CHAPTER V
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study prepared the heterogeneous acid catalysts from the oxidation of crumb rubber. The oxidation was performed by using two different oxidizing agents, i.e. hydrogen peroxide and nitric acid. Then, the oxidized crumb rubbers were applied as the catalysts in the esterification of octanoic acid with long chain alcohols.

Hydrogen peroxide is relatively mild oxidizing agent which has to be assisted with formic acid for the oxidation, resulting in the characteristic changed of crumb rubber. The oxidation of crumb rubber by both nitric acid and hydrogen peroxide with formic acid generated the polar acid groups in the resulting products in which the sulfonic group (-SO_3H) and carboxylic acid (-COOH) were the main acidic functional groups. For the crumb rubber oxidized by hydrogen peroxide mixed with formic acid, the natural rubber as main chain rubber was oxidized to generate aldehydes (-CHO) and ketones at the chain ends. In case of nitric acid, natural rubber and styrene-butadiene rubber were severely oxidized to generate the highly functionalized short-chain polymer. Moreover, the nitro group (-NO_2) was only found in case of using nitric acid as the oxidizing agent. Due to the presence of these functional groups, the polarity and the affinity for the non-polar and the polar solvent were changed. In addition, the presence of polar acid functional groups also increased the acid content of the oxidized crumb rubber. The acid content of CR-H_2O_2 series was lower than the CR-HNO_3 series. From these results, the suitable conditions for preparation of the crumb rubber oxidized by using nitric acid was 6 M in concentration at 80 ^{o}C for 3 h. Moreover, the crumb rubber oxidized by using hydrogen peroxide was unsuitable for using as catalyst because the octanoic acid conversion was decreased. In contrast, the oxidized crumb rubber by using nitric acid was successfully used as the catalyst in the esterification of octanoic acid with octanol or 2-ethyl-1-hexanol. However, the color of reaction mixture was changed from the transparent solution to dark yellow solution due to the dissolution of catalyst into the reaction mixture. As the result, the catalyst recovery was lower than 50%.

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5.2 Recommendations

The oxidized rubbers prepared in this study have not yet been suitable to use as the heterogeneous acid catalysts for the esterification due to their low acidity and high solubility in organic media. To increase the acidity, choosing the rubber with high sulfur content, such as the ebonite rubber as a raw material, probably gained the product with high acid content after the oxidation. Moreover, the dissolution of rubber might be solved by crosslinking the oxidized rubber again. However, the proton nuclear magnetic resonance spectroscopy (^{1}H NMR spectroscopy) should be applied to confirm the dissolution of catalyst in the reaction mixture. In addition, the oxidized rubber should be tried in other applications, for example the adsorption in aqueous media due to its low solubility in water.


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