 hours. After that excess amount of HCl was removed by washing the catalysts using deionized water. Until the water became neutral washing was done. Then washed catalysts were dried in the oven at 800C for 24 hours.

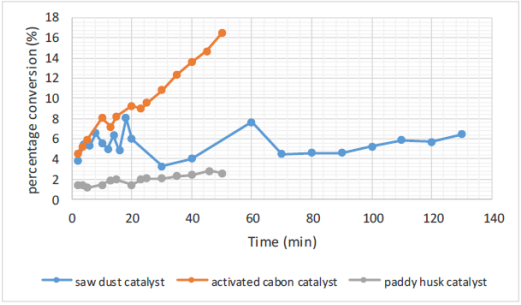

Figure 1. performance of catalysts

The hydrolysis reaction was carried out with 2 g of catalyst loading in the batch reactor at 40 0C. Reagent samples were pipetted out of the reactor at different time intervals. They were titrated against a standard NaOH solution using phenolphthalein indicator. After taking burette values, conversion of each case was calculated and a graph of time vs. conversion was drawn.

RESULTS AND DISCUSSION

Results of the experiment without catalyst shows only 0.05% conversion of ethyl acetate.

According to the results obtained by the experiments, paddy husk, saw dust, and activated carbon based catalyst systems treated with HCL showed 3%, 8% and

17% conversion of ethyl acetate respectively.

A higher conversion shown in activated carbon catalyst could be due to its better catalyst surface properties with porous structure and additional partial carbonization. Hence it is recommended activated carbon as a suitable heterogeneous support catalyst for hydrolysis of ethyl acetate.

CONCLUSION

Hydrolysis of ethyl acetate was studied using three types of heterogeneous catalysts including saw dust, paddy husk and activated carbon treated with HCl. The hydrolysis of ethyl acetate proceeds very slowly with the absence of a catalyst. Carbon based catalysts showed improved characteristics to use as heterogeneous catalysts for hydrolysis of ethyl acetate. Using a batch experimental setup, the percentage conversion of ethyl acetate was analysed for four catalyst reacting systems including system without the catalyst and system with acid impregnated catalysts of 2g/ 220ml of reaction volume. According to the analysed results the sample with activated carbon showed the highest conversion per unit time at 40 ºC constant temperature with a catalyst loading 0.00909g/ml.

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