

Synthesis, Characterization and Evaluation of two Forms of Sulphated Zirconia for Triglyceride Cracking

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Abstract

Catalyst technology has a substantial impact on the sustainability of industrial processes, products, environment, the economics and quality of life of any country. Currently the world is becoming increasingly environmentally aware and renewable transport fuels such as biodiesel are becoming increasingly prevalent. Currently, the major technical challenges in conventional biodiesel production (by transesterification with methanol) are: unwanted side reactions, difficult downstream separations, and ensuring sustainability^[1]. Catalysis can make a significant contribution to improving this process, as the load on downstream processes would be greatly reduced, and the flow sheet greatly simplified, thereby reducing capital cost. However, another catalytic-based solution is heterogeneously catalysed vegetable oil cracking^[2]. This would remove the dependence on methanol, which is seldom produced sustainably and is a significant process cost. Current challenges to catalysis of this reaction include maximizing efficiency and ensuring that the catalyst is chemically and thermally stable, and can be regenerated.

Catalytic properties are strongly affected by preparation method^[3]. Hence it is necessary for researchers to investigate different methods of preparation in order to optimise catalyst performance. Sulphated zirconia is a super-acid catalyst, used for many organic reactions. However, it has been found to be susceptible to leaching, rapid deactivation during catalytic reactions and a relatively small surface area when prepared through the conventional method^[4].

In this study, the conventional wet route of preparing sulphated zirconia (by the precipitation method) was compared to a "direct synthesis" involving simple calcination of a mixture of zirconium oxychloride and ammonium sulphate for thermocatalytic cracking of triglycerides

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at 270°C. The pore sizes and surface areas of the catalysts were 169m²/g, 0.61µm (directly synthesized) and 65m²/g, 0.24µm (conventional method), respectively. XRPD revealed directly synthesized sulphated zirconia as amorphous and the conventionally prepared as polycrystalline.

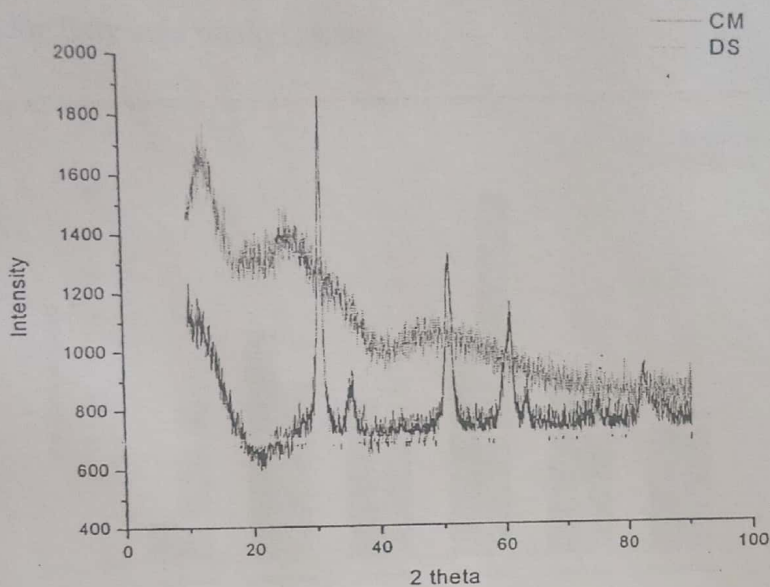


Figure 1: The XRD of the Catalysts

Their FTIR spectra were generally similar, although the area of the 1300 to 920 cm⁻¹ band was larger in the case of direct synthesis, indicating more absorption of sulphate ions on the zirconium cation.

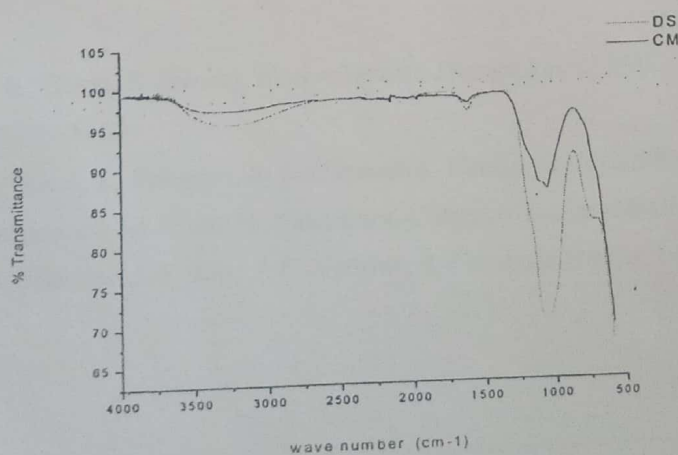


Figure 2: FTIR spectra of the catalysts

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Not only were conversions greater for directly synthesised sulphated zirconia than conventionally produced (63% and 42% after 4h, respectively), but direct synthesis exhibited greater selectivity for fatty acid methyl esters.

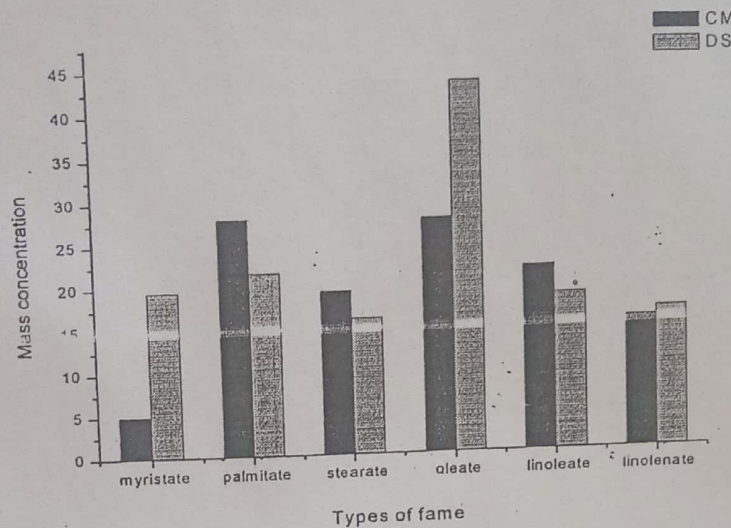


Figure 2: Different composition of fatty acid methyl acid (FAMES) from the catalysts

The percentage yield (after 1h) of methyl esters was found to be 43% using the directly synthesised sulphated zirconia and 15% for the conventional method.

Reference

- [1] S. A. Basha, K. R. Gopal, S. Jebaraj, *Renew Sustain Energy Rev* (2009), doi:10.1016/j.rser.2008.09.031
- [2] M. D. Serio, R. Tesser, L. Pengmei, E. Santacesaria, *Energy fuels* (2008), 22, (1), 207-217
- [3] K. Arata, H. Matsushashi, M. Hino, H. Nakamura, *Catalysis Today* (2003) 81, 17-30
- [4] F. R. Chen, G. Coudurier, J. F. Joly, J. C. Vedrine, *J. Catalysis* (1993), 143, 616-626

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