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Waste Cooking Oil Management in Egypt: Production of Biodiesel-Development of Rapid Test Method

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Abstract. According to the United Nations Office for South-South Cooperation (UNOSSC) report in 2017, Egypt produced 500,000 tonnes of WCO from various resources including food industries, restaurants and hotels. Based on our previous funded project, we have reported unique properties for Egyptian WCO as it has very high range acidity (≈ 18 mg KOH/ g oil) due to the extensive usage of oil in the cooking process. Further, the repeated heating of cooking oil for long time produces carcinogenic compounds that have significant impact on people health. On the other hand, high acidity WCO could be valorised into soap, biodiesel and value added chemicals. However, most of these industries requires costly pre-treatment for the WCO to reduce the acidity prior processing. Accordingly, the WCO should be produced within a guideline of specified properties that could detect if it has been extensively used. Low-acidity WCO will prevent several health consequences for Egyptian people and allow industries to easily valorise it into value added chemicals and fuels. The waste cooking oil was subjected to filtration before being fed to the reactor the produced biodiesel was treated to remove any methanol and the characteristics of the final product was compared with the international standards. This work develops a guideline for WCO quality testing to ensure that it has not been extensively used in cooking process. The work also applies supercritical conditions to convert WCO into biodiesel. The response surface methodology was used to conclude the optimum conditions to produce biodiesel.

Keywords: Biodiesel, Waste management, WCO, Cooking Oil.



1. Introduction

Cooking oil as well as other oils used in eating, along with fats that are produced from households are currently disposed with other public waste or are dispensed into drainage. For a long time, the recycling of this type of waste had been essential from the food industry companies along Europe. Recycling of this type of waste on a household level is still not the great matter even if it occupies a significant part of the overall production, around 19% of the overall production comes from households in South Korea. In some countries, the idea of fat waste recycling is grabbing the interest of the government; in Czech Republic municipalities are forced to enable the separate collection of fats and Waste Cooking Oil (WCO) in 2020. Due to the physical properties and structure of fats, fat waste is a branch of waste management which gets the interest of many. However, there are some challenges as the animal fats and vegetable oils becomes solid at low temperatures and have high melting temperatures. Simultaneously, the previous tend to appear in a semi-solid or as paste at room temperature, while the other has higher viscosity [1]. Unlike the handling of glass, paper and plastic, the handling of fats requires some work. When fat is dumped off to sewage, it combines with other solid wastes in a sewer which causes clogging in the waste system. However, fats and waste cooking oils show a valuable feedstock and commodity which can be used for energy generation [2].

Waste cooking oil is an inland waste that is produced as a result of frying and cooking food using vegetable oil. Waste cooking oil results mainly from frying at elevated temperatures which is dumped to sewage systems after being used, it is municipal solid waste (MSW). Even though the usage of acronym MSW carries some error and is solid evidence that this type of waste is given poor attention in terms of management and collection. In Europe the statistics of the level of WCO collection is between 100,000 and 700,000 tons/year, which is equal to 1 kg/inhabitant/year. Negative environmental impacts and economic loss are created from the uncontrolled dumping of WCO [3]. Altering the improper ways of disposal of WCO increases the lifecycle of the product and the avoids the groundwater's contamination with this liquid waste. Not only for the environment, the collection of the unwanted WCO has an economic point of view too, it is a promising and a cost-effective raw material for the production of synthesized biofuel as well as recovering valuable energy content. Ever since the 70s, the European Commission suggested that it is essential to collect recognizable amounts of waste oils to avoid environmental contamination [4].

When WCO is thrown into water systems, the oil forms a layer on the surface which stops the oxygen from dissolving, therefore causing a drastic extinction in the marine life. Moreover, the mixture of water and oil elevates the chemical oxygen demand (COD) of water and causes it to be poisonous as a result of the production of by-products formed from oil degradation. The sea creatures absorb carcinogenic compounds and are returned back to humans through the food chain [5]. When the temperature reaches above 180 °C in the absence of oxygen, by-products of alkenes, alkanes, lower fatty acids, symmetric ketones, CO, CO₂ and alkanes are formed from the non-oxidative breakdown of saturated fatty acid [6].

An average of 500,000 tons of WCO is produced yearly from Egypt from different sources. It is produced from food factories, restaurants, hotels and houses, but it is not managed efficiently by the previously mentioned entities to be reused and to benefit from it. Conventionally, it is sold to some collectors of waste. The waste collectors filter it and sell it back to the food industry. Not only food industry, but to soap companies too and are sometimes exported to be used in biofuel industry abroad. The idea of using WCO in the production of biodiesel started in 2013 in Egypt, the conversion process takes around 3 hours and results in two products, biodiesel and glycerin. One of the leading tourism companies is currently using the produced biodiesel in its hotel's vehicles, generators and machinery

[7]. The work of this research aims at 2 objectives, first is to propose a systematic method to measure the quality of WCO, second is to convert WCO into useful fuel such as biodiesel.

2. Methodology

2.1 WCO Quality Testing Method

The acid value of cooking oil is measured as a very important parameter determining the quality of cooking oil. Oil is the most essential and widely used ingredient in Egyptian households. It enhances the taste and flavour of the food. But do you know how poisonous it can be to your health if you keep on reusing the same cooking oil for various purposes and for several times. In order to avoid the wastage of oil, many people reuse it. It is very important to understand that this practice is extremely unsafe for families and loved ones and can lead to a number of health problems. Nutritionists explain that the risks in reusing cooking oil depends on different categories. "When someone does not have a deep fryer or uses a normal fryer, it is recommended to use the oil for frying chips only once. When somebody uses a deep fryer such, as common in restaurants, they can reuse the oil three or four times." Reusing cooking oil without using a deep fryer is extremely harmful to your health. The harmful effects of reusing cooking oil:

- 1- A high amount of toxins/chemicals is released such as aldehydes/4-hydroxy-trans-2-nonal associated with many health conditions such as heart-related disorders, dementia cancer, Alzheimer's, which alters the function of DNA, RNA and protein in our body.
- 2- Gives rise to trans fatty level of oil: it increases levels of bad cholesterol.
- 3- Oils become partially or completely oxidised and thus chemical composition changes.
- 4- Blood pressure levels can increase when repeatedly heated cooking oil.
- 5- Can generate compounds including polycyclic aromatic hydrocarbons that may lead to carcinogenic risks.

Acid value of the feedstock is one of the most important parameters which affects biodiesel production and yield. Based on the acid value of the feedstock, the production process as well as the catalyst to be used are determined or selected. Waste cooking oil WCO which contains triglycerides is converted through transesterification with methanol and at the presence of base catalyst into biodiesel. Typically, 1 mole of WCO reacts with 3 moles of methanol to produce 1 mole of methyl ester or biodiesel and 1 moles of glycerol as by product. The acid value test is used twice in biodiesel production. The first time determines how much catalyst to use in the biodiesel subject to the properties of the oil. The second time determines how long the fuel's shelf-life will be subject to the amount of free fatty acids FFAs in the biodiesel. Oils and fats tend to break down through oxidation over time to form free fatty acids. When these feedstocks are used to make biodiesel by transesterification, the FFAs quickly consume some of the catalysts to form sodium and potassium soaps. Too much soap can complicate the separation process and reduce the yield by carrying some of the fuel into the glycerol phase. Soaps must be removed from the biodiesel by water washing or some other purification processes. Residual soap in the fuel can lead to sodium/potassium deposits when burned that are abrasive to the engine. The acid value is used to determine the amount of free fatty acids in the oil/fat, which in turn is used to calculate the exact amount of catalyst needed to produce biodiesel. Having too little catalyst can lead to an incomplete reaction resulting in fuel with too high levels of total glycerides. According to ASTM specification, biodiesel can have no more than 5 ppm of sodium and potassium and no more than 0.24 % total glycerides in the fuel sample. The ASTM D6751 biodiesel acid-number limit was harmonized with the European biodiesel value of 0.50 mg KOH/g fuel. Titration is a chemical procedure that can be used to obtain this number, i.e. acid value for waste cooking oil and biodiesel product. The acid value is the quantity of a known concentration of potassium hydroxide KOH consumed by titrating an oil/fat sample (i.e. neutralise the fatty acids) and is expressed in the units of milligrams of KOH per gram of sample.

In this activity six different samples were collected to investigate the effect of frying oil reuse on its acid value from Egyptian market/location. The First sample was pure oil as reference. While the second, third, fourth and fifth ones are representative samples of oil reused once, twice, three times and four times, respectively. Last sample was collected from an Egyptian fast food restaurant where they reused the oil several times (more than 10). After collecting the samples, the acid values were measured using titration method.

Manual titration requires a colour indicator such as phenolphthalein and organic solvent such as toluene or ethyl alcohol to dissolve the sample in order to obtain a clean end point. The standard method for acid and base number using colour indicator titration (ASTM D974) was implemented to measure the acid value of the samples (waste cooking oil/biodiesel). Titration solvent, colour indicator and KOH are the main chemicals employed in titration. Instructions for standardising the titrant (KOH) are found in ASTM D664. Acid value test of oil samples was carried out by titrating 20 grams of oil in 100 mL ethanol by KOH 0.1 N using phenolphthalein as indicator. Every sample was subject to 3 experiments with equal volumes to measure the acid value and an average number is considered to reduce the uncertainty in values. The procedure is as follows:

- 1) Add 100 mL of the titrating solvent (ethanol) to a 250 mL flask.
- 2) Add approximately 10 drops of phenolphthalein indicator solution.
- 3) Pour weighed sample of approximately 20 grams into the solvent.
- 4) Titrate while stirring with KOH titrant to the end point which is a definite colour change to orange or green brown colour that persists for at least 15 seconds.
- 5) Report the millilitres of titrant consumed.
- 6) Calculate the Acid value from the below equation:

$$\text{Acid Value} = \frac{(V - V_b) \times N \times \text{Eq_wt}}{W_{oil}} \quad (1)$$

where V and V_b are the volumes of 0.1 N KOH needed for titration in mL for both the oil sample and blank sample, N is the normality of KOH which equals about 0.1 geqL^{-1} , Eq_wt is the equivalent weight of KOH = 56.1 and W_{oil} is the oil sample mass in g. This works because the KOH is a basic solution, which reacts with an acidic solution until all of the acid is consumed and the solution is neutral. Any KOH added to the sample after it is neutral causes the solution to become basic and change colour.

- 7) To express the acid value as a percent of free fatty acids (% FFA), divide the acid value by 2 if the fatty acid profile is mostly oleic, palmitic or stearic acids. Oils with high percentages of lauric acids such as coconut, should be divided by 2.8.

$$\% \text{ FFA} = \frac{\text{Acid Value}}{2.0} \quad (2)$$

- 8) Use this same method to measure the acid value of the biodiesel product. Sample size will be larger and a weaker normality of KOH should be used to achieve accurate results.

When oil feedstock acid value is greater than 2, extra catalyst should be added to account for what will be lost in forming soap. It takes 1 mole of catalyst to neutralise one mole of soap. The amount of additional catalyst can be calculated using the following equation:

$$\text{NaOH} = (\% \text{ FFA}) \times (0.144) + 1\% \quad (3)$$

$$\text{KOH} = \frac{(\% \text{ FFA}) \times (0.197)}{0.8} + 1\% \quad (4)$$

2.2 Biodiesel Production: Materials

Waste cooking oil is supplied which originates from sunflower oil by Uptown biodiesel Ltd. Moreover, methanol bought from Fisher scientific in the United Kingdom. The calibration curves were prepared by using standard methyl esters, and the heptadecanoic acid methyl ester was used as an internal standard and bought from Sigma-Aldrich, UK. The liquid CO₂ cylinder (99.9%) equipped with a dip tube was bought from BOC Ltd., UK.

2.3 Biodiesel Production: Experimental Setup

The waste cooking oil was filtered in order to get rid of all of the impurities that originated from cooking/frying. The reaction was carried out in supercritical conditions in a reactor made of stainless steel and has a volume of 100 ml and could withstand high pressure. The reactor's type is 49590, Parr Instrument Company, USA. The reactor is supplemented with a heating mantle, model 4848 controller, J-type thermocouple and a mechanical stirrer. Figure 1 shows a schematic for the experimental setup. The oil and methanol with a specific molar ratio were added to the reactor then heated. After the oil and methanol are mixed with a specific molar ratio, the mixture was fed to the reactor. In order to reach the required temperature, stirring at 300 rpm and heating were continuously applied to the reactor. The needed pressure was achieved by pumping CO₂ using a supercritical fluid pump. Before any pressure is applied by the CO₂, the reactor is left to heat as the heated methanol produces vapor which causes a build-up pressure. The reaction conditions were achieved nearly after 15 minutes; this is when the reaction time starts. After the reaction time is over the reaction is stopped by quenching, which is using an ice bath for the reactor to stop the reaction. The pressure of the reactor is released and a centrifuge is used to separate the reaction's product to glycerol and biodiesel. Furthermore, the biodiesel is heated again to 80 °C to recuperate any methanol that didn't react in the distillation process. Finally, the pure biodiesel is analyzed and its properties is then compared to the European standard (EN14214). The biodiesel's product yield is calculated using the following equation [8].

Equation 1: yield of the produced biodiesel equation

$$BD \text{ Yield (\%)} = \frac{\text{Total weight of methyl esters}}{\text{Total weight of waste oil used}} \times 100 \quad (5)$$

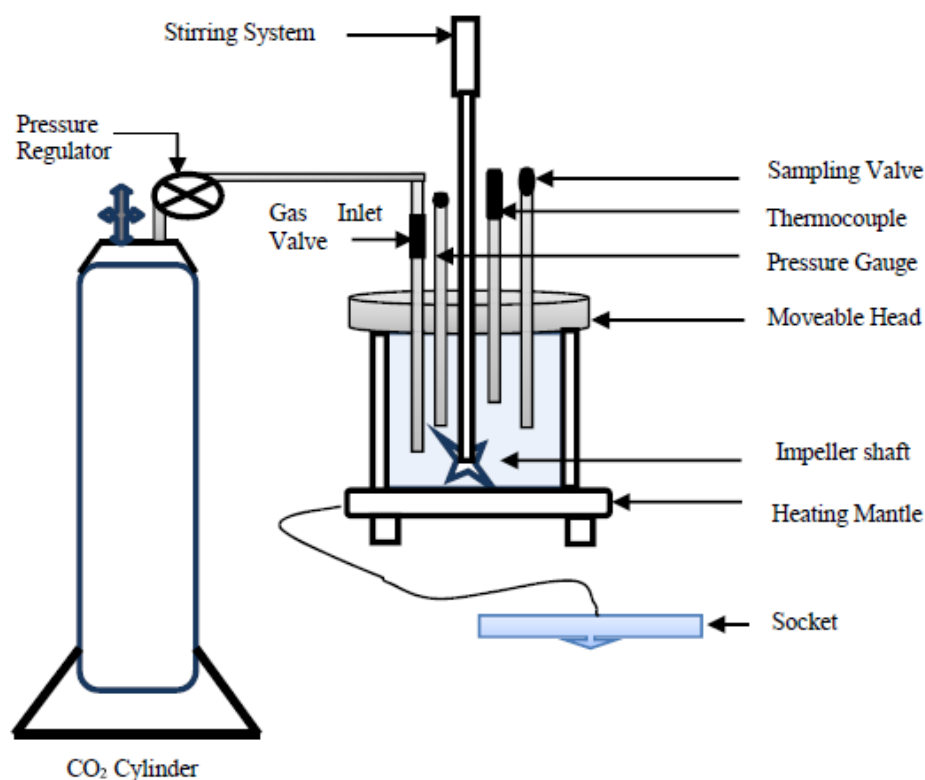


Figure 1. Schematic of the experimental setup



2.4 Experimental Design

Response surface methodology (RSM) is a multi-variable method which is capable of developing a model that shows the response function which depends on the reaction in the experimental studied independent variables [9]. To reach the optimum reaction conditions that are needed for the production of biodiesel RSM is used, this is done by learning the relationship of each of the variables with the response yield. There are four variables on which the experimental runs have been operated; methanol-to-oil molar ratio, temperature, pressure and time, labelled as A, B, C, D respectively. Three levels of each of these variables are coded as -1,0,1 as shown in the table below. BBD is one of the RSM techniques used to study the effect of the process variable on the response. Moreover, it studies the interactions of the variables on the response [10]. The yield of biodiesel is selected as the response for this study. In order to lower the effect of unexplained consistency in the responses, the experiments are completed in a random order [11]. Twenty-nine runs are performed in a random way and their response are calculated from the experimental results using the yield equation.

Table 1. Experimental design variables and their coded levels

Factor	Code	Levels		
		-1	0	+1
M:O (molar ratio)	A	20	31	42
Temperature (°C)	B	240	260	280
Pressure (bar)	C	180	220	260
Time (min)	D	12	22	32

3. Results and Discussion

Results are reported for the main activities of this research, i.e. results of quality test method of WCO, and results of biodiesel production.

3.1 Results of WCO Quality Testing Method

Table 2 reports the measured acid values for the waste cooking oil samples collected previously.

Table 2. Acid value data for WCO samples

WCO Sample	Reuse Cycle	Acid Value (mg KOH/g Oil)
1	0	0.1
2	1	2,5
3	2	4
4	3	7.5
5	4	10
6	>10	25

As indicated from table 2, the acid value increases with the increase of the number of reuse cycles. A possible reason for this increase is the hydrolysis and oxidation of oil due to the introduction of wet food and high temperature. It should be noted that there are another important factors that affect the acidity of the reused oil, which is the type of food being fried, time, frying temperature and the type of used oil (palm, soybean). Also, the number of double and triple bonds already exist in the structure of the oil molecule have a significant influence on its hydrolysis and oxidation in addition to their decomposition to other polar components.

The data obtained were plotted so, the acid value on the ordinate (y-axis) and the number of reuse cycles of WCO on the abscissa (x-axis). Then data are fitted to a proper model and correlation is extracted. The data are shown in Figure 2 below which fit well to a linear interpolation. The correlation derived from the data plot is reported to be:

$$A_v = m \times N_c + c$$

Where A_v is the predicted acid value by the model, N_c is the number of reuse cycles of WCO, and m and c are the model constants ($m=2.4$, $c=.1$). The error of the predicted values by the model with respect to experimental data is calculated from the following equation (N_s =total number of WCO samples), and shown in Table 3:

$$\% \text{ Error} = \frac{\text{Predicted} - \text{Experimental}}{\text{Experimental}} \quad (6)$$

$$\% \text{ Max Error} = \frac{\sum_{i=1}^{N_s} (\text{Predicted} - \text{Experimental})}{N_s} \quad (7)$$

The maximum error of the predicted acid values is calculated to be 5.3. The correlation can be employed to predict the acid value corresponding to a number of reuse cycles, e.g. for a number of 7 times reuse cycles, the predicted acid value will be 16.9 mg KOH/g oil.

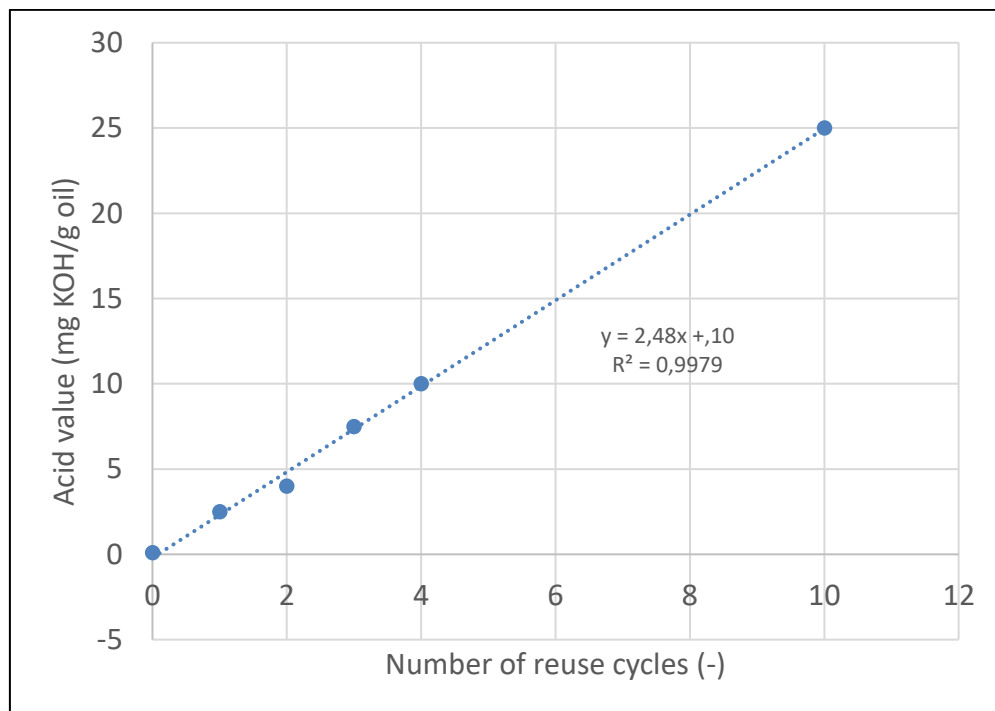


Figure 2. Correlation plot for acid values

Table 3. Experimental/predicted data for acid value data of WCO samples

WCO Sample	Reuse Cycle	Experimental (mg KOH/g Oil)	Predicted (mg KOH/g Oil)	Deviation (mg KOH/g Oil)	% Error
1	0	0.1	0,10	0,00	0
2	1	2.5	2,50	0,00	0
3	2	4	4,90	0,90	22,5
4	3	7.5	7,30	0,20	2,7
5	4	10	9,70	0,30	3
6	>10	25	24,10	0,90	3,6
Average Error %					5,3

The calculations of the extra catalyst concentration and the % FFA for the measured data are shown in Table 4 due to higher values of acid numbers.

Table 4. Experimental/predicted data for acid value data of WCO samples

WCO Sample	Reuse Cycle	Experimental (mg KOH/g Oil)	% FFA	KOH Catalyst amount (%)	Extra KOH catalyst (%)
1	0	0,1	0,05	1,01	0,01
2	1	2,5	1,25	1,29	0,29
3	2	4	2	1,46	0,46
4	3	7,5	3,75	1,86	0,86
5	4	10	5	2,15	1,15
6	>10	25	12,5	3,86	2,86

3.2 Physiochemical Properties

Waste cooking oil feedstock properties are calibrated as shown in the table 5. The purified biodiesel produced from the supercritical trans-esterification reaction using the optimum conditions that were analyzed for evaluating the chemical and physical properties. The obtained results were obtained and compared with the European standards of biodiesel, EN14214. The analysed properties have been repeated two times and the final result have been obtained as an average of the two results. ASTM D4052 method was used to determine the standard density, while ASTM D445 method was used to determine the kinematic viscosity. ASTM D974 method was used to calibrate the produced biodiesel total acid number (TAN) of the produced biodiesel.

Table 5. Properties of WCO feedstock

Test	Calibration Method	Result
Kinematic viscosity at 40 °C	ASTM D-445	54.2 cSt
Density at 15°C	ASTM D-4052	0.883 g/cm ³
TAN	ASTM D-974	0.8 mg KOH/g oil

3.3 Physiochemical Properties of the Produced Biodiesel

The standard density of the produced biodiesel is 887 kg/m³. The produced biodiesel at methanol-to-oil molar ratio 37:1, temperature 253°C, pressure 199 bar and reaction time of 15 minutes has been subjected to examination ensuring that its properties abide by the European Biodiesel Standards, EN14214 [12]. Table 6 shows the properties of the produced biodiesel and the European biodiesel standard acceptable range. The other byproducts produced were discarded.

Table 6. Comparison between produced biodiesel properties and European biodiesel standard EN14214

Test	Unit	Produced biodiesel	Biodiesel (EN14214)
Density at 15°C	kg/m ³	887	860 - 900
Kinematic viscosity at 40°C	cSt	4.63	3.5 - 5
TAN	mg KOH/ g oil	0.09	< 0.5
Pour point	°C	-6	N/A
Flash point	°C	161	> 101
Cetane number		59	> 51

4. Conclusion

Based on our previous projects results, the collected WCOs from restaurants and industries usually have very high acidity due the extensive usage in cooking process. It has been recently reported that the repeatedly usage of cooking oil at elevated temperatures produce polycyclic aromatic hydrocarbons (PAH) that are recognized as carcinogenic compounds. Further, the high acidity obtained from literature review creates several problems for WCO valorization feedstocks, i.e. biodiesel. One biodiesel company (Biodiesel Misr), who are the only operating biodiesel plant in Egypt working to convert WCO into biodiesel, have confirmed the difficulties of processing high acidity WCO where costly pre-treatment steps are vital. Accordingly, the extensive usage of cooking oil from industries and restaurants are affecting the Egyptian people's health and causing problems for WCO valorization processes. The project is aiming to develop guidelines for WCO management including disposal standard regulations to be implemented by households, restaurants, and industries. The guidelines will be submitted to the Egyptian government and a practical implementation method will be also proposed. The project will develop a rapid testing method for onsite inspection of WCO prior collection. The optimum reaction conditions are at methanol-to-oil molar ratio 37:1, reaction temperature of 253°C, reaction pressure of 199 bar and reaction time of 15 minutes.

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