



Characterization Study of Oily Sludge Produced from North Refineries Company Baiji to Determine the Suitability for Conversion into Solid Fuel

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Abstract

Oily sludge (OS) contains toxic compounds such as aromatic hydrocarbons and polyaromatic hydrocarbons; oily sludge is hard to dehydrate due to its high viscosity. Generally, it contains about (40 to 50 wt.%) of water. However, this research's oily sludge sample contains about (50 wt.%) of water collected from North Refineries Company Baiji (NRC). NRC Baiji yields around 3000-3500 m³ annually of oily sludge. This research aims to study oily sludge specifications, thereby prove suitability to undergo the thermochemical process. The physical properties of oily sludge samples have been conducted, it indicated the complex formula of oily sludge, and it has a serious effect on human health and environmental issues. In this study, the oily sludge has a low heating value (LHV) of 6.705 MJ.kg⁻¹ (as received) and a high heating value (HHV) of 9.465 MJ.kg⁻¹ for a dried sample at 105 °C for 24 h, which indicate that it is a rich in carbon content. FTIR has been carried out to obtain the function group characterizations. The TGA of the oily sludge sample as received showed the greatest loss in volume (39%), while the loss in volume for the dried sample was (15%). The thermochemical conversion process eco-friendly, rapid, short time, moderate temperature, and low-cost. It is used for the total reclamation of waste. It is a novel way of totally converting oily sludge for solid carbon fuel production.

Keywords: Oily sludge; LHV, HHV; North Refineries Company Baiji NRC.

1. Introduction

The processing of crude oil generated different kinds of waste, such as oil and oily sludge [1]. Petroleum industries are responsible for producing large quantities of oily sludge, a major source of environmental impacts. Oil companies are burdened with waste oil pollutants due to the oily sludge usually stacked together and generate different sources. Oil processing operations are accompanied by operations called reclamation [2]. The disposal of oily sludge for hazardous waste in landfills is not allowed even to minimize the oily sludge's organic content by different treatment methods [3]. It was proved that petroleum sludge has hazardous effects on humans and the

environment alike [4]. The oily sludge's organic structure analysis proved that it comprises multiple quantities of wastewater, solid sediments, waste oil, sand, and mineral substances [5]. Landfilling is the common way of disposal of the sludge produced from the liquid effluent treatment plants of an oil industry [6]. Waste oil sludge product is one of the by-products of the categorized refineries (oil refinery A) [7]. The huge quantity of oily sludge originates from the upstream and downstream processes [8]. NRC Baiji, produced roughly 3000 - 3500 m³ per year [9].

The oil industry's oily sludge application is two types of application methods that deal with oily sludge (disposal and oil recovery). Disposal methods included incineration, oxidation, biodegradation, and

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stabilization/solidification, while oil recovery methods included pyrolysis, solvent extraction, surfactant, freeze/thaw, centrifugation, microwave irradiation, electrokinetic method, ultrasonic irradiation, and froth flotation. These methods used to deal with oily sludge currently [5]. On the other hand, the application of oily sludge recovery technical processes of oil from petroleum sludge. It is included solvent extraction, mechanical centrifugation, surfactant, freezing/thawing, pyrolysis, electrical method, ultrasonic, flotation, supercritical fluid, combined processing, and emerging methods [10].

It was proved practically that the oily sludge could be a potential source of the oil and hydrocarbon products. However, converting oily sludge into valuable substances needs some pre-treatments steps to facilitate its analysis [11]. The oily sludge concentration, namely the oil and water rate in its chemical composition of 50%, is preferable to be recycled again. The oily recycling sludge generated beneficial outputs of carbonic products [12]. The earlier recycling methods included thermal treatment processes that are pyrolysis, incineration, and gasification, which can be a vital and effective method in recycling the oily sludge and obtain char and carbon materials. The recovery oil from this method under high temperature is considered a preferred fuel for energy recovery [13]. In the same line with the recycling process, various instruments and methods of incineration of oily sludge have been used to deal with it. These instruments include kiln, rotary, rack, fluidized bed combustors, and circulating fluidized bed combustors [14]. From this literature review, it was found that the oil mentioned above sludge treatment methods, pyrolysis, incineration, and gasification are not economically feasible operations and require large areas, high equipment, operation cost, high temperatures, high maintenance, and fuel consumption in order to occur.

Moreover, release high quantities of carbon dioxide polluting the environment [6]. Oily sludge cannot be disposed to the landfill, even if it is remediated [15][16] has been reported the characterization of oily sludge sample from Petronas in Melaka Malaysia the density was found 1.08 g/ml, the black oily sludge has 78.91% moisture, 5.06% ash, 5.52% volatile matter, and 10.51% fixed carbon. Despite the high moisture content, the dried sludge has a higher heating value (HHV) of 23.599 MJ/kg. The oil depletion throughout the world is another

inclination that provoked the researchers to carry out studies on the oily sludge to harvest the maximum benefit from the oil products and obtain marketable and valuable hydrocarbon-based substances as carbon and fuel.

This research looking to find an alternative method for the conversion of oily sludge such as thermochemical conversion process has a high advantage the most important of which is low operating conditions, in particular temperature and pressure, low power, short time, low cost, and produces hydrochar that is rich in carbon materials [17]. This study's key goals were to carry out a screening study of the water content's effect in oily sludge produced from NRC Baiji. It also determines the ability to undergo the thermochemical conversion to the production of different types of carbon, such as solid carbon fuel. The specific objective was to identify the thermochemical features of oily sludge for its suitability to undergo the thermochemical conversion process to produce economic environment-friendly hydrochar solid carbon fuel.

2. Materials and Methods

2.1. Materials

Four different oily sludge samples were evaluated to obtain deeply understanding the specifications and formatted and find and prove which one is older in terms of the operating period. Oily sludge samples were collected directly from fuel oil storage tanks (3107 FA, 3107 FB, 3108 FA, and 3108 FB), numbered 1, 2, 3, and 4, respectively.

The samples were brought from oil depot Salahuddin in the NRC Baiji, Ministry of Oil, Republic of Iraq. All samples were subjected to a series of analyses to determine their primary components; Specific gravity Sp.gr, American Petroleum Institute API, color, water content, and asphaltene content in oily sludge were determined in the laboratories of the Ministry of Oil, Oil Products Distribution Company, Salahuldeen Branch, Tikrit, Iraq.

2.2. Oily Sludge samples Selection

In this research, sample number one has been chosen to proceed with the characterization study. To calculate the thermochemical conversion process's physical and chemical properties, taking such a sample was because it has a longer operation period, higher specific gravity, higher API, higher water content, and

higher asphaltene content as in Table 1. Concerning the color test, all of the samples were black; that is mean it formed from upstream sources [8].

Table 1: Operation period of oily sludge samples from North Refineries Company Baiji

	Oily sludge samples number			
	Sample 1	Sample 2	Sample 3	Sample 4
Tank number	3107 FA	3107 FB	3108 FA	3108 FB
Product	Fuel oil	Fuel oil	Fuel oil	Fuel oil
Operation period	Six years	Five years	Four years	Four years

2.3. Characterization of Oily Sludge Samples

The specific gravity Sp.gr of all oily sludge samples have been calculated according to the standard method (ASTM D 1298) [18]. All experiments were carried out based on the international code and standard American Society for Testing and Materials (ASTM) [19]. The HHV was measured with a Bomb calorimeter (C5001 IKA-WERE) according to (ASTM E711) [20]. The CHN analyser elemental analysis was carried out using a (PerkinElmer, Series II,2400) at FSG in UiTM. The 1 g sample measured the volatile matter was put in a muffle kiln (absence of air) at 950 ± 10 °C for 7 min, the reduced weight was evaporative matter content, according to the standardization method of an experiment for volatile matter content in the analysis of particulate fuels [21]. Briefly, The ash percentage was found of the 1 g sample was set in a muffle furnace (absence of air) at 950 ± 10 °C for 3 h, according to Standard Test Method for Ash in fuel [22]. The fixed carbon was calculated summation of ash and volatile matter that is subtracted from 100 percentages. The effects of moisture and water content for both dry and wet samples have been discussed. On the one hand, Fourier-transform infrared spectroscopy FTIR on a Perkin Element FTIR Spectrometer (model: Spectron one) was utilized to analyse the functional groups. On the other hand, Thermogravimetric Analysis (TGA) and Derivative Thermogravimetric (DTG) analysis were implemented under N₂ gas, the weight of sample 20 mg, the rate of heat 10 °C/min, atmosphere nitrogen

Gas flow rate of 20 ml/min, starting temperature 30 °C, final temperature 1200 °C flow by a (TGAQ/DSCI) instrument.

3. Results and discussion

3.1. Physical Properties of Oily Sludge

All the oily sludge samples were collected and characterized; the physical and chemical properties were calculated to investigate appropriate undergoes to the thermochemical conversion method. Five main aspects of oily sludge samples in terms of the physical properties were noticed. The tests were performed in the Oil Distribution Company's research laboratories, Salahuddin Branch, Ministry of Oil, Iraq. The physical properties of oily sludge comprised specific gravity, American Petroleum Institute API, color, asphaltene content, mass%, and water content mass% were determined, as shown in Table 2. Table 2 shows a varying degree of specific gravity between the samples 1 is 0.9150 maximum result, and sample 4 is 0.9000 minimum result, which indicates that the oily sludge is stacked from different sources and long times [23].

All samples were black like the sample [24] used. This indicates the purity of the samples and the heterogeneity of water with oily sludge samples. The color in the oily sludge samples expresses the purity of the material. It was proved in the previous literature that there were three sorts of sludge; they are black and dark [25]. The color sample used in this study was black.

Table 2: Physical properties of NRC Baiji oily sludge samples 1, 2, 3, and 4

Tests	Oily sludge samples number			
	Sample 1	Sample 2	Sample 3	Sample 4
Specific Gravity (Sp.gr)	0.9150	0.9100	0.9120	0.9000
API	23.15	24.00	23.65	25.72
Color	Black	Black	Black	Black
Water content, mass%	50.0	46.0	40.0	48.0
Asphalt content, mass%	11.6	10.3	10	12

3.2. Proximate and Ultimate Analysis

The proximate analysis offers an appraisal on the relative quantities of fixed carbon (FC) volatile matter (VM), and ash, while ultimate analysis evaluates the relative content of individual rudiments such as C, H, O, N, and S [26]. Oily sludge sample was kiln-dried at 105 °C for 24 h, before proximate analysis [21]. Ultimate analysis has been conducted for oily sludge samples. The outcome of ultimate and proximate compositions of oily sludge samples are shown in Table 3.

The moisture percentage was determined gravimetrically by the oven-drying method (ASTM D3173- 11) [21]. The ash content for both samples

Table 3

Proximate and ultimate analysis for dry and wet oily sludge of Baiji Refineries

Oily sludge samples	Proximate analysis			Ultimate analysis					Calorific value MJ/kg
	M%	VM%	Ash%	FC%	H%	N%	S%	O%	
Wet sample	34.1	53.1	22.3	24.6	-	-	-	-	6.705
Dried sample	12.6	46.3	23.1	30.6	55.1	30.1	0.9	0.009	9.465

3.3. Heating Value

The calorific value is the most critical parameter to describe a material as a fuel. Both higher and lower heating values of oily sludge were resolved by Bomb calorimeter (C5001 IKA-WERE) according to ASTM E711. As in Table 3 the oily sludge has a lower calorific value 6.76 MJ.kg⁻¹ for oily sludge sample (as received) and a higher heating value of 9.460 MJ.kg⁻¹ for dried oily sludge sample at 105 °C for 24 h [28]. A higher heating value (HHV) of 9.465 MJ.kg⁻¹ was gained, and this value is within the scope of HHV of coal [29]. This outcome indicated that the reactions decarboxylation and dehydration will occur, vital for increasing the calorific value of oily sludge [30].

Consequently, the processes that can occur to the oily sludge (dehydration and decarboxylation) through thermal, chemical conversion processes (i.e. hydrothermal carbonization) will contribute to improving the combustion properties of the oily sludge by increasing the calorific value of oily sludge [31]. This indicates the possibility of using it as fuel after recycling and improving quality. Thus, it will release low amounts of pollutants upon combustion, such as carbon dioxide.

is shown in Table 3. It can be seen that the dry-oily sludge shows a higher value, which caused decreases in the calorific value in the fuel. The value of volatile matter of wet, oily sludge is higher than dry, which indicated that long flames, smock could cause it, and declines the calorific value. Table 3 also indicates the individual's components of the ultimate analysis relative content of such as, H, N, C, O, and S. It found that the dry sample contains a high percentage of carbon (55.17%), which facilitates the alteration process of oily sludge into carbon-rich fuel. Furthermore, the lower percentage of sulphur (0.009%) reduced the corrosion processes in equipment and pipes [27].

3.4. FTIR Spectroscopy

The FTIR spectroscopy is shown in Fig. 1. The oily sludge, as a received result, is demonstrated in Fig. 1a. The oily sludge sample dried at 105 °C for 24 h as in Fig. 1b. Numerous practical groups are discovered, like amide bending (C=O) extending at 1630 cm⁻¹, Alkynyl group (C≡C) at 2148 cm⁻¹ Alcohol/phenol stretching (O-H) at wavenumber 3234 cm⁻¹. [32]. At the same time, the oily sludge sample results are shown in Fig. 1b. Several functional groups are detected, such as di-alkyl stretching (C-O-C) at 1075 cm⁻¹ and aromatic bending (C=C) 1589 cm⁻¹. It can be seen that the peaks at 1000 cm⁻¹ was corresponding to the (C-O-

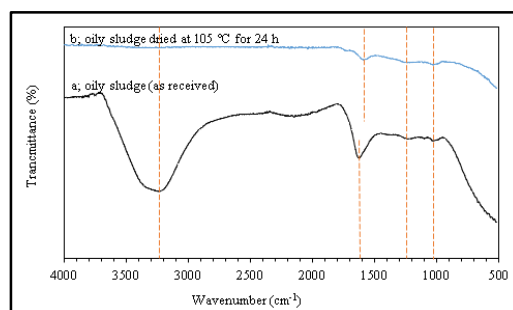


Fig. 1. FTIR spectrum test for oily sludge samples; (a) oily sludge as received; (b) oily sludge dried at 105 °C for 24 h.

C) while, stretching bands of dialkyl at 1589 cm^{-1} was corresponding to the C=C aromatic bending. Fig. 2 presents the consequence of moisture on the oily sludge sample because the oily sludge is rich in hydrocarbons and heavy metal components [32].

3.5. TGA Analysis

The outcome of using the thermogravimetric analysis (TGA) method to decomposition oily sludge samples is shown in Fig. 3. TGA test inspected the effect of elevated temperatures.

The temperature varied (22 to $1206\text{ }^{\circ}\text{C}$). The weight-loss method can be divided into three stages, starting from the TGA curve. The first stage occurred at a lower temperature ($150\text{ }^{\circ}\text{C}$) with a weight loss of 39.25%, 7.84 mg due to moisture content's evaporation, low molecular weight solvents, and oily sludge gas desorption. The second stage occurred at temperatures ranging from (390 to $984\text{ }^{\circ}\text{C}$), with a weight loss of 7.1%, 1.50 mg involved. At this point, the weight loss was due to volatile materials evaporation. The final stage occurs at (982 to $1082\text{ }^{\circ}\text{C}$) involving 10.3 & carbonization of hydro carbonated compounds, which are the residuals produced by 2.05 mg ashes. The sample shows high thermal stability at $1145\text{ }^{\circ}\text{C}$ temperature. As with Fig. 2a.

Fig. 2b shows the result of the TGA of dried at $105\text{ }^{\circ}\text{C}$ for 24 h oily sludge sample. The temperature ranged from (34.25 to $1204.50\text{ }^{\circ}\text{C}$). The weight-loss method can be divided into three phases starting from the TGA curve. The first stage occurred at a below temperature ($147\text{ }^{\circ}\text{C}$) with a weight loss of 1.5%, 0.38 mg, due to the evaporation of moisture content, low molecular weight solvents, and oily sludge gas desorption. The second stage occurred at a temperature ranging from (153.5 to $942.50\text{ }^{\circ}\text{C}$) and involved a weight loss of 12.5%, 2.5 mg. The weight loss at this stage was due to volatile material evaporation.

The final stage, involving carbonated hydro compounds' reaction, occurs from (971 to $969\text{ }^{\circ}\text{C}$). The subsequent residuals were ashes. The sample shows high thermal stability at $1151\text{ }^{\circ}\text{C}$ temperature. Based on the study [33], the upsurges in the rate of water content for oily sludge harmed the oily sludge incineration process. The oily sludge with higher water content (40–50%) had some minor peaks in volatile matter emission and incineration, as shown in Fig. 2a. This kind of oily sludge had a half-solid shape [34].

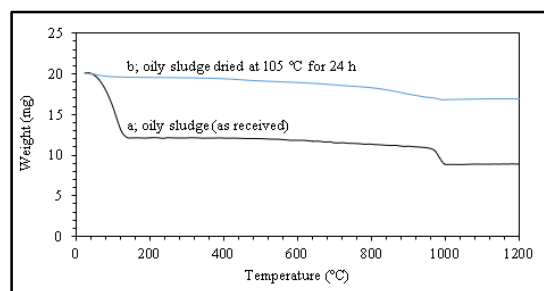


Fig. 2. TGA analysis for oily sludge samples; (a) oily sludge sample as received; (b) oily sludge dried at $105\text{ }^{\circ}\text{C}$ for 24 h.

The DTG test was observed to lift sharply to a height of $120\text{ }^{\circ}\text{C}$ due to a large amount of moisture. Between (120 and $200\text{ }^{\circ}\text{C}$), weight loss is decreased as opposed to the earlier stage (between 70 and $120\text{ }^{\circ}\text{C}$). Even this area is replicated with a sharp fall in the DTG curve. This finding could be attributed to residual moisture evaporation and volatilization of a limited quantity of low-temperature hydrocarbons, as shown in Fig. 4 DTG. Decay was observed from (151 to $451\text{ }^{\circ}\text{C}$), as seen in the TG curve's incremental reduction. The DTG curve was seen to go up and down somewhat since the chemical; organic materials have different boiling points and different concentrations in the oil sludge sample. Moreover, a change in the range of weight loss with reaction temperature is the same throughout the range of oily sludge samples.

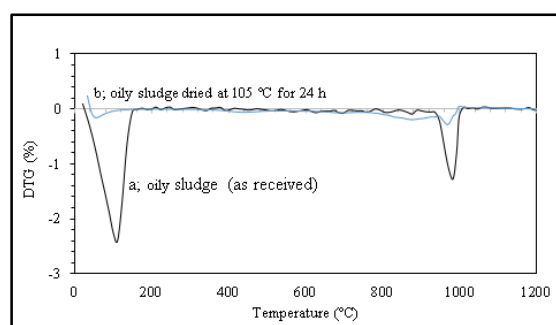


Fig. 3. DTG analysis for oily sludge samples; (a) oily sludge as received; (b) oily sludge dried at $105\text{ }^{\circ}\text{C}$ for 24 h.

4. Conclusion

The results showed that the oily sludge density of Baiji 0.915 was lighter than the oily sludge 1.08 referred to in previous studies, which indicates that the samples in this research is rich in carbon materials. The samples of oily sludge have been detected and determined by physicochemical

properties. The black oily sludge's characterization results have been conducted as follows; 34.1% represents moisture, 22.3% stands for ash, 56.1% volatile matter, 21.6% fixed carbon of oily sludge sample (as received). While the characterization results have been conducted of the dried sample at 105 °C for 24 h oily sludge as follows; 12.6% represents moisture, 22.1% stands for ash, 46.3% volatile matter, 30.6% fixed carbon. Although the LHV 6.705 MJ.kg⁻¹ while, the HHV 9.465 MJ.kg⁻¹. The high value of HHV is perhaps associated with low ash content and high fixed carbon. The carbon content outcome was around 55.17%, which means the oily sludge is rich in carbon components. It can be converted to hydrochar based on carbon .

Moreover, the low sulphur content was about 0.009%, which made it easier to convert the conversion process to high-quality fuel and protected the equipment from corrosion at burned. The TGA results show that the weight-loss temperature was 983 °C at the last stage producing a more stable aromatic carbon. As a result, the inclination of the waste disposal technique should consider the ability and potentiality of conversion of oily sludge for energy production and preserving the environment by producing fuel that releases fewer pollutants CO₂ gas. Therefore, it can be converted into fuel used to power electric production units of the North Refineries Company Baiji, thus reduce pollutant products.

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