

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS AND
CHARACTERIZATION OF
SULFONATED SnO₂ CATALYSTS
FOR ESTERIFICATION OF LOW-
COST PALM FATTY ACID
DISTILLATE FOR BIODIESEL
PRODUCTION**

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

Excessive use of non-renewable fuel in transportation and industry leads to the twin crises of fossil fuel depletion and environmental degradation. As fossil fuels are limited and it takes millions of years to be produced, their availability may be prolonged by decreasing overall consumption. Following this issue, various renewable sources of energy have successfully been tested to replace the dependency on fossil fuel consumption. Since Malaysia is the largest exporter of palm oil, biodiesel derived from palm fatty acid distillate (PFAD) was produced *via* catalytic esterification using sulfonated tin oxide ($\text{HSO}_3^-/\text{SnO}_2$) as a superacid solid catalysts. In this work, the SnO_2 catalysts were synthesized by different synthesis methods such as solid-state method, sol-gel method and self-propagating combustion method. After that, all the SnO_2 catalysts was further integrated with HSO_3^- ions by chlorosulfonic acid. The commercial SnO_2 was also sulfonated and used for comparison. Among all the synthesis methods, sulfonated SnO_2 -SPC exhibited superior physicochemical properties in terms of homogeneous nano-sized particles that covalently anchored greater HSO_3^- ions which is important criteria for the esterification of PFAD. All SnO_2 based catalysts from non-sulfonated and sulfonated groups were preliminarily screened for the esterification of PFAD feedstock. Among them, the sulfonated SnO_2 -SPC catalyst showed remarkable esterification activity. The used of the SPC method produced nano-sized particles with homogenous size and shape, that anchored many HSO_3^- ions resulted in greater acid properties that effectively esterified the PFAD feedstock into FAME (fatty acid methyl ester). At the optimized condition of 9:1 (methanol:PFAD molar ratio), 4 wt.% (catalyst loading), 100 °C (reaction temperature) and 3 h (reaction time), the FFA conversion and FAME yield were 98.9% and 93.8%, respectively. Besides, the sulfonated SnO_2 -SPC catalyst could be reused up to five consecutive cycles with an acceptable esterification performance and minimal sulfur leaching. Additionally, the fuel properties of PFAD biodiesel were investigated and compared with both the American (ASTM D6751) and European (EN 14214) standards. Overall, the production of FAME from low value, cheaper, abundance, and non-edible PFAD feedstock assisted with non-transition metal oxide of sulfonated SnO_2 catalyst met all the requirement standards proposed by ASTM D6751, EN 14214 and diesel.

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