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Microwave process of oily sludge produced at NRC Baiji to micro-char solid carbon production

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ABSTRACT

This research aimed to purify the environment of undesired substances by converting oily sludge (OS) micro-char fuel generation by microwave. The North Refineries Company (NRC) Baiji was the source of the OS sample utilized in this study. Micro-char manufacture, which has improved solid carbon characteristics, uses various microwave power and irradiation times. The parameters conditions at a range of power: feed of 5 g, 10 min of irradiation time, and microwave power ranges of 200, 400, and 600 W, respectively, under a nitrogen pressure of 3 L/min. While at the range of irradiation time: the feed was 5 g, microwave power was fixed at 600 W (the best microwave power discovered), and the range of irradiation duration was 5, 10, and 15 min. The ideal circumstances were (5 g, 600 W, and 15 min, under N_2 pressure). The fuel ratio was increased from 0.70 to 0.97 OS with a 38.57% rise in the percentage of micro-char at 600 W and 15 min of irradiation time, while the HHV was increased from 9.40 to 17.22 MJ/kg at a rate of 83.20% as finding micro-char had improved as a solid carbon fuel. In general, micro-char has a fuel ratio and a higher HHV, making it suitable for solid carbon fuel in the oil industry.

Keywords: Oily sludge; Microwave process; Micro-char; Calorific value; Fuel ratio; North Refineries Company (NRC) Baiji

1. Introduction

Crude oil is a hydrocarbon complex that contains organic and inorganic materials [1]. Oily sludge (OS) is sludge that has been combined with heavy oils such as residual, refined, and crude oils [2]. OS is a combination of soil, water, and oil that may also contain contaminants from the operation of oilfields, refining, transportation, use, and storage. OS is a significant pollutant in the petroleum and petrochemical sectors and is hazardous to people, plants, and aquatic life [3]. As a result, efforts to reduce OS and safely handle it have long been a top focus in the carbon manufacturing industry [4]. As more people learn about the importance of protecting the environment, the risks of OS have become more well-known. If thrown carelessly, OS is solid waste threatening human health and the natural world [3]. OS is a substance made of water, unrefined oil, refined oil, dirt, and other pollutants produced by the petroleum industry throughout extraction, refinement, transportation, use, and storage [5]. On the one hand, maximum resource recovery based on environmentally sound oily sludge treatment is a promising long-term management potential path [6]. Oilfield firms produce a lot of OS during drilling and extraction activities. OS complicates drilling fluid, which reduces pump efficiency, increases water pressure, and increases expenses, making work more difficult [7]. The extraction, storage, transportation, refineries, end product distribution, and upstream, and downstream operations in the petroleum business produce a large quantity of OS [8]. In China, during the cleaning of oil

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storage tank bottoms, approximately a million tons of OS were released [9]. Up to three million tons of OS are produced in China annually [10]. One billion tons of OS have been manufactured worldwide, with 60 million tons generated annually [11]. The Ministry of Oil, North Refineries Company (NRC) Baiji of the Republic of Iraq produced approximately 3,000–3,500 m^3/y of OS [12]. Nearly 90% of the oily muck in Baiji is dumped in the Makhoul mountains—Baiji, which has harmed the environment [13]. The oily sludge generated by NRC Baiji has significant levels of organic chemicals and carbon concentrations, according to earlier studies [14]. Researchers and engineers have put much effort into increasing fuel performance and economy while minimizing size and expense [15]. If OS is not adequately managed, it endangers human health and damages the environment [16].

Unlike conventional heating methods, microwaves use high-frequency electromagnetic radiation. Its energy is transferred via space or a medium as an electromagnetic wave. It's the process through which a dielectric loss in an electromagnetic field causes a material to heat up everywhere. Compared to other traditional heating techniques, microwave heating provides several benefits, including lower energy consumption, greater efficiency, customizable operations, and clean heating connections [17]. The loss and transmission of energy in the magnetic wave give microwaves their "thermal effect" and "nonthermal effect", respectively; this can be used to identify the internal structural condition of the heated object [18]. Those who believe that the quick rise in reaction temperature is the only reason microwaves accelerate reactions support the "thermal effect". In addition, the reaction rate at high pressure and temperature was significantly increased. According to proponents of the "nonthermal effect", when microwaves selectively heat polar materials, the activation energy and pre-exponential factors are reduced. The polarity transition state can be stabilized, activation energy can be decreased, and reaction time may be accelerated by the electromagnetic field's electric field [19]. Microwaves can selectively pyrolyze macromolecular substances in oily sludge, such as colloids and asphaltenes, reduce toxic and harmful polymers in macromolecules, and enhance the state and properties of oil products. These capabilities allow for achieving the goals of resource utilization, reduction, and harmlessness of oily sludge [20]. On the other hand, microwave pyrolysis of oily sludge typically favors the heating medium in a microwave electromagnetic field environment. Due to their dielectric heating capabilities, microwaves may efficiently heat polar materials. Even though nonpolar molecules do not immediately heat up, the effect is poor [21].

In this study, the calorific value, fuel ratio, micro-char yield, and energy densification were used to calculate the energy of the fuel [22]. Because oily sludge is poisonous due to the hydrocarbons it contains, it poses a threat to both workers and the environment, making it a burden for the oil industry to deal with continually [7]. In the oil industry, oily sludges are often stored from numerous sources and in random, ad hoc warehouses exposed to the sun, making the installation of oily sludges more complex [23]. This study aims to manufacture solid carbon fuel micro-char using a microwave process and cleanse the environment of waste materials that pollute the environment; oily sludge was used as the undesirable product in this study. Varying microwave powers and different irradiation times were used. Consequently, micro-char was prepared to be used at NRC Baiji as a solid carbon fuel for electricity production.

2. Materials and methods

2.1. Oily sludge sample preparation

The tank number (3107-FA) residues from the bottom of the fuel oil tank were utilized as the raw material in this experiment to create the micro-char at NRC Baiji. At 105°C for 24 h, the sample was dried [24]. The material was then ground into a powder and sieved to get a homogenous sample. The raw material oily sludge sample's proximate, fuel ratio, final analysis, and calorific value are displayed in Table 1.

2.2. Micro-char production

Three samples of oily sludge were weighed at 5 g each after being sieved and dried at 105°C for 24 h to produce a homogeneous sample. First, three samples of oily sludge weighing 5 g each were adequately measured according to the range of microwave power and placed in crucibles. 5 g of OS, 10 min of irradiation, and the range of microwave power were initially 200, 400, and 600 W, respectively. Micro-200, Micro-400, and Micro-600 were the designations given to the prepared micro-char. On the other hand, three samples of oily sludge weighing 5 g each were irradiated for 5, 10, and 15 min, respectively, according to a range of irradiation times. The three types of micro-char were created. Each had the labels micro-5, micro-10, and micro-15. The microwave procedures were carried out in a microwave oven at a pressure of 3 L/min N_2. Calculations were made for the micro-solid char's yield and energy characteristics. A self-electric heating power source with a 1.0 kW power rating and a 220 V voltage rating was used to heat the microwave oven. The heating rate was adjusted to 15°C/min in the interim. As the irradiation period was specified, the microwave oven was closed and heated to a predefined power auto. To create an anaerobic environment, nitrogen was constantly pumped into the microwave oven beginning 20 min before the microwave procedure and continuing until it began. Hand-milled micro-char was sieved and stored for other tests.

2.3. Calculations

Volatile matter (VM) was determined by heating the sample to 950°C for 7 min. Ash, fixed carbon (FC), and the residue was weighed to estimate the weight difference. VM was then taken into consideration. It took 3 h and 950 °C to thoroughly melt 1 g of the material. The ash content was estimated, and the residue were weighed to determine the weight difference concerning ash. Assuming the total weight is 100%, the FC content was estimated by deducting the VM and ash contents [14]. The fuel ratio was calculated by dividing the FC by the VM [25]. The final study looked at CHNS-O components in accordance with (ASTM D-5672) and S following (ASTM D-1552). A 1 g sample was heated to 950°C for 3 h in a muffle furnace without air for the proximate analysis; the lowered weight represented the volatile matter content [26]. An ASTM E711-87 compliant Bomb calorimeter determined the high heating value. The micro-char upgraded properties by energy properties were investigated via Eqs. (1)–(3) [27].

$$\text{Solid yield} = \frac{\text{micro-char yield}}{\text{raw OS}} \quad (1)$$

$$\text{Fuel ratio} = \frac{\text{VM}}{\text{Fixed carbon}} \quad (2)$$

$$\text{Energy recover} = \frac{\text{HHV of micro-char}}{\text{HHV of the OS}} \quad (3)$$

3. Results and discussion

3.1. Micro-char yield percentage

The micro-char yield was investigated at various microwave power and exposure times. Solid yield products are displayed in Tables 3 and 4 and Figs. 3 and 4 at various microwave power and irradiation times, respectively. As demonstrated in Fig. 3, the micro-char production of OS at different microwave powers reduced weight. The solid yields were 4.20 g or 84.0% at 200 W, 4.28 g or 85.6% at 400 W, and 4.32 g or 86.4% at 600 W. The carbonization weight loss rates were 16.0%, 14.4%, and 13.6%, respectively, due to low to high microwave power (micro-200, micro-400, and micro-600). Similar to other investigations, this carbonization causes weight loss [28]. There are three main phases of mass loss during the OS microwave phase: moisture removal (200 W), low microwave power (200–400 W), and high microwave power (400–600 W) [29,30]. Micro-200 and micro-400 belonged to the low microwave power stage in this study. The evaporation and breakdown of petroleum hydrocarbons and the elimination of all water primarily caused mass loss. As the microwave power was increased from micro-400 to micro-600, the weight loss ratio increased to 31.6%.

As a result, Fig. 3 shows the micro-char yield of oily sludge at different irradiation time can reduce weight and volume from low to high irradiation time (micro-5, micro-10, and micro-15), the solid yields were 4.55 g, 91.0% at 5 min, 4.32 g, 86.4%, at 10 min, and 4.10 g, 82.0% at 15 min, the carbonization weight loss rates were 9.0%, 31.6%, and 18.0%, respectively.

3.2. Proximate and ultimate analysis

Proximate and ultimate analysis of micro-char oily sludge via microwave process at microwave power and irradiation time were investigated. Tables 4 and 5 show proximate and ultimate analysis at the range of microwave power and irradiation time, respectively. As a result, Table 4 shows a proximate and ultimate analysis at the range of microwave power. Proximity analysis provides a rough estimate of the percentages of VM, A, and FC [25]. A high VM % results in extended, smoky flames and reduced calorific value. The HHV in gasoline reduces when the proportion of A is increased. The high carbon content of high-quality gasoline raises the fuel's calorific

Table 1
The proximate, fuel ratio ultimate analysis, and calorific value of OS

Sample	Proximate analysis			Fuel ratio	Ultimate analysis					HHV MJ/kg
	VM%	Ash%	FC%		C%	H%	N%	S%	O%	
Oily sludge	46.3	21.6	32.1	0.62	24.10	4.11	2.80	0.35	68.64	9.41

Table 2
Microwave process, micro-char weight, yield percentage at range of microwave power

Sample	Feed (g)	Power (W)	Residence time (min)	Micro-char weight (g)	Micro-char yield (%)
Micro-200	5.0	200	10	4.20	84.0
Micro-400	5.0	400	10	4.28	85.6
Micro-600	5.0	600	10	4.32	86.4

Table 3
Microwave process, micro-char weight, and yield percentage at range of irradiation time

Materials	Feed (g)	Power (W)	Irradiation time (min)	Micro-char weight (g)	Micro-char yield (%)
Micro-5	5.0	600	5.0	4.55	91.0
Micro-10	5.0	600	10	4.32	86.4
Micro-15	5.0	600	15	4.10	82.0

Table 4
Proximate, ultimate analysis of OS and micro-chars at range of microwave power

Sample	Proximate analysis				Ultimate analysis			
	VM%	Ash%	FC%	C%	H%	N%	S%	O%
OS	46.3	21.1	32.6	25.22	4.21	2.95	0.41	67.21
Micro-200	33.6	35.1	31.3	39.60	3.95	1.75	0.31	54.39
Micro-400	32.1	38.2	29.7	38.50	3.88	1.55	0.31	55.76
Micro-600	31.6	39.5	28.9	38.44	3.55	1.42	0.22	56.37

value. The ultimate analysis study focuses primarily on the physical properties of the oily sludge micro-chars produced. Based on the results, the proximate and ultimate micro-char compositions were compared. Micro-char's chemical make-up shifts, as seen above, in response to the microwave process's temperature reaction. The volatile matter content of oily sludge was reduced by microwaves at increasing temperatures. Because of the conversion process, microwaving oil sludge led to a higher concentration of carbon in the micro-char and a lower concentration of volatile substances [31]. The micro-char FC content was higher than OS; the result showed an increasing trend and higher microwave power.

3.3. Fuel ratio

The fuel ratio determines how quickly a solid fuel may be gasified or transformed through burning, and it is computed using VM and FC. The fuel ratio of OS and micro-char formed at a range of temperatures is illustrated in Table 6 and Fig. 4. The most significant fuel ratio value, 0.85, was purchased after the furnace was heated, while the fuel ratios for the other microwave operations were enhanced [32]. Because of this, the material created by the microwave method was better suited for use as a renewable fuel. The fuel ratio was raised from 0.70 oily sludge at a fixed residence time of 2.0 h to 0.93 at 200 W, 0.92 at 400 W, and 0.91 at 600 W, respectively. According to Fig. 4, the optimal fuel ratio value was 0.85 at 200 W, corresponding to a 32.85% rise in the proportion of micro-char. This fuel will release a modest quantity of carbon dioxide compared with the raw material, oily sludge [14].

Table 7 and Fig. 5 shows the OS's fuel ratio and micro-char produced at a range of irradiation time. Fuel ratios have been increased across the board for microwave procedures, with the highest value ever recorded at 0.97 after the furnace heating phase. This outcome means that the renewable fuel produced by the microwave technique is superior. The fuel ratio increased from 0.70 of oily sludge to 0.88 at 5 min, 0.92 at 10 min, and 0.97 at 15 min, respectively. Fig. 5 depicts the improved fuel quality achieved by increasing the fuel-to-air ratio to 0.97 at 600 W for 15 min [27].

3.4. Calorific value

To evaluate how effective the microwave process is, the amount of energy that was recovered from the micro-char product was measured [33]. This investigation showed that the solid carbon fuel micro-char samples from oily sludge had a much greater calorific value than the raw OS. Because power contributes to micro-char formation, micro-char produced by microwaves operating at higher powers has a higher calorific value. The heating value of micro-char following the order OS for the conversion process at various microwave powers was enhanced to 14.82 MJ/kg at 200 W, 15.64 MJ/kg at 400 W, and 16.23 MJ/kg at 600 W. Calorie calculations can be used to gauge a material's combustibility [34].

Also, for the same amount of irradiation time, a higher treatment power removes more oxygen from the organic material and increases the value of the microwave power

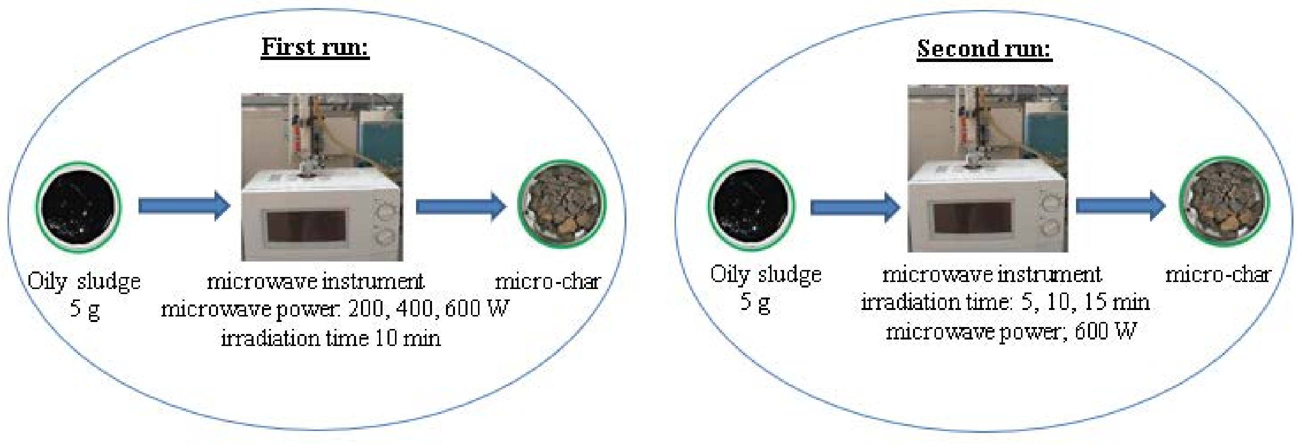

Fig. 1. Schematic illustration microwave process of oily sludge to micro-char production.

char [35]. Since oily sludge is the most common form of hydrocarbon compound, heating efficiency is improved by using a microwave to treat it. The heating value of micro-char at a constant reaction temperature of 600 W, by the order OS, was 9.41 MJ/kg, increasing to 14.22 MJ/kg after 5 min, 16.23 MJ/kg after 10 min, and 16.11 MJ/kg after 15 min of the conversion process at varying microwave power. The results suggest that the micro-char produced from OS can benefit from the microwave procedure in terms of its fuel qualities. This research showed that the micro-char was upgraded due to the enhanced transformation, confirming that the optimal conversion temperature is 600 W with HHV 16.23 MJ/kg, as previously mentioned by the study of Nizamuddin et al. [36]. It has been found that a residency length of 15 min, which may be optimal for all reaction temperatures, increases the ash content while simultaneously decreasing the volatile content of the micro-char.

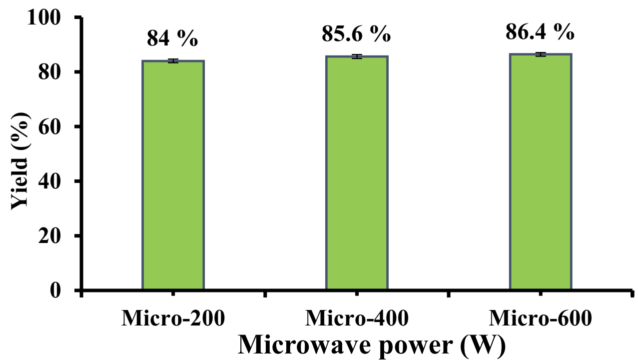

Fig. 2. Micro-char yield percentage at different microwave.

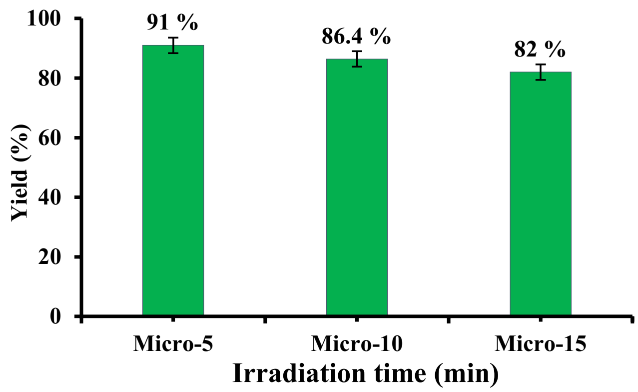

Fig. 3. Micro-char yield percentage at different irradiation time.

3.5. Energy recovery

The energy recovery of the micro-char produced was calculated to evaluate the calorific value recovery after micro-char production. Table 6 and Fig. 8 shows the energy recovery of micro-char produced at a range of microwave

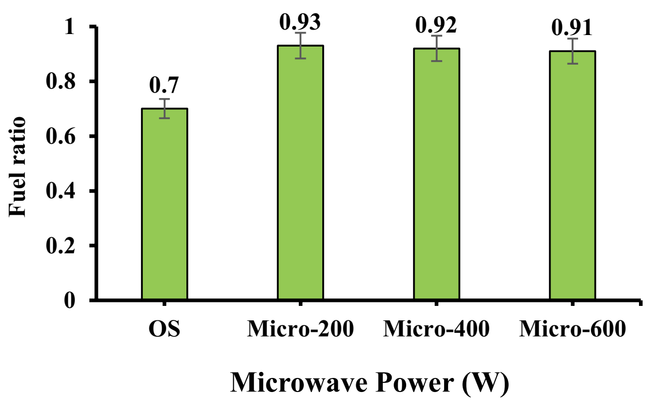

Fig. 4. Fuel ratio of OS and micro-char produced via microwave at various microwave power.

Table 5
Proximate, ultimate analysis of OS and micro-chars at range of irradiation time

Sample	Proximate analysis			Ultimate analysis				
	VM%	Ash%	FC%	C%	H%	N%	S%	O%
OS	46.3	21.1	32.6	25.22	4.21	2.95	0.41	67.21
Micro-5	33.6	36.5	39.9	38.22	3.61	2.11	0.35	55.71
Micro-10	31.6	39.5	28.9	38.44	3.55	1.42	0.22	56.37
Micro-15	33.9	32.9	33.2	37.95	3.99	1.22	0.33	56.51

Table 6
Fuel ration, HHV of raw material OS, and micro-chars at range of microwave power

Samples	Fuel ratio FC%/VM%	Fuel ratio increase %	Calorific value MJ/kg	Energy recovery
OS	0.70	–	9.41	–
Micro-200	0.93	32.85	14.82	1.57
Micro-400	0.92	31.42	15.64	1.66
Micro-600	0.91	30.00	16.22	1.72

Table 7
Fuel ration, HHV of raw material OS, and micro-chars at range of irradiation time

Samples	Fuel ratio FC%/VM%	Fuel ratio increase %	Calorific value MJ/kg	Energy recovery
OS	0.70	–	9.41	–
Micro-5	0.88	25.17	14.22	1.51
Micro-10	0.92	31.42	16.22	1.72
Micro-15	0.97	38.57	16.11	1.71

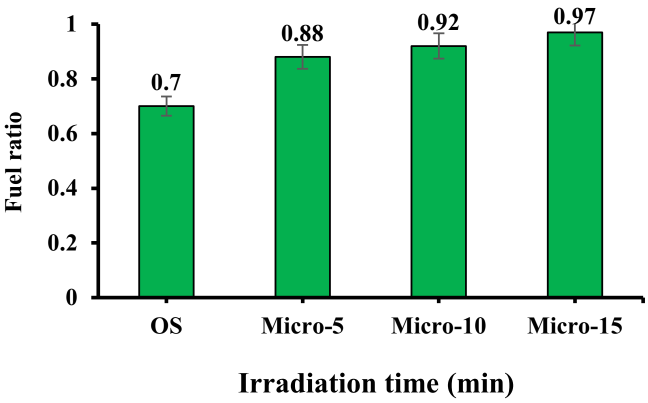

Fig. 5. Fuel ratio of OS and micro-char produced via microwave at various irradiation time.

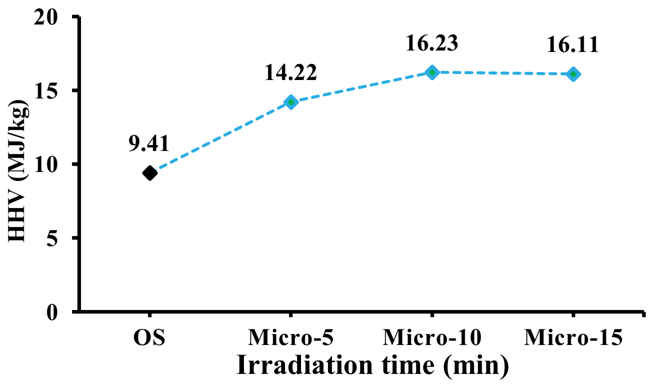

Fig. 7. HHV of OS, micro-char produced via microwave at various irradiation time.

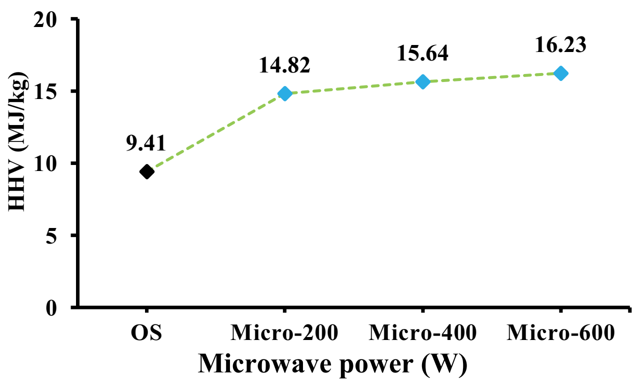

Fig. 6. HHV of OS, micro-char produced via microwave at various microwave power.

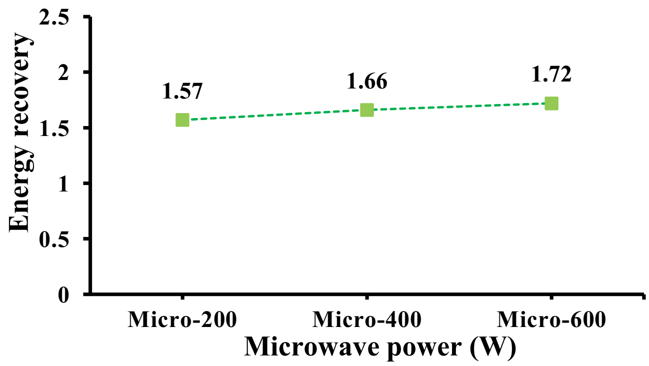

Fig. 8. Energy recovery of micro-char produced via microwave at various microwave power.

power. While Table 7 and Fig. 9 shows the energy recovery of micro-char produced at various irradiation times. Energy recovery via the divided calorific value of micro-char on the oily sludge [37]. As a result, the energy recovery of micro-char produced at a range of microwave power follows the range of 1.2 for micro-200, 1.3 for micro-400, and 1.5 for micro-600. Therefore, the energy recovery of micro-char produced at irradiation time follows the range of 1.2 for micro-200, 1.3 for micro-400, and 1.5 for micro-600. The energy recovery values increased from 1.2 to 1.3 and 1.5 for micro-char samples micro-200, micro-400, and micro-600, respectively. An energy yield of 72.47% was found for the micro-char sample micro-600 (made under the most extreme reaction conditions), with a calorific value of 16.23 MJ/kg [22].

Table 8 compares this study with past studies regarding fuel ratio, HHV, and energy recovery. The results also showed convergence and reasonableness in the results, especially in the fuel values that have been enhanced and upgraded.

4. Conclusion

This study successfully converted OS to solid carbon fuel micro-char using various microwave power and irradiation times. OS samples' calorific value increased from 9.41 to 16.23 MJ/kg, and the oily sludge fuel ratio was improved from 0.68 to 0.85 of micro-char at 600 W and 15 min of residence time. Therefore, a residence period of 15 min was appropriate for turning oily sludge into

Table 8
Compares this study and past studies of fuel ratio, HHV, and energy recovery

Study	Sample	Fuel ratio FC%/VM%	Calorific value MJ/kg	Energy recovery	References
Characterization study of oily sludge produced from North Refineries Company Baiji to determine the suitability for conversion into solid fuel	OS	0.66	9.65	–	[14]
Solid fuel char production via pyrolysis process of oily sludge produced as a resulted in storage tanks at North Refineries Company Baiji	OS Char	0.78 0.97	9.12 16.22	– 1.90	[27]
This study	OS Char	0.70 0.97	9.40 17.22	– 1.72	This study

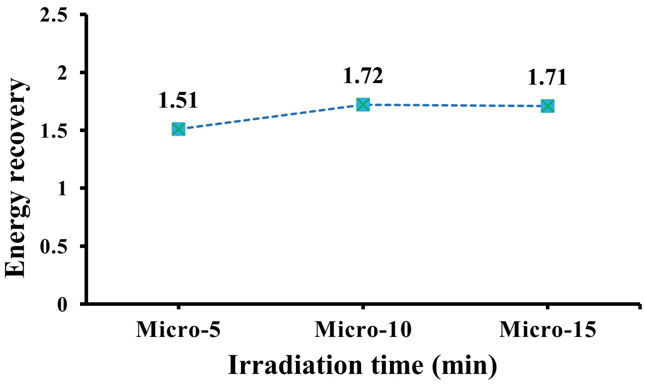

Fig. 9. Energy recovery of micro-char produced via microwave at various irradiation times.

micro-char. Therefore, the best energy recovery under ideal circumstances was 17.2. These results show that solid carbon fuel from oily sludge may be used at North Refineries Company Baiji to generate electricity. Then lessen the harmful effects of waste products produced by the oil sector on the environment.

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