

# Preparation and Characterization of Biodiesel Produced from *Jatropha* Seed Oil Using Sulphated Zirconia as Catalyst

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## Abstract

Biodiesel has become beguiling nowadays for its environmental benefits and it seems an opposite alternative fuel for future. In the current study, the biodiesel was produced from *Jatropha* seed oil using sulphated zirconia as catalyst. The catalyst was characterized using Fourier transform infrared (FTIR), differential scanning calorimetry (DSC), X-ray diffraction (XRD), thermogravimetric analysis (TGA/DTA) and X-ray fluorescence (XRF). The GC-MS result of biodiesel produced shows 8.46% hexadecanoic acid, introduction of

Fossil fuels are one of the major sources of energy in world today. It can be accounted to easy usability and considered as alternatives to fossil fuels. Palm oil, coconut oil among other oil is required in

refined forms to obtained quality biodiesel, in Addition to their food needs, this makes the production of biodiesel from their sources uneconomical [5]. Non-edible oils obtained from plant species such as *Jatropha curcas* L and *Castor* seeds may provide better de-b aties Ade h reay pot nti opiodiesel Orodutioncausa atc

*Jatropha* seed was obtained from Sokoto Energy Research Centre, Usmanu Danfodiyo University Sokoto premises, Sokoto State, Nigeria

## Sample preparation and extraction of oil

The seed was sundried to remove moisture content and ground into powder. The *Jatropha* oil extraction was carried out by soxhlet extraction method as reported by Farag et al. [7].

## Preparation of catalyst

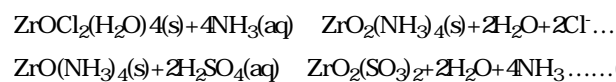
The catalyst was prepared heterogeneously by a method of Ameliorated method below

## Preparation of S-ZrO<sub>2</sub> powder

The fine S-ZrO<sub>2</sub> powder was prepared by ameliorated method which was based on the method reported by Hara and Miyayama. Zirconium oxychloride hydrate (ZrOCl<sub>2</sub>·8H<sub>2</sub>O), ammonia (NH<sub>3</sub>·H<sub>2</sub>O) and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) were used as starting materials. Ammonia was used as precipitating agent and sulphating agent, respectively 20% NH<sub>3</sub> ac gradually dropped into ZrOCl<sub>2</sub>·8H<sub>2</sub>O solution (0.20M) and the value was adjusted to 10 hydrate an

The ZrO<sub>2</sub>·nH<sub>2</sub>O hydrogel was changed into its alcogel by washing several times with anhydrous ethanol. The fine ZrO<sub>2</sub> powder was obtained from the ZrO<sub>2</sub>·nH<sub>2</sub>O alcogel by washing several times with anhydrous ethanol. The fine ZrO<sub>2</sub> powder was obtained from the ZrO<sub>2</sub>·nH<sub>2</sub>O alcogel supercritical drying method at 260°C and 7MPa. The ZrO<sub>2</sub> powder was added to 0.20M H<sub>2</sub>SO<sub>4</sub> under vigorously stirring for 30 minutes, minus

powder:



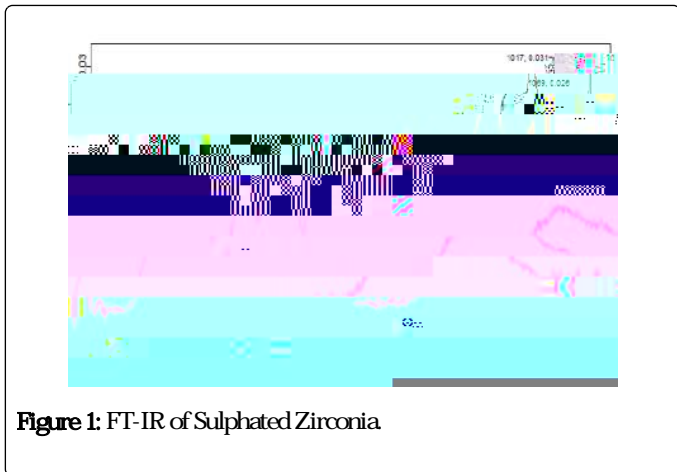
The product will be:

Sulphated Zirconia ZrO<sub>2</sub>(SO<sub>3</sub>)<sub>2</sub>

Characterization of catalyst

T e S-ZrO<sub>2</sub>

represent  $ZrO_2$  the stretching vibration, the peak absorption at and the absorption peaks at 2363 and 2325 and are due to S-H stretching the characteristic absorption of sulphated zirconia observed correspond to the absorption of  $SO_4^{2-}/ZrO_2$  reported by Yunfeng et al. [15].



**Figure 1:** FT-IR of Sulphated Zirconia

**DSC analysis** The DSC (differential scanning calorimetry), analysis of  $S-ZrO_2$  as indicated in Figure 2, show glass transition temperature (the temperature at which the catalyst undergoes change in heat capacity without change of state) as 74.82°C, the crystallization temperature (the temperature at which catalyst amorphous solid change to

biodiesel yield results may be due to the different methods of the catalyst preparations, catalyst loads, temperatures and oil to methanol ratios.

#### Physicochemical properties of biodiesel produced

**Acid value:** The acid value of the biodiesel determines the level of free fatty acids present in the biodiesel. The acid value of biodiesel produced was 2.02 mgKOH/g, as presented in Table 1 is higher than 0.5 maximum ASTM standards. This can be corrected by neutralizing the oil before use to suit engines in order to avoid corrosion and wear in fuel systems and storage tanks.

**Iodine value:** The iodine value of biodiesel produced is 104 gI<sub>2</sub>/100 g as presented in Table 1, which is within the range of 130 maximum ASTM standards. The value shows low degree of unsaturated compound in the biodiesel produced, this is upheld by GC-MS analysis of the biodiesel produced which indicates low degree of unsaturated methyl esters i.e., (82.3%) hexadecanoic acid, methyl ester; (12.14%) 9,12-octadecadienoic acid, methyl ester and (10.82%) 11-octadecanoic acid, methyl ester.

**Saponification value:** The saponification value of *Jatropha* biodiesel is  $186.00 \pm 2.00$  mgKOH/g as shown in Table 1, similar result (190.00 mgKOH/g) was obtained by Singh et al. [18]. The saponification value suggests that the biodiesel has higher molecular weight that may be responsible for its higher specific gravity (0.85) of the biodiesel produced. The high saponification value indicates higher methyl ester contents, as ester value is the

