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Oil Water Separation: Inauguration of Cellulose and Chemically Modified Cellulose-rGO via Graphene Oxide Functionalization

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Abstract:

The expenditure on water remediation is the constant main rationalization. However, the adoption of our lignocellulosic biomass at the highest level might provide a solution. Therefore, our main objective is to enhance the surface area of cellulose on the surface of empty oil palm fruit bunch fibers via a functionalization of graphene oxide (GO) and lamination of GO on the surface of EFB cellulose (EFBC) occurred by the intermolecular hydrogen bonding through a thermal treatment of reduced-graphene oxide. The hydrophobicity of the functionalized cellulose (EFBC_rGO) was improvised by a single layer grafting of graphene in the selectively separate oil for water remediation. The optimization of EFBC_rGO on the selectivity of oil uptake was carried out in different temperature and oil, kinetic sorption and chemical analysis. The modified EFBC_rGO showed distinct morphological and chemical characteristic changes as the surface of cellulose had been coated with rGO. This was supported by the FTIR analysis that showed a diminishing peak of hydroxyl group region of EFBC and that chemical modification has improved the hydrophobicity of the EFBC_rGO. In the contact angle measurement, the EFBC_rGO showed better hydrophobicity compared to EFBC. In the oil uptake study, increasing the temperature up to 80oC has increased the oil uptake up to 94% by EFBC_rGO at 9:1 water-oil ratio. Meanwhile, the kinetic sorption revealed that 60 min treatment was the maximum time for oil sorption.

Keywords: *Absorption, EFB, Kinetics, Water*

I. INTRODUCTION

"Malaysia is not able to achieve Developed nation status if the environment is left to rot," highlighted our Prime Minister, Dr. Mahathir Mohamad, on 20th April 2019. Although the statement was key during the Earth Day celebration, the incident at Sungai Kim Kim might be the trigger for the government to seriously tackle the water remediation issues, especially water pollution coming from oil and hazardous industry. The oil and hydrocarbon content in water pollution have become a crucial problem and captivated worldwide attention. Hence, a well-planned action should be taken to prevent and reduce the effect of this water pollution. One of the main purposes for cleaning water

contamination is the oil spills or oily wastewaters which contain petroleum and its products, including crude oil and its derivatives, such as gasoline, kerosene, diesel and heavier fuels [1]. Recently, many researches have a fascination towards the utilization of natural fibers for remediating polluted waters [2].

The advancement of unique materials with special wettability properties has increased in material research [3]-[5]. This type of materials possesses opposite affinities towards oil and water, providing an alternative method to select hydrocarbon in

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oil-water separation. Specifically, hydrophobic and oleophilic materials are suitable for oil-water separation [6]. The method to achieve this property is by modifying its surface chemistry to boost the surface functionality [3],[6]. Consequently, this material will effectively and selectively remove oil-water mixture, indicating the synergistic effect between surface chemistry and structure [3],[5],[9].

In the approaches of using adsorbent as oil removal, most of them (silica, clay, polymers, cellulose-based materials, etc.) was modified to enhance its oleophilicity [7]-[8]. Essentially, a rough structure on the surface material must be constructed to form hydrophobic materials, followed by chemically modifying it with low surface energy on the surface of the materials [5]. In the recent year, graphene oxide (GO) has been widely used for various applications due to its excellent properties (electronic, thermal, optical and mechanical). Moreover, GO shares one bond with a non-carbon atom/molecule, and consists of a large number of functional groups: hydroxyl, epoxy, carbonyl and carboxyl. It is highly reactive and nominated as active sites for oxidation. These factors help GO to be used in nanocomposite materials which are good for chemical modification and adsorption capacity [9]-[10]. Several studies shown by using GO as grafted composite have enhance the properties of the materials (adsorption, etc.) [11]-[14]. Besides, reduced GO present highly hydrophobic properties on the modified materials [4], [15]-[16].

Malaysia, the second largest producer of crude palm oil after Indonesia, produced 19.6 million tons of crude oil from 5.3 million ha of its plantations in 2014 [17]-[19]. Considering the large production of palm oil, there is about 3.0 million tons of empty oil palm fruit bunch (EFB) fibers produced annually [18]. Despite the abundance of EFB fibers, as they serve as a good resource for lignocellulosic material, the fibers are still underutilized. The utilization of EFB in the industry is limited, and mostly are used as a palm oil mill by-product such solid fuel in boilers and organic fertilizers [20].

Selectivity of petroleum hydrocarbon from aquatic system towards adsorbent can be achieved by transforming the hydrophilicity of natural adsorbent into hydrophobic features. In this study, EFB cellulose consist of large numbers of hydrophilic functional groups (hydroxyl, carboxyl, etc.) and enhancing surface area of the adsorbent is necessary. Natural cellulose obtained from extraction process of oil palm empty fruit bunch (EFB). The utilization of the surface porosity of cellulose is necessary to modify its surface with the interaction of graphene oxide via coating the hydrogen bonding on the surface of cellulose (EFBC). Augmentation of reduced-graphene oxide (rGO) via thermal heating has enhanced the surface hydrophobicity of the cellulose (EFBC_rGO). The selectivity of oil-water was investigated by series of oil and water absorption through optimization of parameters including temperature, ratio of oil-water, kinetic study and contact angle.

II. MATERIALS AND METHOD

A. Materials

Empty oil palm fruit bunch (EFB) fibers were obtained from the Malaysian Agricultural Research and Development Institute (MARDI). Used engine oil was collected at the local automobile workshop nearby Bangi, Selangor. Graphene oxide (GO) was prepared by using graphite flakes (Ashbury, Inc. USA), phosphoric acid (85%) (Merck), potassium permanganate (99.9%) (Merck) and hydrogen peroxide (30%) (Merck).

B. Isolation of Cellulose

Bleaching process of empty fruit bunch (EFB) was conducted using four stages of bleaching sequences (D_{Na} E E D_{Na}) where DNa is bleaching process and E is alkaline treatment where both processes were used to remove the lignin and hemicelluloses. During the D_{Na} step, buffer solution was prepared which consist of 27 g of NaOH, 75 ml of acetic acid and distilled water and 1.7 wt% of sodium chlorite solution was prepared. The ratio of buffer solution:sodium chlorite solution:distilled water is 1:1:1 used in D_{Na} step at 80 ^{o}C for 2 h in water bath. For E step, 2 % of NaOH was used at 80 ^{o}C for 2 h in water bath and the ratio of EFB to the solution is 1:20 for each step [21]-[22]. After every single stage was performed, the sample was washed until neutral using phenolphthalein to remove the bleaching chemicals and dissolved lignin from the sample prior to entering the next stage. Then, the sample was dried at 105 ^{o}C for 24 h.

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C. Preparation of Graphene Oxide

Graphene oxide (GO) was fabricated following modified Hummer's method. The simple one-pot oxidation process without the tedious step of temperature control and mixing was carried out. Briefly, graphite flakes (3 g) were added into a 9:1 H_2SO_4:H_3PO_4 (360:40 mL) solution. Potassium permanganate was gradually added (18 g). The oxidation process was stopped by adding ice (~400 mL) and 30 wt% H_2O_2 (27 mL). Subsequently, the GO produced was washed 20 times until a stable pH (~4-5) was achieved via centrifugation and deionized water to remove the excess chemical [12]. For the EFBC-GO modification, the EFBC was deposited on GO aqueous solution (0.5 - 1.5 mg/L) and stirred for 1h. Immediately, the EFBC-GO was dried in an oven (180 - 300 ºC) for 4h to reduce the GO and form reduced graphene oxide (rGO). The rGO coating serves as the hydrophobic layer where it can absorb and repel water at the same time. Fig. 1 shows the mechanism on the oil water selectivity of cellulose and inauguration of EFBC-rGO which has proven in increasing the hydrophobicity/oleophilicity of EFBC after being chemically modified.

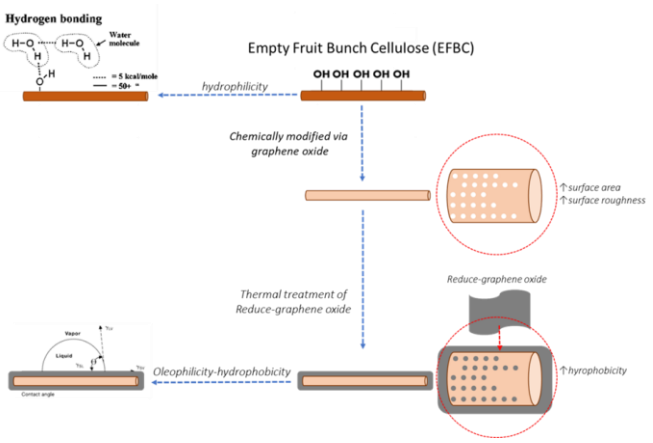

Figure 1: Mechanism of oil-water selectivity on cellulose

D. Oil-water Selectivity Study

For the oil-water separation, the oil selectivity was measured by the amount of oil absorbed by the EFBC_rGO at different oil to water ratio, temperature, and time. Briefly, 10g of oil-water solution was prepared by mixing oil with distilled water at the ratio of 1:9. The mixture was stirred by using magnetic stirrer at 200 rpm for 10 minutes in room temperature (30°C). Then, 0.01g of EFBC_rGO sample was dropped into the mixture and stirred for 10 minutes at 200 rpm. The sample was drawn out from the mixture and dried at 105°C for 24h to remove water. The dried sample was then re-measured and the percentage of weight gain (%) was calculated by using Equation 1. The experiment was repeated for the oil-water ratios of 1:9, 2:8, 3:7, 4:6 and 5:5.

$$\text{Weight gain (\%)} = \frac{(\text{Final weight } (W_f) - \text{Initial weight } (W_i))}{(\text{Initial Weight } (W_i))} \times 100\% \quad (1)$$

Where W_i is the initial weight and W_f is the final weight of the absorbed sample.

The effect of temperature on the adsorption capacity was studied by preparing the oil-water mixture at 1:9 ratio and the mixture was stirred for 10 minutes by using magnetic stirrer. The EFBC_rGO sample was placed into the mixture when the temperature of the mixture reached 30 ºC and the temperature was maintained for 30 min to allow the adsorption process. Modified EFBC_rGO underwent adsorption kinetics study of oil in polluted water. The absorption capacity of hydrophobic EFBC_rGO was investigated via several batch tests. The experiment was repeated whereby the temperature and the total oil content were monitored by a kinetic study for several contact time intervals (10, 20,30, 40, 50 and 60 min). All the adsorbed samples were removed from the mixture and dried at 105°C for 24h. The dried sample was then remeasured and the percentage of weight gain (%) was calculated by using Equation 1.

E. Contact Angle Measurement

The contact angle of the oil on EFBC and EFBC_rGO was measured by the Sessile Drop Method for the wettability and hydrophobicity evaluation. Figure 2 shows an image of the oil droplet on the samples taken by a camera. The contact angle of the oil droplet is the angle between a horizontal line and tangential line of the droplet at a contact point of an edge of the oil droplet [23],[24]. In this study, θ/2 method was used to analyse the profile of a sessile drop and the analysis of liquid drop is assumed to be part of a shape. Scientifically, the contact angle can be calculated by measuring the diameter of the drop, *d* and the height of the apex, *h*

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(Figure 8(b)) and the equation used to measure the value of θ/2 is represented by Equation 2 [23]:

$$\theta/2 = \tan^{-1} (h/d) \quad (2)$$

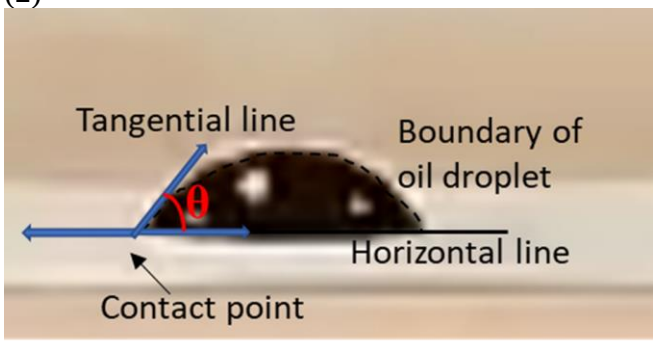

Figure 2: Oil droplet image for contact angle measurement

F. Characterization

The analysis was conducted on the formation of EFBC_rGO from EFB and oil-water selectivity. The increment of the porosity (surface area) and surface roughness of EFBC was observed by morphological analysis using field emission scanning electron microscope, FESEM (Merlin Compact, Zeiss Pvt. Ltd.). The morphological changes were observed by Attenuated Total Reflectance Fourier transform infrared spectroscopy (ATR-FTIR) (ALPHA FTIR Spectrometer, Bruker). The characterization for the functional groups of the samples was performed in the resolution of 1 cm^{-1} in the wavenumber range of 4000 to 650 cm^{-1}. In order to determine the surface hydrophobicity of chemically modified EFBC_rGO, physical analysis of the surface wettability of EFBC_rGO was measured by contact angle system via sessile drop technique.

III. RESULTS AND DISCUSSION

A. Characterization of Modified EFB cellulose

Morphological analysis

The morphological structure of EFBC fibers observed by FESEM showed single fiber and compact structure (Figure 3(a)). Furthermore, the rGO coated on the surface of the EFBC (Figure 3(b)) showed more porous structure of the EFBC_rGO. This may be due to the addition and interaction of GO with cellulose, creating a more porous structure of the cellulose-GO network during the surface modification process [4].

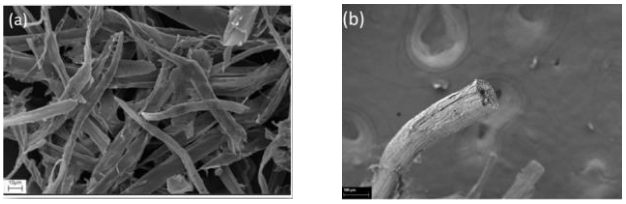

Figure 3: FESEM morphological image of a) EFBC and b) EFBC-rGO

Functional group analysis

The changes in the functional group for engine oil, EFBC, EFBC-absorbed engine oil, EFBC_rGO and EFBC_rGO-absorbed engine oil are shown in Figure 4. In the FTIR spectrum of engine oil, a sharp peak at 2929 cm^{-1} and 2854 cm^{-1} was assigned to the stretching vibration of CH_2 group respectively, indicating a —CH_2 structure within the molecules in engine oil. The band at the range of 1310 and 1380 cm^{-1} was attributed to H-O-H bending mode due to the absorption peak of hydroxyl group in carboxylic acid, indicating that the engine oil was oxidized and acidized. The oxidation reactions took place during operating, leading to the generation of similar compounds in the used oil [25]. EFBC showed a broad peak between 3100-3600 cm^{-1} corresponding to the stretching and bending vibration of OH groups of water molecules [20]. However, the broad absorption peak of the hydroxyl groups of EFBC diminished after being adsorbed in engine oil (EFBC-engine oil) while the existence of a peak at 2929 and 2854 cm^{-1} of EFBC-engine oil proved that EFBC successfully absorbed the engine oil. Considering that the hygroscopy of the EFB cellulose was mainly bound by the hydroxyl groups in the lignocellulosic fibers, the reduction of the O-H bond of the hydroxyl group would promote the hydrophobicity of the treated cellulose [10],[20]. The inexistence of an alkane group in EFBC and the existence of alkene in EFBC-engine oil showed that the alkane group has been attached to the EFBC during the oil absorption process [26].

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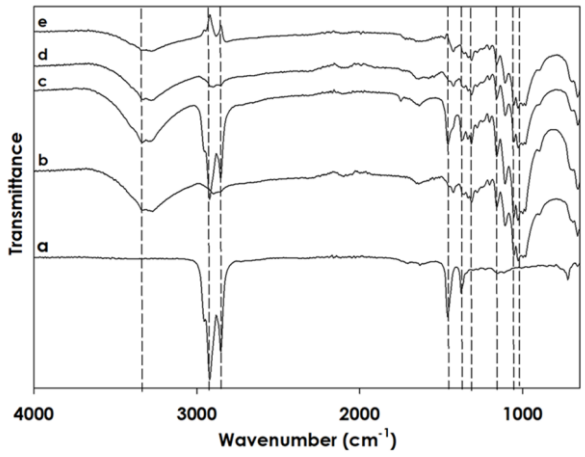

Figure 4: FTIR Spectrum for a) engine oil, b) EFBC, c) EFBC-engine oil, d) EFBC-rG and e) EFBC_rGO-engine oil

B. Oil-water Selectivity Study

Effect of oil-water ratio

In the oil-water separation study, the percentage of oil absorption was carried out at different oil to water ratio: 1:9, 2:8, 3:7, 4:6 and 5:5. As a result, the increment of oil in the water content has enhanced the oil uptake by the EFBC_rGO up to 107.6% of oil absorption at 5:5 oil to water ratio (Figure 5). Theoretically, the addition of oil in water provided more surface area of the EFBC_rGO to uptake and retain the oil absorbed [4]. Moreover, increasing oil concentration (2 to 10 wt%) in oil-water emulsion showed an immense incline in oil uptake with an increment from 38.5% at 1:9 of oil to water ratio to 107.6 % at 5:5 of oil to water ratio.

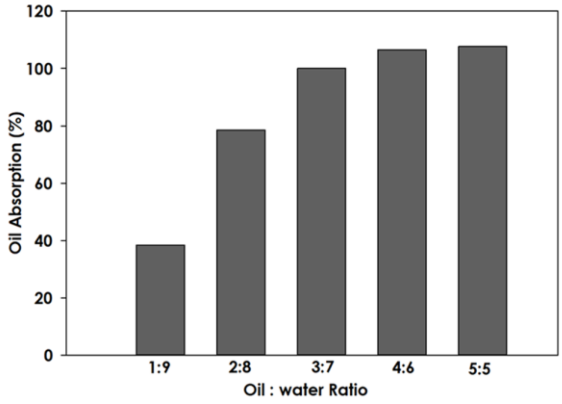

Figure 5: Percentage of oil absorption at different oil:water ratio

Effect of temperature

The reaction temperature plays a significant role in oil absorption. Increasing the absorption temperature significantly increases the percentage of oil absorbed by EFBC_rGO from room temperature (30 ^{o}C) up to 80 ^{o}C as shown in Figure 6. At room temperature, the percentage of oil absorption obtained was 67.1%; it gradually increased up to 79.2% at 40oC of absorption temperature. Increasing the temperature from 40 to 60 and 80 ^{o}C led to an increment in the percentage of oil absorption consistently by 7-8% up to 94.7 % at 80 ^{o}C. The phenomena happened because of the swelling capacity of the cellulose and oil which was benign when it was exposed to high temperature. Therefore, the breakage of hydrogen bonding by the hydroxyl group in the EFBC_rGO samples resulted to the diffusion of oil into the samples. Eventually, it enhanced the absorption rate and capacity, improving the hydrophobic properties of EFBC_rGO [27]. The theory of collision also stated that when a reaction temperature increases, the kinetic energy of the oil particles also increases. Thus, the oil particles will collide more frequently and break down, making them easier to be absorbed. Therefore, the rate of oil absorption increased.

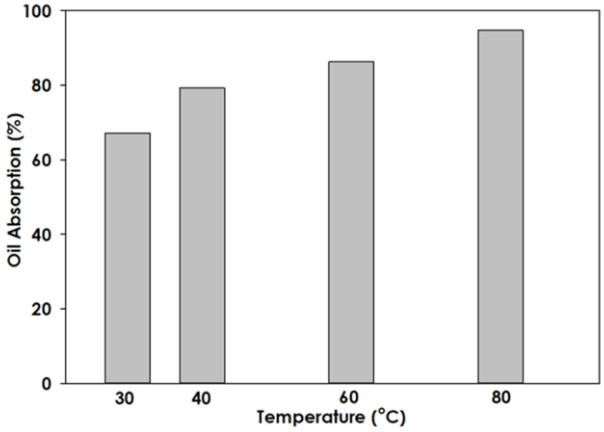

Figure 6: Percentage of oil absorption at different absorption temperature

Kinetics study

Figure 7 shows the kinetic study of the percentage of oil adsorption by EFBC_rGO at different contact time. Similar to the effect of temperature, the pattern of the oil adsorption by EFBC_rGO increased by time. At the first 10 min of the reaction time, the oil adsorption was 105% and it increased by 30 to 40% by each 10 min increment. This is due to the favorable effect of time on diffusion and sorption of reactants. However, at 50 min of reaction time, the oil adsorption achieved equilibrium where increasing the reaction time had no effect on the oil adsorption.

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The engine oil has fully diffused on the surface of EFBC_rGO and the optimum oil adsorption of EFBC_rGO in the engine oil was 230% at 60 min of reaction time.

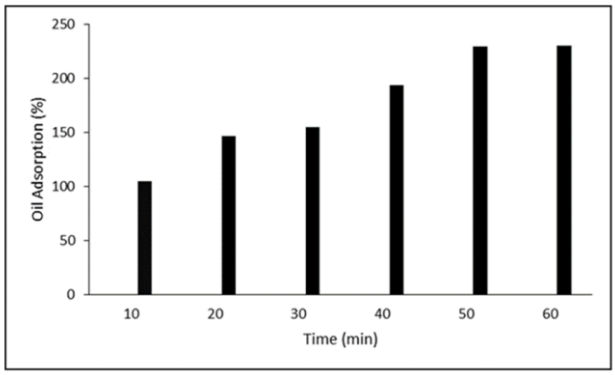

Figure 7: Kinetics study on the percentage of oil adsorption

C. Contact angle measurement

Figure 8 shows the relationship between the contact angle of engine oil when a droplet of engine oil was dropped on the surface of EFBC and EFBC_rGO. As shown in Figure 8(a) and Figure 8(b), the engine oil was dropped on EFBC and EFBC_rGO; the contact time between the surface of the EFBC and EFBC_rGO and the droplet was immediately observed on its contact angle [23],[28]. At the beginning of the dropping process, the droplet shape was spherical and immediately changed to hemi-spherical on the EFBC and EFBC_rGO. Consequently, the droplet slightly disseminated and reached equilibrium (stabilized) after 10 seconds of a collision (contact time). The contact angle observed between the tangential line and horizontal line was the highest when the droplet shape was spherical and decreased as the time increased until the shape became semi-sphere and finally flattened (0°) after 10 s of contact time. When the droplet of engine oil was dropped onto the EFBC_rGO, the droplet took a longer time to turn from sphere to flattened at 0° proving that the surface of EFBC_rGO had better hydrophobicity compared to the surface of EFBC. Then, it slightly spread and reached equilibrium after 10 s from the collision. It was found that the contact angle decreased at the instant of the collision with the plate and became stable 10 s after the collision [24].

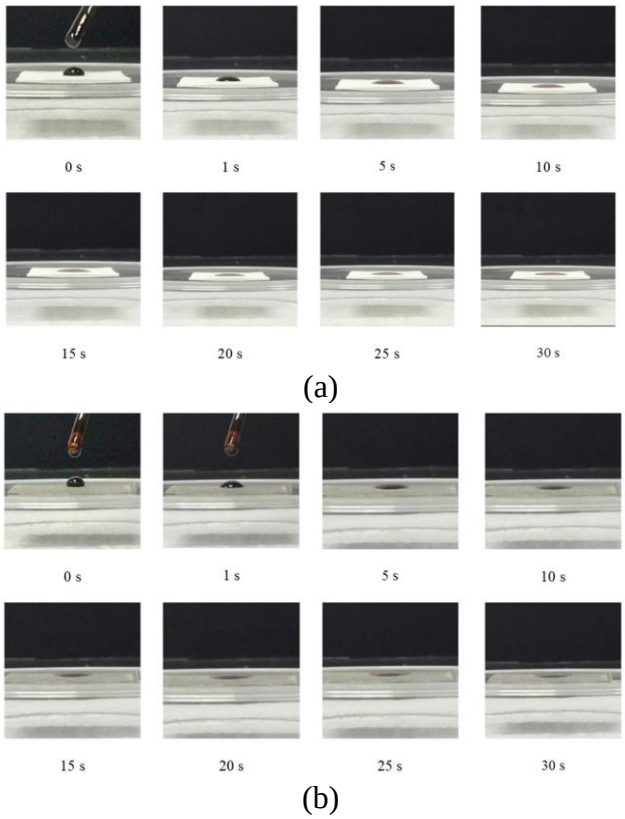

Figure 8: Droplet of engine oil on a) EFBC and b) EFBC_rGO for contact angle measurement.

Exquisitely, the liquid drop shape hinge on the execution of both interfacial and gravitational forces. Surface tension tends influence on minimization of the surface area enforcing the drop into spherical shape. Meanwhile gravitational force distorts the drop in two steps, i.e; by elongating a pendant drop and/or flattening a sessile drop. Therefore, by implementation of θ/2 method in measuring contact angle as can be seen from Table 1, the value of the contact angle decreasing as the time of dropping increasing for both EFBC and EFBC-rGO. In comparison of angle dropping percentage, at 1 second of dropping, the contact angle has decreased by 52.6 for EFBC, while EFBC_rGO decreased 33.0 % at the same dropping time. By increasing dropping time, the value of angle dropping percentage starts achieve equilibrium at 25 s of dropping time for both EFBC and EFBC_rGO up to 88.5 % and 84.9 %, respectively. This proof the relationship between dropping time increase linearly with contact angle value and dropping angle percentage summarized in Table 1 and the Figure 8 . This also proof that EFBC_rGO perform better hydrophobilicy compared to EFBC.

Table 1: Contact angle value for EFBC and EFBC_rGO

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Sample	Dropping time (s)	Height of apex (cm)	Drop Diameter (cm)	θ/2 value (°)	θ value (°)
EFBC	0	2.14	2.49	40.7	81.4
	1	1.26	3.59	19.3	38.7
	5	0.98	3.99	13.8	38.7
	10	1.00	4.33	13.0	26.0
	15	0.73	4.02	10.3	20.5
	20	0.48	3.62	7.6	15.1
	25	0.33	3.82	4.9	9.9
	30	0.33	3.99	4.7	9.5
EFBC-rGO	0	2.63	1.87	54.6	109.2
	1	2.05	2.76	36.6	73.2
	5	0.85	4.03	11.9	23.8
	10	0.80	4.40	10.3	20.6
	15	0.80	4.40	10.3	20.6
	20	0.80	5.13	8.9	17.7
	25	0.80	5.49	8.3	16.6
	30	0.64	4.42	8.2	16.5

CONCLUSION

In this study, the modification of EFB cellulose with rGO showed oleophilicity/hydrophobicity property and better oil selectivity in the oil-water emulsion. However, since the composition of lignocellulosic was naturally hydrophilic, the additional hydrophobic rGO properties gave the complex mechanism of the oil uptake which was supported from the contact angle measurement. The utilization of EFB cellulose as an adsorbent in oil-water separation would help solve the water pollution by oil, especially for water security, expanding the chain of high value-added products and the green growth for sustainability in line with the key principles of the National Biomass Strategy 2020 and Sustainable Development Goals 2030 by United Nations

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