Pyrolysis Characteristics of Indonesian Oil Sand in a Fixed Bed
Chunxia Jia, Hao Yu, Hongpeng Liu,* Hong Qin, and Qing Wang

ABSTRACT: In this research, pyrolysis experiment of Indonesian oil sand was performed in a fixed bed. Results show that the temperature and heating rate had significant effects on the pyrolysis products. Data studied include yields of products and the gaseous product composition of carbon monoxide, hydrogen, methane, and other hydrocarbons. Four functional group bands (aromatic hydrocarbon, oxygen, aliphatic, and hydroxyl functional groups) are observed from Fourier transform infrared spectroscopy of oil sand. The influence of pyrolysis temperature on the total pore volume and the Brunauer–Emmett–Teller (BET) specific surface area of char was also studied. With increasing temperature, the BET specific surface area showed an increasing trend in the overall temperature range. The specific surface area and the total pore volume showed similar trends except the tiny difference of 5 °C/min from 200 to 300 °C. The heating rate did not influence the development of the pore structure obviously. Mainly, three different reaction regions were observed during pyrolysis of oil sand. This study provides a theoretical foundation for further effective exploitation and economical application of oil sand.

1. INTRODUCTION
Soaring oil demand has led many countries to seek alternative sources of energy. Oil sand is an unconventional oil resource; it is mainly used to extract tar sands to produce synthetic crude oil. Organic solvent extraction was the traditional separation process used to recover bitumen from the inorganic sand matrix. The production efficiency of this method is relatively high, and it is suitable for extracting oil sand from oil–water emulsions. Also, the extracted asphalt is an oil–water emulsion and polluted with fine clay particles.1 Pyrolysis method is used for the separation of oil and sand, which can improve the quality of oil; pyrolysis directly produces dry tailing and is environmentally friendly; it can avoid consuming a large amount of water resources and can effectively use oil sand resources, which will greatly improve the economic value of oil sand industry. Therefore, pyrolysis has great potential for development. If it can be applied on a large scale, it will be of great significance to alleviate the shortage of natural oil supply. Hence, it is necessary to study the thermal conversion of oil sands; also, it is important to analyze the organic matter precipitation and oil-forming characteristics.

Much of research on oil sand pyrolysis was conducted with thermogravimetric analysis,2–8 and the thermal behavior of oil sands has also been studied.9–11 Oil sand pyrolysis products include evolved liquids, gases, and char contained in a carbonaceous residue. Pyrolysis process development studies can provide data on the influence of experimental conditions, product distribution, product yields, and qualities of the oil products. The distribution characteristics of the pyrolysis products are connected with the type of materials present in the oil sand and the experimental conditions, such as temperature, atmosphere, particle size and so forth. The volatile released during the pyrolysis process of Indonesian oil sands was studied by the author’s group using the Fourier transform infrared (FTIR) method. Various functional groups
of oil sand and coke were obtained from the FTIR curves.\textsuperscript{12} Liu et al.\textsuperscript{13} studied the pyrolysis characteristics of Indonesian oil sand in a thermogravimetric analyzer and then in a fixed bed reactor. The effect of temperature on the thermal behavior of pyrolysis liquid, gas, and coke was studied. The components of pyrolysis gas and their properties were determined. Fletcher et al.\textsuperscript{14} determined the effect of residence time, gas flow rate, and gas composition on the product properties. The product oils produced in pyrolysis reactors were much more upgraded than the bitumen extracted directly from oil sand. Gishler\textsuperscript{15} applied the fluidization technique to study the distillation of oil from Alberta bituminous sand, a 76% oil recovery was obtained at 500 °C, and the yield of oil decreased above 500 °C. Gao et al.\textsuperscript{16} studied the reaction characteristics of Monglia oil sand in a fluidized bed. The influence of the reaction temperature and time on product distribution and yields was examined.

The objective of this work is to characterize the products produced by pyrolysis of Indonesian oil sand through a fixed bed. The aim is to determine the influence of temperature and heating rate on product distribution and yields. In addition, in order to obtain a better understanding of the evolution of volatiles and the sample’s internal structure, variations in the char pore structure were studied.

2. EXPERIMENTAL PROCEDURES

2.1. Sample Material. The oil sand comes from Indonesia. The fuel properties of oil sand include proximate and ultimate analysis. Table 1 presents the characteristics of the oil sand.

2.2. FTIR Spectroscopy. FTIR spectroscopy experiment for oil sand was carried out on an infrared spectrometer (VERTEX 70, Bruker, Germany). The infrared spectra were recorded between wave numbers 400 and 4000 cm\(^{-1}\), and the baseline was corrected.

2.3. Pyrolysis System. The whole oil sand pyrolysis device includes a gas supply system, a fixed bed reactor, a bed heater and temperature control, a gas condensing and sample collecting system, and a data acquisition system. The reactor is made from a stainless-steel tube with an inner diameter of 50 mm and 1.2 m height. High-purity nitrogen was used, and the heating rates were 5, 10, and 20 °C/min. The fixed bed temperature was 100, 150, 200, 250, 300, 350, 400, 500 or 550, 600, and 700 °C. The pyrolysis gas from the reactor is purified and enters the cooling system. There is an oil collecting device at the bottom of the condenser. Then, the purified gas from condenser goes through a flow meter and enters a gas collecting device. The volume of gas was measured by a gas flow meter, and the gas composition was analyzed by a gas chromatograph (GC-9560-HQ). The pore structure of the char was measured on a Gemini2380 micropore analyzer.

3. RESULTS AND DISCUSSION

3.1. FTIR Analysis and Functional Groups of Oil Sand. FTIR spectroscopy provides an efficient method for determining the functional groups in oil sand. The absorption peaks at about 700–900 cm\(^{-1}\) are due to the aromatic stretching bonds. The bands at 800–1800 cm\(^{-1}\) reflect the presence of oxygenated functional groups in the oil sand. The peaks for aliphatic stretching bonds are at 2800–3000 and 3200–3600 cm\(^{-1}\), which are considered to be the absorption peaks for hydroxyl functional groups. FTIR curve for every functional group was effectively separated into multiple peaks, as shown in Figure 1.

![FTIR spectrum from oil sand at 4000–400 cm\(^{-1}\) and multipeak fitting for (a) aromatic hydrocarbon functional groups at 900–700 cm\(^{-1}\), (b) oxygenic groups at 1800–1000 cm\(^{-1}\), (c) aliphatic groups at 3000–2800 cm\(^{-1}\), and (d) hydroxyl groups at 3600–3000 cm\(^{-1}\).](https://doi.org/10.1021/acsomega.2c01479)

Figure 1. FTIR spectrum from oil sand at 4000–400 cm\(^{-1}\) and multipeak fitting for (a) aromatic hydrocarbon functional groups at 900–700 cm\(^{-1}\), (b) oxygenic groups at 1800–1000 cm\(^{-1}\), (c) aliphatic groups at 3000–2800 cm\(^{-1}\), and (d) hydroxyl groups at 3600–3000 cm\(^{-1}\).
The absorption peak at 1448 cm\(^{-1}\) has the maximum width and area. It is attributed to the stretching vibration of ether C–O and hydroxyl benzene. It is followed by the peak for symmetric bending vibration of –CH\(_2\). The absorption peaks at 1033 cm\(^{-1}\) are caused by the C–O–C vibration in Ar–O–Ar, but this peak may also in part be from the Si–O vibration absorption peak of silicate minerals (including quartz and clay minerals).

3.1.3. Aliphatic Groups (2800–3000 cm\(^{-1}\)). Figure 1d shows the peak fitting for the aliphatic groups. A strong peak near 2920 cm\(^{-1}\) is caused by the –CH\(_3\) stretching vibration of the cycloalkane and aliphatic hydrocarbons. Another strong peak near 2860 cm\(^{-1}\) is attributed to the –CH\(_2\) stretching vibration of cycloalkane or aliphatic hydrocarbons. The absorption peak at 2900 cm\(^{-1}\) is due to the CH vibration of aliphatic hydrocarbons which provides an important parameter for further research on the structure of oil sands because the proportion of aromatic hydrocarbon to lipid hydrogen reflects the main structure of the macromolecules in the oil sand. The band at 2800–3000 cm\(^{-1}\) is due to the CH\(_2\) stretching vibration of the aliphatic functional groups. The fitting parameters for the aliphatic group FTIR peaks are shown in Table 4.

### Table 2. Fitting Parameters for Aromatic Group Peaks in the FTIR Spectrum From Oil Sand

| peak number | location/cm\(^{-1}\) | breadth/cm\(^{-1}\) | height/cm\(^{-1}\) | attribution                                                                 | area proportion/% |
|-------------|----------------------|--------------------|---------------------|-------------------------------------------------------------------------------|-------------------|
| 1           | 711.47               | 7.41               | 0.13                | single substituted aromatic hydrocarbon (five hydrogen atoms)                  | 9.30              |
| 2           | 795.22               | 47.66              | 0.03                | ortho second substituted aromatic hydrocarbon (four hydrogen atoms)            | 13.64             |
| 3           | 872.60               | 47.20              | 0.11                | benzene ring fifth substituted aromatic hydrocarbon (one hydrogen atom)        | 51.58             |
| 4           | 875.05               | 10.18              | 0.25                | benzene ring fifth substituted aromatic hydrocarbon (one hydrogen atom)        | 35.48             |

3.1.1. Aromatic Hydrocarbon Functional Groups (700–900 cm\(^{-1}\)). Four peaks fitted from the infrared spectrum of aromatic hydrocarbon functional groups are shown in Figure 1a. The absorption peaks at 700–900 cm\(^{-1}\) are due to the aromatic stretching bonds, a wide band attributed to the aromatic C–H surface bending vibration. The peaks of aromatic C–H bond stretching bonds at 850–921 cm\(^{-1}\) are considered to be the absorption peak of outside surface deformation vibration (two adjacent hydrogen atoms). The peaks at 730 and 670–780 cm\(^{-1}\) are attributed to the aromatic C–H external surface deformation vibration (four to five adjacent hydrogen atoms). The absorption band depends on the substitutions on benzene. The peak parameters for the aromatic group absorption peaks in this multipoint fitting for this oil sand are shown in Table 2. The bands at 795.22 and 711.47 cm\(^{-1}\) are caused by a second substituted aromatic and a single substituted aromatic, respectively. The benzene ring fifth substituted aromatic has the maximum peak width and area.

3.1.2. Oxygenic Groups (1000–1800 cm\(^{-1}\)). For the oxygenic groups, four peaks are observed according to the second derivative of the infrared spectrum; the multipoint fitting results for this spectrum are shown in Figure 1b. Oxygenic groups are divided into carbonyl, carboxyl, hydroxyl, and ether oxygen. The infrared absorption band at 1000–1800 cm\(^{-1}\) is mainly attributable to oxygen-containing functional groups, such as C–O–R and C=O (including hydroxyl, ether, etc.), but also includes the deformation vibration of CH\(_2\), CH\(_3\), and aromatic C=C. The multipoint fitting parameters for the oxygenic groups in the FTIR spectrum are shown in Table 3.

### Table 3. Fitting Parameters for an Oxygenic Group in the FTIR Spectrum From Oil Sand

| peak number | location/cm\(^{-1}\) | breadth/cm\(^{-1}\) | height/cm\(^{-1}\) | attribution                                                                 | area proportion/% |
|-------------|----------------------|--------------------|---------------------|-------------------------------------------------------------------------------|-------------------|
| 1           | 1033                 | 66.43              | 0.09                | C–O–C vibration of Ar–OC, r–O–Ar                                            | 3.35              |
| 2           | 1091.6               | 194.56             | 0.13                | C–O stretching vibration of secondary alcohol, ether                          | 14.12             |
| 3           | 1439.1               | 77.53              | 0.30                | CH\(_2\) symmetric bending vibration                                          | 33.44             |
| 4           | 1448                 | 265.37             | 0.33                | C–O stretching vibration of hydroxyl benzene, ether                          | 46.78             |

### Table 4. Multipeak Fitting Parameters of the Aliphatic Group in the FTIR Spectra for Oil Sand

| peak number | location/cm\(^{-1}\) | breadth/cm\(^{-1}\) | height/cm\(^{-1}\) | attribution                                                                 | area proportion/% |
|-------------|----------------------|--------------------|---------------------|-------------------------------------------------------------------------------|-------------------|
| 1           | 2850.49              | 11.56              | 0.006               | symmetric R\(_2\)CH\(_2\)                                                    | 1.73              |
| 2           | 2868.49              | 48.65              | 0.031               | symmetric R\(_2\)CH\(_2\)                                                    | 42.21             |
| 3           | 2923.06              | 30.97              | 0.034               | antisymmetric R\(_2\)CH\(_2\)                                               | 29.00             |
| 4           | 2965.31              | 39.50              | 0.025               | antisymmetric RCH\(_2\)                                                     | 27.06             |

The peak-fitting routine applied to the second derivative of the hydroxyl group infrared spectrum defines three peaks, as shown in Figure 1d. As the main functional group for hydrogen bonds, hydroxyl is closely related to the pyrolysis of oil sands, so it affects all main functional groups of the oil sands. The absorption band at 3300–3600 cm\(^{-1}\) is attributed to the hydroxyl groups. The absorption peaks for the hydroxyl group partial peak fitting are shown in Figure 1d. The multipoint fitting parameters are shown in Table 6.

### Table 5. Band Assignments for the Hydroxyl Groups in the FTIR Spectrum

| hydrogen bond types       | absorption peak position/cm\(^{-1}\) |
|---------------------------|--------------------------------------|
| hydroxyl–π                | 3516                                 |
| self-association hydroxyl | 3400                                 |
| ether oxygen of hydroxyl  | 3300                                 |
| closely associated hydroxyl ring | 3200                              |
| hydroxy–N                 | 3100–2800                            |

Hydroxyl groups include hydroxyl ether oxygen bonds, self-association hydroxyl, and hydroxyl–π hydrogen bonds, as shown in Table 6. A few free hydroxyl groups are caused by the presence of water. There are many hydroxyl–π hydrogen
The hydroxyl functional groups are much more abundant than the other three functional groups.

### 3.2. Pyrolysis of Oil Sand in a Fixed Bed

The oil sand pyrolysis experiments were conducted in a fixed bed to investigate the effects of temperature on product conversion, gas and oil yields, and components. The influences of pyrolysis temperature and heating rate on the products are shown below in Figure 2.

The overall trend shows that the liquid yields and gas products increase with higher temperatures; however, the char yield decreases. There is an initial increase in the liquid yield but a decrease at a temperature of 400 °C and then an increase after 500 °C. There is a faster increase stage at temperatures 300–450 °C, where the majority of volatiles were emitted, and a slow increase stage in the range of 450–700 °C. There is an increase in the secondary cracking reactions of the volatile at higher temperatures that contributes to the decomposition of organic components into smaller components. In that case, more gas products were released from the produce of thermal cracking volatiles during the pyrolysis process. The higher weight loss of the solid yield from 300 to 450 °C is caused by the initial large amount of volatile evolution with increasing temperature.

The gas and liquid product yields also increase with an increased heating rate as char production decreases. At higher heating rates, the pyrolysis reactions in the oil sands are accelerated, and thus the products produced by those reactions increase. The reason for this behavior is mainly because of two competitive reactions: tar cracking and char production.

At a heating rate of 5 °C/min, the liquid product yield increases with the pyrolysis temperature in the range of 200–500 °C and reaches its maximum of 18% at 500 °C. The liquid yield decreases to 15% at 600 °C before reaching 18% at 650 °C. Qing et al. described the chemistry of the oil produced by pyrolysis of this Indonesian oil sand at different temperatures. The heating rate and the composition of the oil change with variations in the heating profile. The data presented here show that gas yields increase with the pyrolysis temperature up to 350 °C and then decrease slightly before continuing to increase, finally reaching 12% at 700 °C. In contrast to the oil and the gas, char production decreases with increasing temperature but begins to increase at temperatures over 500 °C. The char yield of 80% is achieved at 600 °C. The oil yield reaches a maximum at 450 °C at a heating rate of 10 °C/min and a maximum at 650 °C with a 20 °C/min heating rate. The gas yield reaches its maximum value at 700 °C as does the yield for char.

#### 3.2.1. Composition of the Gas Product

The effects of temperature on the gas composition are shown in Figure 3.

| peak number | location/cm⁻¹ | breadth/cm⁻¹ | height/cm⁻¹ | attribution | area proportion/% |
|-------------|----------------|--------------|--------------|-------------|-------------------|
| 1           | 3291.05        | 189.71       | 0.049        | hydroxyl ether oxygen bond | 31.66            |
| 2           | 3404.48        | 115.51       | 0.072        | self-association hydroxy   | 28.23            |
| 3           | 3515.38        | 123.63       | 0.097        | π hydrogen bond of hydroxyl | 40.11            |

**Figure 2.** Pyrolysis product yields from oil sand in a fixed bed at different temperatures and for different heating rates of 5, 10, and 20 °C/min. (a) Liquid product yield; (b) solid product yield; (c) gas product yield.
shows that temperature has a significant influence on the gas composition. Because the carbonyl group starts to break down at 200 °C, and in the oil sands, the volume fractions of CO and CO are associated with carbonyl, at temperatures 200–250 °C, the volume fraction of CO and CO has a sharp decline, but the overall content represents a downward trend when the temperature increases. The O2 content first represents a growth trend and begins to decline after reaching a maximum at 400 °C. The oil sand bitumen contains carbon (about 80%) and small amounts of nitrogen, sulfur, and oxygen (plus a few trace elements), so the gases produced are mainly CO, CO2, H2, and hydrocarbons.

During pyrolysis, the CH4 content decreases from 48% at 200 °C, reaches a minimum of about 30% at 250 °C, and then increases to about 43% at 500 °C. The volume fraction of CO at temperatures 200–250 °C shows a dramatically sharp decrease and decreases to a minimum value at 450 °C; then, both in the ranges of 250–400 and 450–500 °C, the CO volume fraction has a slight increase with increasing temperature but represents a decreasing trend in the overall temperature range. The yield of O2 increases first and then decreases with a temperature increase, reaching a maximum of about 1.3% at 400 °C and then decreasing to about 0.54% at 500 °C. Hydrogen mainly comes from polycondensation. The volume percentage of H2 yield decreases from 32.5% at 200 °C to a minimum of about 20% at 400 °C and then increases to about 32% at 500 °C.

The volume fractions of C2+ hydrocarbon gases are relatively stable in the 250–450 °C range. Carbon dioxide is mainly associated with carboxyl functional groups. In addition, at a high temperature, secondary decomposition of C2+ hydrocarbon gas occurs readily. Carbon monoxide is mainly generated from the fracture decomposition of carbonyl and ether bonds; carbonyl starts to break down at 400 °C, but ether bonds only break above 700 °C. Thus, for the temperature range investigated by this study, the pyrolysis gas yield increases continuously with increasing temperature. Because the volume fractions of CO2 and C2+ hydrocarbon gases are reduced in the pyrolysis gas. The CO evolution at low temperatures was ascribed to the break of C–O–C and C=O functional groups, such as carbonyl and ether groups, while CO produced in the high temperature stage was the result of the reduction reaction of CO2 and nascent coke.18,19

However the change in the volume fractions is not obvious. Methane is generated by the decomposition of alkyl groups, char polycondensation, secondary reaction of tar, and hydrogenation reactions of free radicals and volatiles. With increasing pyrolysis temperatures, the CH4 yield increases. The rate of CH4 increase is greater than that of the total pyrolysis gas, so the volume fraction of CH4 in the pyrolysis gas increases.

3.2.2. Influence of Pyrolysis Temperature on the Specific Surface Area and Pore Structure in Char. The influence of temperature on the total pore volume and the Brunauer–Emmett–Teller (BET) specific surface area of char is shown in Figure 4. The pores in the char are relatively small and their distribution is relatively uniform until the temperature exceeds 400 °C. Above that temperature, the pore structure becomes more complicated and the pores increase in size and number. With increasing temperature, the BET specific surface area represents an increasing trend in the overall temperature range. The specific surface area increases rapidly in the 300–450 °C range because at those temperatures begin the decomposition of organic matter and that releases a large amount of oil vapor and gas to increase the pore specific surface area. At about 550 °C, the specific surface area shows a downward trend because less oil vapor and gas are generated at this temperature.20,25 Above 600 °C, the specific surface area increases again. The total pore volume and the total BET specific surface area show

Figure 3. Content of gas product composition during the pyrolysis process of oil sand in a fixed bed at different pyrolysis temperatures.

Figure 4. Influence of temperature on the total pore volume and the BET specific surface area from N2 adsorption for oil sand chars. (a) Graph of final pyrolysis temperature vs. char pore volume and (b) graph of final pyrolysis temperature vs char BET specific surface area.
similar trends during pyrolysis. Micropores contribute more to the BET specific surface area than the mesopores and macropores but constitute less of the total pore volume. Thus, it can be concluded that the microporous structure does not change obviously during pyrolysis; the mesopores and macropores develop mainly from the decomposition of asphaltene.

The influence of heating rate on pore structure development is not obvious. The specific surface area for heating at 5 °C/min is larger than the value for heating at 20 °C/min in the 500–600 °C range. In the high-temperature section after 500 °C, the heating rate is too fast to make the actual temperature not reach the target temperature, and there is much hydrogen-rich cross-linking removal at a lower heating rate than that at a higher heating rate. The other reason is that the obturator effect caused by the thermoplastic material is slowly removed. The molecular chains in thermoplastics are linear or branched, and there is no chemical bond between the molecular chains. When the thermoplastic material is heated, the molecular chains soften and flow, and the internal pores become smaller or even closed. According to the literature, oil shale would form some less porous thermost in the heating process. Because the organic matter of oil shale and oil sand are similar, we conclude that the actual temperature of oil sand at the higher heating rate cannot reach the target temperature. In that case, thermoplastic material is removed slowly, so the specific surface area of 5 °C/min is larger than that of 20 °C/min.

4. CONCLUSIONS

Four functional group bands are observed from FTIR spectroscopy of oil sand. The absorption peaks at about 700–900 cm−1 are attributed to the aromatic stretching bonds. The bands at 800–1800 cm−1 reflect the presence of oxygenated functional groups. The peaks for aliphatic stretching bonds are at 2800–3000 and 3200–3600 cm−1, which are considered to be the absorption peaks for hydroxyl functional groups. For all pyrolysis products, char had the largest yield in all temperature ranges. Liquid and gas yields increase with increasing temperature and heating rate, but chars decrease as the heating rate increases. During pyrolysis, the volume fraction of CO2 and CO represents a downward trend. The O2 content first presents a growth trend and then declines after 400 °C. The CH4 content reaches a minimum at 250 °C. The volume fraction of CO at temperatures 200–250 °C decreases dramatically and reaches a minimum value at 450 °C; the yield of O2 increases first and then decreases, reaching a maximum of about 1.3% at 400 °C. The volume percentage of H2 decreases from 32.5% at 200 °C to a minimum of about 20% at 400 °C and increases to about 32% at 500 °C. The effect of temperature on the total pore volume and the BET specific surface area of char is significant. With an increase in the final temperature, the specific surface area first increases but then decreases. The total pore volume and the BET specific surface area show similar trends during pyrolysis, but the structure of the micropores does not change.

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Notes

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