Improvement of Residual Oil Recovery from Oil Palm Biomass Using High Pressure Water Steam System for Biodiesel Production

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Various applications of oil palm empty fruit bunches (OPEFB) would be hindered by the presence of residual oil. This study aimed to remove and recover the residual oil from OPEFB using an integrated system, high pressure water spray system (HPWSS). The performance of the HPWSS was evaluated at different temperatures and water pressures, and the residual oil collected was recovered through water shaking method and tested for biodiesel application. A maximum of 84.9% of residual oil was removed by HPWSS at 60 °C and 8960 kPa and the highest residual oil recovery of 58.8% was observed at 95 °C, using power shaking 5 and 90% of dilution. The following ranges of deterioration of bleachability index (DOBI), free fatty acid (FFA), and peroxide value (PV) for the residual oil were 1.21 to 2.67, 7.11 to 10.4%, and 4.85 to 7.56, respectively. Biodiesel with different blends of recovered residual oil (5%, 10%, and 15%) showed lower values (9.87, 9.57, and 9.56 Nm) of brake torque as compared with diesel (10.89 Nm). Overall, this study showed the potential of HPWSS to obtain an acceptable quality of residual oil from OPEFB to be used in any value-added product generation.

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INTRODUCTION

As one of the major agricultural plantation industries in Malaysia, oil palm plays a significant role in heightening the economy. Up until 2019, there were approximately 5.58 million hectares of oil palm plantation in Malaysia, making Malaysia one of the biggest exporters of oil palm products in the world (MPOB 2019). Owing to the huge plantation areas and active palm oil extraction, huge amounts of biomass are generated continuously. Oil palm empty fruit bunch (OPEFB) is one of the major oil palm biomasses produced, which accounted for up to 23 million tons annually (Anuar *et al.* 2019). These numbers will keep increasing as a result of high demands of oil palm products worldwide.

As a by-product from palm oil mill, OPEFB is categorized as herbaceous biomass. It has shown unclear potential applications and suffer poor monitoring (Tursi 2019). Being a renewable lignocellulosic material, OPEFB has been subjected to various bioconversion products and materials. Its physicochemical composition has been explored and exploited for many great purposes. Latip *et al.* (2019) and Mensah *et al.* (2019) investigated the usage of OPEFB fibers for reinforced biocomposites, while Padzil *et al.* (2020) reviewed the potential application of nanocelluloses from OPEFB for hydrogels production. Due to its high holocelluloses and calorific content, OPEFB has proved to be a great source for sugar and bioenergy production (Ibrahim *et al.* 2015a; Hassan *et al.* 2020). However, all these bioconversion processes will be hindered because of the presence of residual oil on the surface of OPEFB. Residual oil has been referred to as any oil that is lost during palm oil mill processing such as fresh fruit bunch pressing, threshing, and stripping processes that occurred earlier in the mill. It has been proposed that it will hold up the penetration of enzymes, chemicals, and affect the products of the bioconversion (Gomez *et al.* 2014; Ibrahim *et al.* 2015b). Hence, removal of residual oil is a crucial step before OPEFB can be digested and utilized.

Removal of residual oil from OPEFB surfaces has not yet been implemented commercially in Malaysia because of the current practices that usually abandon OPEFB on the ground to be composted or used as mulching and bulking agents in the oil palm plantation (Noah 2022; Chiew and Shimada 2013). Rather than being thrown away, the residual oil on OPEFB can be recovered and re-used. Recovering this residual oil not only allows it to be used for biodiesel, but it can also act as one of the strategies to increase the national oil extraction rate (OER). Suwanno *et al.* (2017) and Yusoff *et al.* (2021) studied the conversion of the residual oil from oil palm biomass for biodiesel and were able to produce palm biodiesel that met the ASTM D570-098 (2010) standard. In another study by Gomez *et al.* (2014), 15.2 to 28.5% of residual oil were detected on outer surface of several sizes of OPEFB, while Xiang *et al.* (2016) recorded that 13.1% of residual oil was detected on spikelet of OPEFB. Hence, recovering this residual oil is compelling. Ignoring this residual oil is a tremendous loss, since its potential in increasing the OER rate and palm-based biofuel is financially rewarding.

Removal of residual oil from OPEFB can be very challenging because of its almost round to polygonal shape, making it always mobile and causing it to have many hidden corners. The bulky OPEFB and the sharp spikelets on the surface also contribute to the difficulty in handling them. Numerous studies have been conducted to remove residual oil from oil palm biomass. However, until this current research there have been no studies attempting to remove residual oil from OPEFB as a whole bunch. A summary of residual oil removal techniques from OPEFB is shown in Table 1. Gomez *et al.* (2014) attempted to remove residual oil from spikelet and was able to achieve 82% oil removal by using HYSASE (hydro solvent assisted steam extraction) technique. However, this method required one to cut the spikelets individually in order to achieve higher oil recovery. This method was considered impractical for commercialization because of the difficulty and because it is time-consuming and laborious. In another study by Yunos *et al.* (2017), OPEFB were subjected to high pressure water and steam spray to remove and collect residual oil. This method was able to remove up to 94.4% of residual oil from OPEFB at 150 °C. Yet, this method was performed on a small scale where individual bunches were collected and subjected to high pressure water spray system (HPWSS). Because of the almost round shape of OPEFB, it has to be manually turned in order to remove residual oil at every nook and cranny of the bunch. Again, this method can be judged to be impractical for industrial scale and to pose dangerous hazards because of the high pressure and hot steam. In another recent study by Ng *et al.* (2020), a crystallization technique was performed on OPEFB fibers where both liquid nitrogen and standard refrigerator system were adopted. Though the proposed technique has some potential, the usage of liquid nitrogen is not cost-effective and the developed crystallization cooling system is inappropriate for bulky OPEFB bunches.

Table 1. Residual Oil Recovery Techniques from Oil Palm Empty Fruit Bunches

No.	Biomass	Recovery Oil Techniques	References
1	OPEFB fibers	Integrated water-steam process by proposed Hydro Solvent-Assisted Steam Extraction (HYSASE)	Gomez *et al.* (2014)
2	OPEFB bunches	Manually spraying pressurized water and water-steam, skimming floating residual oil and centrifuged to remove water and impurities	Yunos *et al.* (2017)
3	OPEFB spikelet	Batch sub-critical water extraction chamber	Kurnin *et al.* (2016)
4	OPEFB fibers	Crystallization technique with liquid nitrogen and standard refrigerator system	Ng *et al.* (2020)

In this paper, the authors designed and developed a machine that is suited for residual oil removal and recovery from whole bunches of OPEFB, without any prior maceration or pulverization process on the OPEFB. This machine was improvised from previous work (Yunos *et al.* 2017), with the addition of specific features such as nozzles, heating element, rotary drums, and belts, aiming to efficiently remove residual oil from OPEFB continuously. Until this paper was written, there was no machine having similar function and design reported for OPEFB processing, thus highlighting the novelty of this study. In this paper, the design of the machine is thoroughly discussed, and the removal and recovery of the residual oil is investigated in order to show its efficiency.

EXPERIMENTAL

Materials and Reagents

Oil palm empty fruit bunches and raw crude palm oil (CPO) were obtained from Felda Trolak Palm Oil Mill (Sungkai, Perak, Malaysia). Palm oil tester reagents for free fatty acids (FFAs), peroxide value (PV), and deterioration of bleach ability index (DOBI) were purchased from PLT Scientific Sdn Bhd (Selangor, Malaysia). Analytical grade *n*-hexane was purchased from Merck (Darmstadt, Germany).

Design and Fabrication of Prototype

The prototype of the machine consisted of a twin mechanical rotating drum with dimension of 1.6 m length and diameter of 0.51 m and powered by 1.5 kW single phase induction motor (DEPRO, Bryne, Norway). Rotation speeds was controlled with worm gear reducer with ratio 1:60 (WPA-60, China) coupled with 4 and 7 inches (10.16 and 17.78 cm) single groove V-belt pulley (ESCO, Osaka, Japan). Linear track for Kärcher high pressure nozzle size 65 (25065, Winnenden, Germany) movement was controlled by ST100 Stepper motor drive coupled with stepper motor (17HS4401, NEMA, China) and adjusted to spray at angle of 90°. The mechanical structure of the machine prototype was fabricated using mild steel and welded together. Pressurized hot water and steam were supplied by a Kärcher high pressure washer (HDS 13/20/4S, Winnenden, Germany).

OPEFB Physical Characteristic Measurement

Sphericity and aspect ratio

A shape of non-uniform object can be categorized by using Sphericity and Aspect ratio index based on linear dimension detail. The sphericity index of OPEFB was calculated using Eq. 1,

$$S_c = (abc)^3 / a \times 100 \tag{1}$$

while the aspect ratio (R_a) was calculated using Eq. 2,

$$R_a = b/a \times 100 \tag{2}$$

where a refers to OPEFB length (major diameter), b refers to OPEFB width (intermediate diameter), and c refers to OPEFB thickness (minor diameter).

Center of gravity

The center of gravity of OPEFB is a hypothetical point in which the force of gravity may be considered to act. The center of gravity value will be affecting object stability towards rolling and process machinery design to handle OPEFB. The center of Gravity could be calculated using the formula recommended by Lemaire *et al.* (1991) as shown by Eq. 3,

$$R_{cog} = [R_1 - (R_1 W_{sc} - R_2 W_{bp}) / W_{EFB}] - R_4 \tag{3}$$

where R_{cog} refers to the distance from the stalk to the OPEFB center of gravity (cm), R_1 refers to the distance between two pivot points (platform length, cm), R_2 refers to the distance from the pivot point to the balance platform center of gravity (cm), R_3 refers to the distance from the pivot point to the OPEFB center of gravity (cm), R_4 refers to distance from pivot point to stalk (cm), W_{sc} refers to the weight reading from the scale (kg), W_{bp} refers to the weight of balance platform (kg), and W_{EFB} refers to the weight of OPEFB (kg) (Fig. 1).

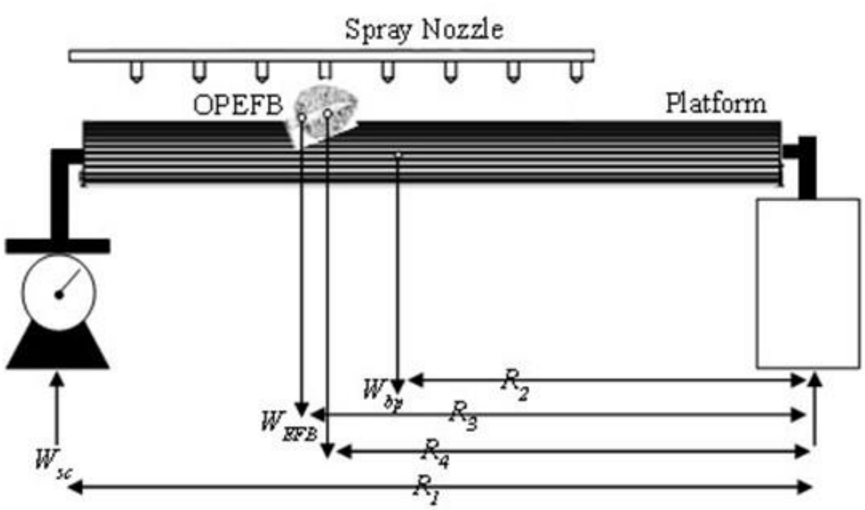


Fig. 1. Setup of center of gravity measurement

Removal of residual oil from OPEFB

Residual oil removal from OPEFB was improvised from Yunos *et al.* (2017), where pressurized water/steam was sprayed onto the surface of the rotating OPEFB. A variation of temperatures (30 and 60 °C) and pressures (3447 and 8963 kPa) were adopted at different cycles. The OPEFB was placed on the twin drum conveyer, and with the rotation action of the twin mechanical rotating drum, the OPEFB was rotated, exposing the multiple surfaces of the OPEFB. As the OPEFB rotated and moved towards the end of the drum, pressurized water/steam was sprayed onto the OPEFB. Finally, the water with oil resulted from the spraying was collected and drained for oil analysis and stored in a freezer (-20 °C) for subsequent use.

Recovery of residual oil by water shaking method

Residual oil was recovered through the water shaking method, where the water-oil separation process was performed by mechanical force. The effluent from the spraying experiment was thawed at room temperature. About 200 mL of the thawed effluent was placed in a 500 mL beaker and securely placed in a water bath (Thermo Fisher, Waltham, MA, USA). The experiments were run for 10 h where the accumulated oil layer on top was extracted using hexane at 2 h intervals. Temperatures (65, 75, 85, and 95 °C), shaking power (1, 3, and 5) and sample dilution (40% and 90% v/v) were investigated, and data were collected.

Residual oil content determination

Residual oil content determination was performed according to Abd Majid *et al.* (2012). A soxhlet extraction apparatus (Bibby Scientific, Staffordshire, UK) with hexane as the solvent was adopted. Initially, the fiber samples were oven dried overnight at 105 °C. About 150 mL of hexane as solvent was used to extract residual oil from 10 g of samples. The extraction was performed continuously for 6 h, and finally the hexane was removed by evaporation using a rotary evaporator (RV10 Digital, IKA, Staufen, Germany). Residual oil content was calculated using Eq. 4,

$$\text{Oil content (\%)} = \text{weight of oil} / \text{weight of sample} \times 100 \qquad (4)$$

Residual oil characterization

Deterioration of bleachability index, FFA, and PV values were determined according to MPOB Test Methods (2004). All results were performed in three replicate experiments.

Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy was performed on residual oil and OPEFB fibers using Nicolet iZ10 (Thermo Fischer Scientific, Waltham, MA, USA). The wavelength range of 400 to 4000 cm^{-1} at 4 cm^{-1} resolution was used with IR spectra, where 32 scans of each sample were performed. The OMNIC spectrum software (Thermo Fisher Scientific, Waltham, MA, USA) was used to analyze and construct the FTIR spectra. The reflection of spectra was determined using a golden gate diamond attenuated total reflectance (ATR) 10 500 modules with germanium crystals.

Residual oil transesterification

Biodiesel was produced from residual oil using a transesterification process in which the ethanolysis of oil to its ester/biodiesel with the addition of 1.0 wt% NaOH catalyst was conducted in a 1 L of separating funnel with methanol in a molar ratio of 1:5 (methanol: oil) at 60 °C for 60 minutes and allowed to separate into distinct layers (Efavi *et al.* 2018). The glycerol layer was decanted, and the oil ester/biodiesel was dried at 60 °C to allow for the complete removal of methanol and for the water to remain in the solution.

Test engine setup

The engine tests were conducted using a 4-cylinder diesel (CI) engine model L48N6 (Yanmar, Harayana, India). The engine was then connected to the eddy current dynamometer model GFA174A-5 (Landtop, Fujian, China). The dynamometer was used to measure the engine speed, engine power and engine torque and was run for a few times until the exhaust gas temperature, cooling water temperature, and lubricating oil temperature attained steady-state values. The analysis of brake torque and nitrogen oxide (NO_x) level were recorded using 5%, 10%, 15%, and 20% biodiesel blended with petrol notated with B5, B10, B15, and B20 and compared to the petroleum diesel and commercial manufacturer diesel.

RESULTS AND DISCUSSION

OPEFB Physical Characteristics and Residual Oil Distribution

Assessment of physical characteristics of OPEFB is crucial as it serves as foundational knowledge on the OPEFB bunches which are used to design the HPWSS machine. A total of 100 OPEFB bunches were freshly selected from the mill. Table 2 shows that the average weight and length of the collected OPEFB bunches was around 7.27 kg and 53.8 cm, respectively. This was considered as large bunches as according to Gomez *et al.* (2014). Spikelet and stalk weights were around 30% and 70% from overall OPEFB bunches, respectively. All of spikelet, stalks, and OPEFB have high moisture content (more than 60%) which is caused by the water and steam sterilization process during crude palm oil extraction in the mill (Arbaain *et al.* 2019).

Table 2. Physical Characteristics and Oil Content of Oil Palm Empty Fruit Bunch

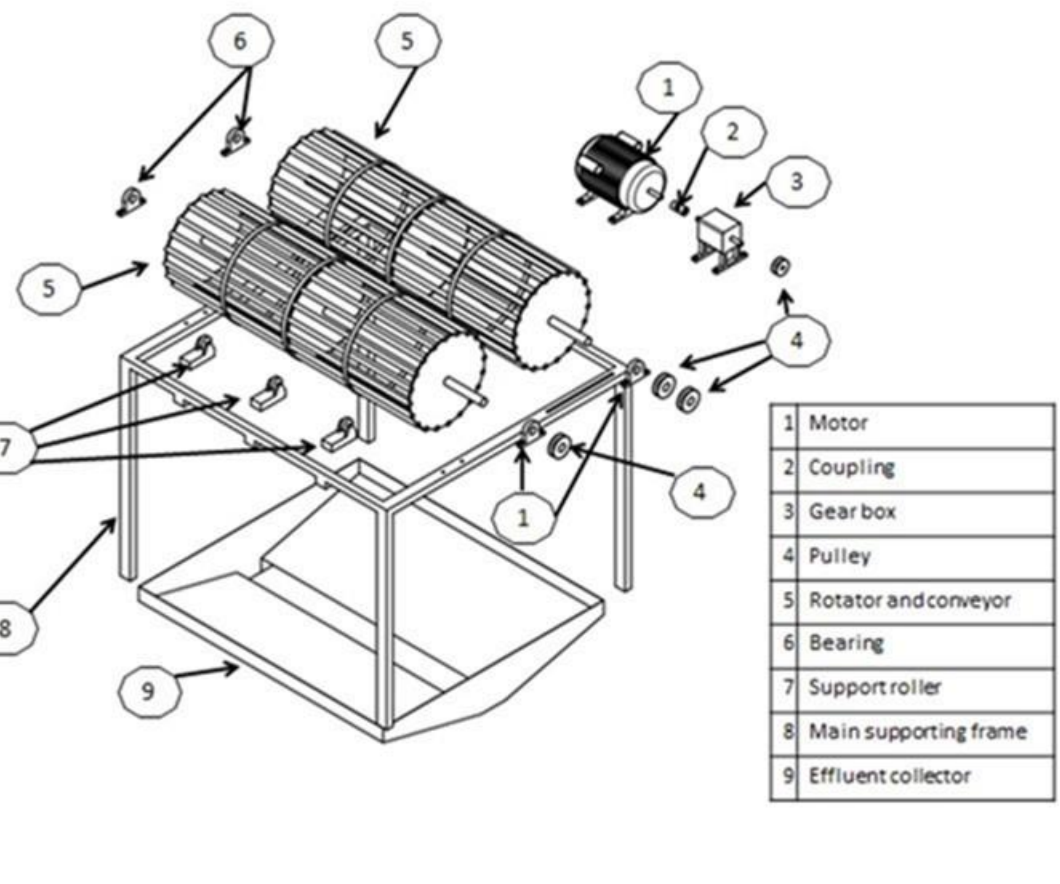
Samples	Weight (kg)	Length (cm)	Moisture Content (%)	Oil Content (%)
EFB	7.27 ± 3.32	53.81 ± 2.61	61.74 ± 1.49	9.32 ± 3.98
Spikelet	2.13 ± 0.31	11.78 ± 3.01	74.19 ± 0.4	13.99 ± 1.66
Stalk	5.14 ± 0.44	43.73 ± 3.56	81.16 ± 2.7	0.44 ± 0.22
Major axis (cm)	70.78 ± 7.14			
Minor axis (cm)	54.32 ± 6.27			
Sphericity (%)	72.60 ± 4.97			
Center of gravity (CoG) (%)	46.93 ± 5.05			

Residual oil content was also determined, where it refers to the total oil remaining on the biomass after the palm oil mill process. Spikelet contained the highest residual oil content (14.0%), followed by OPEFB shredded bunches (9.32%), and stalk containing the least amount of residual oil (0.44%). A similar trend was observed by Yunos *et al.* (2015) and Xiang *et al.* (2016), where the former studied the oil attachment on the fibers surface and observed its attachment through Sudan dye colorization. The highest percentage of residual oil in spikelet might be due to its adjacent localization with the fruitlets where the source of the oil came from. Stalk had a very minute oil residue because of it was enclosed at the core of the OPEFB bunch, abstaining from any contact with oil. Table 2 also shows the major and minor axis of OPEFB which was 70.78 and 54.32 cm, respectively, while sphericity and center of gravity were calculated as 72.60% and 46.93%, respectively. A similar trend for sphericity and aspect ratio was reported by Hazir and Shariff (2011), who studied oil palm fresh fruit bunch.

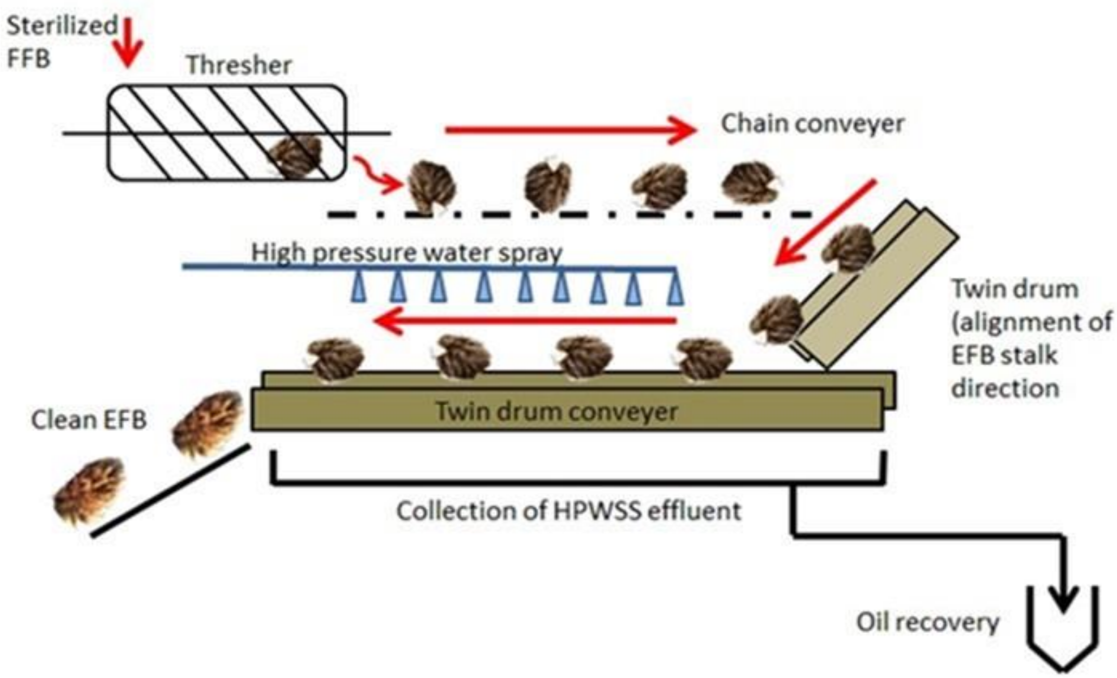
Design and Fabrication of High Pressure Water Spray System

The design and fabrication of HPWSS were made based on the current authors' preliminary study (Yunos *et al.* 2017). In this study, the authors designed a twin cylinder conveyer system to aid the alignment of OPEFB bunches along the conveyer system by its stalk direction (Figs. 2a and 2b). The mechanism was based on its center of gravity properties and assisted by the twin cylinder. Because of the similar approach, the concept of the design was improvised from the egg rollers that are being widely used in mills. Such rollers action affected the axial and turnover motion, depending on the center of gravities and the major and minor axis lengths (Jiang *et al.* 2014; Cheng *et al.* 2021). The continuous rolling motion of the twin cylinders projected the rotational of the individual OPEFB bunches and movement of OPEFB towards the end of the cylinders. The spaces between the longitudinal twin cylinders were maintained similarly in order to provide guides for OPEFB to move as well as to protect the OPEFB from being hindered to be individually rotated.

(a)



(b)



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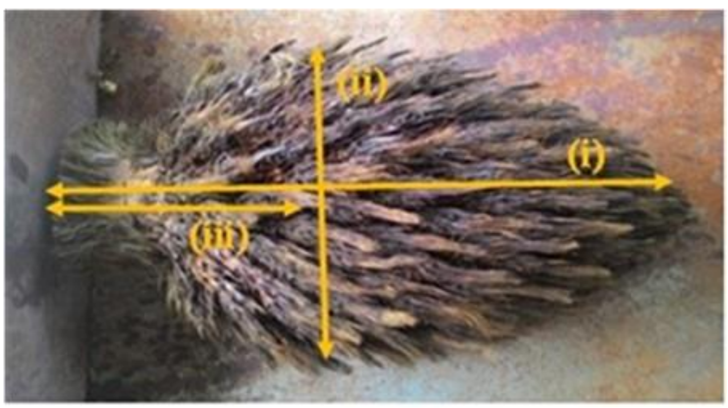

Fig. 2. (a) Schematic assembly view of HPWSS, (b) process flow of HPWSS in the palm oil mill, and (c) a fresh OPEFB with measurement of (i) major axis, (ii) minor axis, and (iii) center of gravity

As the OPEFB bunches moved along the longitudinal twin cylinders, they were cleaned using high-pressured hot water that were channeled through multiple nozzles. It is worth mentioning that this design of HPWSS has been submitted for patent application that can be referred with patent file number PI2017703897.

Residual Oil Removal by High Pressure Water Spray System

Efficiency of HPWSS for oil removal from OPEFB was assessed at different temperatures and water pressures. The physical effects of untreated and HPWSS treated OPEFB can be observed in Figs. 3a and 3b, where obvious differences on color and structures were recorded. As shown in Fig. 3c, the structure of OPEFB was a little bit ruptured, indicating the penetration of HPWSS treatment on every surface of OPEFB.

Figure 3d shows fungal growth on the surface of OPEFB after 3 days of HPWSS treatment, suggesting that the OPEFB was clean and had minimal residual oil hence the favor of the microorganism's growth.

Fig. 3. Physical effect of OPEFB bunch for (a) untreated OPEFB, (b) HPWSS treated OPEFB, (c) closed-up of HPWSS treated OPEF, and (d) fungal growth on HPWSS treated OPEFB after 3 days

Table 3 shows the effects of temperatures of HPWSS on oil removal and the oil quality. In this experiment, the authors used temperatures of water (30, 60, and 90 °C) and water-steam (120 and 150 °C) at 3447 kPa to treat OPEFB. Table 3 summarizes the maximum oil recovered from OPEFB at 150 °C (94.1%), and the lowest oil recovered (62.6%) was achieved at 30 °C. It is obvious that as the temperature rose, more residual oil could be removed. These results were in good agreement with Gomez *et al.* (2014) and Yunos *et al.* (2017) where higher oil removal from OPEFB can be achieved at higher temperatures.

The water must be hot enough (reach its critical point) in order to lessen the oil's dynamic viscosity (Gomez *et al.* 2014). However, as the temperature increased, the oil quality decreased. The FFA percentage increased as the temperature increased where the maximum FFA content (10.39%) was recorded at 150 °C, and the minimum FFA content (7.11%) was recorded during HPWSS operated at 30 °C. The FFA content refers to the acidity of oil and is usually caused by the oxidation from enzymatic action and microbial lipases. As suggested by Godswill *et al.* (2017), it indicates the oil degradation rate of the oil. Its value can be affected from enzymatic action, extreme conditions of the oil extraction process, longer storage time, high moisture, and bruised oil palm fruitlets. It is noteworthy that the maximum FFA content set by Palm Oil Refiners Association of Malaysia in crude palm oil is 5%, where beyond that, values may require further refining process, which is costly and time consuming (Azeman *et al.* 2015).

Table 3. Effect of Temperatures of High Pressure Water Spray System on Oil Removal and its Residual Oil Quality

Temperature (°C)	Processing Media	Oil Removal (%)	FFA (%)	DOBI	PV (meq/kg)
30	Water	62.57	7.11	2.67	4.85
60	Water	77.64	7.81	2.21	5.53
90	Water	85.44	8.64	2.05	6.42
120	Water-steam	92.54	9.47	1.87	7.09
150	Water-steam	94.08	10.39	1.21	7.56

The highest PV was recorded (7.56 meq/kg) during the 150 °C condition, while the lowest PV (4.85 meq/kg) was obtained during the 30 °C condition. Table 3 shows a linear correlation between PV and temperature, where similar observations have been recorded previously (Gomez *et al.* 2014; Yunos *et al.* 2017). As mentioned by Teh *et al.* (2020), PV refers to the level of lipid oxidation in the oil where the maximum acceptance values for edible oil is no more than 10 meq/kg according to CODEX STAN 210-1999 (1999). Hence, the PV values in Table 2 were within the range and met the acceptance values. As for DOBI, the values decreased as the temperature increased. Residual oil samples obtained at 150 °C had the lowest DOBI value (1.21), while the highest DOBI value (2.67) was obtained at 30 °C. The DOBI value refers to the numerical ratio of carotene and secondary oxidation products, signifying the rate of the oxidation of the oil (Yunos *et al.* 2017). Like FFA, DOBI values are highly affected by the degree of ripeness, quality of fruitlet, processing extreme conditions, storage time, temperatures, aeration, and contamination (Jusoh *et al.* 2013). Such classification of DOBI (< 1: Bad, 1 to 2: Poor, 2 to 3: Average and > 4: Good) was suggested by Yunos *et al.* (2017). In the current case, only residual oil that obtained during 30, 60, and 90 °C were considered as average, while the rest were poor. Henceforth, only temperatures of 30 and 60 °C were selected to be studied further in order to maintain a good quality of residual oil.

Based on the results presented in Fig. 4, more than 50% of oil removal from OPEFB was recorded at the first 120 s at all conditions. The highest (84.9%) and the lowest (58.1%) oil removal percentage during the first 120 s were observed at both 60 °C and 8963 kPa and 30 °C and 3447 kPa, respectively. As mentioned earlier, this study was a continuity study from an earlier study (Yunos *et al.* 2017), where in this study the authors designed and fabricated HPWSS to remove and recover residual oil from OPEFB continuously. A slight lower oil removal value from this study (84.9% at 60 ℃) was observed as compared with Yunos *et al.* (2017) (94.4% at 150 ℃). However, this study offers an automated technology that can hold and rotate the OPEFB bunches during the cleaning, while the manual approach was used by Yunos *et al.* (2017). Additionally, an elevated temperature used by Yunos *et al.* (2017) may lead to the higher energy requirement and might contribute to higher operating cost.

One can see that there was a linear correlation between the temperature and pressure with the oil removal (Fig. 4). As suggested by Tunio *et al.* (2011), the heat transfer between the hot water to the oil may spread the oil molecules and lead to the decrement of the surface tension and hence increase the oil solubility. Water at critical conditions (high temperature and high pressure) is able to dissolve nearby hydrophobic compounds by receding the electrostatic bonding between the water molecules and any nearby ion molecules (Plaza and Turner 2015). In another study by Gomez *et al.* (2014), about 83%

of oil removal from OPEFB spikelet cuttings were recorded for a proposed process called hydro solvent-assisted steam extraction (HYASES). The rate of oil removal from OPEFB was very little after 180 s. This may indicate the almost completion of oil removal from OPEFB by HPWSS. Overall, an improvement of 37% of oil removal rate was obtained using the integrated HPWSS system as compared with the non-integrated manual system.

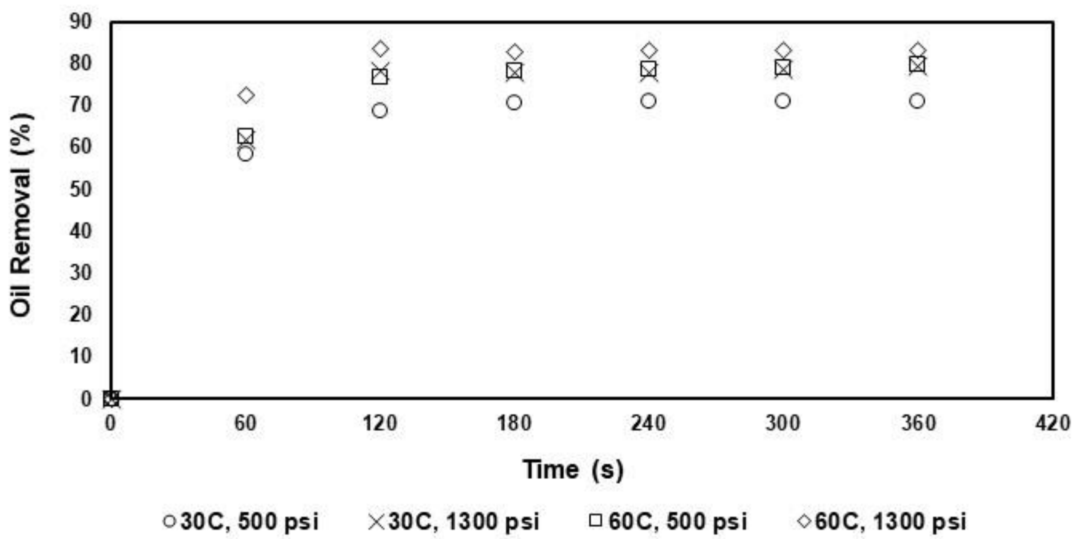

Fig. 4. Oil removal from OPEFB using the fabricated HPWSS at different temperatures and pressures. All data are the mean of three experiments

The FTIR analysis was performed to discover the changes of any functional groups in HPWSS treated fibers. The FTIR spectra (Fig. 5) show the differences of both untreated and HPWSS treated fibers. Obvious broad absorbance bands were observed for both samples at 3500 to 3300 cm^{-1} indicate O-H groups stretching between cellulose and water while peaks occurred at 2800 to 2900 cm^{-1} attribute to the typical saturated aliphatic C-H stretching (Xiang *et al.* 2016). The bands from 1800 to 600 cm^{-1} usually are attributed to lignocellulosic constituents such as cellulose, hemicellulose, and lignin.

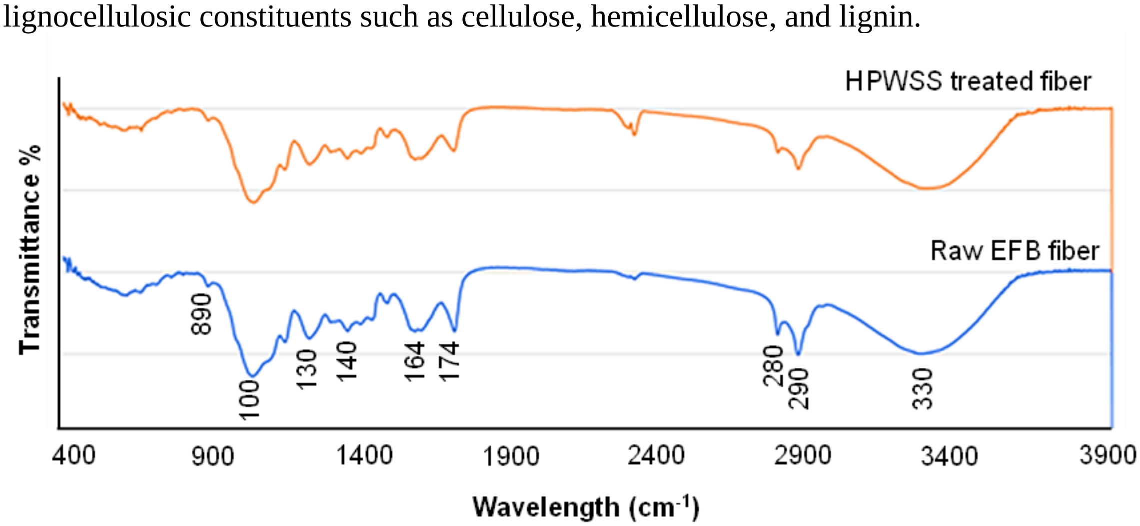

Fig. 5. FTIR spectra of untreated (blue) and HPWSS treated (orange) OPEFB

These bands were also reported previously in other lignocellulosic samples: 1715 to 1740 cm^{-1} (C=O unsaturated ester stretch and C=O stretching aldehydes); 1610 to 1640 cm^{-1} (C=C stretching of phenyl and aromatic monomers); 1420 to 1400 cm^{-1} (C-H and O-H plane deformation vibrations); 1300 to 1200 cm^{-1} (C=O stretch in primary alcohols); and 897 to 890 cm^{-1} (C-H deformation in cellulose) (Ishola *et al.* 2014; Xiang *et al.* 2016; Latip *et al.* 2019; Nandiyanto *et al.* 2019). From the data here, it can be said that the harsh HPWSS treatment could remove the majority of the residual oil from OPEFB, while preserving the treasured lignocellulosic content, suggesting that the HPWSS treated fiber can be used for any bioconversion process.

Residual Oil Recovery by Water Dilution Method

Residual oil was collected with the effluent collector (Figs. 2a and 2b) and was skimmed and centrifuged to remove impurities and dirt. Oil recovery was performed using simple techniques on the grounds that there are restricted workspace areas in the mill. Hence, in this experiment the authors explored the potential to recover oil by water and shaking methods at different temperatures. As shown in Fig. 6a, oil recovery increased with the temperature increment. It can be seen that maximum oil recovery was achieved within 30 min at all temperatures where the highest oil recovery (29.7%) was achieved during 95 °C and the lowest was oil recovered was achieved at 65 °C (6.7%). Similar observations have been reported elsewhere (Gomez *et al.* 2014; Yunos *et al.* 2017). Residual oil is a mixture of oil and water, which has been obtained from the HPWSS treatment previously. At elevated temperature, electrostatic interactions between hydrophobic water molecules and oil will be lessened, causing the oil to be less viscous and more soluble, thus making the oil recovery easier (Plaza and Turner 2015). As the temperature increased, the oil and water molecules vibrate faster, increasing the Brownian motion and promoting heat transfer, causing the coalescence by increasing momentum between oil droplets (Hajivand and Vaziri 2015).

Figure 6b depicts the effect of shaking power to recover residual oil from HPWSS. The experiment was conducted using water bath that used linear shaking motion with a temperature controller. Oil recovery percentage increased with the shaking power. It is noticeable that all the maximum oil recovery were achieved within 30 min of experimentation. The highest oil recovery (29.8%) was achieved during shaking power 5 while the lowest oil recovery (7.6%) was achieved during shaking power 1. The plateau trends of oil recovery beyond 30 min indicated the completion of oil recovery from the HPWSS residual oil. As suggested by Motin (2015), shaking involves the lateral movement that can cause resultant inertia force and inertia moment, hence droplet coalescence may occur when the droplets are subjected to shear forces. The higher the shaking power, the more vigorous the lateral movement, hence the more pronounced the hydrodynamic effect and shear stress of the liquid (Azmi and Yunos 2014).

The effect of dilution of residual oil from HPWSS with water on oil recovery is illustrated in Fig. 6c. The experiment was conducted at 95 °C using shaking power 5. The maximum oil recovery (58.8%) was obtained by water dilution 90% after 1 h of experiment. The higher dilution was able to recover more oil where all the oil recovery was completed within 30 min of the experiment as evident in Fig. 6c. The high dilution (90%) was able to recover almost 20% more oil as compared to the low dilution (40%). The separation of oil and water through dilution has been performed in most refinery mills where the addition of water causes a barrier that is able to sediment all the fine particles, resulting in small floating oil droplets to re-surface (Abd Manaf and Chung 2018). To the authors' knowledge, there are no studies on using the water dilution to separate palm oil. Hence, it is a great opportunity to understand and explore more of its fundamentals and principles.

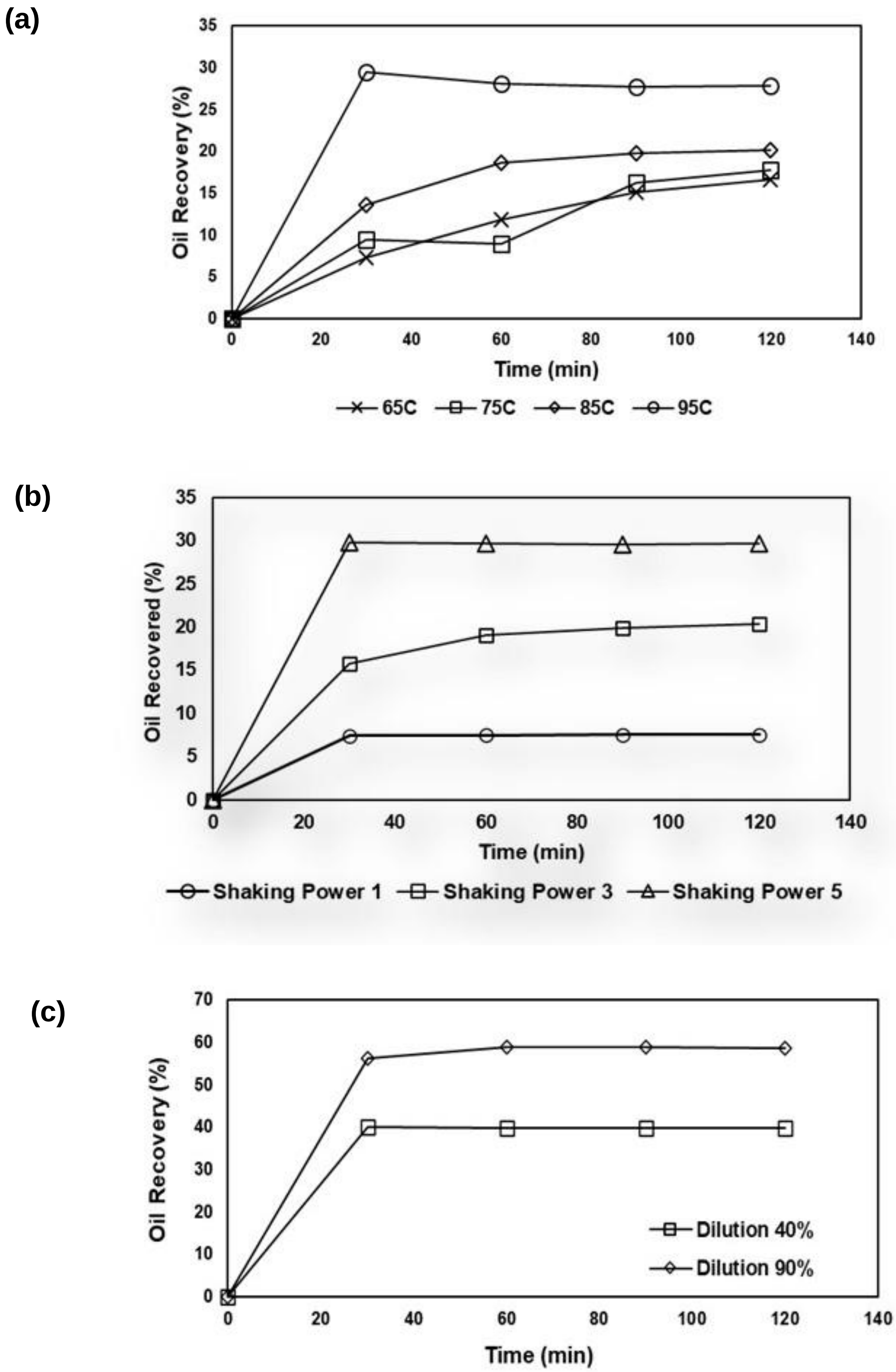

Fig. 6. Recovery of residual oil from OPEFB at different temperature, shaking power, and water dilution. All data are mean of three experiments

The functional group changes of the recovered oil from HPWSS were analysed using FTIR spectra (4000 to 400 cm^{-1}) (Fig. 7). Generally, all the absorption peaks were almost similar for both oil samples. Peaks at 3008 cm^{-1} can be attributed to the cis double bond of unsaturated fatty acids, while both 2840 cm^{-1} and 2920 cm^{-1} refer to the stretching of C-H from fatty acids hydrocarbon chains. The presence of fatty acids methylene groups in residual oil is evident from the aforementioned peaks (Yunos *et al.* 2017). The peaks that occurred at 1793 cm^{-1} indicate the stretching of C=O of carbonyl functional groups in the triglycerised fats (Hashim *et al.* 2017), while the broad peaks at 1450 cm^{-1} correlate to the bending stretching vibrations of CH_2 and CH_3 in aliphatic groups. The small peak at 711 cm^{-1} is associated with the overlapping vibrations in aliphatic groups and cis-disubstituted olefins (Yunos *et al.* 2017). From the FTIR spectra, it is safe to assume that both residual oil recovered by HPWSS was chemically comparable with normal crude palm oil.

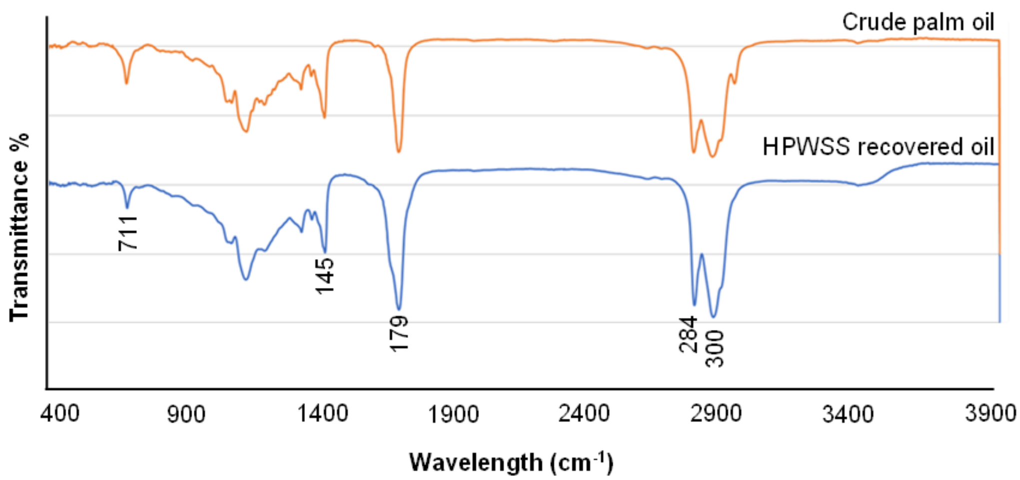

Fig. 7. FTIR spectra of recovered oil from HPWSS (blue) and crude palm oil (orange)

Residual Oil for Biodiesel

Advanced biodiesel production from agricultural and crop residues has shown tremendous increasing trends globally (Aghbashlo *et al*. 2021), while various strategies are adopted to actualize the dream. In this study, the low quality of the oil recovered from the HPWSS process for edible oil seems promising to be utilized as a feedstock for biodiesel production. Preliminary data reported by Yunos *et al.* (2017) stated that FAME-base bioprospection profile of oil recovered from empty fruit bunches showed a cetane number (CN) at 60.55, which was comparable to crude palm oil (60.48). Higher CN values are accompanied with better cold start, shorter ignition time, and less formation of white smoke. Figure 8a shows the effect of biodiesel percentage on the brake torque of the compression ignition (CI) engine at various engine speed ranging from 2000 to 3400 rpm. The maximum brake torque of 10.89 Nm for diesel was obtained at 3000 rpm speed whereas biodiesel blend of 5%, 10%, and 15% shows brake torque of 9.87, 9.57, and 9.56 Nm, respectively, with the engine speed of 2600 Nm. The increase in brake torque was due to proper atomization and high combustion efficiency concerning the decrease in viscosity and density of the fuel particularly at lower biodiesel blend (Tat *et al.* 2021). Meanwhile, the minimum brake torque was 9.05 and 9.15 Nm at 2000 rpm engine speed for biodiesel blends of 5% and 10%, respectively. The value of brake torques decrease slightly along with the increase in engine speed and amount of biodiesel in the fuel blend. Izani *et al.*

(2011) reported on the decrement of heat content in the fuel with high percentage of biodiesel. However, high oxygen content with high lubricity properties of biodiesel results in the reduction of friction loss and leads to improved brake torque efficiency.

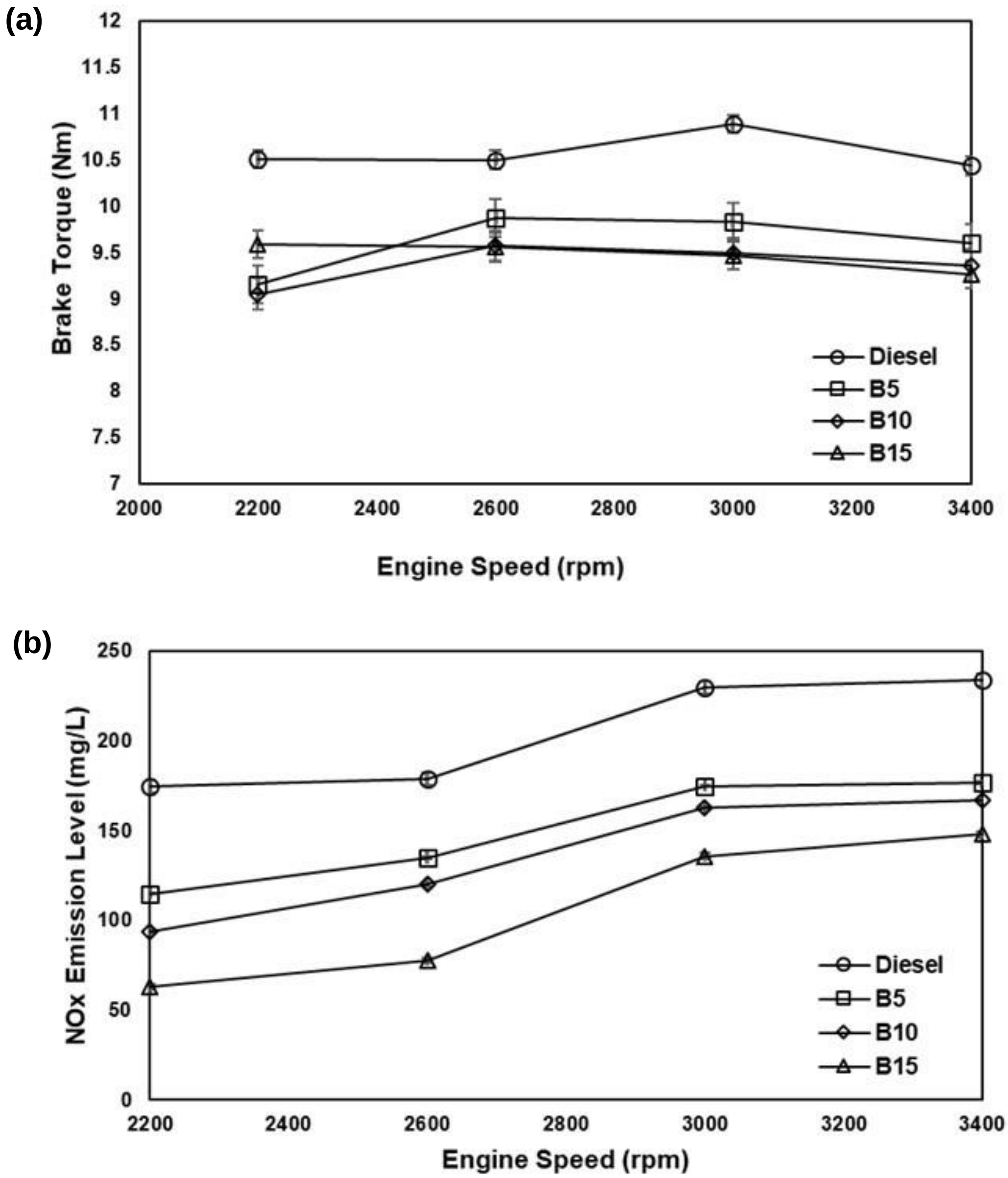

Fig. 8. Graph of (a) variation of brake torque with engine speed and (b) NOx emission for various biodiesel blend

Biodiesel fuel has a potential to enhance the performance of CI engine; however, the emission of carbon dioxide (CO_2) and nitrogen oxide (NO_x) is reported to increase. Nitrogen oxide is generated in a very high combustion temperature (Karpagarajan *et al.* 2021). The profile of NO_x emission with different biodiesel blends and engine speeds is shown in Fig. 8b. The change trend shows the elevation emission of NO_x concentration (235 mg/L) was higher using commercial diesel at 3400 rpm engine speed. In comparison, there was slightly lower emission of NOx for the fuel with biodiesel blend range from 148 to 177 mg/L at the same engine speed. An increase in the CI engine power for B5 biodiesel blend was accompanied with the high NO_x emission. However, it was shown that the high biodiesel blend (B15) led to the reduction of NO_x emission by 16% as compared to B5 biodiesel blend and up to 36.7% in comparison with diesel. Commercial diesel with higher combustion temperature increases flame temperature and emits more NO_x (Ghanbari *et al.* 2021) as compared to the low heat content of fuels with biodiesel blends.

In-depth studies of the biodiesel production from residual oil of OPEFB should have been performed in future research. Further explorations on the detailed composition of biodiesel produced from this residual oil can be carried out in order to determine its quality and suitability. Parameters such as cetane number, heating value, cloud point, flash point, acid and iodine value, density, viscosity, and oxidation stability are crucial to know, as they will be affected by origins of feedstocks and will affect the refining process (Aghbashlo *et al*. 2021). Another critical issue that can be study is the sustainability of the biodiesel produced from HPWSS. The biodiesel sustainability and its impact on the planet and human health can be investigated through life cycle assessment (LCA), as well as energy and exergy analysis. Such an approach is preferable, as it offers a cradle-to-grave approach by assessing and evaluating all materials and energy used during the process and taking into account all the wastes or gases emitted as well (Rosen 2018). Although HPWSS has shown some promising potential, location of an HPWSS to be in the oil palm mill and have accessible to OPEFB is preferable in order to minimize additional costing for OPEFB transportation. The open-system of HPWSS can be improvised in the future with a closed one in favor for entrapment of heat from the hot water and steam for HPWSS usage. In pursuance of environmental sustainability, emission of heat and gases can be avoided and HPWSS can reduce its dependency to oil palm mill for its hot water and electricity.

CONCLUSIONS

1. High pressure water spray system showed a promising usage in the oil palm industry, since it was able to remove 84.9% of residual oil from OPEFB at 60 °C and 8960 kPa with acceptable range values of DOBI, FFA percentage, and PV.
2. The water shaking method has shown that it is able to recover residual oil from OPEFB as evidenced from the experimental data, where 58.8% of residual oil was recovered at 95 °C using 90% water dilution and maximum power shaking.
3. The ability of recovered residual oil to be used as biodiesel was observed where a few biodiesel blends have lower brake torque values and lower NOx emission as compared to diesel.

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