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Producing Refuse Derived Fuel with Refining Industry Oily Sludge and Mushroom Substrates

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Abstract: The sludge in this study was obtained from refinery crude oil storage tanks. It contained a high proportion of hydrocarbon composition and harmful substances (such as polycyclic aromatic hydrocarbons and benzene). Through the microwave irradiation treatment process, the harmful substances were removed from the sludge which was then recycled and combined with agricultural waste mushroom substrates to produce refuse derived fuel (RDF). The results showed that the calorific value of RDF was 7279 cal/g when the blending ratio (*wt*/*wt*) of oil sludge and mushroom substrates was 5:5. On the other hand, when the portion of the mushroom substrates was increased, the sludge became easier to ignite with better combustion reaction. When the blending ratio (*wt*/*wt*) was changed from 8:2 to 5:5, the ignition index and comprehensive performance index were increased by 51.9 and 50.2%. Therefore, mixing the sludge with agricultural waste mushroom substrates is in line with the concept of waste recycling and circular economy.

Keywords: oily sludge; refuse derived fuel; circular economy

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1. Introduction

The refining industry produces a large amount of sludge during crude oil exploration, production, storage, and the refining process [1]. The global petrochemical industry produces about 60 million tons of oil sludge every year. According to the estimation of literature, there has been more than 1 billion tons of oily sludge accumulated in the world [1,2]. Oily sludge contains a variety of aromatic hydrocarbons which are carcinogens, such as BTEX (benzene, toluene, ethylbenzene, xylene) and polycyclic aromatics hydrocarbons (PAHs), and heavy metals with harmful microorganism attachment (siltation) [3]. Due to the complex source and composition, and the global increase in oily sludge, many countries have classified oily sludge as hazardous waste [1].

With the continuous increase in the consumption of petroleum products in recent years, the technological development for the detoxification, recycling, reduction, and reuse of oily sludge has become the main topic of current research. The detoxification of oily sludge includes incineration, solidification, oxidation treatment, and biological treatment [4]. These technologies have the disadvantages of low efficiency, high cost, waste of resources, and high risk of environmental pollution. The recycling of oily sludge can effectively lower the volume and pollution level of the hazardous solid waste. The recycling technologies include solvent extraction, mechanical centrifugation, surfactants, freeze/thaw, pyrolysis, electronics, ultrasound, flotation, and supercritical [3,5]. For example, Zhao et al. used smoldering combustion and found that the solid residue was odorless and granular. The composition of the recovered oily sludge was similar to that of coker diesel, and its heat value was higher than that of kerosene, showing the possibility of reuse [6]. Zhong et al. studied the conversion of harmful sludge into a shale stabilizer product (SS-OS) used in drilling fluids. The results showed that SS-OS can effectively inhibit the expansion and dispersion of shale, and has excellent sealing performance [7].

The characteristic of microwave irradiation heating is to make molecules move by inducing ion migration and dipole rotation, but the molecular structure cannot be changed. When the pollutants are irradiated by microwave energy, the temperature increases due to the movement of molecules. The rate of temperature rise is related to the dissipation factor (tangent δ) of the heated material which is defined as the ratio of the dielectric loss factor (ε'') to the dielectric constant (ε') [8,9]:

$$\tan\delta = \varepsilon''/\varepsilon' \tag{1}$$

The dielectric constant is a measure of the sample's ability of blocking microwaves from penetrating while the dielectric loss constant is a measure of a sample's ability to disperse energy. In other words, when microwave energy penetrates a sample, the rate of microwave energy being absorbed depends on its dissipation factor (δ) [10]. The biggest difference between microwave heat transfer and traditional heat transfer is that microwave energy is directly transferred by the interaction between molecules and electromagnetic fields, and the generated electromagnetic energy is converted into heat energy. Microwaves do not transfer energy by the diffusion of heat on the surface of materials, so it can also achieve rapid and uniform heating for thicker materials [9,11]. Microwaves provide an alternative way of handling non-degradable materials to reduce the environmental impact by improving the physical properties of substances. Its economic advantages include rapid heating, selective heating, reduced processing time, and improved energy efficiency which the traditional ways of substance treatment do not have.

Refuse derived fuel (RDF) is the homogeneous fuels converted from urban, agricultural, and industrial waste, etc., through physical or thermochemical methods. For example, Yuliarningsih et al. studied the use of oily sludge mixed with wood chips and PE (polyethylene) as a binder to produce RDF of heat value 5976 kcal/kg [12]. Karpan et al., studied the mixing of five industrial hazardous wastes (rubber waste, mixed waste, paint sludge, palm oily sludge, and sewage treatment plant sludge) and three biomass types (sawdust, rice husks, and nut husks) to produce RDF of heat value 18,652 kJ/kg [13]. Li et al., studied the use of automotive stamping gaskets and electronic packaging waste (56%), anaerobic digestion sludge (24%), and industrial desiccant residue (20%) to produce RDF of heat value 6.8 MJ/kg [14].

The oily sludge is rich in petroleum substances with high viscosity and harmful substances. With proper pre-treatment procedures, it can provide great recycling value. It has relatively high economic benefits of being converted into derived fuels. This research is based on the reuse of resources and the production of high-value RDF fuel energy. The focus of this study is as follows: (i) Industrial hazardous sludge can be heated rapidly and evenly by microwaves coupling with a high dielectric constant medium in the sludge and transform the absorbed electromagnetic energy into thermal energy diffused from the inside out. In this way, the content of the toxic substances in the sludge can be lower than the legal standard to achieve the goal of recycling. (ii) In addition, blending detoxified sludge with agricultural waste to produce derived fuels can provide fuels in the industries, and completely recycle the troublesome sludge waste while solving the follow-up treatment of agricultural waste. It can achieve the best effect of waste resource reuse and the purpose of a circular economy.

2. Materials and Methods

2.1. Experimental Materials

The oily sludge used in this study was the hazardous sludge from the crude oil tank in the refining industry. The sludge was pre-treated by microwave to make its benzene content under the legal standard, which is 0.5 mg/L set by the Environmental Protection Administration (EPA) in Taiwan, and reduce the water content and viscosity before being mixed with agricultural waste mushroom substrates to make RDF. The mushroom substrates were mixed well and sampled by quarter method to dry in batches to make the water content less than 10%.

The microwave oven (SAMPO Co.) was equipped with a proportional integral derivative (PID) to control the output power. The microwave frequency was 2.45 GHz and the maximum output power was 750 W. A 50 mL quartz reactor with 20 holes at the bottom was also used. The sludge sample (10 g, semi-liquid, water content 18 wt%, heat value 10,968 cal/g) was placed in the reactor then put into the microwave oven (Figure 1).

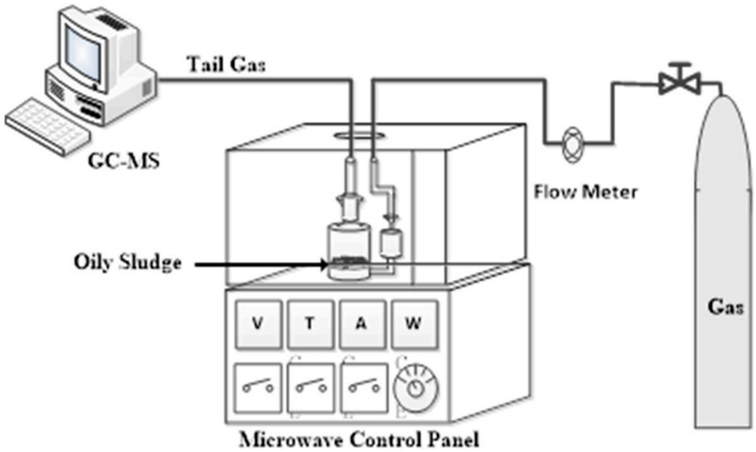

Figure 1. Experimental equipment.

2.2. Experimental Methods

A layer of insulation cotton (about 2 mm thick) was laid on the bottom of the reaction apparatus to avoid blockage of the air venting from the oily sludge. Sludge (10 g) was evenly spread on the insulating cotton, and the reactor was placed in the microwave oven. The flow rate of the gas (nitrogen) introduced into the bottom was maintained at 10 mL/min. The main purpose is to make the gas flow inside the sludge during the experiment, which is beneficial to the gas analysis. The microwave condition was as follows: the microwave power was set to 300 W with an irradiation time of 10 s, a stop time of 10 s, and a total irradiation time of 900 s to calculate the removal rate of BTEX in the sludge. The mixing ratios (*wt*/*wt*) of oily sludge and waste mushroom substrates were 8:2 (O8M2), 7:3 (O7M3), 6:4 (O6M4), and 5:5 (O5M5), respectively.

The steps of producing RDF are as follows: (1.) Detoxification of the oil sludge by using microwave treatment: A layer of 2 mm insulation cotton was laid at the bottom of the reaction bottle to prevent sludge from blocking the ventilation holes. Then, 10 g of sludge was evenly spread on the insulation cotton and placed in a microwave oven with the gas (nitrogen) introduced into the bottom by the flow rate of 10 mL/min. The main purpose was to make the gas flow inside the sludge during the experiment, which was beneficial to the gas analysis. Microwave conditions: microwave power 300 W, irradiation time 10 s, stop time 10 s, total irradiation time 900 s. The removal rate of BTEX in sludge was calculated. (2.) Drying of waste mushroom substrates: 10 g of mushroom substrates was placed in a 100 °C oven for 30 min. (3.) Mixing to produce RDF: In order to mix the samples evenly, the blending temperature was maintained at 70 °C. The blending ratios (*wt*/*wt*) of oil sludge and waste mushroom substrates were 8:2 (O8:M2), 7:3(O7:M3), 6:4(O6:M4), and 5:5(O5:M5), respectively.

On the other hand, the oily sludge was mixed with waste mushroom substrates with different proportions to produce RDF. The analysis of three components (moisture, combustible content, and ash) and the heat value was conducted. The change in heat value per unit volume and energy yield were calculated to evaluate the energy value and to analyze the heavy metal content in the ash after RDF combustion.

2.3. Analyses

Two grams of oil sludge was put into a 50 mL Teflon centrifuge tube followed by adding 10 mL of n-hexane and anhydrous sodium sulfate (0.5 g), sulfuric acid (1 mL), and saturated brine (2 mL) in sequence. After that, extraction was carried out by an ultrasonic vibration machine, and the BTEX in the sludge was dissolved in n-hexane by ultrasonic vibration. After centrifugation, the supernatant was obtained and put into a 2 mL brown sampling bottle and another 2 μL was obtained for material analysis by GC/MS. The concentration of BTEX was calculated by comparing the calibration curve and ion fragments.

An HP 6890 gas chromatography (GC) equipped with a capillary column (HP-5MS) and coupled with an HP 5973 mass selective detector (MSD), was used for identifying and quantifying intermediates and final products. The carrying gas (He) flow rate was maintained constant at 10 mL/min. The oven temperature was programmed to vary from 100 °C to 280 °C at a ramp rate of 20 °C/min; it was then held at 280 °C for 10 min. A thermocouple, K-Type with error 0.3% of full scale, was used for temperature measurement. The heat value was according to NIEA R214.01C for heat value; the heavy metal content analysis was based on NIEA R317.11C and NIEA R201.15C methods; the three components (moisture, combustible content, and ash) analysis was based on the NIEA R205.1C method.

On the other hand, for combustion characteristics analysis, thermogravimetric analysis (TGA) of oily sludge, waste mushroom substrates, and RDF was performed with a high-performance simultaneous thermogravimetric enthalpy analyzer (TGA/DSC). The reaction gas (O_2) flow was set at 50 mL/min, the protective gas (N_2) flow was set at 20 mL/min, the heating rate was 20 °C/min, and the maximum temperature was 800 °C.

3. Results and Discussion

3.1. BTEX Removal from Oil Sludge through Microwave Induction

The sludge absorbs MW energy and penetrates into the sludge under microwave irradiation. Through the interaction between the sludge constituent molecules and the electromagnetic field, the generated electromagnetic energy is converted into heat energy for diffusion, and the sludge temperature rises to decompose the heavy hydrocarbons into light hydrocarbons. In addition, due to the fact that microwave heating generates hot zones, the alkyl side chains of alkanes and aromatic hydrocarbons are destroyed to result in cracking, while the composition is physically volatilized [15]. Among them, polar substances (heavy chain molecules) can better absorb microwave energy, thereby destroying the structure of a low energy chain (C-H). The degree of cracking depends on the heat generated in the microwave process. For sludge, the dielectric loss of the water phase is higher than that of the oil phase. It can absorb more microwave energy to expand the water molecules and thin the oil/water interface film, thereby separating the oil and water [16]. Therefore, the main mechanism of sludge receiving microwave energy is a thermal effect rather than non-thermal effect. The results show that the microwave power was set 300 W with the 10 s irradiation, and the interval was 10 s per cycle. A total of 90 cycles with 900 s irradiation time was conducted. The removal rates of benzene, toluene, ethylbenzene, and xylene were 90.3%, 59.2%, 48.2%, and 27.4%, respectively.

3.2. Substance Analysis of Oily Sludge and Waste Mushroom Substrates

The substance analysis of oily sludge and waste mushroom substrates are summarized in Table 1. The heat value of sludge is from the composition of hydrocarbons which are flammable. Therefore, the heat value of sludge will increase as the flammable composition increases. Microwave treatment of sludge can effectively destroy the water-in-oil emulsion in sludge, and make its water volatilize during the microwave process. Microwaves also reduce the lighter hydrocarbons in the sludge through the rise of temperature while increasing the calorific value. The results show that after the microwave treatment of sludge, the water content was reduced by 50% while the weight per unit volume was reduced and the heat value of sludge was increased. Meanwhile, ash of the waste mushroom substrates was higher than 6%. Therefore, when using oily sludge to produce RDF, the mixing ratio of the waste mushroom substrates should not be too high to avoid making excessive bottom ash after combustion and increasing the final disposal cost.

Table 1. Substance analysis of oily sludge and waste mushroom substrates.

	Sludge before MW Treatment	Sludge after MW Treatment	Waste Mushroom Substrates
Moisture (%)	17.58	8.04	9.07
Ash (%)	1.65	2.72	6.24
Combustible content (%)	80.77	89.24	84.69
Heat value (HHV) (cal/g)	9044	10,277	4173
Unit volume weight (g/cm^3)	0.95	0.94	0.21

3.3. Producing RDF with Different Proportions of Oily Sludge and Waste Mushroom Substrates

The oily sludge with high heat value has the advantage of producing RDF. However, it is not easy to ignite. Therefore, adding waste mushroom substrates with low heat value but easy to ignite can improve the combustion property and adjust the heat value. In this experiment, the microwave-treated oily sludge was mixed with waste mushroom substrates in the weight ratio of 8:2 (O8M2), 7:3 (O7M3), 6:4 (O6M4), and 5:5 (O5M5). Meanwhile, in order to make the samples well-mixed, it was heated to about 70 °C during the blending process. The results show that as the mixing ratio of waste mushroom substrates increased, the RDF became less coagulable, as shown in Figure 2.

Figure 2. Appearances of RDF.

Table 2 summarizes the substance analysis of RDF produced by oily sludge and waste mushroom substrates. The moisture and ash of waste mushroom substrates are higher than those of oily sludge while the combustible content is lower than that of oily sludge. The results show that as the ratio of waste mushroom substrates increased, so did the moisture and ash of RDF. However, the combustible content of RDF decreased. On the other hand, as the ratio of waste mushroom substrates increased, the heat value and unit volume weight also decreased. Although the calorific value decreased (9176 vs. 7279 cal/g) as the proportion of oil sludge and mushroom substrates (*wt*/*wt*) increased from 8:2 to 5:5, it was still higher than the calorific value of bituminous coal (5000~5500 cal/g).

Table 2. Substance analysis of RDF produced by oily sludge and waste mushroom substrates.

	Mixing Ratio (W/W)			
	O8M2	**O7M3**	**O6M4**	**O5M5**
Moisture (%)	8.52	8.47	8.78	8.59
Ash (%)	4.05	4.16	4.44	4.66
Combustible content (%)	87.43	87.37	86.78	86.75
Heat value (HHV) (cal/g)	9176	8,452	7871	7279
Unit volume weight (g/cm^3)	0.90	0.84	0.71	0.54

3.4. Combustion Property Analysis of RDF Produced by Oily Sludge and Waste Mushroom Substrates

In order to evaluate combustion property of RDF, ignition index (D_i) and comprehensive performance index (S) is used as a criterion and based on Equations (2) and (3) [11–13,17–19].

$$Di = DTGmax/(T_i \times T_p) \tag{2}$$

where DTG_{max} (%/min) is the maximum weight loss rate, T_i (°C) is the ignition temperature, and T_p (°C) is the corresponding temperature. D_i reflects the level of difficulty and speed the fuel is ignited.

$$S = DTGmax \times DTGmean/({T_i}^2 \times T_b) \tag{3}$$

where DTG_{mean} (%/min) is the average weight loss rate, and T_b (°C) is the temperature at 98% total mass loss defined as the burnout temperature. It is generally considered that the higher the value of S, the higher the reactivity of fuels and the more intense the combustion.

The combustion property analysis of RDF is summarized in Table 3. The results show that as the waste mushroom substrates increased, the combustion property of RDF also increased, meaning that waste mushroom substrates can improve the ignition index of oily sludge. The more the waste mushroom substrates were added, the more the ignition index was increased. On the other hand, the comprehensive performance index showed higher reactivity of the RDF and more intense combustion as the waste mushroom substrates increased.

Table 3. Combustion property of RDF produced by oily sludge and waste mushroom substrates.

	Mixing Ratio (W/W)			
	O8M2	**O7M3**	**O6M4**	**O5M5**
DTG_{max} (%/min)	8.14	8.05	8.07	9.23
DTG_{mean} (%/min)	2.50	2.47	2.46	2.47
T_i (°C)	284.85	275.33	271.39	258.84
T_p (°C)	427.67	418.00	357.67	351.33
T_b (°C)	591.45	571.57	569.40	534.24
D_i (10^{-5})	6.68	6.99	8.31	10.15
S (10^{-7})	4.23	4.59	4.73	6.37

3.5. Heavy Metal Analysis of RDF Produced by Oily Sludge and Waste Mushroom Substrates

After high temperature (800 °C) combustion on producing RDF, the residual ash content of heavy metals analyzed is summarized in Table 4. The waste mushroom substrates contained heavy metals such as selenium, chromium, lead, and barium, which came from the fertilizers added in the mushroom substrates. The results showed that the concentration of heavy metals contained in the RDF produced by the blending ratio of oil sludge and waste mushroom substrates, O7M3 and O6M4, all met the standards set in the regulations.

(The heavy metal concentration standards for Hg, Se, Cr, Pb, Cu, and Ba are 0.2, 1.0, 5.0, 5.0, 15, and 100 ppm, respectively, according to the Taiwan Environmental Protection Agency.)

Table 4. Heavy metal analysis of RDF produced by oily sludge and waste mushroom substrates.

Heavy Metal (ppm)	Oily Sludge	Waste Mushroom Substrates	Mixing Ratio (W/W)			
			O8M2	O7M3	O6M4	O5M5
Hg	0.21	N.D.	0.20	0.17	N.D.	N.D.
Se	N.D.	1.07	0.60	0.76	0.79	0.86
Cr	N.D.	3.12	2.17	2.31	2.66	3.04
Pb	0.67	0.37	N.D.	N.D.	N.D.	N.D.
Cu	0.47	N.D.	N.D.	N.D.	N.D.	N.D.
Ba	5.45	1.20	0.59	0.64	0.77	1.05

The results of analyzing and comparing various data of producing RDF by different blending ratios of oil sludge and mushroom substrates showed that all RDF products produced by different blending ratios had high calorific value, low moisture, and ash content. Considering unit volume weight, ignition index, comprehensive combustion characteristic index, and TCLP analysis: the blending ratio O8M2 had better performance only in terms of unit volume (0.90 g/cm^3). The low ignition index made it difficult to ignite, and the overall combustion performance was poor. The concentration of mercury dissolved in the ash after incineration exceeded the legal standard. The ignition index and comprehensive characteristic combustion index of the blending ratio O6M4 were improved compared with that of the blending ratios O8M2 and O7M3. Meanwhile, the concentration of heavy metals in the ash after incineration was in line with the regulatory standards. Although the blending ratio O5M5 had the best ignition index and combustion characteristic index, its unit volume weight was the lowest. When the unit volume weight was low, it greatly increased the space required for future transportation and storage. In addition, the elution concentration of selenium in ash exceeded the legal standard after incineration. In short, based on the various analyses mentioned above, including heat value, combustion property, and heavy metal analysis, the best mixing ratio of using oily sludge and waste mushroom substrates to produce RDF was O6M4.

4. Conclusions

In this study, the oil sludge from the refining industry was combined with waste mushroom substrates to produce refuse derived fuel (RDF). The results showed that the calorific value decreased (9176 vs. 7279 cal/g) as the proportion (*wt*/*wt*) of oil sludge and mushroom substrates increased from 8:2 to 5:5. However, it was still higher than the calorific value of bituminous coal (5000~5500 cal/g). On the other hand, when the portion of mushroom substrates was increased, the sludge became easier to ignite with better combustion reaction. The results showed that when the blending ratio of oil sludge and waste mushroom substrates increased from 8:2 to 5:5, the ignition index and comprehensive performance index of the production of RDF were 6.68×10^{-5} vs. 10.15×10^{-5} and 4.23×10^{-7} vs. 6.37×10^{-7}. In addition, the benzene content concentration for the produced RDF was lower than the standards set by the Taiwan Environmental Protection Agency. Therefore, mixing oily sludge with agricultural waste to produce RDF can fully recycle the troublesome oily sludge as well as solve the problem of agricultural waste treatment to achieve the best effect of waste resource reuse and the purpose of circular economy.

Author Contributions: The first author (C.L.L.) was responsible for performing the data analysis and curation of the oily sludge, carrying out literature searches and collation, and providing valuable information on refinery sludge composition and treatment, including the preparation of refuse derived fuel. He wrote the Introduction, Materials and Methods, and Reference sections of this study. The second author (C.-J.G.J.) wrote Sections 3 and 4 of the manuscript as well as coordinating and directing the research structure. All authors have read and agreed to the published version of the manuscript.

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