



Synthesis and characterization of activated carbon as a heterogeneous catalyst derived from palm leaves for production of biofuel

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Abstract

In this study, activated carbon was prepared from palm leaves as renewable source. The prepared activated carbon was characterized using different spectroscopic and surface analysis to determine its chemical and morphological structures. The prepared activated carbon was used as a heterogeneous catalyst at different ratios in catalytic cracking of castor oil to produce biofuel. The parameters controlling the production of the biofuel were determined and the reaction condition was optimized. The fuel properties of the produced biofuel were determined including: viscosity, density, flash and fire points, pour point and smoke points.

Keywords: activated carbon; heterogeneous catalyst; catalytic cracking; biofuel.

Received; 24 June 2019, Revised form; 1 Aug. 2019, Accepted; 1 Aug. 2019, Available online 1 Jan. 2020.

1. Introduction

Vegetable oil is an attractive renewable resource for producing biofuel in particular, the tropical countries which have vast areas for growing plant oil under suitable climate [1]. Triglyceride (TG), the main component found in vegetable oils, contains of various hydrocarbons that are related to several types of biofuel [2]. The chain length of fatty acids composted in natural TG is varied but usually contains carbon atom range from 2 to 28 and is always an even number. This led to the idea that the chain of fatty acids can be changed to new structures and has a carbon atom number shorter than the original compounds and exposed their characteristics as a fuel [1]. At present, the most successful class of biofuels is biodiesel, which is produced from plant oils or animals fats by a liquid-phase catalyzed transesterification process at low temperature or a solid catalyzed catalytic cracking process at high temperature. However, the process requires large investments for the production units in order to ensure high efficiency. Also, biodiesel product is not stable compared with the petroleum fuel because of its low oxidation stability and poor cold flow properties.

Conversion of plant oils and animal fats into biofuel products has been studied by using a catalytic hydrotreating process similar to in the oil and gas industry. In addition, hydrocracking reaction is one of the promising way to produce renewable kerosene which is widely known in several names such as hydroprocessed kerosene, green kerosene, hydroprocessed renewable jet (HRJ), hydroprocessed esters and fatty acids synthetic paraffinic kerosene (HEFA-SPK), synthetic paraffinic kerosene (SPK) and bio-hydrogenated kerosene (BHK) [3, 4]. Basically, hydrocracking, a sub-reaction of hydroprocessing process, is a chemical reaction that can

convert a larger molecule of hydrocarbon into a smaller one. It has been used in a petroleum refinery in the second step of crude oil refining for upgrading heavy oil [5]. During the process, a dual function catalyst composed of a metallic part and amorphous mixed oxides of acidic nature or proton exchanged crystalline zeolites is required, where metallic sites are required for hydrogenation and dehydrogenation reactions and the acid sites are necessary for isomerization and cracking activities. Therefore, it is very important to design the acidic sites and metal components as well as tailor the balance between the metal and acid for the product selectivity, catalyst activity and stability [6].

Recently, two types of catalysts have been reported as effective hydrotreating catalysts in converting vegetable oils to biofuels, especially green diesel: supported noble metal catalysts (Pd and Pt) and sulfided bimetallic catalysts (usually Mo- or W-based sulfides promoted with Ni or Co). However, there are disadvantages of using these catalysts. On one hand, the rarity and high price of noble metal catalysts has made the process economically unfeasible [7]. Furthermore, since noble metal catalysts are very sensitive to catalyst poisons, impurities (such as sulfur, heavy metals and oxygenated compounds) in feedstock can cause significant deactivation of the catalysts. Therefore, it is necessary to remove impurities from the biomass feedstock before the reaction. On the other hand, conventional γ -Al₂O₃ supported sulfide bimetallic catalysts (usually Mo or W based sulfides promoted with Ni or Co) as presently used for desulphurization of fossil diesel streams need to be operated under high energy consumption conditions, such as high temperature, high pressure, and large amount of hydrogen consumption. The process is costly, and the yield

of product can be low because of formation of coke. The products obtained in the mentioned processes over the bimetallic aluminum oxide supported catalysts are mainly n-paraffin's (n-C₁₅ up to n-C₁₈) which solidify at low temperatures, so, they are unsuitable for high quality diesel fuels, kerosene and gasoline compounds. More importantly, the transition metals in these hydro treating catalysts need to be maintained in the sulfide form in order to maintain the activity at process conditions. Therefore, a sulfurization co-feed needs to be added to the biomass feedstock. Gu et al. (2015)[8] reported that transition metal carbide catalyst, Mo₂C, showed high activity and selectivity for one-step conversion of vegetable oils into branched diesel-like hydrocarbons. Nitrides of molybdenum, tungsten and vanadium supported on γ -Al₂O₃ were also used for hydrodeoxygenation of oleic acid and canola oil.

The most widely used feedstocks for biodiesel production are vegetable oils. Among them, castor oil has two interesting phenomena as biodiesel raw material; on one hand, it does not compete with edible oils; on the other, its cultivation does not need high inputs[9].

Therefore, this study aims to produce biofuel from castor oil via the catalytic cracking process using activated carbon collected from the date palm leaves as catalyst. In addition to investigate the properties of the obtained biofuel by varying the catalyst ratio and different conditions.

2. Materials and Methods

2.1. Materials

All material used as received Date palm leaves, castor oil, sulfuric acid (25%) (Aldrich, German), Sodium bicarbonate (Aldrich, German)

2.2. Preparation of activated carbon derived from date palm leaves

The collected date palm leaves were rinsed thoroughly with distilled water, dried and then crushed into small fragments. The obtained fragments were steeped in a sulfuric acid solution (25%) for 24 h at 25°C [10]. The treated fragments were rinsed thoroughly with deionized water and air dried. Next, the dried samples were calcined at 250°C in a muffle furnace for 24h. The obtained activated carbon samples were washed several times with deionized water then with sodium bicarbonate solution (1%) until the pH became steady at a value around 6.0. Eventually, the acquired activated carbon samples were dried at 100 °C overnight, grinded and finally sieved.

2.3. Catalytic cracking of Castor oil

Catalytic cracking procedures were performed as follows: 150 mL of *Castor* oil was charged in 500 mL two necked flask and metal salt [activated carbon] was added individually at different ratios of 0.01, 0.02, 0.04, 0.06, 0.08, 0.1 and 0.2% by weight relative to castor oil. The mixture was mixed and allowed to thermal agitation for 4 h at 250 °C. The experiment setup was connected to nitrogen flow (5 mL/min) and stirring rate at 150 rpm. The reaction product was distilled and collected by a condenser and its volume was determined. The obtained biofuel was settled in a separating funnel to separate the produced water and then centrifuged to remove any contaminated or dispersed water. The reaction was completed using Activated carbon

and the products were: 85% biofuel, 10% water, 3% solids, and the rest were vapors [11].

2.4. Catalyst Characterization measurements

SEM images of the AC (activated carbone) samples were obtained using a JOEL 5410 that was operating at 30 kV. Surface texture parameters were determined on a NOVA 3200 system instrument by nitrogen adsorption-desorption at -195.8 °C. Before each measurement, the sample was degassed at 200 °C for at least 2 h. FTIR measurements were executed using a Nicolet FTIR IS-10 spectrometer (Waltham, MA, USA) with KBr discs.

2.5. Biofuel specification

The obtained biofuels from the catalytic cracking reactions were characterized by determining the following properties: viscosity, density, specific gravity ASTM D-4052 (ASTM International United States, 2013), carbon residue ASTM D-445 (ASTM International, 2000), ash content ASTM D-4530 (ASTM Standard, 2013), sulfate content ASTM D-482 (ASTM, 2015), pour point ASTM D-4294-16 (ASTM D97-11, 2011), flash point, fire point ASTM D-97 (ASTM International, 2015), ASTM distillation ASTM D-93 (ASTM Standard D86, 2008) and cetane number ASTM D-4737 [12]

2.6. Reusability test of catalyst

The reusability of the catalyst was determined by repeating the cracking reaction using one catalyst sample for several rounds and the properties of the obtained biofuel after each round were determined[13]. Before each experiment, the catalyst was washed by benzene and dried to remove the formed contaminations on its surface. The reactions were stopped after decreasing the comparable fuel properties of the obtained biofuels.

3. Results and discussions

3.1. Catalyst Characterization

The FTIR spectra of activated carbon sample of date palm leaves are shown in Figure 1. These spectrum show abroad stretching vibrational band of hydroxyl (-OH) at about 3400 cm⁻¹, vibrational bands of aliphatic C-H at 2926 cm⁻¹, stretching vibrational band of carboxyl group (C=O) in carboxylic acid or quinone type structure at 1710cm⁻¹, stretching vibrational bands of C=O (-COO-) or C=C centering at 1616 cm⁻¹, bending vibrational band of C-H at 1442 cm⁻¹[14]. Vibrational bands at 1105, 800, and 473 cm⁻¹ are the normal vibration modes of the SO₄²⁻ tetrahedral configuration [15]. Since the bands at 3400, 2926, and 1713cm⁻¹ are ascribable to ν (O-H), ν (C-H) and ν (C=O) vibrations, this suggests that date palm leaves were oxidized by sulfuric acid.

The adsorption-desorption isotherms of the activated carbon sample derived from date palm leaves can be classified as types II according to IUPAC classification. The isotherms exhibit hysteresis loops widening down to P/Po~ 0.15. The values of specific surface area calculated using BET-equation, A_{BET}, are presented in Table 1 together with the values of total pore volume, V_p, taken at P/Po = 0.95, and average pore radius ($r = 2V_p/A_{BET}$) [16]. The pore size distribution curves for activated carbon samples derived from date palm leaves were obtained using BJH calculation method [17] as displayed in Figure 2. The curve of activated carbon sample derived from date palm

leaves confirms the presence of two sizes of pores, a peak at 1 nm and a small broader one centered at 10 nm.

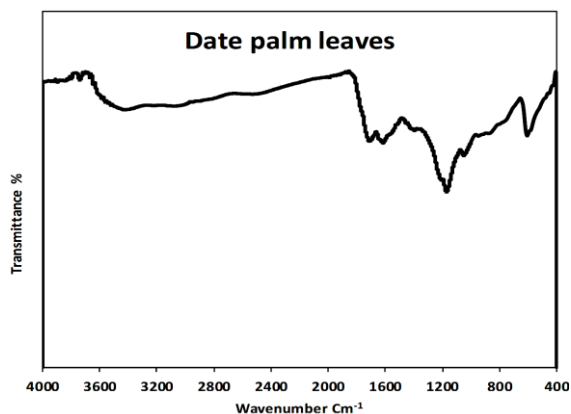


Fig (1): FTIR spectrum of activated carbon sample of date palm leaves.

Table (1): Texture data for activated carbon samples derived from date palm leaves

AC source	A_{BET} (m^2/g)	V_p (ml/g)	$r=2V_p/A_{BET}$ (Å)
Date palm leaves	12.879	0.017	13.199

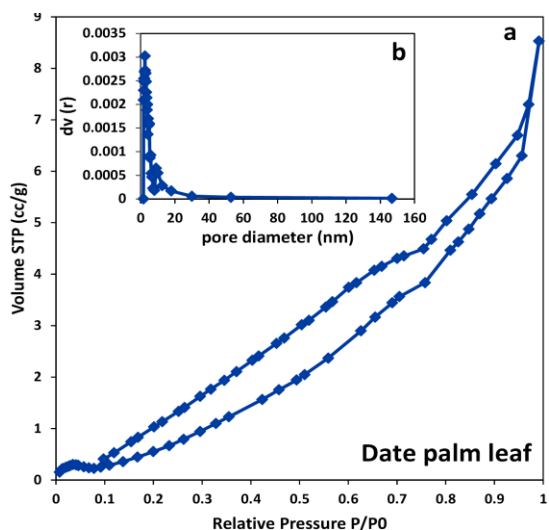


Fig (2): (a) N_2 adsorption-desorption isotherm of AC samples (b) Pore size distribution of AC samples.

The SEM images of the activated carbon samples derived from date palm leaves are shown in Figure 3. It can be observed that the samples of activated carbon particles have

a rough surface with several large pores and cracks on their surface. The existence of these pores is attributed to release of volatile organic and inorganic compounds during the activation process using sulfuric acid. The inclusion of mesopores and micropores is in good agreement with the mean pore diameter values, 19.84 and 26.39 Å for activated carbon samples derived from date palm leaves, respectively obtained using BET measurements.

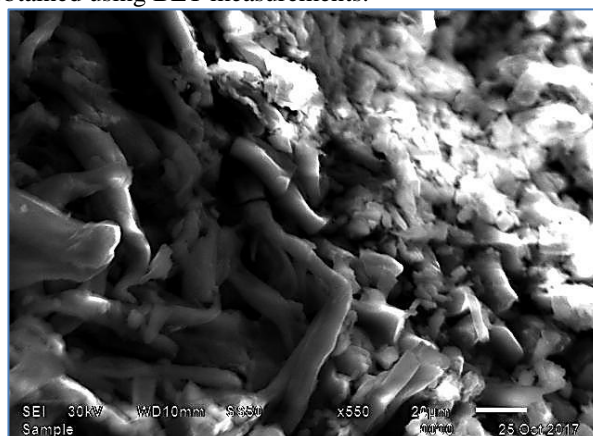


Fig (3): SEM image activated carbon sample derived from date palm leaves.

3.2. Characterization of the castor oil and obtained biofuel.

The fatty acids profile describes the different fatty acids incorporated in the chemical structure of the oil molecules (triglycerides) and their percentage. The fatty acid composition of the castor oil was analyzed in Regional Center for Microbiology and Biotechnology. The oil sample was methylated before GC injection. The analysis was conducted using GC-7890A equipped with DB-23 column, 60 mm x 0.25 mm, i.d. of 0.25 μm . Peaks in the chromatograms were identified by comparison of the retention times of commercial standards. Standard curves for converting peak areas to mass were constructed using methyl ricinoleate (Sigma–Aldrich), as ricinoleic acid (18:1:OH) is the predominant acid of *castor* oil (89%) and the remaining fatty acid esters have similar acyl chain lengths. The retention time and the signal response of methyl ricinoleate did not vary during the analysis. It is clear from **Table 2** that, castor oil contains 92% of ricinoleic acid and oleic acid (monounsaturated fatty acids), 5% of linoleic acid (diunsaturated fatty acid), and about 3% of different saturated fatty acid.

Table (2): Fatty acid profile of Castor oil

Fatty acid	Fatty acid composition (wt %)		
	Molecular Formula	g/mole	Castor oil
Palmitic acid	$C_{16}H_{32}O_2$	256.43	1.00
Oleic acid	$C_{18}H_{34}O_2$	282.47	3.00
Linoleic acid	$C_{18}H_{32}O_2$	280.46	5.00
Linolenic acid	$C_{18}H_{30}O_2$	278.44	1.00
Ricinoleic acid	$C_{18}H_{34}O_3$	298.46	89.00

The obtained products of the catalytic cracking reaction were of three types listed in (Table 3). The physical properties of the obtained biofuel were determined and compared to ASTM limits specifications. The measured

properties were: density, viscosity at 40 °C, flash point, cloud point, pour point, cetane number, sulfur content, ash content, carbon residue, Table 4).

Table (3): Reaction products of catalytic cracking of castor oil using activated carbon

Compound	% Yield	
	activated carbon	
Biofuel	85	
Water	10	
Solids	3	

Table (4): Properties of biofuel obtained from castor oil cracking

Property	ASTM specification	ASTM limits	activated carbon						
			0.01%	0.02%	0.04%	0.06%	0.08%	0.1%	0.2%
Density, g/cm ³ (@15.6 °C)	ASTM D-4052	0.860-0.900	0.8667	0.8663	0.8663	0.8657	0.8651	0.8636	0.8634
Specific Gravity (@15.6 °C)	ASTM D-4052	0.989-0.984	0.8679	0.8675	0.8669	0.8663	0.8659	0.8644	0.8642
Viscosity, at 40°C	ASTM D-445	1.6 - 7.0	4.62	4.62	4.61	4.59	4.58	4.24	4.03

The density of the fuel represents the weight of one gram of it. The density is an important factor during the fuel processing and ignition, because the fuel represents an extra weight on the vesicle. Consequently, higher density of fuel will consume larger amount of fuel during the automotive work. The obtained values of the density of Castor oil cracking using Activated carbon as catalyst at 0.01% was 0.8667 g/cm³, which are located within the range of ASTM D-4052 specification of biofuel. Increasing the amounts of the used catalysts to 0.02% - 0.2% decrease the density of the obtained biofuel considerable to reach to 0.8634. That indicates the increasing of the catalyst amount decreases the density of the obtained fuel (Figure 4). Most of biofuels densities obtained from non-edible oils such as castor oil, rapeseed oil, and linseed oil [18] are high due to the presence of high unsaturation and oxygen contents in their chemical structures (linolenic acid and recinoleic acid), which is overcome by the mixing with short chain aliphatic alcohols such as ethanol or propanol to decrease their density.

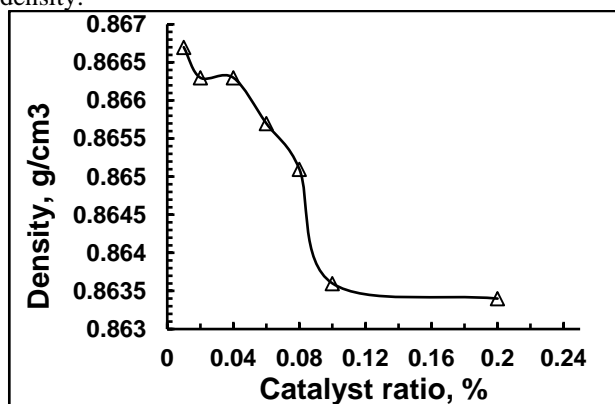


Fig 4: Variation of Density of the obtained biofuel by changing the catalyst ratio.

The viscosity of the fuel represents the degree of fluidity of the fuel in the tubes and in the circulating system of the

engine. It is an important characteristic due to high viscosity fuel will requires pumping into the ignition or compression chambers of the various engines. The viscosity of the obtained biofuels after the catalytic cracking of Castor oil was in the range of 4.62-4.03 cSt using Activated carbon as a catalyst (Figure 5). The obtained values are within the ASTM D-445 limit of 1.6 to 7.0 cSt. Increasing the amount of the catalyst used from 0.01% to 0.2% has a decreasing effect on the viscosity of the obtained biofuel. That can be attributed to the catalytic effect of Activated carbon on the Castor oil. Increasing the catalyst amount increases the cracking extent of the fatty acid chains in the oil and consequently decreases the produced carbon chain length, which decreases their viscosity[19].

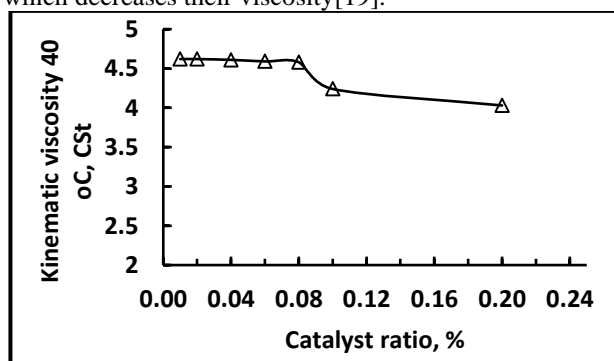


Fig (5): Variation of kinematic viscosity of the obtained biofuels by changing the catalyst ratio of the

The pour point of the fuel represents the temperature at which the fuel becomes solid before it and liquid after it. Pour point is important characteristic during the transportation of the fuel at elevated low temperatures. In cold climate countries of low temperatures, high pour point fuels freeze. The pour points of the obtained biofuel are ranged between -9 and -12 °C using Activated carbon as a cracking catalyst. These values are located within the specific ASTM standard limit of -5 to 15 °C (Figure 6). Low

pour points of the obtained biofuels at the various ratios of the used catalysts indicate that the fuels are composed of high amount of relatively short chain hydrocarbons[20]. The cloud point of the fuel represents the temperature at which the regular crystal of the hydrocarbons starts to form. Low cloud points of the biofuels are important property due to the formation of regular clusters of the hydrocarbons lowers the fluidity of the biofuel which decreases its transportation through pipes and tubes. The approved cloud

point values of ASTM D-97 are -3 to 15 °C, and the obtained values for the produced biofuels were 4 to -1 using Activated carbon as catalyst. These values are considered as acceptable values for the biofuel at all catalysts ratios[20]. Increasing the ratio of the used catalysts decreases the cloud point of the produced biofuel, and the lowest cloud points were -1 and -3 °C at 0.2% catalysts ratios (Figure 6).

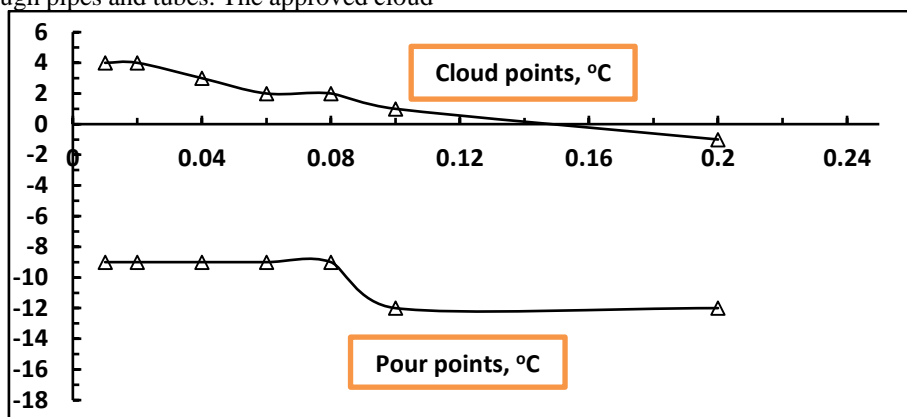


Fig (6): Variation of pour points and cloud points of the obtained biofuels by changing the catalyst ratio.

The flash point of the fuel represents the temperature at which the fuel starts to ignite. The obtained biofuel from catalytic cracking of Castor oil using Activated carbon as catalytic cracking catalyst have flash points range of 37 to 28 °C using Activated carbon at different ratios (0.01% to 0.2%) (Table 5). The flash points of the biofuels obtained in the presence of the catalyst in the ratio of 0.01% and 0.2% are not located in the ASTM specification of biofuel (Figure 7). In the presence of 0.01% to 0.2% of the catalyst, the flash points of the obtained biofuels are lower than those of

the biofuel [21], but in the range of the bio-gasoline. The lower flash points of the obtained biofuels at higher catalysts ratios were attributed to the low hydrocarbon chains produced by the cracking reaction. The using of these ratios of the catalyst causes a strong destructive reaction on the fatty acids of Castor oil, which produces comparatively short chain hydrocarbons with low flash points. The fire points of the obtained biofuels also showed similar trend as in case of flash points.

Table (5): Fuel properties of the obtained biofuel from catalytic cracking of castor oil

Property	ASTM specification	ASTM limits	activated carbon						
			0.01%	0.02%	0.04%	0.06%	0.08%	0.1%	0.2%
Pour point, °C	ASTM D-97	-5 to 15	-9	-9	-9	-9	-9	-12	-12
Cloud point, °C	ASTM D-2500	-3 to 15	4	4	3	2	2	1	-1
Flash point, °C	ASTM D-93	52 (min.)	37	37	36	34	33	30	28
Fire point, °C	ASTM D-93	54 (min.)	39	39	38	36	35	32	30
Sulfur content, wt%	ASTM D-4294	0.0-1.0 (max)	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Carbon residue, wt%	ASTM D-4530	0.01 (max.)	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Ash content, wt%	ASTM D-482	0.01 (max.)	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Cetane number	ASTM D-4737	48 – 65	38.79	38.76	38.51	36.72	35.12	32.19	30.25

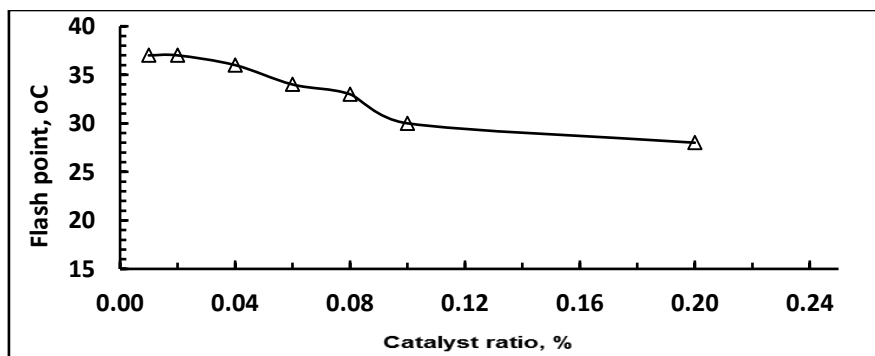


Fig (7): Variation of flash points of the obtained biofuels by changing the catalyst ratio

Sulfur content, ash content and carbon residue

Petroleum fuel generally contains amount of sulfur in the form of free sulfur, thiols, di-sulfides and mercaptans [22]. The ignition of the fuel produces sulfur dioxide gas, which is highly toxic and corrosive when combined with water (Equation 1).



Biofuel contains less amounts of sulfur, as the biological sulfur in the non-edible oils is low. Hence, it is expected to obtain low sulfur content in case of biofuel compared to the petroleum fuel. The sulfur content of the used Castor oil was 0% by weight. The obtained sulfur content of the obtained biofuel was traces compared to the virgin oil using activated carbon (Table 5). On the other hand, the carbon and ash residue as a result of the fuel ignition play an important factor on the life time of the engine components and also on the environment. Solid particulates are considered as polluting threats for the environment. Decreasing the produced solid particulates from the fuel

ignition is one of the important aims of using biofuels. The approved limits of the carbon and ash residue after biofuel ignition are 0.01 wt% according to ASTM D4530 and ASTM D-482, respectively (Table 5). The obtained carbon and ash residues of the produced biofuels were traces using activated carbon as a cracking catalyst. These values are comparatively lower than those obtained in case of petroleum fuel.

4. Conclusions

The study represents the preparation and characterization of activated carbon as efficient heterogeneous catalyst for catalytic cracking of vegetable oils to produce biofuels. The reactivity of the catalyst showed high percent product of the biofuel compared to the commercial and reported heterogeneous catalysts. The properties of the obtained biofuels were ranged between the standard specifications of gasoline and diesel fuel.

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