Bio-aviation fuel via catalytic hydrocracking of waste cooking oils

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Abstract

Background: Biomass fuels (bio‑jet fuel) have recently attracted considerable attention as alternatives to conventional jet fuel. They have become the focus of aircraft manufacturers, engines, oil companies, governments and researchers alike. This study is concerned with the production of biojet fuel using waste cooking oil (WCO). Batch reactor is used for running the experimental study. The catalytic cracking products are investigated by GC mass spectra. Final products from different reaction conditions are subjected to fractional distillation. The (Bio kerosene) fraction was compared with the conventional jet A-1 and showed that it met the basic jet fuel specifications. Optimum reaction conditions are obtained at (450 °C), pressure of (120 bars), catalyst dose (2.5% w/v), reaction time (60 min) and hydrogen pressure 4 atmosphere. The aim of this study is to produce bio aviation fuel according to specifications and with a low freezing point from waste cooking oil in one step using a laboratory prepared catalyst and with a low percentage of hydrogen to complete the process of cracking and deoxygenation in one reactor, which is naturally reflected positively on the price of the final product of bio aviation fuel.

Results: The results indicated that the product obtained from WCO shows promising potential bio aviation fuels, having a low freezing point (−55 °C) and that all bio kerosene’s specifications obtained at these conditions follow the international standard specifications of aviation turbine fuel.

Conclusion: Biojet fuel obtained from WCO has fairly acceptable physico‑chemical properties compared to those of petroleum‑based fuel. Adjustment of the hydro catalytic cracking reaction conditions was used to control quantities and characteristics of produced bio aviation fuel. Taking into consideration the economic evaluation WCO is preferable as raw material for bio aviation fuel production due to its low cost and its contribution in environmental pollution abatement. Blend of 5% bio aviation with jet A-1 (by volume) can be used in the engine without any modifications and a successful test of blended aviation fuel with 10% bio aviation has been achieved on Jet-Cat 80/120 engine.

Keywords: Bio aviation fuel, Hydrocracking, Catalytic, Waste cooking oils

Background

Aviation sector is one of the strongest growing transport sectors. Global airline operations consumed around 1.5 billion barrels of Jet A-1 fuel. Jet fuel consumption of 705 million metric tons (Mt) generates significant greenhouse gas emissions, accounting for about 2% of global CO₂ emissions each year (www.climatesolutions.org/programs/aviation-biofuels-initiative). For Egypt from 1980 to 2016, the average consumption value during that period was 19.32 thousand barrels per day, with a minimum of 7.29 thousand barrels per day in year 1980 and a maximum of 38.11 thousand barrels per day in 2010 (https://www.theglobaleconomy.com/Egypt/jet_fuel_consumption/). Awareness of greenhouse gas emissions and climate change push researchers and airlines sector to develop renewable jet fuel alternatives.

Biomass fuels (bio‑jet fuel) have recently attracted considerable attention as alternatives to conventional jet fuel. They have become the focus of aircraft manufacturers, engines, oil companies, governments and
researchers alike. IATA identified biofuels as the most promising strategies to reduce carbon dioxide emissions from the aviation sector in the foreseeable future (Yang et al. 2019).

The high dependence of the bio-jet production cost would be on the following parameters: (1) feedstock's composition and cost, (2) process design, (3) product yield or conversion efficiency, (4) co-product's valorization; and (5) energy conservation. Hence, decreasing the production cost of bio-jet fuel depends on significant hard work in all areas, including enhancements in the areas of feedstock productivity, the extracted yield of oil or sugar yield from the crops, process conservation energy, and balance between jet fuel product and co-products added value (Wang and Tao 2016).

Derived jet fuels from renewable feedstock can reduce the dependency of the aviation industry on one single energy source. Also this can decrease the petroleum prices, and essentially lowering greenhouse gas (GHG) emissions. The use of aviation biofuels could reduce greenhouse gas emissions in the life cycle of aviation by a range of 68.1% in 2050 (Staples et al. 2018; Blakeley 2012; Tao et al. 2017).

Investigators from different academia and organizations (the oil-refining industry, the aviation industry, government, bio fuel companies, agricultural organizations), tried to develop new commercially practicable and sustainable processes that produce renewable low cost jet fuels with low greenhouse gas emissions. Produced jet fuels must meet (ASTM) international standards and can be a 100% drop-in replacement for the current petroleum jet fuel. The emissions after combustion and engine tests show up the benefits of running the aero plane with bio-jet fuels. Technologies for producing renewable jet fuels from different pathways containing alcohols-to-jet, oil-to-jet, syngas-to-jet, and sugar-to-jet, are studied. The main challenges for each technology pathway, including feedstock availability, process conceptual design and economics, life-cycle assessment of greenhouse gas emissions, and commercial willingness, are discussed (Wang et al. 2016).

Several types of feed stocks for bio aviation fuel production are listed as (a) oil-based feedstocks, such as vegetable oils, waste oils, algal oils, and pyrolysis oils; (b) solid-based feedstocks, such as lignocellulosic biomass (including wood products, forestry waste, and agricultural residue) and organic portion of municipal waste or (c) gas-based feedstocks, such as biogas and syngas (ICCT 2019).

Using Camelina, jatropha and algae, as raw materials for Jet fuel production reduce the fuel's carbon foot print by80% relative to jet fuel without competing for food sources (Bailis and Bake 2010).

Basically, kerosene is a straight-run distillate of petroleum fraction with boiling temperature ranging from 205 to 260 °C. Increasing jet fuel production decreases obviously the production of other products (Sun 2012).

Also safety and security of supply criteria change and developed the specifications of aviation kerosene (Pires et al. 2018; Corporan et al. 2011; Hileman and Stratton 2014). Strategy of both environmentally and economically feasibility of bio fuel production is related to the development of conversion technologies and feed stock resources so that the cost-competitive production of bio fuels is compatible with the use of sustainable low cost, and diverse feed stocks (Sathaye et al. 2011; Skreiberg et al. 2013).

Most contaminants that result in bio oil yields can be traced back to the feedstock, such as residual solids (char), alkali metals, and high water content (Zacher et al. 2014a).

Common technologies used is catalytic hydride oxygenation at high pressure (Mante et al. 2017) which generates a hydrocarbon liquid suitable for co-processing in a petroleum refinery or blending into finished fuel (Tal-madge et al. 2014). To achieve IATA goals, higher blending rates may eventually be required. In fact, this product may not be available everywhere during the growing period. The ability to mix higher ratios in some locations can help achieve the establishment objectives, taking into account the local availability of the product (Chuck and Donnelly 2014). The first task of the High Biofuel Blends in Aviation (HBBA) study was to establish the relevant range of properties of conventional kerosene properties, and to identify sources for the supply of suitable samples (Prussi et al. 2019). The target set by the European Union Flightpath for the aviation sector focuses on all methods of production, but in particular, takes into account artificial paraffin or biologically derived biomass. Aviation biofuels must be fully compatible and can be combined with standard fossil fuels (such as Jet-A fuel). These fuels should also be tested and approved before commercial use, and emissions are verified. In addition, production and use should not only be developed and verified, but also the entire transport and distribution chain (Chairamonti 2019).

Conventional biofuels cannot be mixed even in very few percentages with fossil kerosene, as the fuel standards are too strict for aircraft engines. This also applies to distilled fractions of conventional biofuels (Chuck and Donnelly 2014).

The first track relies on vegetable oils and fats, and therefore on raw materials such as oilseeds or fat-rich algae, residues such as cooking oil used or animal fat, or even common products such as tall oil from the paper-making industry (Chuck and Donnelly 2014; Prussi et al.
Esterification is not a vital option for the production of aviation fuel: the so-called "biodiesel", is a mixture of fatty acid methyl esters (FAME) to be used as a low component (5–7%) in the land transport mix. On the contrary, hydrogen treatment of fats can be applied to the production of jet fuel, as has already been done by many companies (such as Neste Oil, Petrobras, ENI/UOP, etc.) to obtain high-quality biomass-derived fuel, compared with FAME (Chairamonti 2019).

It is clear that if biodiesel is to be used for aviation then it will have to contain short-chain esters rather than long-chain polyunsaturated. While hydrogen-treated triglycerides are accepted as synthetic aviation fuels, a number of studies have shown biodiesel, alkyl esters of fatty acids produced by the glyceride transformation process, to be a suitable fuel for gas turbine engines. Biodiesel typically contains straight-chain fatty acid esters ranging from 16 to 18 carbon atoms that can contain 3 double bonds. Biodiesel is suitable for aviation if composed solely of short chain saturates (Chuck and Donnelly 2014; Wang et al. 2016). However, the melting points of long-chain saturated and unsaturated esters are too high to be suitable for flight. Reducing the length of the chain or increasing the level of saturation significantly reduces the freezing point (Wahyudi 2018; Holladay 2015a; Gawron and Białecki 2018).

Numerous literatures have examined the effect of hydro treating process on bio-oil characteristics (Zacher et al. 2014b; Howe et al. 2015). The produced hydrocarbons via zinc aluminate as catalyst was mainly branched alkanes and cycloalkanes with a maximum yield $\approx 89\%$ after upgrading and distillation processes (Hawash et al. 2017). Overall, thermo-chemical conversion is an important route for the production of biojet fuel using vegetable oils (Xu et al. 2016).

The present investigation is undertaken to study the effect of change of operating conditions in the hydro thermal catalytic cracking process for converting waste cooking oils to bio aviation fuel through the determination of different parameters representing the experimental results based on the rate of depression in freezing point of the produced bio kerosene with different reaction conditions and the test proved that the obtained thrust of using 10% blend of the produced bio aviation fuel is in a good agreement with jet A-1 specifications.

**Methods**

**Materials**

- (WCO) feed stock was collected from local Egyptian fast food restaurants.
- Nano (Zinc Aluminate) catalyst was freshly prepared through the present study.

**Raw materials preparation**

**Preparation of waste cooking oil**

The WCO was filtered to remove any solid impurities prior to the analysis.

**Preparation of catalyst \( \text{Zn Al}_2\text{O}_4 \) nano particles**

The used Zinc aluminate as a catalyst was prepared by co-precipitation method using 48 g H$_2$O, zinc nitrate Zn(NO$_3$)$_2$, 6 H$_2$O (96% purity) and 120 g aluminum nitrate Al(NO$_3$)$_3$,9H$_2$O (98% purity) (Diwani et al. 2009; Battiston et al. 2014), LOBA CHEMI company. (ZnAl$_2$O$_4$) nano particles were investigated by using transmission electron microscope (TEM) which operates on the same basic principles as the light microscope but uses electrons instead of light, small amount of powder was dispersed in ethanol using an ultrasonic bath for 30 min. Then few drops were placed onto a carbon coated copper grid. Specific surface area was calculated by BET method using N$_2$ adsorption experiments.

BET surface area analyzers determine the total surface area of a solid by suspending a powdered analyte in an inert gaseous bath and measuring the adsorption of gas molecules to the surface and its porous structures. This can be measured volumetrically or via a continuous flow.

**Preparation of thermal hydro-catalytic cracking process**

The experimental work was carried on floor-stand reactor under pressure (Parr reactor) with 400 ml volume. The reactor includes stainless steel vessel, pressure gauge and a heating jacket. The temperature inside the reactor was controlled by proportional integral derivative panel (PID).

The reactor vessel was charged with oil (100 ml) and (2.5% w/v) of ZnAl$_2$O$_4$ catalyst. Applied process parameters were varied: the reaction temperature from 400 to 450 $^\circ$C, starting hydrogen pressure of 4 atm then the pressure increased with heat and time to reach a range of (59–130 bass), and the reaction time from 20 to 60 min. Operating conditions of different experiments are shown in Table 1 and the process flowchart is illustrated in Fig. 1. the reaction operating conditions were changed as follows:

- Time from 20 to 60 min.
- Temperature from 350 to 450 $^\circ$C.
- Hydrogen pressure of 4 bars.

**Preparation of fractional distillation steps**

Scaling-up of the thermal catalytic cracking reaction is achieved in a reactor of 1 gallon (3.7 L), at the optimum operating conditions obtained in the 400 ml batch reactor. Product of this run is fractionally distilled at...
temperature range from 82 to 350 °C for separation of products which are subjected to further analysis and testing, Fig. 2. The fractional distillation was achieved for the hydrocracking product to obtain the different distilled fractions: 1—bio gasoline (< 120 °C), 2—bio kerosene (120–245 °C), 3—biodiesel (245–350 °C), 4—heavy product (> 350 °C) as shown in Fig. 3.

Preparation of GC/mass analytical procedure

Gas chromatography Mass (GC/MS) was performed for determination of the product fuel constitution using Thermo Scientific, ISQ Single Quadruple MS, TG-5 MS fused silica capillary column (30 m, 0.251 mm, 0.1 mm film thickness). For GC/MS detection, electron ionization system with ionization energy of 70 eV (electron volt) was used. Helium was used as a carrier gas at flow rate 1 ml/min, column temperature was kept at 240 °C for 30 min.

Preparation of (10%) biojet fuel blend testing

Blend of jet A-1 (kerosene) with 10% of produced biojet from WCO was subjected to a jet engine test (Jet-Cat 80/120) Fig. 4 and the resulted thrust was compared with that obtained from jet A-1.

Results and discussion

Physicochemical properties of waste cooking oil

The chemical composition (wt%) and physical characterization of WCO are given in Table 2 from which it is clear that the composition and properties of WCO indicated that the fuel produced from the feedstock are regarded as environmentally benign green fuels; the acidity of the oils is within the acceptable range, insuring that oil does not contain any corrosive materials which confirm high quality of oil. The iodine value of WCO was 76 g of I₂/100 g oil, indicating that the oil contained many unsaturated C=C bonds as linoleic acid.

Elemental analysis and high heating value (HHV) calculation

Elemental composition of crude oils (C, H, N, O, S) were determined using Agilent model elemental analyzer (7800 ICP-MS) and HHV was calculated using modified Dulong equation (Bousdira et al. 2014):

$$\text{HHV (kJ/kg)} = 4.18 \times (78.4 \times \text{C} + 241.3 \times (\text{H} - \text{O}/8) + 22.1 \times \text{S})$$

Gas chromatography (GC)

The profiles of WCO was determined by GC with capillary column DB-5 (60 m: ID 0.33 mm). Helium was used as a carrier gas at flow rate 1 ml/min, column temperature was kept at 240 °C for 30 min.

Table 1 Experimental conditions for batch biojet fuel preparation (catalyst: 2.5 vol%)

| Run no. | Temperature (°C) | Time (min) | Final pressure (bar) |
|--------|-----------------|------------|---------------------|
| 1      | 435             | 60         | 115                 |
| 2      | 440             | 60         | 143                 |
| 3      | 450             | 60         | 150                 |
| 4      | 450             | 20         | 75                  |

Fig. 1 Hydrocatalytic cracking process of WCO bio jet production flowchart
From elemental analysis, oxygen content was small and nitrogen was negligible and no presence of sulfur was detected. Magnitude of HHV was 13.7 MJ/kg for WCO as shown in Table 2 which indicated that it is quite good for fuel production.

### Table 2 Physical characterization and chemical composition of WCO feed stock

| Parameter                        | Waste cooking oil (WCO) |
|----------------------------------|-------------------------|
| Viscosity at 40 °C mm²/s         | 39                      |
| Density g/cm³                    | 0.9                     |
| Acidity KOH/goil                 | 0.5                     |
| Iodine value gI₂/100 g           | 76                      |
| Pour point °C                    | −9                      |
| Cloud point °C                   | −4                      |
| Elemental analysis               |                         |
| C (wt%)                          | 76.8                    |
| H (wt%)                          | 11.6                    |
| O (wt%)                          | 10.6                    |
| N (wt%)                          | 1                       |
| S (wt%)                          | Nil                     |
| HHV (MJ/kg)                      | 13.7                    |
| < C5 (%)                         | –                       |
| C5–C7 (%)                        | –                       |
| C8–C16 (%)                       | 39.8                    |
| C17–C19 (%)                      | 60.1                    |
| > C19 (%)                        | –                       |

#### Catalyst characterization

In thermal catalytic cracking (TCC) process the type of catalyst is one of the most important factors that affect the composition of the product (Prussi et al. 2019). Zinc Aluminate (ZnAl₂O₄) is the catalyst in this study due its effectiveness in decreasing H/C ratio less than other metallic oxide catalyst (Xu et al. 2016). This indicates that the product of thermal cracked oil via
ZnAl₂O₄ was not only aromatic compound but more saturated alkanes (Hanafi et al. 2015). BET surface area and particle size of the used ZnAl₂O₄ freshly prepared is shown in Table 3.

The particle size and morphology of ZnAl₂O₄ nano particle were investigated by using transmission electron microscope (TEM). It was noticed that the particle size possess a narrow size distribution and the average particle diameter was about 7.6 nm. The specific surface area measured by the BET method is relatively high (85 m²/g) which is beneficial due to its catalytic activity. These data are in good agreement with previous work (Weng et al. 2015) where Ni-HZSM-5/SBA-15 catalyst of diameter 8.7 nm was effective in converting large molecular biomass (sorbital) into small molecule hydrocarbons so the co-precipitation method confirms that it is the best method for the preparation of ZnAl₂O₄ as solid nanoparticles.

The overall process for the preparation of the ZnAl₂O₄ from Al³⁺ and Zn²⁺ nitrate using ammonium hydroxide is illustrated by the following equation:

\[
\text{Zn(NO}_3\text{)}_2 + 2\text{Al(NO}_3\text{)}_3 + \text{NH}_4\text{H}_2\text{O} \rightarrow \text{Zn(OH)}_2\text{Al(OH)}_2 + 3\text{HNO}_3
\]

\[
\Delta \text{Calcination}
\]

\[
\text{ZnAl}_2\text{O}_4 + 4\text{H}_2\text{O}
\]

### Table 3 Physicochemical properties of ZnAl₂O₄ catalyst

| BET surface area (m²/g) | Particle size diameter (nm) | Shape |
|-------------------------|----------------------------|-------|
| 85                      | 7.5                        | Sphere |

### Table 4 Effect of reaction temperature on product freezing points (reaction time: 60 min, catalyst 2.5% wt/vol and 120 bar)

| Reaction temperature (°C) | Biofuel product freezing point (°C) |
|---------------------------|-----------------------------------|
| 400                       | −48                               |
| 430                       | −49                               |
| 450                       | −52                               |

**Effect of reaction time**

The results showed a linear relationship between time and freezing point Fig. 5, where the freezing point reached its lower value after (60 min) reaction time. The freezing points of the products was considered as a good indication of the reactant conversion to the biojet fuel, the reaction can be represented by the Eq. (a) which are linear relationships between freezing point (Fp.) and time (t) for WCO hydro catalytic cracking.

\[
\text{Fp.} = 0.46t + 40.1
\]  

From the previous data, it can be concluded that the acceptable freezing points for fuel product are obtained within 60 min. This result gave a promising potential in the bio aviation manufacture starting from WCO due to economic and environmental purposes, being of lower cost than vegetable oils and environmentally positive way for using oil wastes.

**Results of the distillation steps**

Fractions obtained from distillation procedure are shown in Fig. 3 which illustrated that the bio gasoline product was collected up to 150 °C while biojet fuel range was obtained from 150 to 290 °C then finally bio diesel fuel was produced from 290 to 350 °C.

 Thermal cracking of WCO under optimum conditions gave about 96% crude bio fuel. After the distillation step of crude bio fuel, about 49% of bio aviation fraction was obtained and the rest (51%) were biodiesel and heavy fuel with calorific values suitable as bio fuel to be used in other purposes. These results are in good agreement with ASTM standards (Dai et al. 2018).

**GC/mass spectra results**

The formation of alkanes, cycloalkanes, iso alkanes and alkyl benzene during thermal catalytic cracking of WCO was investigated by GC/mass to classify hydrocarbons present in the product feedstock. The results showed that: the hydrocarbon mixture contained n-paraffin, iso
paraffin, cyclo paraffin, olefins and aromatics. It can be confirmed that typical ideal carbon length of C_8–C_{16} in jet fuel is from 70 to 85%. Total yield of paraffin content in bio aviation fuel obtained was close to 83% as shown from Fig. 6.

GC–mass spectra data illustrated that most of iso and cyclo-paraffins have one /two branched chain of alkanes, as methyldecane, alkyl cyclohexane and iso octane (2,2,4-trimethylpentane) in the total product. This is in agreement with the literature (57.01%) (Holladay 2015b).

It is interesting to say that the obtained unrequired olefins is near about minimum (5% wt%) as in ideal jet fuel. Also GC/mass spectra data illustrated that most of iso, cyclo-paraffin have one or two branches as methyldecane, and straight chain alkanes or branched chain cyclo-alkane as iso octane.

Molecules as methyldecane (iso-paraffin) are much more represented in petroleum jet fuel component than iso-octane (branched alkane). Jet fuel range iso-paraffin is the most expensive and difficult component to obtain, was detected in good ratio in the prepared bio aviation fuel.

Pure fuel is highly dependent on the amount of branched alkanes because increase in branching suppresses the fuel ignition point (Eldeeb 2007).

**Biojet blend (10%) test**

Specifications of 5% blend of biojet from WCO are illustrated in Table 5 and compared with the standards specifications results were within the required limit. The test proved that the obtained thrust of using 5% blend is in
a good agreement with jet A-1. Figure 7. Blue points are different thrust values of the petroleum jet fuel (kerosene jet fuel) where red points are different thrust of the blend kerosene jet fuel and 5% biojet obtained in this work at different time during the testing experiment and it shows the higher thrust values of blend biojet than the pure petroleum kerosene.

The submitting of biofuels into aviation sector must not alter the aircraft engine performance and the fuel must keep its properties during the wide range of atmospheric conditions during flights (ASTM International 2015).

**Conclusions**

From the study it is concluded that:

- Renewable bio aviation fuel can be produced via thermal catalytic cracking of vegetable oils where conversion of WCO to hydrocarbon range alkanes has been successfully performed under high temperature and pressure.
- Biojet fuel obtained from WCO has fairly acceptable physico-chemical properties compared to those of petroleum based fuel.
- Adjustment of the hydro catalytic cracking reaction conditions was used to control quantities and characteristics of produced bio aviation fuel.
- Results of this study revealed that hydrothermal catalytic cracking of WCO gave convenient freezing point (−55 °C) for use as biojet fuel.
- Taking into consideration the economic evaluation WCO is preferable as raw material for bio aviation fuel production due to its low cost and its contribution in environmental pollution abatement.
- Bio aviation fuel fraction from (C₈–C₁₆) was obtained by fractional distillation of hydrocracking product from temperature from 120 to 250 °C.
- Blend of 5% bio aviation with jet A-1 (by volume) can be used in the engine without any modifications and a successful test of blended bio aviation fuel with 10% bio aviation has been achieved on Jet-Cat 80/120 engine.

**Abbreviations**

WCO: Waste cooking oil; IATA: International Air Transport Association; GHG: Greenhouse gas; FAME: Fatty acid methyl esters; ASTM: American Society for Testing and Materials; HBBA: High biofuel blends in aviation; BET: Brunauer–Emmett–Teller; TEM: Transmission electron microscope.

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**Authors’ contributions**

RE, EA, SI: experimental part and writing the research paper. SI, GE, RE, EA: preparing experiments work plan. GE: reviewing and contributing in writing the research paper. All authors have read and approved the manuscript.

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**Availability of data and materials**

Data sharing is not applicable to this article as no datasets were generated or analyzed during the current study.
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