

CHARACTERISTICS OF JATROPHA SEED OIL FROM YEMEN

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Abstract

Yemeni Jatropha oil was investigated with respect to its applicability in biodiesel production. The extracted oil (~20% of the seed weight) has a low acid value of 0.86 mg KOH/g Oil corresponding to free fatty acid content (FFA) less than 0.5%. Accordingly, no extra preesterification with methanol is required to remove the FFA. Moreover, the preesterification using H₂SO₄ or solid natural bentonite was found to reduce the acid value only slightly down to 0.6-0.8 mg KOH/g Oil. Conventional homogeneous transesterification of (preesterified) Jatropha oil with methanol at 60°C using KOH as a catalyst was found to convert the Jatropha oil quantitatively into the corresponding methyl esters (FAME). Different forms of Mg(OH)₂-MgO phases were also investigated as potential transesterification catalysts in an attempt to replace the homogeneous environmentally unfriendly KOH catalyst with a solid base. Thermal treatment of Mg(OH)₂ for two hours at 350°C (MgO-350) was found to give the best Mg(OH)₂-MgO form being capable to convert almost 70% of the Jatropha oil into biodiesel. MgO-350 seems thus very promising and is the subject of further investigation. The specifications of the biodiesel produced in this work match the standards. Sulfated ash is a little bit high but this was attributed to the poor filtration of the oil before sending it to transesterification. Our results also suggest that the IR spectroscopy is an easy way to monitor the progress of transesterification and can be applied as a control technique in biodiesel production.

Keywords: Jatropha Seeds, Heterogeneous Catalysts, Biodiesel

INTRODUCTION

The potential benefits of the spread of Biodiesel Crops to society are far more than just reducing environmental damage. Through adopting technologies that reduce energy, water and resource usage, societies will increase their productivity, their global competitiveness and drive local economic development and employment. The ultimate goal of Bio-energy is to integrate it into everything we do, and, in the words of Vinod Khosla (one of the world's leading green energy venture investors), to become 'maintech' (Jain et al 2009, Lang et al 2001). The category will then not be something special: it will just be the way

things are done.

Clean Technology investments therefore not just about providing the scientific foundation for future technologies. Green energy is an essential part of the transition to a world in which efficiency improves, productivity and economic growth improves, and communities function more effectively.

The utilization of edible food crops (corn, soya, etc.) for the production of biofuels are expected to

Create a short supply of food for human consumption. The utilization of non-edible and renewable crops such as *Jatropha* is expected to minimize this problem. *Jatropha* is a valuable multi-purpose crop that can help to alleviate soil degradation, desertification and deforestation, and it can be used for bio-energy to replace petro-diesel, for soap production and climatic protection, and hence deserves specific attention. *Jatropha* can help to increase rural incomes, self-sustainability and alleviate poverty for women, elderly, children and men, tribal communities, small farmers. It can as well help to increase income from plantations and agro-industries (Kazi et al 2006, Le Tu Thanh et al 2012, Teresa et al 2016).

For industrial biodiesel production, homogeneous basic catalysts, including potassium hydroxide, sodium hydroxide, as well as potassium and sodium alkoxides, are commonly used for the process suffers from several drawbacks, such as difficulty in recycling catalyst and environmental pollution. To circumvent homogeneous process problems attempts to use heterogeneous catalysts in the transesterification of Yemeni *Jatropha* oils have been made.

The objective of the present research is to evaluate the characteristics of Yemeni of seed oil extracted from the *Jatropha* seeds collected from Ibb (Wadi Jannat) Yemen. The outcome of this study revealed that the *Jatropha* oil may have a potential as a biodiesel feed stock.

Material and Methods

Seed Material

Jatropha fruits were obtained from Ibb Governorate (Wadi Jannat), where the plant is growing. The fruits were peeled off to obtain the seeds which were found to constitute ~ 60 % of the fruit mass. The seed were selected in such a way, the damaged seeds were discarded and the seeds in good condition were cleaned, de-shelled and dried at a temperature of 100-105°C for 35 min. seeds were ground using a grinder before oil extraction.

Oil Extraction

Jatropha seeds were finely ground by means of a standard kitchen grinder. n-hexane or cyclohexane was then added with the ratio of 1 L hexane/1 Kg seeds. The obtained slurry was mixed at 2500 rpm for 1 hr at room temperature. The liquid phase was then separated by means of suction filtration and hexane was consequently removed from the liquid phase by evaporation under reduced pressure in a rotor evaporator (Büchi Rota evaporator R110).

Chemical Analysis of Seed Oil

Acid Value, % FFA

The acid value was carried out in laboratories, of Chemistry Department, Yarmouk University.

The acid value (A.V.) was determined according to the standard method II.D.1 adopted by the IUPAC [79]. The method is based on titrating the free fatty acids present in oil using ethanolic KOH solution and phenolphthalein as indicator. The oil sample was dissolved in a 1:1-solvent mixture of 96% ethanol and diethyl ether. The acid value is given as the number of mg of KOH needed to neutralize 1g of oil and can be calculated according to equation :

$$A.V = \frac{56.11 \times N \times V}{m} \quad (1)$$

Where "56.11" is the molecular weight of KOH, "N" is the exact normality of standardized KOH solution, "V" is the volume of KOH solution needed to reach the end point of titration and "m" is the mass of oil in gram.

Iodine Value

The iodine value of the seed oil was determined according to AOAC (Association of Official Agriculture Chemists) Official Method 993.20 .

Saponification and unsaponification Value

The saponification and unsaponification value were determined according to the Manual of Methods of Analysis of foods Oils and Fats” Food Safety and Standards Authority of India Ministry of Health and Family Welfare Government .of India, New Delhi 2012, p.20-22

Peroxide Value

The peroxide value was determined according to AOAC Official Method 965.33

Fatty Acid Compositions

Fatty acid composition of the seed oil was determined in Royal Jordanian Society Laboratories, using gaschromotography (GC) connected with FID model SHIMADZU 2010.

Physical Analysis of Oil

Kinematic Viscosity ,Flash point, Copper Corrosion, Sulfated ash, Cloud point, Cetane Number analysis has been don in the laboratories of Chem. Dept of Science Faculty of Yarmouk University and Aden Refinery Company, according to ASTM analysis.

Transesterification

Homogeneously base-catalyzed transesterification was carried out for 3 h at 60°C using KOH. 40 g of preesterified oil were loaded into a 250 ml round bottom flask, heated to the designated temperature, then methanol (6:1 methanol/oil mole ratio) and KOH (1.2% w/w KOH/Methanol ratio) were added. The flask was then equipped with a reflux condenser and the magnetic stirrer turned on. Samples were taken at different time intervals. Each sample consists of 2 layers, the upper layer is the oil/biodiesel phase and the lower layer is the glycerin and methanol containing layer. The reaction was followed by measuring the ¹H-NMR after washing with water and drying up the upper layer with anhydrous Na₂SO₄. (this analysis was carried out in the laboratory- Yarmouk University- Jordan

1. Characterization of the raw oil with respect to free fatty acid content (acid value), fatty acid composition, insoluble impurities in hexane, Acidity as oleic acid, Acid value, Peroxide number, Iodine number, Saponification value and the Unsaponifiable matter content. Refining the raw oil by pre-esterification using H₂SO₄ (homogeneous catalyst).

2. Preparation of natural Yemeni bentonite catalysts. Different acid and thermal treatments has been performed to modify their properties. The prepared catalysts has been characterized by XRD. Their catalytic activities has been tested in the preesterification reaction.

3. Preparation of MgO catalyst. The structure of prepared catalyst has been characterized by XRD and their basicity were investigated by means of CO₂ chemisorption and FTIR. The

catalytic activity of the prepared solid base then tested in the transesterification reaction. The catalytic performance compared with that of the homogeneous KOH catalyst.

The magnesium oxide was prepared by adding a small excess of KOH to an aqueous solution of magnesium nitrate hexahydrate $Mg(NO_3)_2 \cdot 6H_2O$ (BDH Chemicals Ltd.). The precipitated magnesium hydroxide was centrifuged and washed with water until the washing water become neutral. The white gel was then dried in oven at $120^\circ C$ and powdered. The resulting magnesium hydroxide, $Mg(OH)_2$, was subjected to different thermal treatments. These include two hours of heating under flowing oxygen at $250^\circ C$, $350^\circ C$ or $500^\circ C$. The effect of thermal treatment was evaluated by means of X-ray diffraction (XRD) using Shimadzu XRD-6000 LabX device with a $Cu-K_\alpha$ -tube ($\lambda = 1.5418 \text{ \AA}$) in the Faculty of Archeology at Yarmouk University labs.

The MgO catalysts were characterized with respect to their surface basic properties. This was accomplished by chemisorbing CO_2 on the basic centers of the catalyst applying a pulse flowing technique. 0.20 g of MgO was placed in the reactor R and heated under He for 20 min at the temperature at which it was prepared with the purpose of removing any adsorbed water and freeing the surface for adsorption. The catalyst was then allowed to cool down to $20^\circ C$. 250 μl Pulses of CO_2 were then injected into the reactor. If injected CO_2 is chemisorbed, then no CO_2 will reach the thermal conductivity detector (TCD) and no change of the detector signal is observed. When the surface, however, becomes saturated with CO_2 , the CO_2 injected passes over the catalyst unaffected and reaches the detector producing a signal. From the number of the CO_2 injections missing at the detector, the total amount of chemisorbed CO_2 can be determined and the number of basic centers at the catalyst surface can be calculated.

The strength of chemisorption was determined by means of Thermal Desorption Spectroscopy (TDS) in which CO_2 on the surface is desorbed by increasing the temperature of the catalyst linearly (i.e. with a constant heating rate). Desorbed CO_2 is detected by the TCD as a function of the catalyst temperature.

Results and Discussion

The physicochemical properties of Jatropha oil extracted from the seeds of Yemen are given in Table 1. The oil content obtained from the seeds of different country lies in the range 25 % of Yemen. The observed amounts of oil are less than from seed of Jatropha in (Thailand, 64.23% , Malaysia, 63.16% and Indonesia 61.36%). (A. Emil et al 2010) While the observed oil yield is higher than such as from soybeen (18.35%) reported elsewhere

(Gunstone FD 1994). This less amount of oil contents seems depend on the kind of *Jatropha* plant which is growing in Yemen.

The iodine value is a measure of the unsaturation levels in fats and oils. A high iodine value is an indication of the presence of high unsaturation levels in the oils (Knothe G 2003). The determined iodine values of *Jatropha* seed oil was 110.1 for the samples from Yemen. The high iodine value of *Jatropha* oil is due to the presence of high amounts of unsaturated fatty acid such as oleic and linoleic acid

(Table 1.). The iodine value results of sample of *Jatropha* is well within the value of 120 (as specified in EN14214) which is an indication of its potential for use as a biodiesel feedstock (Knothe G, Gerpen JV, Krahl J (2005), Mittelbach M, Remschmidt C (2004). The obtained iodine value of *Jatropha* seed oil has been found satisfactory for exploring its use in the production of alkyd resin, shoe polish, varnish, ect. (Aintaya ET (2004)

Test Type	results	Unit	According to
% FFA	0.86	mg KOH / g Oil	
Oil content	25	%	
Insoluble impurities in hexane	0.01	%	ISO 663, 2007
Acidity as oleic acid	0.45	%	SOP:03/01/01-004
Acid value	0.86	mg KOH/ g oil	SOP:03/01/01-004
Peroxide number	0.7	Milliequiv O ₂ /Kg oil	SOP:03/01/01-001
Iodine number	110.1	g I ₂ / 100 g oil	SOP:03/01/01-002
Saponification value	189.96	mg KOH/ g oil	ISO 3657, 2002
Unsaponifiable matter	7.23	g/ Kg oil	ISO 3596, 2000
Unsaponifiable matter (after preesterification)	7.63	g/ Kg oil	ISO 3596, 2000

Table 1. The physicochemical characteristics of *Jatropha* oil extracted from seeds from Yemen

The *Jatropha* oil is characterized by its low acidity (originating mainly from the presence of free fatty acids). This is valuable in biodiesel production since high acidity requires a preesterification step in order to remove the free fatty acids that, otherwise, lead to soap and foam formation impairing thus the washing and separation processes of subsequently transesterified oil (biodiesel). Such a preesterification step is usually necessary if the acid value exceeds 2 mg KOH/g-oil i.e. acid concentration should be less than 1% (Berchmans, S. Hirata, 2008) .

Unsaponifiable matter corresponds to non-fat components of oils. The very low value for unsaponifiable matter (~0.7%) in Table 1. indicates the high value of Jatropha oil applied in this work since almost all of it can be used in the biodiesel production. After preesterification, the content of unsaponifiable matter didn't change. This is because it is already very small and/or because the unsaponifiable matter present in Jatropha oil is not affected or extracted by the acidic medium used in the preesterification step. The iodine number is defined as the weight of iodine that "adsorbs" on 100 g oil. Iodine undergoes thereby an addition reaction at the unsaturated double bonds. The value reported in Table 1 is rather high (110 g I₂ / 100 g oil) in accordance with the high fraction of unsaturated fatty acids (~80%) as noted from Table 2. It is higher than that of olive oil (79 - 88g I₂ / 100 g oil), comparable to that of cottonseed oil (108 - 110 g I₂ / 100 g oil), but lower than that of sunflower oil (119 - 135 g I₂ / 100 g oil) and of soy bean oil (137 - 143 g I₂ / 100 g oil) (Lewkowitsch).

Test type/ Fatty Acid Composition	%	According to
C14:0	0.08	COI/T.20/Doc. No. 24 (2001) AOCS Ch 2- 91 (1997)
C16:0	13.23	
C16:1	0.90	
C17:0	0.09	
C17:1	0.06	
C18:0	4.47	
C18:1	33.50	
C18:2	47.13	
C18:3	0.26	
C20:0	0.16	
C20:1	0.06	
C22:0	0.02	
C24:0	0.04	

Table 2: fatty acid composition of Jatropha oil

Test type/ Fatty Acid Composition	%	According to
C14:0	0.08	COI/T.20/Doc. No. 24 (2001) AOCS Ch 2- 91 (1997)
C16:0	13.20	
C16:1	0.90	
C17:0	0.09	
C17:1	0.06	
C18:0	4.39	
C18:1	32.99	
C18:2	47.7	
C18:3	0.27	
C20:0	0.16	
C20:1	0.06	
C22:0	0.02	
C24:0	0.04	

Table 3: fatty acid composition of biodiesel

The peroxide number is a measure of the rancidity of the oil produced by autoxidation reactions in which double bonds were found to play a significant role. It is usually associated with the taste of edible oils but was determined in this work with the purpose that there could be a correlation between the peroxide number and the biodiesel properties. Peroxide values of fresh oils are less than 1.0 milliequivalents /kg.

The results of the analysis of hexane extracted raw Jatropha oil are listed below in Table 2 and Table 3 The fatty acid profile shows that the Jatropha oil consists mainly of C18 fatty

acids (~85%), the majority of which is unsaturated either with one double bond (~34%) or two double bonds (~47%). The oil contains also significant amounts of saturated C16 fatty acid with a percentage of ~13%.

Figure (1) represents the X-ray diffraction patterns of $Mg(OH)_2$ and of the various forms of magnesium oxide, MgO-250, MgO-350 and MgO-500, prepared by the thermal treatment of $Mg(OH)_2$. $Mg(OH)_2$ is characterized by diffraction lines at $2\theta=32.76^\circ$, 37.86° , 50.66° , 58.50° , 62.0° and 68.22° , in agreement with the X-ray characteristics of $Mg(OH)_2$ as reported in X-ray data banks. There are, however, rather small diffraction features that are still not identified, for example at 34.01° , 45.48° and 55.45° . The diffraction lines of $Mg(OH)_2$ observed in Figure (1) are rather broad. Line broadening in XRD takes place as a result of having a particle size below 100 nm and/or due to deviation from perfect crystallinity. The origin of this line broadening in this work wasn't however further investigated.

Thermal treatment at $250^\circ C$ and $350^\circ C$ cause the diffraction lines of $Mg(OH)_2$ to decrease, and new diffraction features appear at 42.78° , 74.49° and 78.51° . These diffraction lines were found to correspond to the formation of MgO as they agree with those of magnesium oxides reported in X-ray data banks. The intensity of these lines was larger in the $350^\circ C$ treatment than in the $250^\circ C$ treatment indicating that the fraction of formed MgO is larger as the temperature at which the thermal treatment was performed is increased.

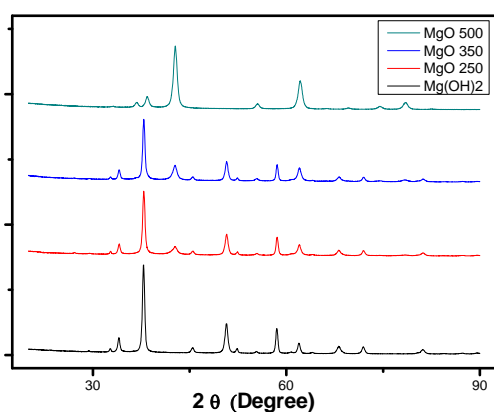


Fig.1: XRD for MgO thermal treatment at 120, 250, 350 and 500 °C

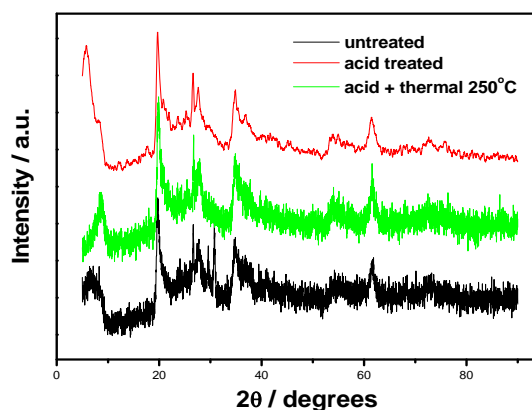


Fig. 2 : XRD patterns of untreated natural bentonite, acid treated bentonite and thermally treated bentonite

Thermal treatment at $500^\circ C$ causes on the other hand drastic changes. The major diffraction lines corresponding to $Mg(OH)_2$ disappear completely and those of MgO are dominant (small unidentified lines are still observed). So we can conclude that the formation

of pure MgO did take place only in this thermal treatment at 500°C. It is however still not perfectly crystalline as can be concluded from the rather large line broadening.

The bentonite samples used as acid catalysts in the preesterification reaction were also examined by XRD as shown in (Fig.2). It is difficult to determine the phases involved in these samples because the clay is natural, i.e. not pure, and various substances may be included. In addition, slight changes in composition may affect the diffraction pattern. A good match found in the X-ray data bank is the montmorillonite-21A (reference code 00-029-1499) with the chemical name sodium magnesium aluminum silicate hydroxide hydrate and with the chemical formula $(\text{Na}_{0.3}(\text{Al},\text{Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot 8\text{H}_2\text{O})$. An agreement with the major peaks in figure 6 was observed, namely those at 8.3°, 19.74°, 34.91°, 54.25° and 61.6°. The presence of other diffraction lines indicates the presence of other phases. The illite montmorillonite (reference code 00-007-0330) with the chemical name potassium aluminum silicate hydroxide hydrate and with the chemical formula $\text{K}_x\text{Al}_4(\text{SiAl})_8\text{O}_{20}(\text{OH})_4 \cdot x\text{H}_2\text{O}$ is suspected to be present in minor amounts.

The presence of this mineral is suggested by the diffraction lines at 26.7°, 27.7° and 36.9°. Also, untreated bentonite had a diffraction feature at 30.8° and a less intensive one at 29.6°. These lines disappeared upon acid treatment, the reason why they are believed to belong to calcite which has a rather large diffraction line at 29.455° (reference code 00-024-0027). Other carbonate minerals are not excluded.

The basicity of MgO was determined by chemisorbing CO₂ at the MgO surface. CO₂ binds to basic centers (surface -O⁻) forming a carbonate. The number of CO₂ molecules chemisorbed corresponds thus to the number of basic centers on the MgO surface.

Figure (3) represents the destiny of CO₂ injections allowed to pass over MgO-500 at 20°C. The first two injections are completely missing at the detector indicating that they have been adsorbed by the MgO surface. With increasing number of injections, the amount of CO₂ (i.e. peak area) appearing at the TCD detector becomes larger and larger, apparently due to the fact that the surface becomes gradually saturated with CO₂. When the surface becomes completely saturated with CO₂, no change of the CO₂ signal intensities at the detector is observed (last three injections in Figure 3).

The thermal desorption spectra (TDS) of CO₂ chemisorbed on MgO-350 and MgO500 are shown in figure (4). Although MgO-350 and MgO-500 have both the same concentration of basic centers, they differ significantly in the strength of these basic centers. In the case of MgO-500, CO₂ desorbs at much lower temperatures than in the case of MgO-

350, indicating that in the latter CO₂ is more strongly adsorbed to the surface. This in turn means that the basic centers that “captured” the CO₂ molecule are stronger in their basicity.

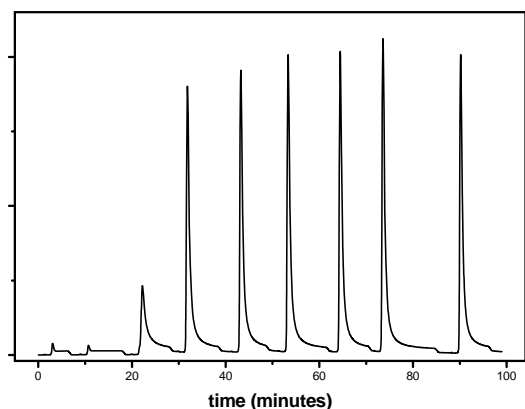


Fig 3: CO₂ pulses passing over MgO-500 at 20°C

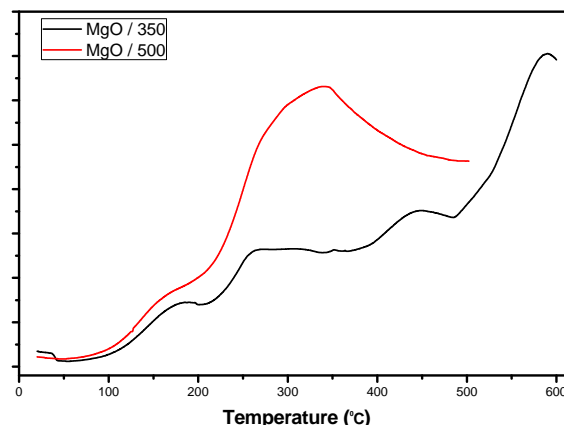


Fig.4 : (TDS) of CO₂ chemisorbed on MgO-350 and MgO-500

ATR-FTIR was used to characterize the MgO catalysts. Figure (5) shows the (OH) stretching band at 3694 cm⁻¹ for the different MgO forms applied in this work. The band is rather sharp and corresponds thus to surface OH groups and not to probably condensed water. Obviously, the band decreases with increasing the temperature of calcination, indicating that surface OH groups are gradually removed upon thermal treatment.

Normalization of the absorption band at 1435 cm⁻¹ against its area when the oil was cooked under reflux temperature (64°C) with KOH as a catalyst (KOH with 1.2% w/w of methanol added, and 6:1 methanol/oil molar ratio) for 3 hours enables us to calculate the conversion of raw oil into biodiesel (Table 4).

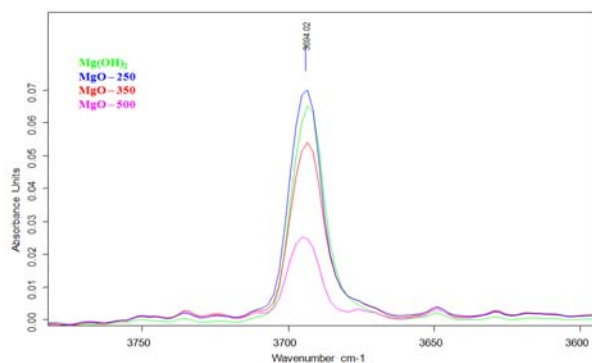


Fig.5: ATR-FTIR spectra of different forms of MgO catalyst

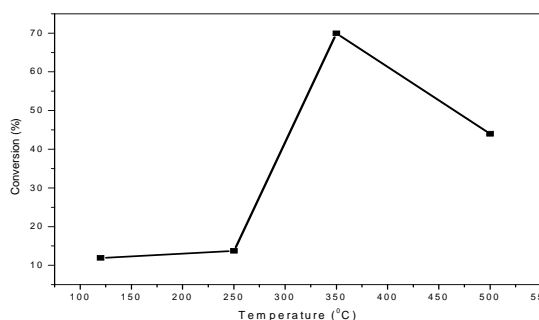


Fig. 6: Effect of the thermal pretreatment of MgO on its catalytic behavior in the heterogeneous transesterification of Jatropha oil

Carrying out the thermal treatment at 500°C was found to decrease the catalytic activity (Figure 6). This observation can be explained by the fact that the strength of basic sites in MgO-350 is larger than that in MgO-500. Furthermore, we can conclude from the fact that

both MgO-350 and MgO-500 have almost the same number of basic centers that it is the strength of the basic sites that matters in the transesterification reaction not just its total amount.

Sample Description	Peak Area	Normalized Peak Area	Conversion%
Raw oil	0.005	0.00276	0.276
pre-estrified oil	0.135	0.0745	7.45
KOH	1.812	1	100
Mg(OH) ₂	0.216	0.119	11.9
MgO-250	0.249	0.137	13.7
MgO-350	1.267	0.699	69.9
MgO-500	0.797	0.440	44.0

Table 4: Conversion of raw oil into biodiesel using different MgO forms.

The good catalytic performance that MgO-350 shows is actually very encouraging and suggests that heterogeneous catalysts based on MgO/Mg(OH)₂ phases can indeed replace the homogeneous environmentally non-friendly alkalis. Conversions better than 70% can in principle be achieved either by running the reaction for a longer time, increasing the mass of heterogeneous catalyst or modifying its surface properties. The latter requires however further research work.

Figure(7) shows the ¹H-NMR spectrum of Jatropa oil. The various peaks are assigned to the different H atoms in the oil molecule according to the insert in figure (7). Upon preesterification, a new singlet peak appears at 3.65 ppm (j peak) corresponding to the hydrogen atoms of the methoxy group. It appears in low intensity, obviously because of the low percentage of free fatty acids in fresh Jatropa oil. No other significant change in the spectrum of raw oil is observed.

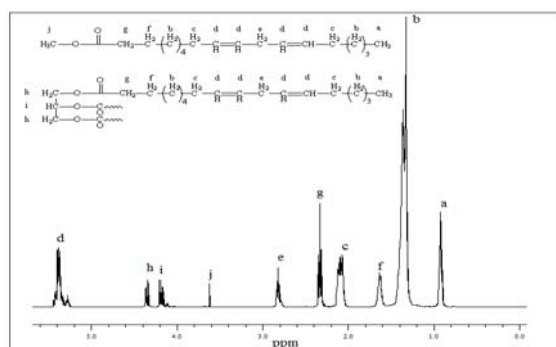


Figure 7: The ¹H-NMR spectrum of preesterified Jatropa oil (1% acid/oil w/w ratio with 5%methanol/oil w/w ratio and reaction time of 2hrs)

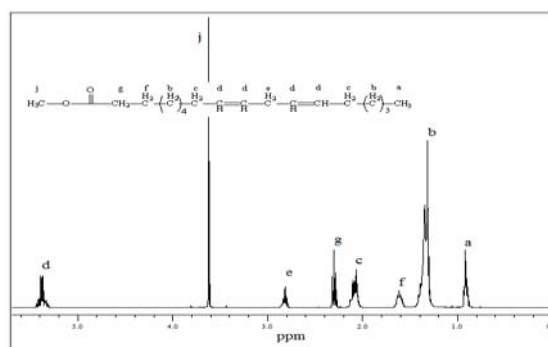


Figure 8: the ¹H-NMR spectrum of transesterified Jatropa oil (KOH with 1.2% w/w of methanol added, and 6:1 methanol/oil molar ratio)

By transesterification (Figure 8), the intensity of the j-peak increases corresponding to an increase in the fraction of methyl esters, whereas the h- and i-peaks corresponding to the triglyceride hydrogen atoms disappear indicating complete transesterification. The intensity of the j-peak can thus be taken as a measure to the extent of reaction progress. To eliminate the effect of oil concentration in the analyzed samples, the intensity of the j-peak relative to that of the a-peak (which corresponds to terminal CH₃ groups of FAME or oil molecules) is considered. The reason why the a-peak was chosen for this purpose is it is not supposed to undergo any change during preesterification or transesterification reactions. The intensity of the j-peak relative to that of the a-peak is shown in Table (5) depending on the type of chemical reaction the oil underwent. By calibrating the areas of peaks from NMR with those of IR we will get the calibration curve that allows us to use the IR instead of NMR for monitoring of the reaction Figure (9).

The biodiesel resulting from the 3-hour transesterification of Jatropha oil with 1.3% w/w KOH/Methanol ratio and 6:1 methanol/oil molar ratio were analyzed in the Royal Scientific Society labs. The results of the analysis of biodiesel are listed in Table 3 and Table (6), the fatty acid profile shows that the biodiesel consists mainly of C18 fatty acids (~85%), the majority of which is unsaturated either with one double bond (~34%) or two double bonds (~47%). The biodiesel contains also significant amounts of saturated C16 fatty acid with a percentage of ~13%.

Sample Description	I _j /I _a	relative to KOH [*]	Conv.%
Raw oil	0	0	0
Preesterification with H ₂ SO ₄ ^a	0.15	0.156	15.6
Transesterification with KOH ^b	0.96	1	100
Transesterification with Mg(OH) ₂ ^c	0.17	0.177	17.7
Transesterification with MgO-250 ^c	0.16	0.167	16.7
Transesterification with MgO-350 ^c	0.72	0.750	75.0
Transesterification with MgO-500 ^c	0.45	0.469	46.9

Table (5): The intensity of the j-peak relative to that of the a-peak
a: preesterified oil with H₂SO₄
b: transesterified oil with KOH
c: transesterified oil with different MgO forms

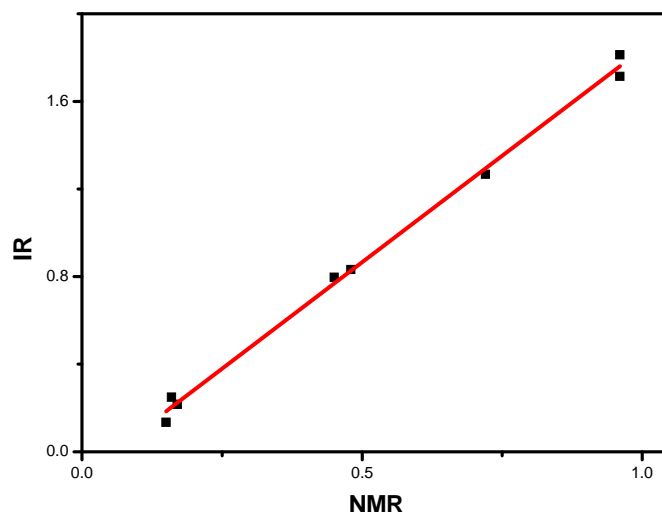


Figure (9): Correlation between the I_j/I_a -NMR data and the normalized IR peak at 1435 cm^{-1}

Conclusions

In this paper, Yemeni Jatropha oil was investigated with respect to its applicability in biodiesel production. Conventional homogeneous transesterification of (preesterified) Jatropha oil with methanol at 60°C using KOH as a catalyst was assumed to convert the Jatropha oil quantitatively into the corresponding methyl esters (FAME). This assumption is based on the fact that the specifications of the product of the above described transesterification match the standards of pure biodiesel (B100).

Test Type	Results	ASTM specifications	According to ASTM
Cetane number	51	45-67	Calculated
Flash point / °C	93	93 min.	D 93 – 10
Kinematic viscosity @ 40°C / cSt	5.7	1.9 – 6	D 445 – 09
Sulfated ash / %wt	0.09	0.02 max.	D 874 – 07
Copper corrosion, 3hrs @ 50°C	1a	1	D 130 – 04
Saponification value mg KOH/g oil	187.66	-	ISO 3657, 2002
Iodine number g I_2 / 100 g oil	108.7	120 max.	SOP:03/01/01 – 002
Cloud point / °C	7	-	D 2500 – 05
Distillation @ 90% / °C	353.3	360 max.	D 86 – 10a

Table (6): Some characteristics of produced Jatropha biodiesel and comparison with ASTM standards

Different forms of $\text{Mg}(\text{OH})_2$ -MgO phases were also used as trans-esterification catalyst in an attempt to replace the homogeneous environmentally unfriendly KOH with a

solid base. Thermal treatment of $\text{Mg}(\text{OH})_2$ for two hours at 350°C (MgO-350) was found to give the best $\text{Mg}(\text{OH})_2$ -MgO form being capable to convert almost 70% of the Jatropha oil into biodiesel. Annealing $\text{Mg}(\text{OH})_2$ for two hours at 500°C (MgO-500) was found to reduce the strength of the surface basic centers and convert $\text{Mg}(\text{OH})_2$ almost completely into MgO leading to a drop in the efficiency of the catalyst in biodiesel production down to 44%. MgO-350 seems thus very promising and is the subject of further investigation.

The specifications of the biodiesel produced in this work match the standards. Sulfated ash is a little bit high but this was attributed to the poor filtration of the oil before sending it to transesterification. Our results also suggest that the IR spectroscopy is an easy way to monitor the progress of transesterification and can be applied as a control technique in biodiesel production.

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