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Solid Fuel Char Production via Pyrolysis Process of Oily Sludge Produced as a Resulted in Storage Tanks at North Refineries Company Baiji

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Abstract

The oily sludge contains a toxics material. It has an impact effect to the environment and worker's health. Therefore, treatment the residue oily sludge in the refineries storage tanks and convert it to useful product, is an important task. Oily sludge (OS) sample was obtained from North Refineries Company (NRC) Baiji which produced about 3000-3500 m^3/ year. In this study, different range of pyrolysis temperatures have been applied (300, 500, 700, and 900 °C). The parameters have been investigated the efficiency of char produced from OS. The operation conditions of (20 g, 700 °C, and 1.5 h, under N_2 pressure) are investigated. The calorific value was enhanced from 9.125 to 17.247 $MJ.kg^{-1}$ with an increased rate ~ 89.0 %. The fuel ratio was increased from 0.78 of OS to 0.97 around 24.35 % increased percentage of char at 700 °C. Finally, the energy recovery was enhanced at maximum value 1.909 % of char at 700 °C. The results show the fuel properties were upgraded to burn with a small amount of CO_2.

Keywords: Oily Sludge, Pyrolysis Process, Char, Calorific Value.

1. Introduction

The dangers of oily sludge have increasingly gained interest as public understanding of environmental protection has grown. The indiscriminate dumping of oily sludge, which is a form of hazardous solid waste, would affect both the environment and humans [1]. Oily sludge is a mixture of water, crude oil, refined oil, soil, and other contaminants created by the petroleum industry at various points, such as crude oil extraction, refining, transportation, utilization, and storage [2]. On one hand, oil can be recovered as valuable new renewable fossil fuels due to its high quality. On the other hand, the light hydrocarbons in the oily sludge are flammable, non-

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biodegradable, and extremely bio-toxic. As a result, oily sludge has been observed by environmental organizations such as China's "National Hazardous Waste List" (HW08). The environmentally appropriate treatment of oily sludge is the promising potential path from the stand point of sustainable management [3]. In China, over a million tons of oily sludge were discharged from the cleaning process of oil storage tanks bottom [4]. In China, upward three million tons of oily sludge was produced annually [5]. Sixty million tons of oily sludge has produced per year accumulated over the world, one billion tons of oily sludge has been accumulated worldwide[6]. In the Republic of Iraq Ministry of Oil NRC Baiji, produced about 3000 – 3500 m^3/year of oily sludge [7]. Pyrolysis is a promising method for extracting char, oil, or gas from oil sludge [8].

In Baiji, about 90 % of oily sludge discharged to landfill in the Makhoul mountains Baiji, that is caused adverse in the land [9]. Previous study stated that the oily sludge produced from NRC Baiji was rich in organic compounds and carbon content [10]. During the extraction, storage, transportation, refineries, and final products to distribution, upstream, and downstream processes in the petroleum industry, a significant amount of oily sludge is generated. [11]. Oily sludge contains a percent of substances that are dangerous to humans and the environment. It was listed in several countries as toxic, prevented from being disposed of to landfills [12]. The oily sludge generated in the oil industry is collected together from different sources, which caused an obstruction in storage yards, obtaining complex oily sludge that caused different composition, in specifications.

The oil industries are quite burdened by the problem of dealing with the large quantities generated from oily sludge continuously because it is a very dangerous waste and threatens workers and the environment because of the toxicity of the hydrocarbons it contains [13]. Oilfield companies produce large quantities of oily sludge during drilling and extraction operations. oily sludge presence makes drilling fluid more complicated, which impedes the work of pumps, increases water pressure and increases cost, which causes a burden to work [13].

During the pyrolysis of oily sludge, three reactions occur (alkanes, oxygenates, heavy fractions) [14]. Liquid oil, syngas, and char are the three most common pyrolysis products. The most common fuels are oil and syngas, but char can be recycled in a variety of ways. Because of the existence of toxic substances such as heavy metals, the use of char needs further study [15].

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The aims of this research are to the conversion of oily sludge waste (harmful materials) to (useful materials) char solid carbon fuel, rid the environment of oily sludge waste, then produce solid carbon fuel char via pyrolysis process, the char will be used as a solid carbon fuel for power generation at NRC Baiji.

2. Materials and Methods

2.1. Oily Sludge Sample Preparation

The oily sludge waste was collected from a fuel oil storage tanks bottom of North Refineries Company Baiji. It was collected from tank number (3107 FA). It was selected as the raw material for the preparation of char. The sample was dried at 105 °C for 24 h [16]. Subsequently, it crushed in powder to obtain the homogeneous form. The proximate analysis, ultimate contents, and calorific value of raw oily sludge were calculated, as in Table (1).

Table (1) Proximate and ultimate analysis of raw material oily sludge

Raw	Proximate analysis			Ultimate analysis					Calorific value
	VM%	Ash%	FC%	C%	H%	N%	O%	S%	MJ/Kg
OS	41.3	26.4	32.3	28.551	1.015	1.20	69.234	0.0	9.125

2.2. Pyrolysis Process

Fresh sludge was first sorted to extract large inorganic particles, then dried at 105 °C for 24 h, and sieved. Four 20 g of oily sludge samples were weighed accurately and put in four crucibles, respectively. The pyrolysis processes were performed in a horizontal furnace tubular reactor (CARBOLITE GERO 30-3000 °C - S/N 21-802840). It was working under N_2 at range of temperature 300, 500, 700, and 900 °C, respectively, and the prepared char were recorded as OS300, OS500, OS700, and OS900 accordingly, at Petroleum Research Development Center PRDC Iraqi Ministry of Oil, as in Figure (1).

The reactor was heated by an electric heating jacket power supply with a power of 1.5 kW and 220 V. Meantime the heating rate was set at 20 °C /min. Then the reactor was sealed and heated up to preset temperature with holding time for 1.5 h. The prepared char was milled by hand, sieved, and stored in a desiccator for later use. To ensure an anaerobic atmosphere, nitrogen was introduced into the horizontal tube furnace 20 minutes prior to the start of the pyrolysis process and continuously fed until the pyrolysis carbonization was completed. The mass of oily sludge before and after pyrolysis was determined using an analytical balance (PRACTUM224- 1S). In

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this research, the average value of multiple replicates was registered. All of the measurements were done at least three times.

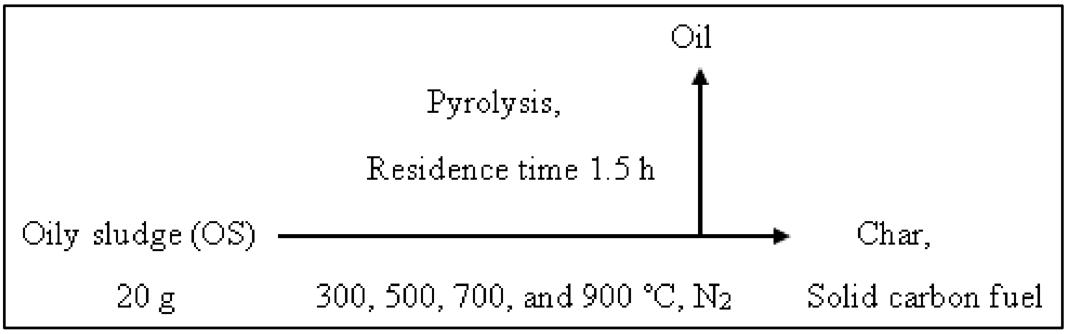

Fig. (1): Schematic illustration of the pyrolysis procedure of char produced from oily sludge

2.3. Analytical Procedure

The high heating value were measured with a Bomb calorimeter according to (ASTM E711-87 standard). A CHN analyzer according to (ASTM D-5672) and S according to (ASTM D-1552). For the proximate analysis, 1 g sample was set in a muffle furnace (absence of air) at *950 ± 10* °C for 3 h, the decreased weight was volatile matter content [17]. The moisture content was determined by placing each sample in a porcelain crucible, weighing it, measuring it again after heating at 100–105 °C for 2 hours, and calculating the weight difference. A dried sample was placed in a porcelain crucible and weighed to determine the volatile matter content. After closing the lid, it was heated at *950 ± 20* °C for 7 minutes and weighed again to calculate the weight difference. The ash content was calculated by subtracting the weight measured when a completely dried sample was placed into the porcelain crucible from the weight measured after the sample was heated at 850 °C for 3 hours. The fixed carbon content was calculated by assuming a completely dried sample had a fixed carbon content of 100 percent and subtracting the volatile matter and ash contents (percent).

2.4. Calculations of Char

The production of solid carbon fuel char was evaluated at the calculate of the energy and properties of the fuel [18]. The energy properties of fuel included; the char yield, fuel ratio, energy recovery, energy yield, and HHV improvement (higher heating value) improvement were calculated using equations from1 to 4 [19].

$$\text{Char yield (\%)} = \frac{\text{Char weight}}{\text{Raw matiral weight}} \times 100 \qquad \text{……1}$$

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$$\text{Fuel ratio} = \frac{\text{Fixed carbon}}{\text{Volatile matter}} \quad ......2$$

$$\text{Energy recovery} = \frac{\text{Calorific value of char}}{\text{Calorific value of oily sludge}} \quad ......3$$

$$\text{Energy yield} = \text{Char yield} \times \text{Energy recovery} \quad ......4$$

3. Results and Discussions

3.1. Mass Reduction

As shown in Table (2), pyrolysis of oily sludge at various temperatures can reduce weight and volume. From low to high pyrolysis temperature (OS300, OS500, OS700, and OS900), the weight yields were 8.32 g at 300 °C, 9.10 g at 500 °C, 11.46 g at 700 °C, and 11.82 g at 900 °C, the carbonization weight loss rates were 41.6 %, 45.5 %, 57.3 %, and 59.1 %, respectively. This carbonization weight loss, like to the previous works [20]. There are three major stages of mass loss in the pyrolysis phase of oily sludge, the removal of moisture stage (<200 °C), low-temperature pyrolysis stage (200 – 550 °C), and high-temperature pyrolysis stage (550 – 800 °C) [21]. In this study, OS300, and OS500 belonged to the low temperature pyrolysis stage. The mass reduction was primarily due to the volatilization and decomposition of petroleum hydrocarbons at this point, in addition to the complete removal of water. In comparison to OS500, the weight loss ratio improved at 700 and 900 °C at 1.03 % and 1.02 %, respectively, while the temperature was continuously increased.

Table (2) Pyrolysis process parameters, char weight, and char yield percentage

Sample	Feed amount (g)	Temperature (°C)	Residence time (h)	Char weight (g)	Char yield (%)
OS300	20.021	300	1.5	8.321	41.6
OS500	20.010	500	1.5	9.105	45.5
OS700	20.015	700	1.5	11.462	57.3
OS900	20.014	900	1.5	11.823	59.1

3.2. Proximate and Ultimate

The pyrolysis treatment with rising temperatures decreased the volatile matter content of the raw material oily sludge. Pyrolysis process of oily sludge resulted in the decrease of volatile matter content and the rise in carbon content in the hydrochar due to the conversion process [22]. The char fixed carbon content was higher than that of oily sludge, and their content showed an

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increasing trend along with higher pyrolysis temperatures. The elemental analysis showed that pyrolysis had significantly decreased the contents of H and O atomic of oily sludge.

Thus, the combustion efficiency of the hydrochar produced from oily sludge was increased via the hydrothermal carbonization process. The proximate analysis provides an estimation on the relative proportions of volatile matter (VM), fixed carbon (FC), moisture (M), and ash (A) [23]. Increased moisture content leads to the lower calorific value, increase the cost of transport, a considerable amount of heat is lost evaporation.

The percentage of volatile matter is high the value causes smock, long flames and decreases the calorific value. Increase percentage of ash caused decreases the calorific value in the fuel. the high carbon percentage given good quality coal, increase it is calorific value as given in Table (3). The ultimate analysis assesses the relative content of individual elements such as C, H, O, N, and S [23]. Ultimate analysis study mainly focuses on the physical characters of the oily sludge. Compared the proximate and ultimate compositions of char based on the result. As can be seen, the composition variations among char depend on the temperature reaction of pyrolysis process.

Table (3) Proximate and ultimate analysis of raw material and chars

Sample	Proximate analysis			Ultimate analysis				
	VM%	Ash%	FC%	C%	H%	N%	O%	S%
OS	41.3	26.4	32.3	28.551	1.015	1.20	69.234	0.0
OS300	30.8	39.7	29.5	44.981	1.297	1.12	52.602	0.0
OS500	30.5	41.2	29.3	31.933	1.201	1.21	65.626	0.0
OS700	30.3	40.1	29.6	38.149	1.514	1.24	59.097	0.0
OS900	30.8	40.6	28.6	39.606	1.322	1.22	57.852	0.0

3.3. Fuel Ratio

The fuel ratio measured on the basis of volatile matter and fixed carbon, specifies the ease with which a solid fuel can be gasified or combustion all converted. The highest fuel ratio value was 0.97 which was purchased after the furnace heating, while the other pyrolysis processes have received improved fuel ratios. Therefore, the produced from pyrolysis process was more suitable for use as a renewable fuel. The best value of fuel ratio was 0.97 around 24.35 % increased percentage of char at 700 °C this shows the fuel was upgraded as shown in Table (4).

This fuel will be burning with release small amount of CO_2 compare with raw material oily sludge. That is mean the char fuel properties was enhanced as in study [24].

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Table (4) Fuel ration, fuel ratio increased percentage and calorific value of raw material and chars

Sample	Fuel ratio FC%/VM %	Fuel ratio increase %	Char yield %	Calorific value MJ/Kg	Energy recovery	Energy yield
OS	0.78	-	-	9.125	-	-
OS300	0.95	21.70	41.6	16.355	1.792	74.547
OS500	0.96	23.07	45.5	16.521	1.810	82.355
OS700	0.97	24.35	57.3	17.247	1.909	109.385
OS900	0.92	17.94	59.1	16.987	1.861	109.985

3.4. Calorific Value

To evaluate the pyrolysis process after char production, the recovered energy of the char product was calculated [25]. The combustibility of materials can be determined by calculating the calorific value of the burned fuel [26]. This research proved that the oily sludge derived solid carbon fuel char samples were improved with a noticeable increase in the calorific value compared with the raw material (waste oily sludge). The temperature adversely affects the char, so that the lower the pyrolysis reactor temperature, the higher the calorific value of the char product. HHV for OS was 9.125 $MJ.kg^{-1}$ it was upgraded to 16.355 $MJ.kg^{-1}$ at 300 °C, 16.521 $MJ.kg^{-1}$ at 500 °C, 17.247 MJ.kg-1 at 700 °C, and 16.987 $MJ.kg^{-1}$ at 900 °C. This study is proved that the char has been upgraded, so it has been proven that the best conversion temperature is 700 °C with HHV 17.247 $MJ.Kg^{-1}$, which was previously mentioned by S. Nizamuddin et al. (2017) [27]. The reaction temperature effect on the heating value of char was shown in Figure (2). The residence time 1.5 h may be suitable for all reaction temperatures, which leads to an increase in the ash content and a decrease in the volatile content of the char. The heating value of char followed the order, the HHV 17.247 $MJ.kg^{-1}$ at 700 °C > HHV 16.987 $MJ.kg^{-1}$ at 900 °C > HHV 16.529 $MJ.kg^{-1}$ at 500 °C and > HHV 16.355 $MJ.kg^{-1}$ at 300 °C, and the highest heating value was 17.247 $MJ.kg^{-1}$ at reaction temperature 700 °C.

Similarly, for identical residence times, as the treatment temperature increased, the degree of oxygen removal in the organic matter increased, resulting in an increase in the heating value of the char. Because oily sludge is the most abundant compound in hydrocarbon compounds, pyrolysis process improves the heating value of the solid yield. Finally, the oily sludge with an HHV 9.125 $MJ.kg^{-1}$ produced maximum hydrochar yield with maximal an HHV 17.247 $MJ.kg^{-1}$, upgraded to

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89.0 %, at 700 °C. These results showed that the optimum temperature of pyrolysis process for producing a more energy-rich solid fuel hydrochar is approximately 700 °C. Based on the results, it can be concluded that pyrolysis process can upgrade the fuel properties of char from oily sludge.

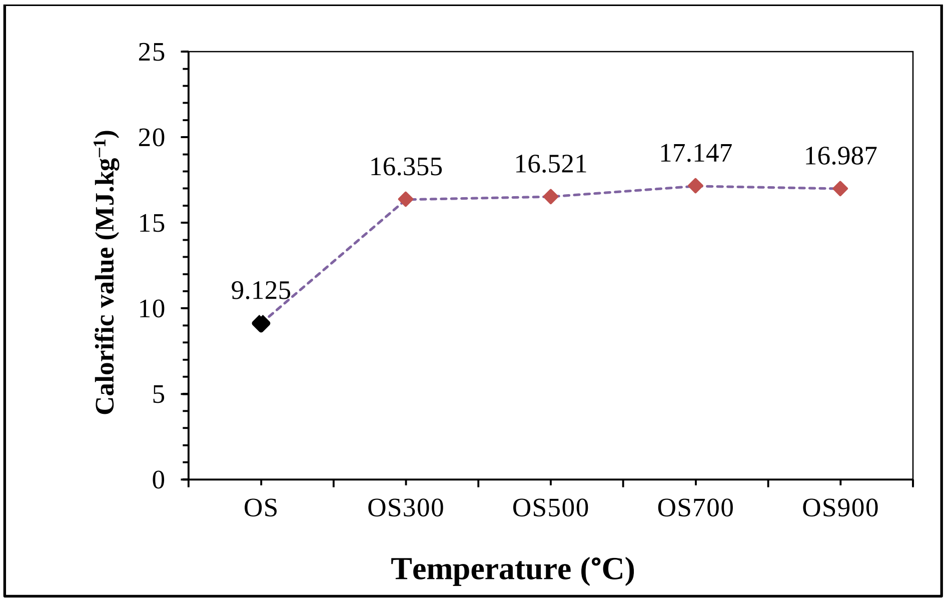

Fig. (2): Calorific value of hydrochar produced from oily sludge with different temperature

3.5. Energy Recovery Efficiency

To evaluate the pyrolysis process efficiency, after the char production, the recovered energy of the char product was calculated [28]. Therefore, the pyrolysis process was evaluated by energy recovery efficiency ERE, which was affected by the reduced product yield and increased heating value [29]. Table (3) shows best of ERE was 90.98% of at 700 °C. As shown in Figure (3) the effect of reaction temperature on ERE was studied.

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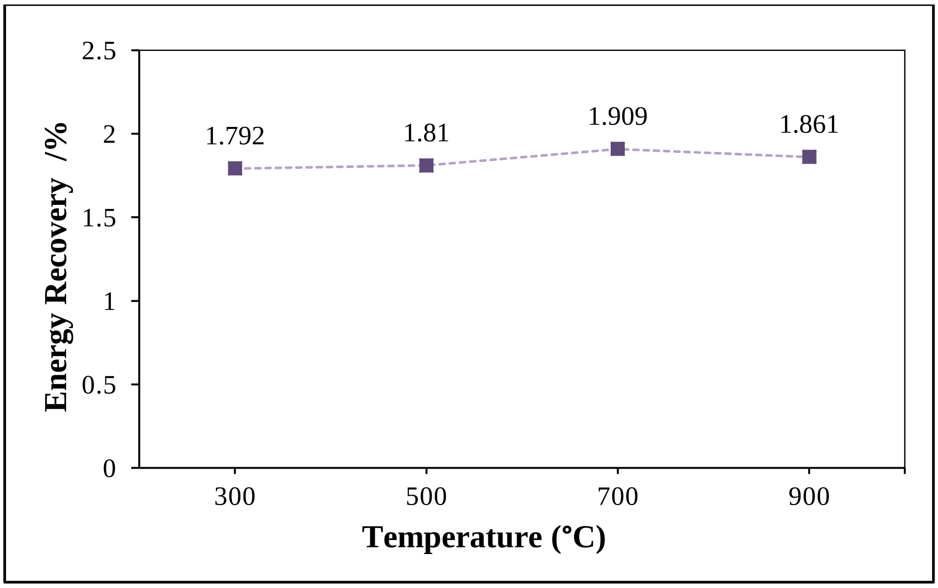

Fig. (3): Energy recovery of char produced from oily sludge by different temperature

3.6. Mechanism Reaction

According to the findings, three major fractions (alkanes, oxygenates, and heavy fractions) were detected in oily sludge. Alkanes were initially dealkylated to form hydrocarbons (C_{20}–C_{30}), and the resulting hydrocarbons (C_{20}–C_{30}) could then be aromatized to form monocyclic aromatics and polycyclic aromatics [30].

4. Conclusions

Pyrolysis process has long been used to treat oily sludge and reduce the environmental impact of radioactive and hazardous compounds. After years of growth, the pyrolysis technology for oily sludge is relatively mature, ensuring that it can be promoted on a wide scale in the future. The calorific value of oily sludge samples was developed from 9.125 $MJ.kg^{-1}$ to 17.247 $MJ.kg^{-1}$ of char at 700 °C. The fuel ratio of oily sludge was upgraded from 0.78 to 0.97 of char at 700 °C around 24.35 % was increased with maximum solid yield. The energy recovery was enhanced at maximum value 1.909 % at 700 °C. These results show the oily sludge converted to the solid carbon fuel and in future can be used as a fuel for power generation at NRC Baiji. Then reduce environment of waste materials produced during oil industry.

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