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Synthesis and characterization of calcium methoxide as heterogeneous catalyst for trimethylolpropane esters conversion reaction

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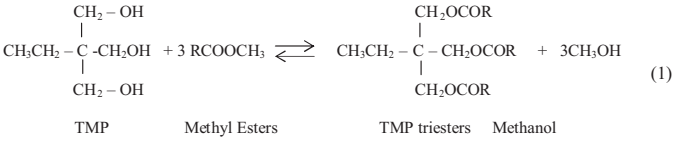
ABSTRACT

Trimethylolpropane (TMP) esters are potential biodegradable basestock for biolubricant. In order to attain environmental benignity, attention has been focused on utilizing heterogeneous catalysts for production of TMP esters. Alkaline homogeneous catalysts tend to react with free fatty acids to produce unwanted soap, thus reducing the overall product yield. This study had focused on the synthesis of calcium methoxide and investigating its potential as heterogeneous catalyst for the transesterification of TMP and palm oil methyl esters (POME) to TMP esters. The performance of synthesized calcium methoxide as a catalyst was examined by characterizing it through some instrumental techniques. X-ray diffraction (XRD) showed calcium methoxide has been successfully synthesized. Scanning electron microscopy (SEM) displayed thermally resistant surface structure with good porosity; BET showed high surface area; particle size analysis evidenced reasonable size of catalyst particles; and thermogravimetry (TGA) revealed good thermal stability of synthesized calcium methoxide. Moreover, the catalyst was found to possess mesoporous surface by pore size analysis through Barrett–Joyner–Halenda (BJH) method. The results of transesterification reaction indicated satisfactory catalytic activity of synthesized calcium methoxide and the TMP triesters yield obtained was 80.35% after 2 h, 87.48% after 4 h, 91.30% after 6 h and 92.38% after 8 h reaction time.

1. Introduction

The concern and awareness of the harmful impact of mineral based lubricants on the environment have pushed the research towards the production of environmental friendly lubricants from vegetable oils. Due to some performance limitations, vegetable oils are not considered suitable to be used directly as lubricants [1]; nonetheless, the limitations can be minimized by means of chemical modification of vegetable oils through transesterification with polyhydric alcohols [2]. In similar fashion, researchers have performed transesterification of vegetable oil methyl esters with trimethylolpropane (TMP) to generate esters of TMP, which are generally utilized as base oil for various types of lubricating oils [3–9].

The transesterification reaction involves three consecutive reactions in the presence of a catalyst. Monoesters (ME) and diesters (DE) of TMP are formed as intermediate products towards the completion of the reaction producing TMP triesters. The overall reaction stoichiometry requires one mole of TMP and three moles of methyl esters. The overall reaction scheme is shown by Eq. (1).



(1)

Catalyst holds a prominent role in the transesterification reaction of TMP and vegetable oil methyl esters, in order to obtain a better yield of TMP triesters in less time. Reported catalysts for the production of TMP esters include homogeneous bases such as sodium methoxide [10], homogeneous acids such as sulfonic acid [11], enzymes such as lipases [4,5] and heterogeneous base calcium methoxide [6]. Alkaline homogeneous catalyst was highly

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