UNIVERSITI TEKNOLOGI MARA

EFFECT OF SODIUM OXIDE MOLAR RATIO ON BENTONITE SUPPORTED CATALYST FOR THE TRANSESTRIFICATION OF WASTE COOKING OIL

IZYAN BINTI YUSOF

Thesis submitted in fulfilment
of the requirements for the degree of
Master of Science
(Chemistry)

Faculty of Applied Science

March 2024

ABSTRACT

Presently, in addition to the feedstock and solvent, very expensive catalyst materials are used in the production of biodiesel. The aim of this study is to convert waste cooking oil (WCO) into biodiesel via transesterification with Na_2O/bentonite catalyst. Six series of modified Na_2O/bentonite were prepared at different NaOH-bentonite molar ratio (1:1, 2:1, 3:1, 4:1, 5:1, and 6:1) via wet impregnation method. The synthesized catalyst was successfully characterized using CO_2-Temperature Program Desorption (CO_2-TPD), BET surface analysis, Powder X-Ray Diffraction (PXRD), Field Emission Scanning Microscopy with Energy Dispersive X-Ray Spectroscopy (FESEM-EDX), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR). The WCO feedstock had undergo pretreatment analysis to fit the transesterification process. The screening test of the Na_2O/bentonite catalysts was conducted via transesterification reaction and optimized using Response Surface Method (RSM) with Box-Bechken Design (BBD). In addition to the reusability test and spent catalyst analysis, the heterogeneous Na_2O/bentonite catalyst's catalytic activity was evaluated. The crystallinity of the synthesized catalyst proved to be a semi-cryatalline materials via PXRD analysis. Successful impregnation of Na_2O compound into bentonite was highly proved as the important peak, Na_2O appeared at 2θ = 26.581, 35.693, 39.501, 43.275, and 53.883°. CO_2-TPD analysis results indicate that Na_2O/bentonite catalyst have strong and very strong basic site based on the temperature desorption. In general, the total basicity increases when the molar ratio of Na_2O increase. The decreasing surface area from 57.44 m^2/g (raw bentonite) to 2.67-5.56 m^2/g (Na_2O/bentonite catalysts) indicated successful impregnation of Na_2O molecules into the bentonite's porous structure. The WCO pretreatment shows that the free fatty acids (FFA) value, saponification value, and average molecular weight are 0.99 wt%, 156.21 mg KOH/g, and 1090.51 g/mol, respectively. The screening test of Na_2O/bentonite catalysts shows that 5:1 molar ratio found to be the best ratio with FAME yield of 85.4%. ANOVA analysis shows that a quadratic polynomial model had been developed with R^2=0.9583, and insignificant lack-of-fit. By using RSM, 55.81% (residual standard error, RSE: 2.74%) of FAME yield was achieved at the optimum reaction condition of 17.9:1 methanol-to-oil molar ratio, reaction temperature of 50.6 °C for 11.9 hours, with 4.7 wt% of a 5:1 Na_2O/bentonite catalyst loading. The catalyst reusability test found that the catalyst can be used up to three cycles. The CO_2-TPD shows that the catalyst starts to degenerate at 4th cycle as the total basicity drops from 913.30 to 509.70 μmol CO_2/g. The leaching process had caused the increase in BET surface area (2.9734 to 3.4420 m^2/g) after the 6th cycle due to the sodium moving out from the bentonite's and decrease of sodium atomic percentage from 6.03 % to 3.56 % from the FESEM-EDX analysis. For recommendation, alternative support catalysts like graphitic carbon nitride can improve catalyst stability and reduce sodium leaching as one study success to produced over 90% biodiesel in ten cycles.

ACKNOWLEDGEMENT

Firstly, I wish to thank Allah S.W.T for giving me the opportunity to embark on my MSc and for completing this long and challenging journey successfully. My gratitude and thanks go to my supervisor Assoc Prof ChM Dr. Norani Hamzah for her guidance, encouragement, and assistance throughout my MSc research work. Special thanks also dedicated to Assoc Prof Dr. Mohd Lokman bin Ibrahim and Dr Nazrizawati binti Ahmad Tajuddin as my co-supervisors for their kind supervision and moral support when carrying out my research.

I am also thankful and grateful to my family who supporting me since the beginning of my MSc research. I would also like to express my appreciation to my close friends for their endless support, encouragement, and prayer, which has helped me able to complete this research.

Not forgotten, special thanks to my MSc friends, staffs, and technicians at the Institute of Science (IOS), Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, Selangor.

TABLE OF CONTENTS

CHAPTER ONE

INTODUCTION

1.1 Research Background

Any substance that generates energy through combustion can be categorised as a fuel. It comes either in solid or liquid forms; for example, solid material such as charcoal and peat. Diesel, gasoline, ethanol, and liquefied petroleum gas (LPG) are some examples of fuels in the liquid phase (Muhammad *et al.*, 2018). Fossil fuel can be divided into three categories which are petroleum or oil, coal, and natural gas (Fuels and Fuel Chemistry, 2020). Fossil fuels, which include coal, oil, and natural gas, have been supplying most of the world's energy for more than 150 years (Environmental and Energy Study Institute (EESI), 2021). This is due to the fact that diesel engines were utilised in the production processes of the majority of industries.

The sustainability of petroleum-based fuel supply has gained broad attention from the global community due to increased usage in various sectors, depletion of petroleum resources, and uncertainty around crude oil market prices. Furthermore, it has been accepted as a fuel and fuel additive worldwide and registered with the U.S. Environmental Protection Agency (EPA) (Zahan & Kano, 2018).

The diminishing petroleum reserve is a problem that is always attached to this type of fossil energy. The main reason for finding alternative diesel engine fuels is the high price. The cost of the catalyst, which is high-priced, together with specialised machinery including separators, pumps, reactors, mixers, pipes, and heaters have an impact on the price of making biodiesel (Hamid *et al.*, 2022). To face the challenges in this energy sector, it is necessary to utilize the potential of new and renewable energy. One alternative energy source that has the potential to be developed is biodiesel.

Biodiesel, which is also synonymous with biofuel, is a renewable energy source that possesses the capacity to supplant fossil fuels. Due to the fact that it is biodegradable, bio-renewable, and nontoxic, biodiesel may be regarded as a viable and prospective energy source. The transesterification reaction, which involves the chemical conversion of triglycerides and alcohol in the presence of a catalyst to