Esterification of acrylic acid using a catalyst prepared from waste bone- Catalyst characterization and Influence of operating parameters on yield

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Highlights

- Characterization of solid activated carbon catalyst prepared from waste bone
- Effect of temperature and catalyst concentration on the esterification of acrylic acid using the prepared catalyst.
- Catalyst activity/Comparison with other commercial catalysts.
- Catalyst reusability

1. Introduction

Esterification involves the reaction of alcohol with carboxylic acid, resulting in the formation of an ester and water as products. Esters are widely used as flavours and perfumes. Esterification is a reversible and slow reaction, and the presence of a catalyst is essential to accelerate the reaction rate and lower the activation energy. To increase reaction yield, industrially, homogeneous catalysts such as sulphuric acid or hydrochloric acid are used, but they suffer from issues such as waste generation, difficult separation, and the corrosive nature of the homogeneous catalysts. This leads research to focus on the use of heterogeneous catalysts (Aafaqi et al. 2004). However, the major challenges associated with commercial heterogeneous catalysts are their high costs and reusability (Farnetti et al., 2009). In view of this, in the present work, a solid catalyst from waste bone was prepared and employed for the esterification of acrylic acid using ethanol. The effects of temperature and catalyst concentration on esterification were studied. The prepared catalyst was compared with conventional catalysts, and catalyst reusability was also investigated.

2. Methods

Esterification studies were carried out in a magnetically stirred (500 rpm) three necked ball glass flask fitted with a reflux condenser. Reactants were heated to the desired temperature before being added to the reactor. The experiment has been carried out at 50, 60 and 70°C and catalyst concentration in the range of 8-12% (vol) of the reaction mixture with a molar ratio of 1:1. Waste bones were collected from a local slaughterhouse and dried at 80°C temperature in a hot air oven for two days until oil extraction ceased completely. Moisture-free bones were then calcined in a muffle furnace at 700°C & kept for 1 hour to prepare crushed and sieved activated carbon. Sulfonation was performed at 100°C for 12 hours to induce sulfonic acid groups into the pores of the activated carbon.

3. Results and discussion

FTIR spectra of the prepared catalyst, resulted in band near 3416 and 1630 cm−1, assigned to -OH vibration, and at 1124 cm–1 for S-O stretching modes, because sulphate ions are present in the prepared catalyst. The region between 500 and 700 cm-1 shows hydroxyapatite [Ca10(PO4)6(OH)2] which is the major constituent of the mineral phase of bone. The bands at 2854 and 2926 cm-1 in the FTIR spectrum are assigned to asymmetric and asymmetric CH2 stretch modes. XRD reflections were obtained at 2θ = 25.45, 31.37 and 40.82, which corresponds to the diffraction planes of 002, 211 and 310, respectively. The XRD of the catalyst shows the presence of peaks of compounds namely hydroxyapatite, calcium

oxide (CaO) and tri-calcium phosphate. SEM image of the catalyst prepared from bone shows that particles have irregular shapes and images revealed, the non-uniform deposition of fine particles on catalyst surface which acts as active sites for the reaction.

An increase in temperature (50-70°C) resulted in a conversion increase from 34.4% to 51.0% . The rate of reaction increases with temperature because, at higher temperatures, more collisions between molecules occur (Jong et al. 2009). Catalyst concentration affects the reaction rate because, as catalyst concentration increases in the mixture, more H+ ions are available. For different amounts of catalyst (8, 10 and 12% (w/w) of the reaction mixture), conversions of acrylic acid were obtained as 44.9, 48.5 and 49.5% after 7 hours, respectively. As expected, the conversion of acrylic acid increases with increased catalyst concentration due to a larger number of active catalyst sites, resulting in high reaction rates to produce a higher yield of product. The performance of the prepared catalyst was compared with other heterogeneous catalysts, namely, Dowex 50WX and Amberlyst 15. The conversion of acrylic acid using prepared catalyst, Dowex 50Wx and Amberlyst 15 was found to be 48.5%, 47.6% and 41.2%, respectively. These results showed that the activity of the prepared catalyst was more than that of commercial catalysts. To assess the catalyst's recyclability, the catalyst was first separated from the reaction system, washed with n-hexane, and then dried in a hot-air oven after subsequent reaction cycles. Experiments were performed under reaction conditions, including a temperature of 60 °C, an initial molar ratio of reactants of 1:1, and a catalyst concentration of 10% (w/w), to study the reusability of the solid acid catalyst. It was observed that a loss of activity occurred after three consecutive runs. The conversion of acrylic acid decreased by 32% after the third run during esterification with ethanol. The conversion of acrylic acid decreased from 48.51 to 33.2% after the third reaction cycle using the prepared catalyst. Deposition of reactants and/or products on the active sites, along with the leaching of active acidic sites from the surface of the solid acid catalyst, leads to a decrease in catalytic activity (Chandane et al. 2017).

4. Conclusions

The prepared activated carbon catalyst from waste bone was employed for estrification of acrylic acid using ethanol. The catalyst was characterized using FTIR, XRD and SEM. To study the reactor performance, the effect of temperature and catalyst concentration was investigated. For commercialization of the prepared catalyst, its performance was compared with the conventional catalysts and reusability studies were also carried out. The result signifies the potential use of a prepared catalyst for the esterification reaction for higher yields of esters.

References

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Keywords

Esterification, Characterization, Temperature, Catalyst Concentration