Preliminary evaluation of the effectiveness of moisture removal and energy usage in pretreatment module of waste cooking oil for biodiesel production

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Abstract. Waste Cooking Oil (WCO) is a plausible low cost biodiesel feedstock but it exhibits few unfavorable parameters for conversion into biodiesel. One of the parameter is the presence of high moisture content which will inhibit or retard catalyst during the acid esterification or base transesterification causing lower purity and yield of biodiesel. This will effect the post processing and escalate production cost making WCO a not favorable biodiesel feedstock. Therefore, it is important to have an effective moisture removal method to reduce the moisture content below 0.05%wt or 500 ppm in WCO for an efficient biodiesel production. In this work, the effectiveness of moisture removal and the energy usage of a newly develop innovative pretreatment module has been evaluated and reported. Results show that the pretreatment module is able to reduce up to 85% to effectively reduce the moisture content to below 500ppm of the initial moisture content of WCO and only consume 157 Wh/l energy compared to conventional heating that consume 386 Wh/l and only remove 67.6% moisture in 2 hours.

1. Introduction
Biodiesel is predominantly produced from vegetable oils and animal fats using a chemical process known as “transesterification” which involves interchanging of glycerol in the triglyceride (vegetable oil) molecules with alkyl alcohol molecules to form monoalkyl esters (biodiesel) in the presence of acid or base catalyst [1]. The base catalyzed transesterification is usually favored for industrial scale biodiesel production for unused vegetable oils are used as feedstock as it is more efficient and faster[2]. In recent years, waste cooking oil (WCO) which is 2-3 times cheaper than edible vegetable oils has been considered as feedstock. However, the use of WCO as biodiesel posses feedstock quality issues and challenges since WCO exhibit high percentage of Free Fatty Acid percentage (%FFA) and high moisture content compared to unused vegetable oils [3].

The favorable level of FFA and moisture content for base catalyzed transesterification are below 1%wt and 0.05%wt respectively [4]. However, WCO generally exhibit FFA more than 2%wt and moisture content beyond 0.05%wt. This will cause formation of soap or saponification reaction process resulting in consumption of catalyst which further will cause emulsion problems during post processing through water washing which is require for removing excess catalyst, alcohol, mono, diglycerol and free glycerol and also reduce the biodiesel yield and purity significantly [5].

The objective of this work is to evaluate the effectiveness of a newly developed innovative pretreatment module for WCO feedstock moisture that hybrids low energy heating and vacuum with a static dehydrator is evaluated. The evaluation of energy consumption has been conducted by
measuring the total energy (heating + vacuum) consumed to reduce the moisture content per liter of WCO feedstock and compared with conventional heating over two hour period.

2. Material and Method
WCO samples were collected from local food outlet Mawa De Village, Segamat. Moisture Content is determined using automated Coulometric Karl Fisher titration. The chemical use for moisture content determination is Aquamicron CXU and Aquamicron AXU with purity of 99.99% from Merck. The moisture content of WCO is determined using ASTM D1796 test method as prescribed in ASTM D6751 standard. The testing is conducted at TNBR Qats Sdn. Bhd in Kajang. The energy usage is determined by using single phase Efergy™, energy meter with ±0.1Wh accuracy.

A pretreatment module with working volume of 20 liter has been used in the moisture removal experiment where the closed system module is equipped with 1 kW heater in the main tank and preheat tank respectively. The module consisted of two section; filtering section consisting filter size of 5µm, 1µm and 0.5µm and water removal section consisting of nozzle system with 60 numbers of 1.2 mm diameter orifices. The closed system pretreatment is equipped with a vacuum pump and condenser system to reduce the moisture vapor temperature which also has a conical shape static dehydrator section located at the bottom of the tank. For conventional heating method, the experiment is conducted at atmospheric pressure by keeping the heater on with the tank cover and dehydrator removed. The pump recirculation is only deployed during filtering in conventional heating.

2.1. Sample preparation and Measurement of Moisture, FFA and Energy Consumption
A 10 liter WCO sample is filtered using 1mm sieve in order to remove large particulate and preheated up to 100°C before re-filtered in 5.0 µm, 1 µm and 0.5 µm in filtering section to remove smaller particulate at 90°C for about 20 minutes (approximately 5 cycles) without deployment of the vacuum.

The Pretreatment module with nozzle size spray size of 1.2 mm without the presence of dehydrator is first operated for 20 minutes filtering, before the flow is changed to moisture removal section. All measurement is repeated 3 times and the average is calculated for reporting. The energy usage is recorded using the energy meter and the heater temperature is maintained at 100°C. Subsequently, the pretreatment module is equipped with the dehydrator and same procedure is repeated. As for conventional heating the sample is heated up to 100°C in the pretreatment module without closing the top cover (open system). Upon reaching 100°C, 75 ml sample is drawn into sampling bottle and the vacuum pump is deployed first sample was drawn and experiment continued for 120 minutes with samples drawn every 15 minutes for moisture content analysis.

3. Result and discussion
Frying activity using triglycerides (cooking oil) at temperatures more than 180°C promotes hydrolysis results in the fatty acid of triglycerides being disengaged from the glycerol backbone of the triglyceride molecule and become FFA [6]. The increase FFA further attract more water molecules to form hydrogen bond. Commonly, heating is deployed up to the boiling temperature of water at 100°C for removing moisture at atmospheric pressure, which is not energy efficient and also time consuming. In the conventional heating, the bound water/moisture molecules have to overcome the surrounding bulk forces by the triglycerides molecules before gaining enough kinetic energy to liberate out of the oil due to convectional heat transfer and evaporate out through top triglyceride surface. A study by Felizardo et al (2006), proposed the use of vacuum distillation (0.05 bar absolute) to remove moisture from crude WCO at temperature as low as 30–40°C, which consumes less energy for the feedstock heating [7]. However, the disadvantage of this method is the high vacuum instrument consumes high energy and also safety concern in working with vacuum tank. The moisture removed and energy usage from the pretreatment module and for comparison the conventional heating has been summarized in Table 1. The result shows that moisture content for all samples after 2 hours meet the requirement for base catalyzed transesterification which is below than 500 ppm. Sample 3 shows the highest moisture removed which is 87.4% with least energy consumption which is 157 Wh/l. Compared to conventional
heating, the energy used is double (386 Wh/l) and only 67.6% of moisture removed. Figure 1 and 2 shows that the moisture content from sample 2 and 3 are already achieve the moisture target (<500 ppm) in 35 minutes while conventional heating required longer time (60 min). Table 1, shows average temperature of only 90°C is required during the whole moisture removal process for sample 2 and 3 because of the pressure in the tank is below the atmospheric pressure which reduces the boiling point of water. This results is in agreement with the work done by Felizardo et al., (2006) but eliminate the need for using pressurize tank where the vacuum pressure in this work is at 0.8 bar absolute [7].

Table 1. Comparison between conventional heating and pretreatment module

| Sample no | Initial Moisture (ppm) | Method of moisture removal | Final Moisture (ppm) | Average WCO Temperature (°C) | Amount of Moisture Removed (%) | Energy Consume/ liter (Wh/l) |
|-----------|------------------------|----------------------------|----------------------|-----------------------------|-------------------------------|-----------------------------|
| 1         | 870                    | Conventional heating       | 282                  | 100                         | 67.6%                         | 386                         |
| 2         | 1243                   | Pretreatment without dehydrator | 305              | 90                          | 75.5%                         | 157                         |
| 3         | 2392                   | Pretreatment with dehydrator                      | 302                  | 90                          | 87.4%                         | 157                         |

Figure 1. WCO Moisture Content over Time

Another method of moisture removal is by using chemical and desiccants such as magnesium sulphate and silica gel but these methods requires a fine filtering system for the feedstock before entering the base catalyzed transesterification stage [7,8,9,10]. Disadvantages of using chemical and desiccant is the increase of biodiesel production cost due to regeneration limit of the chemical. Figure 1 and 2 have shown that the hybrid use of nozzle spray and static dehydration method enhanced the moisture removal from 75.5% to 87.4% with the same energy consumption. The oil droplet formed after the nozzle spray under vacuum condition enables the water molecules to be easily liberated due to inward heat transfer from the triglycerides molecules to the water molecule which undergoes outward mass transfer to surface of droplet and induced out of the closed tank by the vacuum. Also in the smaller droplet form the influence of hydrogen bond between the water molecule and the triglyceride molecule especially with FFA, di- and mono- glyceride presence in the WCO is less stronger compare to being in the bulk form. Subsequently the droplet of oil that impinges on the dehydrator due to the gravitational force further gets spread into a thin layer over the inclined plane of the conical shaped static dehydrator which further enables remaining moisture trapped within.
triglyceride to be liberated and also induced out by vacuum. Despite, the starting moisture for the conventional heating being the lowest of 870 ppm the time taken to achieve 282 ppm is two (2) hours compared to the pretreatment module that had higher moisture content, up to 2,392 ppm and need the same two hour to reach 302 ppm. This indicates that the use of static dehydrator is more energy efficient compare to conventional heating better than chemical and desiccant for moisture removal enhancement.

4. Conclusion
The pretreatment module is able to reduce moisture content in WCO below 500ppm and has effectiveness of removing up to 85% of the initial amount of moisture in WCO using 1.2 mm nozzle size with the presence of static dehydrator. The whole process only used 157 Wh/l which is less than half of energy used by conventional heating method. Therefore, the newly develop pretreatment module can be integrated in biodiesel production system that uses WCO as feedstock where it is more energy and cost efficient. Hence, the cost of biodiesel production using WCO as feedstock can be reduced significantly.

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