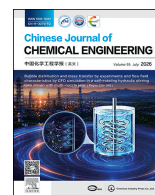




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Full Length Article

Impacts of drying treatment on pyrolysis of oily sludge: Kinetic analysis and product evaluation

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ABSTRACT

Drying pretreatment is a widely adopted preprocessing method prior to the pyrolysis of oily sludge (OS). In this study, a tube furnace, thermogravimetric–infrared spectroscopy (TG-IR), and gas chromatography–mass spectrometry (GC-MS) were employed to evaluate and compare the pyrolysis behaviors of original and dried OS, and to analyze product distributions under various conditions. The results indicate that both original and dried OS undergo pyrolysis in five distinct stages, among which stages 2, 3, and 4 are the most significant. Kinetic analysis reveals that dried OS exhibits lower activation energies of 56.032, 152.166, and 211.462 kJ·mol^{−1} for these three stages, corresponding to reaction mechanisms of D6 (3D diffusion), F4 (chemical reaction) and F4, respectively. In contrast, original OS exhibits higher activation energies of 81.481, 235.933, and 214.194 kJ·mol^{−1}, associated with the mechanisms A8 (random nucleation and growth), F4, and F1 (order of reaction), respectively. TG-IR analysis identifies H₂, CO₂, CO, and CH₄ as the primary gaseous products from dried OS, with the optimal methane-rich gas yield achieved at temperatures below 724.61 °C. Furthermore, GC-MS analysis of the pyrolysis oil obtained from dried OS at 600 °C reveals higher concentrations of hydrocarbons and oxygenated compounds, along with reduced levels of nitrogen-containing and hazardous constituents, suggesting its superior suitability and cost-effectiveness as a recyclable fuel.

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1. Introduction

Oily sludge (OS), a hazardous solid waste generated during petroleum extraction, production, transportation, storage, and refining [1,2], comprises hydrocarbons, water, inorganic solids, and chemical additives. It typically contains toxic polycyclic aromatic hydrocarbons (PAHs) such as benzene, phenols, naphthalene, and anthracene, many listed in *China's National List of Hazardous Wastes* [3,4]. Despite its environmental risks, OS possesses significant energy potential due to 20% to 30% (mass) recoverable petroleum constituents [5]; improper disposal thus causes both ecological damage and resource waste. Global OS generation exceeds 60 million tons annually [6], with China contributing >5 million tons per year—a figure rising with industrial expansion [7].

Pyrolysis technology is an effective method for the harmless treatment of OS, involving the conversion of heavy components in OS

into gases, tars, and other recoverable products through heating under an inert atmosphere [8,9]. In the pyrolysis process, the light components and water in OS are initially separated as the temperature rises. As the temperature continues to increase, heavy components such as macromolecular hydrocarbons begin to undergo pyrolysis, generating pyrolysis gas. This gas is then condensed to recover pyrolysis oil, while the non-condensable gases are directed to the recycling system and used as fuel for the pyrolysis reactor. The remaining solid material is coked and transformed into char, which is finally discharged from the pyrolysis equipment [10,11]. Gong *et al.* [12] investigated the distribution and composition of pyrolysis products from OS using a tube furnace reactor. The results showed that optimal organic matter recovery (>85% (mass)) occurs at around 600 °C and the recovered pyrolysis oil exhibits lower viscosity and higher H/C ratio. Several studies have reported that the calorific value of certain products derived from OS pyrolysis is even higher than that of the original OS, and that the pyrolysis oil can be recovered and utilized as a fuel [9,13,14].

In practical industrial application, however, due to the wide range of sources of OS, its highly variable moisture mass content (>70%) and heterogeneous composition, the traditional pyrolysis

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reactor cannot operate continuously for extended periods, so pre-drying of OS before pyrolysis has become a widely chosen option [13,15]. After prolonged accumulation and storage, moisture in the OS can facilitate the release of harmful substances into the atmosphere or allow them to seep into the surrounding soil, leading to serious secondary pollution. Drying treatment can quickly reduce the volume of OS and significantly lower the risk of secondary pollution during transportation, storage, and treatment [16,17]. Magdziarz and Werle [18] demonstrated *via* thermogravimetric analysis (TGA) that the primary pyrolysis of dried sludge occurs between 200 °C and 540 °C, yielding H₂, CO, CO₂, and CH₄ as dominant gaseous products. Gao *et al.* [19] investigated the pyrolysis process of dried sewage sludge from a micro perspective by thermogravimetric–Fourier transform infrared analysis (TG-FTIR) and differential scanning calorimetry (DSC). From the FTIR test results of the pyrolysis gas, it was observed that as the pyrolysis temperature increased from 450 °C to 650 °C, the functional groups changed significantly, and the concentration of pyrolysis gases increased steadily with the exception of CO₂, and secondary cracking of pyrolysis vapors occurred. Relevant studies have shown that the pyrolysis products of dried OS have high recycling value, and in order to improve the quality and yield of the products even further, it is necessary to study the reaction mechanism and kinetic behavior of the pyrolysis process of OS. Crucially, pyrolysis kinetics research serves as the cornerstone for unraveling the intricate reaction mechanisms of OS pyrolysis, as it quantifies key parameters such as activation energy (E_a) and pre-exponential factors (A), thereby elucidating the rate-controlling steps and decomposition pathways that govern product distribution and process efficiency. The research of Ali *et al.* [20] and Zhu *et al.* [21] reported complex mechanisms for dried OS pyrolysis, determining kinetic parameters (E_a , A , mechanism) *via* model-free and model-fitting methods. Nonetheless, a comprehensive kinetic study linking these parameters to product distribution for both original and dried OS remains lacking, creating a knowledge gap for industrial parameter optimization.

In this work, we employ thermogravimetric–infrared spectroscopy (TG-IR), gas chromatography–mass spectrometry (GC-MS), and tube furnace experiments to elucidate the critical role of reaction kinetics in optimizing OS pyrolysis. We demonstrate that drying pretreatment fundamentally reshapes the kinetic landscape: for original OS, stages 2, 3, and 4 follow A8 (random nucleation), F4 (third-order reaction), and F1 (first-order reaction) mechanisms with activation energies of 81.48–235.93 kJ·mol⁻¹; after drying, these shift to D6 (3D diffusion), F4 (chemical reaction), and F4 mechanisms, reducing activation energies in stages 2 and 3 by an average of 33.37%. This mechanistic transition—from nucleation-dominated to diffusion and chemical reaction control—not only accelerates reaction kinetics but also enhances product selectivity, as evidenced by TG-IR and GC-MS analyses: dried OS yields methane-rich gas (optimal below 724.61 °C) with 22% higher CH₄ concentration, while its pyrolysis oil at 600 °C contains 38% more hydrocarbons and oxygenates yet 60% lower nitrogen content *versus* original OS. Our findings establish the correlation between moisture-induced kinetic pathway evolution and product distribution in OS pyrolysis, providing a theoretical foundation for optimizing industrial reactor parameters to maximize fuel-grade oil/gas production while reducing operational costs.

2. Materials and Methods

2.1. Materials

A representative sample of OS provided by SINOPEC Jinling Petrochemical Company (China) was studied in this work, and the

OS was collected from the bottom of crude oil storage tanks. The collected OS was divided into two groups, one group was the original oil sludge (original OS), and the other group was treated by a vacuum disc dryer [15] (105 °C for 24–48 h to eliminate the moisture to ensure that its content was less than 10% [20]) to obtain the dried oil sludge (dried OS). Proximate and ultimate analyses of the OS were performed using an elemental analyzer (EA2400 II, PerkinElmer, USA) in accordance with GB/T 212-2008, which outlines the proximate analysis of coal. The calorific value was determined utilizing a combustion heat test device (SHR-15, Xi'an MINSKS, China), following the GB/T 213-2008 standard for the determination of calorific value in coal. Additionally, the oil content of the sample was assessed through the Soxhlet extraction method. The experimental results are shown in Table 1.

2.2. Tube furnace experiment

The experimental setup of the pyrolysis system was shown in Fig. 1. Before each experiment, a certain flow rate of nitrogen gas was used to sweep out the residual air in the tube furnace, after which ± 1.5 g of dried OS sample was taken into the quartz boat and relocated to the heating zone of the tube furnace (GKD, Shandong Xianke Instrument Co., Ltd., China), paying attention to retaining the loose space when placing the sample to ensure that the pyrolysis gas could quickly detach from the surface of the OS sample. The tube furnace was heated at a rate of 10 °C·min⁻¹ until the desired temperature was reached (450, 500, 550, 600, and 650 °C). Shielding gas (N₂) was flowed at rates of 50, 100, 150, and 200 ml·min⁻¹ to purge the reactor and maintain an inert atmosphere. Various retention times (10, 20, 30, 40, 50, and 60 min) were applied at the final pyrolysis temperature. To ensure reproducibility, each pyrolysis condition was replicated at least twice. After the experiment, the equipment was turned off, and upon cooling to room temperature, the condensed pyrolysis oil and non-condensable pyrolysis gases in the gas pocket were collected for subsequent infrared (IR) and GC-MS analyses.

2.3. TG-IR experiment

The TG-IR coupler enables simultaneous monitoring of the mass loss of OS and the formation of typical products in the pyrolysis gases. For experiments using the TG instrument (Mettler TG/DSC 3+, Switzerland), a sample of dried OS was heated in an inert atmosphere at a heating rate of 10 °C·min⁻¹ from 30 °C to 1200 °C. The mass of the sample was ± 5 mg in order to avoid possible temperature gradients in the sample and to ensure kinetic

Table 1
Ultimate, proximate, calorific value, and oil mass content of original and dried OS.

	Original OS	Dried OS
Ultimate analysis (dry basis)/%		
C	31.81	29.26
H	6.12	5.63
N	1.80	1.65
S	0.51	0.47
O [⊖]	27.10	30.33
Proximate analysis (air dried basis)/%		
Moisture	31.38	7.52
Ash	22.41	30.20
Volatiles	43.60	59.04
Fixed carbon	2.61	3.24
Calorific value and oil content analysis		
Calorific value/kJ·kg ⁻¹	13354	16025
Oil by solvent extraction/%	28.95	36.67

[⊖] By difference: O% = 100% – (C% + H% + S% + N% + Ash_{db}%), Ash_{db} = ash/(100 – moisture) × 100%.

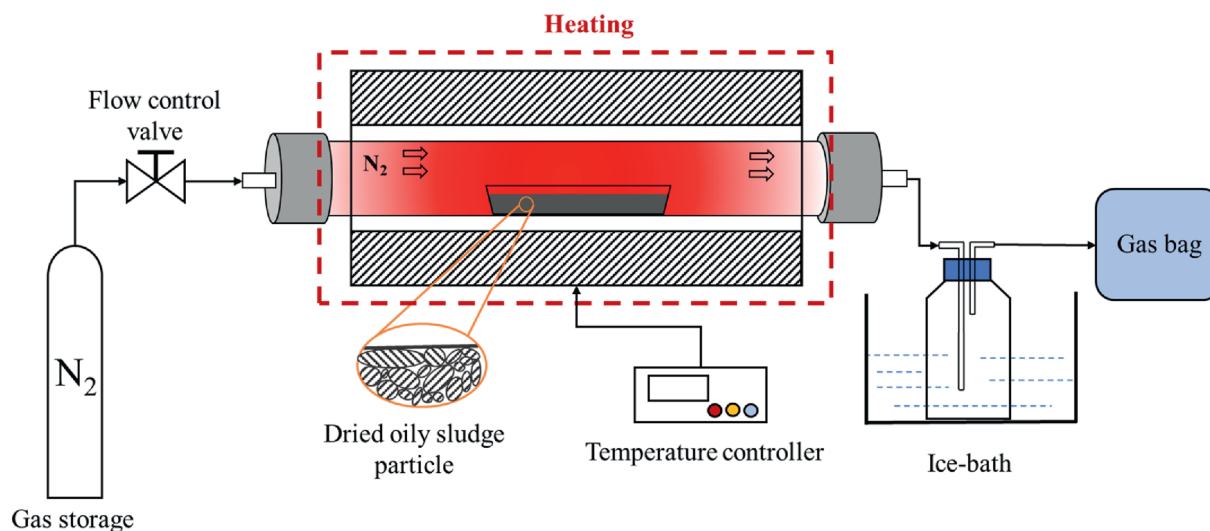


Fig. 1. Schematic of the tube furnace experimental setup including cold trap and gas bag collection system.

control of the process. Nitrogen gas with a flow rate of $50 \text{ ml} \cdot \text{min}^{-1}$ was passed through to maintain an inert atmosphere for sample pyrolysis during the experiment. An infrared spectrometer (IR, Thermo Scientific 50 iS50, USA) was connected to the TGA via a heat-holding channel which was heated to 180°C to prevent condensation of the generated gas on the walls. The IR spectrometer was operated within a wavenumber range of $4000\text{--}400 \text{ cm}^{-1}$. Data were collected during the pyrolysis process and subsequently post-processed to generate absorption spectra along with the corresponding gas formation curves. The spectrometer was initiated prior to the TGA experiments, with a 2-min delay observed between the TGA data and the corresponding spectral readings.

2.4. GC-MS experiment

A single quadrupole GC-MS analyzer (SHIMADZU-GCMS-QP2020-NX, Japan) was used to analyze the components of pyrolysis gas and pyrolysis oil generated from the pyrolysis of the OS. The shielding gas used for the experiment was helium of 99.99% purity, with an inlet temperature of 180°C , a vaporisation temperature of 290°C , and an agent delay of 1.5 min. The pyrolysis gas was collected by a gas bag in the pyrolysis experiment, which was briefly heated to prevent the gas from being adsorbed on the surface of the gas bag before sample injection. The pyrolysis oil was collected in a cold trap, briefly pre-heated to make it flowable and then a small amount of pyrolysis oil was dissolved in methanol and diluted, and injected into the analyzer using a manually injected liquid syringe. After data analysis, the retention indices of the components were compared with the NIST MS Search database to identify the components of pyrolysis gas and pyrolysis oil.

2.5. Pyrolysis kinetics

OS was a typical heterogeneous system consisting of water, oil, and solids [22]. Based on the standard form of the Arrhenius equation for heterogeneous reactions, the kinetic expression for the pyrolysis of OS could be written as follows [23–25]:

$$\frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \cdot e^{-\frac{E_a}{R_u T}} dT \quad (1)$$

where E_a represents the apparent activation energy, $\text{kJ} \cdot \text{mol}^{-1}$; T is absolute temperature, K; A is pre-exponential factor, min^{-1} ; R_u is universal gas constant with a value of $8.314 \times 10^{-3} \text{ kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$; Define $\beta = dT/dt$, $\text{K} \cdot \text{min}^{-1}$; $f(\alpha)$ denotes the mechanism function, which is a function of the conversion α , and could be expressed as:

$$\alpha = \frac{m_o - m_i}{m_o - m_e} \quad (2)$$

where m_i , m_o , and m_e denote the mass at given temperature T , initial temperature and end temperature, respectively.

The pyrolysis kinetic expression of the final OS could be written as Eqs. (3) and (4) through mathematical transformation as follows:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \cdot e^{-\frac{E_a}{R_u T}} f(\alpha) \quad (3)$$

$$F(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_{T_0}^T e^{-\frac{E_a}{R_u T}} dT = \frac{AE_a}{\beta R_u} G(y) \quad (4)$$

In this equation, T_0 represented the initial temperature of pyrolysis. The objective of studying pyrolysis kinetics was to determine the kinetic triplet E_a , A , and $F(\alpha)$ from the equation, which described the specific reaction. Mathematically, an exact analytical solution for $G(y)$ didn't exist. By selecting an appropriate form for the mechanism function $f(\alpha)$, the E_a and A could be determined from the slope and intercept of the fitted line. When the oily sludge was dried, its pyrolysis could be treated as the pyrolysis of solid materials. The potential mechanism functions governing this process, along with the corresponding fitted results, are provided in Supplementary Material, Tables S1–S3 [26,27].

3. Results and Discussion

3.1. Dried OS pyrolysis product distribution

Fig. 2 showed the yield distribution of pyrolysis products of dried OS under different conditions. Fig. 2(a) indicated the distribution of pyrolysis products after 30 min of pyrolysis at different constant pyrolysis temperatures, which in this work were chosen

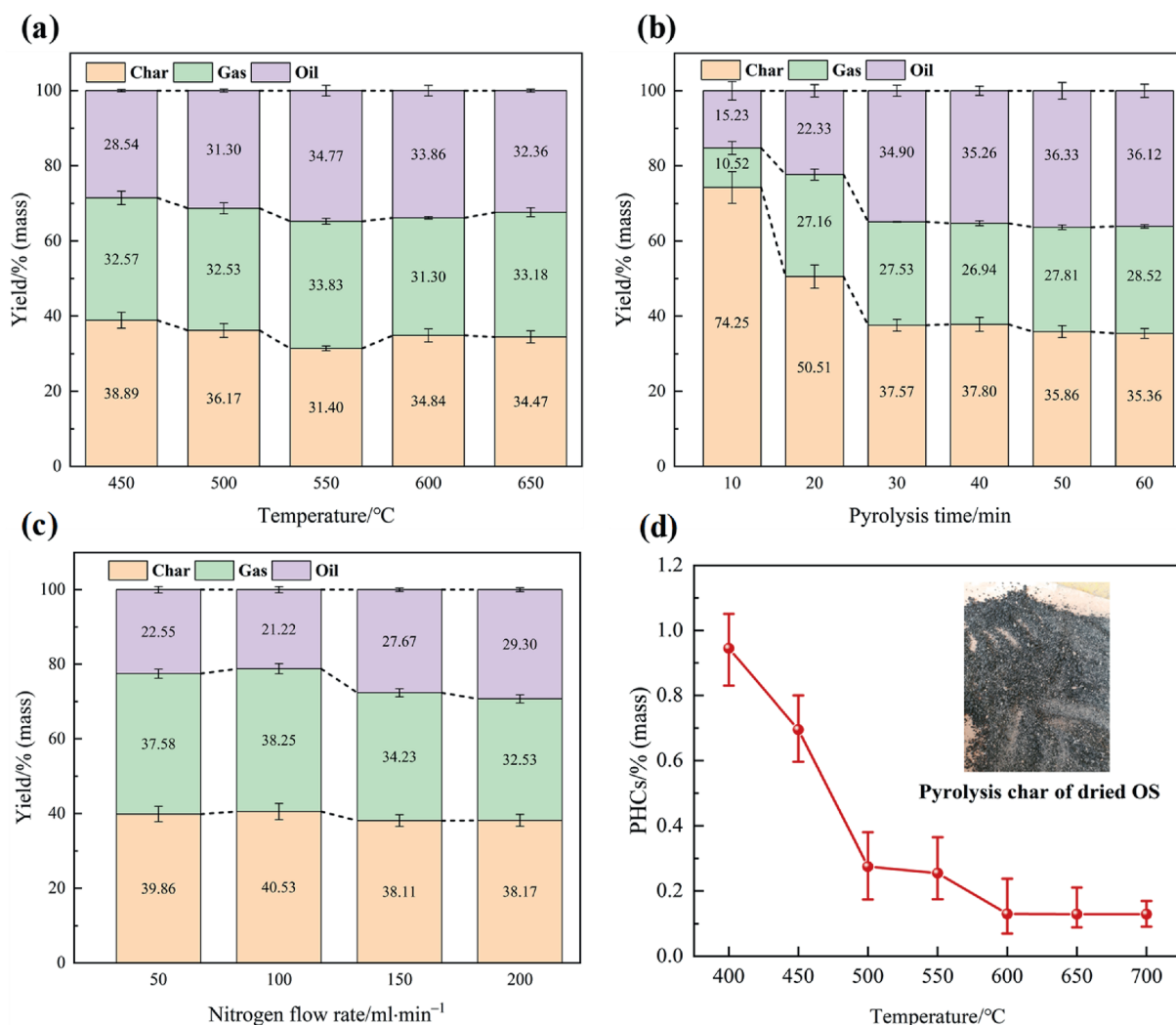


Fig. 2. Product yields under different pyrolysis conditions: (a)–(c) different pyrolysis temperatures, times and shielding gas flow rates, (d) the PHCs in char generated by OS pyrolysis.

to be in the range of 450 °C–650 °C, the temperature range covering the entire decomposition temperature of petroleum hydrocarbons. With increasing pyrolysis temperature, the char yield decreased from 38.89% at 450 °C to 31.40% at 550 °C, which indicated more release of oil and gas. While the pyrolysis oil yield after condensation increased slightly between 450 °C and 550 °C, when the pyrolysis temperature was 550 °C, the pyrolysis oil yield reached the maximum value of 34.77%, and the temperature corresponding to the maximum yield was consistent with the results of Gao *et al.* [19]. This tendency was probably attributed to the secondary reactions in pyrolysis oil, such as the cracking of organic bonds, which became active at 550 °C or higher. The decrease in pyrolysis oil yield with further increase in pyrolysis temperature was due to the enhancement of secondary cracking of volatiles with increasing temperature, which inhibited pyrolysis oil production [28]. Fig. 2(b) indicated that at pyrolysis temperature of 550 °C, the oil yield increased from 15.23% to 36.12% as the pyrolysis time increased from 10 to 60 min. This indicated that longer pyrolysis time is beneficial to the recovery of pyrolysis oil. Meanwhile, it could be seen that the fluctuation of pyrolysis oil yield is less than $\pm 2\%$ after the pyrolysis time is more than 30 min, which could be considered that the dried OS has been fully reacted, proving that the time selection of constant temperature pyrolysis

experiment is reasonable. Fig. 2(c) indicated the yield of pyrolysis products under different shielding gas flow rates, and the results showed that the shielding gas flow rate has little effect on the results.

In practical engineering applications, the selection of pyrolysis temperature considers multiple factors including harmful substance generation, equipment manufacturing constraints, and energy consumption. According to China's national standard for OS char disposal (GB 4284-2018), the petroleum hydrocarbon content (PHC) in residues must remain below 0.3% to be classified as general solid waste [29]. To ensure compliance with this standard while optimizing process efficiency, a series of experimental temperatures (400, 450, 500, 550, 600, 650, and 700 °C) were selected based on the PHC trends as shown in Fig. 2(d). The analysis revealed that PHC in pyrolysis char decreased below the 0.3% threshold at temperatures above 500 °C. Further examination showed that PHC levels at 650 °C and 700 °C were nearly identical to those at 600 °C, indicating diminishing returns at higher temperatures. Considering energy consumption optimization, the temperature range was consequently narrowed to 500 °C–600 °C, where significant PHC reduction occurred without excessive energy input. This medium temperature range aligns with findings in relevant literature, where both high and low temperatures are

typically avoided for OS pyrolysis treatment [9,30]. The experimental data presented in Fig. 2(d) thus confirms the appropriateness of selecting 500 °C–600 °C as the optimal pyrolysis temperature range.

3.2. Thermogravimetric analysis

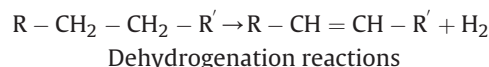
Tables 2 and 3 summarized the key pyrolysis characteristic parameters for the original and dried OS, which were derived from the TGA/DTG curves. The parameters include: the initial (T_o), peak (T_p), and endpoint (T_e) temperatures for each stage, °C; the maximum mass loss rate ($-R_m$), %·min⁻¹; and the stage mass loss (WL), %.

Fig. 3 showed the TG/DTG test results of the original and dried OS, in which four obvious peaks appear. The comparison of the pyrolysis characteristic parameters of the dried and original OS indicated that the pyrolysis process could be roughly divided into five stages:

- (1) Water evaporation stage (<200 °C). In this stage, the OS primarily absorbed heat to rapidly evaporate surface water, without significant chemical reactions occurring. The mass loss rate of the dried OS in this phase was noticeably slower than that of the original OS, and distinct mass loss peaks were still observable on the DTG curve. Water in OS was generally divided into four forms, including interstitial water, capillary bonded water, surface adsorbed water, and chemically bonded water. In this stage, dried OS still contained part of the water bonded to the oil, which required higher temperature for evaporation and separation, and this was the reason for the smaller mass loss rate.
- (2) Light component volatilization stage (200 °C–400 °C). During this stage, the volatilization of light components within the OS primarily occurred. Since the temperature had not yet reached the threshold for deep pyrolysis, the volatilization of light components with lower boiling points was confined to the surface of the OS. As the temperature increased, a portion of the unstable organic matter underwent decomposition, generating small molecular gases such as CH₄, H₂, and others [31]. The mass loss of original and dried OS were 22.54% and 20.97%, respectively, both of which were the stages of maximum mass loss, indicating that there were more light components in the samples of

this experiment, and this stage was the main stage of the OS pyrolysis process.

- (3) Heavy component pyrolysis stage (400 °C–600 °C). The heavy components in the OS began to undergo pyrolysis during this stage. Due to the complex composition of the OS, various organic materials and heavy oils participated in intricate reactions. At elevated temperatures, the heavy oils experienced thermal cracking, resulting in the formation of smaller organic molecules and non-condensable gases. Furthermore, certain polymerization and organic decomposition processes took place, leading to the conversion of heavy oil into lighter fractions. This transformation modified both the type and content of the oil components, thereby influencing the characteristics of the pyrolysis process [32]. When the temperature reached 393 °C, long-carbon chain in the dried OS began to break, fracture, undergo dehydrogenation, secondary condensation, secondary polymerization, and other reactions, generating alkanes, olefins, alkynes, aldehydes, benzene, and other organic substances, while generating a certain amount of char [19]. The above reactions required H₂ to be generated during pyrolysis, and as the pyrolysis process advanced, greater yields of H₂ were produced, which further promoted the reaction. H₂ was mainly generated through the water–gas shift reaction and thermal decomposition/cracking of organic components in the OS, as shown in the following reaction equations [33,34]:



The characteristic peaks of C=C, CH₄, CO and CO₂ could be found in the infrared spectrograms. This stage was the main stage of OS pyrolysis.

- (4) Secondary reaction stage (600 °C–800 °C). In this temperature range, the difficult-to-decompose long-chain heavy oils in OS and the volatile gases in stage 2 had secondary reactions, and the stabilized substances formed by the condensation reaction of organic macromolecules in the low

Table 2
Original OS pyrolysis characteristic parameters.

Sample	$\beta/\text{K} \cdot \text{min}^{-1}$	$T_o/^\circ\text{C}$	$T_p/^\circ\text{C}$	$T_e/^\circ\text{C}$	$-R_m \times 10^{-3}/\% \cdot \text{min}^{-1}$	WL/%
Original OS	Water evaporation	30	71.21	176	−0.97	17.74
	Light component volatilization	176	340.06	457	−1.30	22.54
	Heavy component pyrolysis	457	535.29	660	−1.01	6.73
	Secondary reaction	660	751.45	896	−1.47	13.16
	Final pyrolysis	896	—	1200	—	0.83

Table 3
Dried OS pyrolysis characteristic parameters.

Sample	$\beta/\text{K} \cdot \text{min}^{-1}$	$T_o/^\circ\text{C}$	$T_p/^\circ\text{C}$	$T_e/^\circ\text{C}$	$-R_m \times 10^{-3}/\% \cdot \text{min}^{-1}$	WL/%
Dried OS	Water evaporation	30	79.27	180	−1.00	9.92
	Light component volatilization	180	300.00	393	−1.56	20.97
	Heavy component pyrolysis	393	444.41	590	−1.06	12.28
	Secondary reaction	590	724.61	775	−1.66	12.03
	Final pyrolysis	775	—	1200	—	6.80

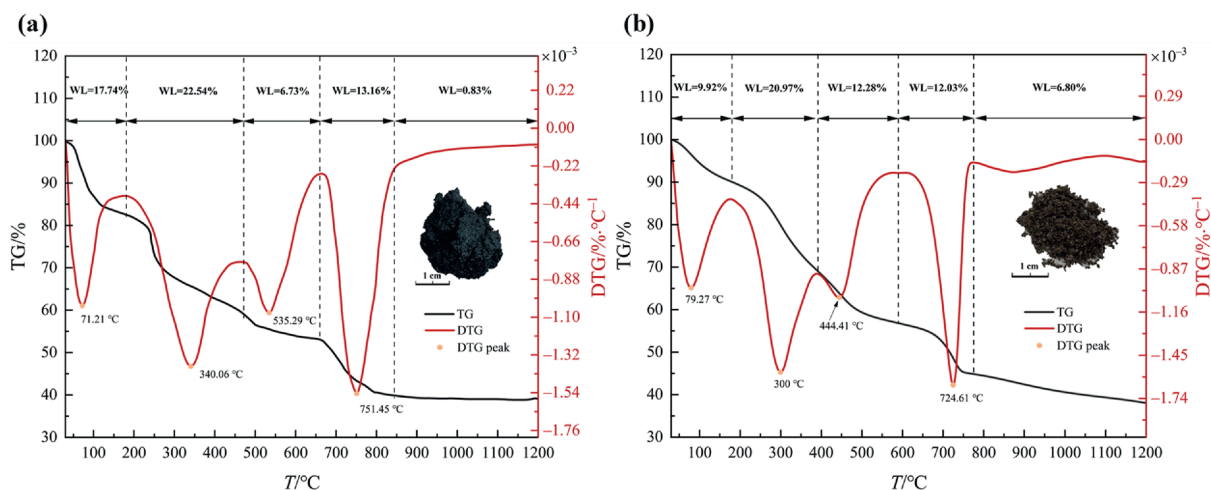


Fig. 3. TG/DTG curves of original and dried OS pyrolysis: (a) original OS and (b) dried OS.

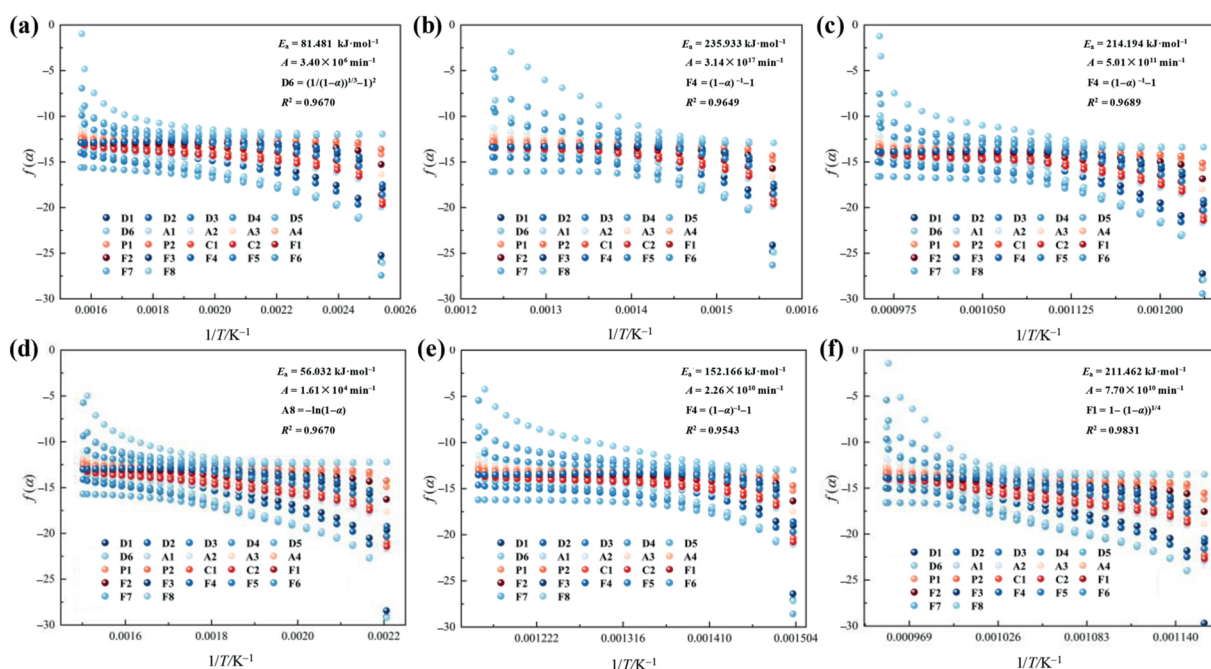


Fig. 4. The fitting results of pyrolysis kinetics of different models: (a)–(c) main pyrolysis stages of the original OS, (d)–(f) main pyrolysis stages of the dried OS.

and middle temperatures were further decomposed at high temperatures. Due to the various sources and components of OS, different temperature ranges had been reported in the references. The study in this paper found a mass loss peak in the 600°C – 800°C temperature range. This stage was also the main stage of OS pyrolysis.

- (5) Final pyrolysis. The pyrolysis reaction was nearly complete at this section. The TG curve decreased slowly, and the DTG curve stabilized, indicating the finalization of the pyrolysis process. This stage represents the combined effects of inorganic matter decomposition and residual char transformation, as small amounts of heavy oil and inorganic matter remaining in the oily sludge undergo thermal decomposition at elevated temperatures [35,36].

It was important to point out that the temperature of each stage was divided into approximate temperature ranges, and there was an

obvious overlap between the different stages, which was consistent with the rest of the solid OS pyrolysis research results [37].

3.3. Pyrolysis kinetic analysis

The activation energy and mechanism function of the main pyrolysis stages of OS calculated were shown in Fig. 4, and the selection of the mechanism model was presented in Table S1. The high correlation coefficient indicated a good agreement between the main reaction model and the experimental data.

Comparing the original and dried OS, it was found that the activation energies of each stage of the original OS were 81.481, 235.933, and $214.194 \text{ kJ}\cdot\text{mol}^{-1}$, which were higher than those of the dried OS, which were 56.032, 152.166, and $211.462 \text{ kJ}\cdot\text{mol}^{-1}$, respectively. Correspondingly, the pre-exponential factors (A) for the three main pyrolysis stages of the original OS were 3.4×10^6 , 3.14×10^{17} , and $5.01 \times 10^{11} \text{ min}^{-1}$, while those for the dried OS

were 1.61×10^4 , 2.26×10^{10} , and $7.70 \times 10^{10} \text{ min}^{-1}$. The significant decrease in both activation energies and pre-exponential factor values after drying pretreatment indicates a fundamental change in the reaction kinetics and molecular collision frequency during pyrolysis. Notably, the dramatic reduction in the pre-exponential factor for the second stage (from 3.14×10^{17} to $2.26 \times 10^{10} \text{ min}^{-1}$) suggests that drying pretreatment substantially reduces the frequency of effective molecular collisions during the main decomposition phase, which aligns with the removal of light volatile components that facilitate rapid reaction kinetics.

It could be seen that as the pyrolysis progressed, the pyrolysis mechanism functions of the three main pyrolysis stages changed from Model 6,19,19 to Model 8,19,7. This was because the drying pretreatment removed the light components involved in the pyrolysis of the internal part of OS at 105°C , changed the reaction path of the pyrolysis process, significantly reduced the pyrolysis activation energy of OS, and made the reaction more likely to take place at a given temperature [38,39]. Meanwhile, the activation energy changed significantly with the progress of the reaction, and the activation energies of stages 3 and 4 were significantly larger than that of stage 2, which was caused by the decomposition of long-chain hydrocarbons and other heavier organic components. This phenomenon also reflected the change of pyrolysis stages from superficial to intensive. In practical engineering applications, drying pretreatment of OS made the pyrolysis more likely to react and could reduce the financial expenses and the difficulty of equipment manufacturing.

3.4. IR test analysis

Fig. 5 showed the three-dimensional infrared spectra of the gases during the pyrolysis of original and dried OS, in which the Z-axis was the transmittance, the X-axis was the temperature, and Y-axis was the wave number. The experimental temperature ranged from 30°C to 1200°C with a heating rate of $10^\circ\text{C} \cdot \text{min}^{-1}$, with a shielding atmosphere of N_2 and a gas flow rate of $50 \text{ ml} \cdot \text{min}^{-1}$.

Comparative analysis of the infrared spectra revealed significant differences between the original and dried OS. The characteristic water peak observed in the original OS spectra was completely eliminated after drying, demonstrating the effectiveness of moisture removal during the drying process. Similarly, the

distinct absorption peak at $2900\text{--}3000 \text{ cm}^{-1}$, corresponding to C–H stretching vibrations of methyl groups and observed across 30°C – 650°C in the original OS, disappeared in the dried OS spectra, indicating that drying successfully removed most of the volatile light components. Notably, the dried OS exhibited a remarkably enhanced characteristic absorption peak of alkenes during pyrolysis, attributed to C=C stretching vibrations from dehydrogenation reactions, while the original OS showed only minimal alkene formation below 300°C . This significant increase in alkene production in the dried sludge suggests that the removal of light volatile components during drying concentrates the hydrocarbon matrix, promoting more efficient dehydrogenation reactions at elevated temperatures. The enhanced alkene generation in dried OS represents a beneficial outcome, as it indicates improved thermal conversion efficiency and potentially higher yields of valuable hydrocarbon products during pyrolysis, thereby validating the superior performance of dried OS for thermal treatment processes.

Analysis of the TG/DTG curves from the dried OS revealed peaks at 79.21 , 300.00 , 444.41 , and 724.61°C , respectively, and the superposition of the IR spectra obtained at these four temperature points was shown in Fig. 6(a). It could be observed from the figure that at 79.21°C , the O–H stretching vibration of $3500\text{--}4000 \text{ cm}^{-1}$ reflected the release of water and alcohol, which was inferred to be the release of H_2O considering that the temperature was not high and it was in the stage of water evaporation. With the increase of temperature, a series of peaks located between 670 and 830 cm^{-1} were mainly the characteristic peaks for the neighboring substituents, meso substituents, and para substituents of benzene ring, which indicated that the dried OS contains a certain amount of benzene, which coincided with the absorption peaks of C–C stretching vibration and benzene ring skeleton vibration region at $1450\text{--}1620 \text{ cm}^{-1}$ [19].

Fig. 6(b) presented the magnified infrared spectra in the range of $1450\text{--}2200 \text{ cm}^{-1}$, the characteristic peaks of C=C in the benzene ring could be seen near 1600 and 1500 cm^{-1} [40], and the content first increased and then decreased with increasing temperature. This may have been attributed to the sample itself containing aromatic substances, and the monocyclic aromatic hydrocarbons were produced by the pyrolysis of the rest of the organic matter at high temperatures. The peak at position 1747

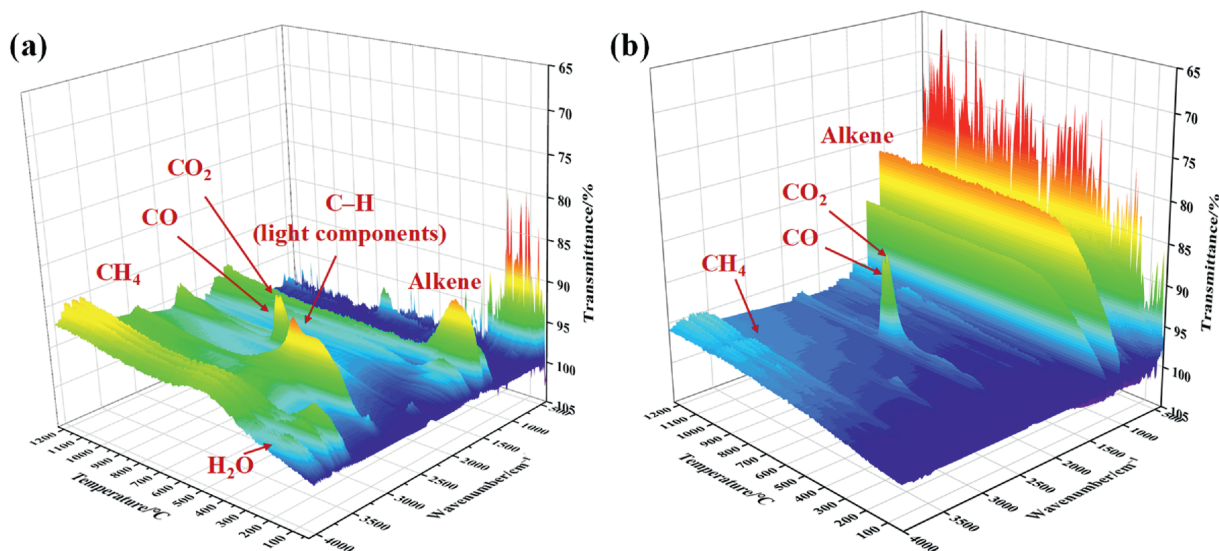


Fig. 5. Original (a) and dried OS (b) infrared spectrogram.

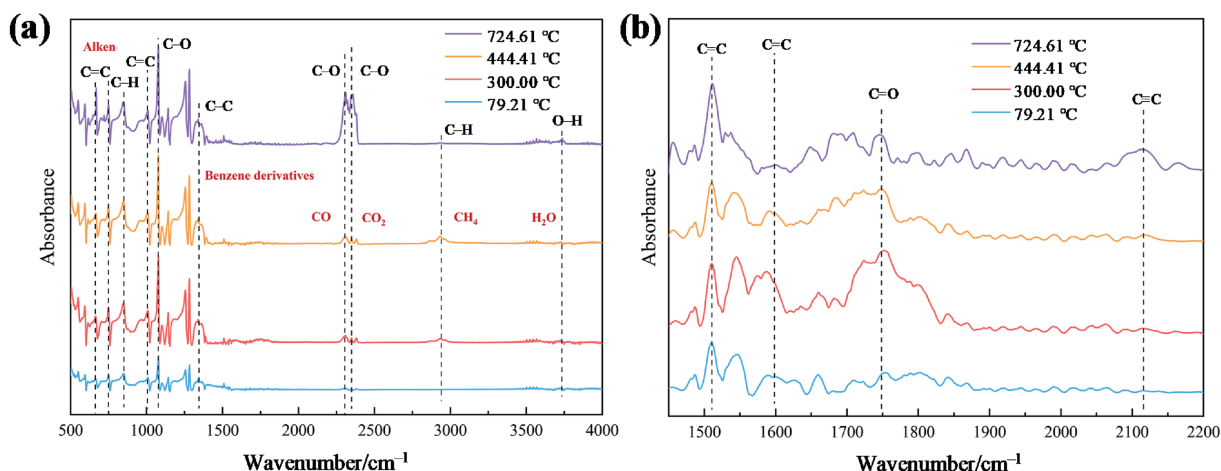


Fig. 6. Infrared spectra of OS: (a) infrared spectra at the peak of DTG and (b) 1450–2200 cm^{-1} localized magnified figure.

cm^{-1} corresponds to the characteristic peak of $\text{C}=\text{O}$ stretching vibration in aldehydes (1755–1665 cm^{-1}) and was found to be highest at 300 °C, where the organic compounds in the samples were pyrolyzed to form aldehydes. The characteristic peak of $\text{C}\equiv\text{C}$ at 2115 cm^{-1} appeared only at 724.16 °C, which could be attributed to the condensation reactions of $\text{C}-\text{C}$ and $\text{C}-\text{O}$ in this temperature range [21], indicating a further pyrolysis progress of the dried OS particles and the regeneration of alkynes from the organic matter after chain breaking and dehydrogenation reactions.

Comprehensive analysis revealed that the main gas products of pyrolysis of dried OS were CO_2 , CO , CH_4 , and organic matter such as benzene hydrocarbons were also observed. Fig. 7 shows the main gas distribution from pyrolysis gas of dried OS.

Below 800 °C, the yield exhibited an initial increase followed by a decrease. Above 800 °C, temperature had minimal impact on the yield of each component. The yields of CO and CO_2 increased significantly with the increase of the pyrolysis temperature. This was attributable to the continued pyrolysis of hydrocarbons in dried OS under elevated temperature conditions. Additionally, high temperatures facilitated the dehydrogenation, decarboxylation, and decarbonylation of organic compounds [41]. The yield of CH_4 reached its maximum at about 600 °C, and higher pyrolysis temperatures inhibited the production of CH_4 , which was consistent with the results of Zhu *et al.* [21]. To mitigate the environmental impact of pyrolysis products, it was essential to take into account the presence of trace amounts of benzene hydrocarbons when selecting the pyrolysis temperature.

3.5. Analysis of pyrolysis gas components

There was a steep increase of the peak value near 2225–2400 cm^{-1} in the infrared spectrum at 724.61 °C, indicating that the intensity of chemical reaction increases at this point of temperature, the number of substances produced increases. To determine the specific composition of the gas products at this point, 1 g of the dried OS sample was taken and pyrolyzed in a tube furnace at a temperature of 724.61 °C for 30 min at a constant temperature, and the pyrolysis gas products were collected through a gas bag for GC-MS testing.

Fig. 8 demonstrated the GC-MS test results of the pyrolysis gas of the dried OS at 724.61 °C, which revealed a totally of 15 species of organic substances. The generation of $\text{C}_5\text{H}_8\text{O}_2$, $\text{C}_4\text{H}_8\text{O}$, and $\text{C}_{10}\text{H}_{18}\text{O}_3$ corresponds to the vibration of $\text{C}=\text{O}$, indicating the generation of aldehydes at this temperature. $\text{C}_5\text{H}_8\text{O}_2$, C_5H_6

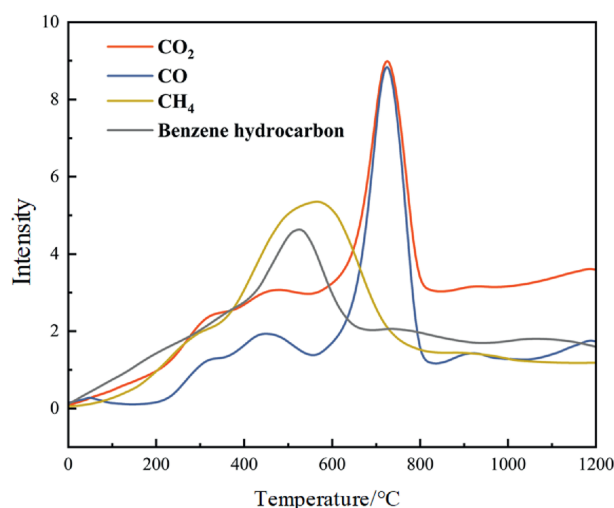


Fig. 7. Product distribution from pyrolysis gas of dried OS.

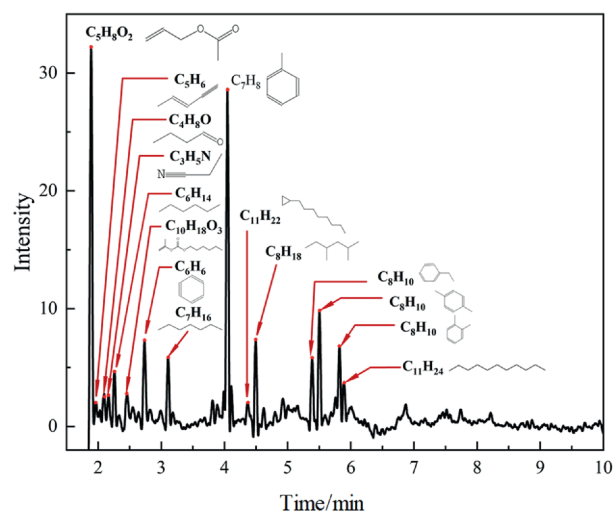


Fig. 8. GC-MS test results of pyrolysis gas at 724.61 °C.

corresponded to the vibrations of $C=C$ and $C \equiv C$, indicating that dehydrogenation and chain-breaking reactions took place within the pyrolysis [42,43]. The generation of aromatic hydrocarbons such as C_6H_6 , C_7H_8 , C_8H_{10} and $C_{11}H_{22}$ indicated the cyclization reaction of long carbon chain organics after breaking the chain during pyrolysis. The production of saturated alkanes, such as C_6H_{14} , C_7H_{16} , C_8H_{18} , and $C_{11}H_{24}$, not only provided insight into the pyrolysis pathway of hydrocarbons but also yielded reusable products.

The experiments demonstrated that for dried OS, it was necessary to control the temperature below 724.61°C in order to obtain as much combustible gases such as CH_4 as possible, while avoiding toxic and harmful substances such as benzene, naphthalene and anthracene.

3.6. Analysis of pyrolysis oil components

The oil obtained from pyrolysis could be recovered as a fuel to achieve the resource utilization of OS. In this section, the oil from the cold trap after pyrolysis at constant temperature for 30 min at 500°C and 600°C was collected for GC-MS testing, and the results were shown in Fig. 9.

According to relevant studies, pyrolytic oil had potential as a high-quality fuel [44]. Components such as esters, alcohols, ketones, and saturated fatty acids were considered favorable due to their high energy density and combustion efficiency [2,45]. Conversely, polycyclic aromatic hydrocarbons and compounds containing nitrogen and sulfur adversely affect combustion efficiency and posed environmental risks [46,47]. In this work, pyrolysis oil at 500°C contained 8.70% alcohols, 24.58% ketones, 1.75% alkanes, 12.46% esters, and 2.46% benzenes, with nitrogen compounds comprising 7%. At 600°C , the pyrolysis oil consisted of 1.56% alcohols, 16.76% ketones, 8.45% alkanes, 18.99% esters, and 25.67% saturated fatty acids, with nitrogen compounds accounting for 4.03%. The decrease in ketones and increase in fatty acids with temperature rise could be attributed to the instability of oxygen-containing groups at high temperatures, where $C=O$ bonds were prone to deoxidation and decarboxylation [48]. The increase in alkanes enhances the potential for using pyrolysis oil as fuel, resulting from the dehydrogenation and cracking of olefins, alkynes, and other chemically active substances [49]. At 500°C , the highest concentrations in pyrolysis oil were 11.64% 2-cyclopenten-1-one, 8.70% dibasic phthalate, and 5.71% butanediol. Additionally,

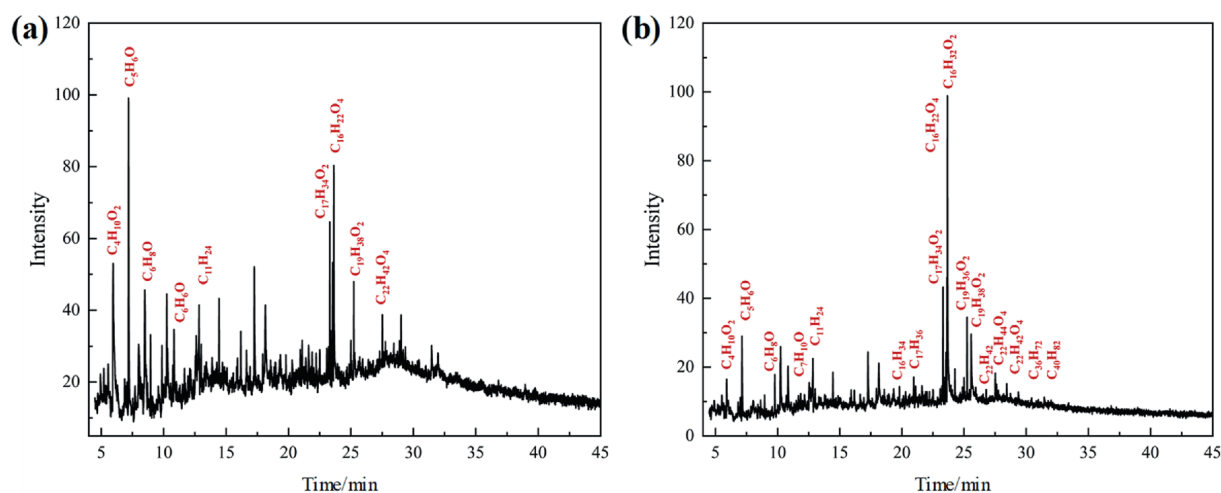
toxic substances such as benzene, anthracene, and naphthalene were present. At 600°C , the highest concentrations were 20.13% hexadecanoic acid, 5.73% dibutyl phthalate, and 5.67% 2-cyclopenten-1-one. Notably, hexadecanoic acid replaced 2-cyclopenten-1-one as the predominant fatty acid in the pyrolytic oil with increasing temperature. Hexadecanoic acid, a saturated fatty acid, was a common component in oils and fats and also frequently found in pyrolytic oil [11,50].

Compared to the 500°C pyrolysis oil, the 600°C variant had a greater proportion of components conducive to combustion, lower levels of nitrogen and heterocyclic compounds, and absence of harmful benzene compounds, making it more suitable for industrial applications as a fuel.

4. Conclusions

In this work, the pyrolysis characteristics of dried and original OS were systematically compared through tube furnace experiments, TG-IR, and GC-MS analyses. The feasibility of drying pretreatment of OS for industrial applications was evaluated based on variations in the chemical composition of pyrolysis products and theoretical analysis of pyrolysis kinetics. The main conclusions are as follows:

- (1) The distribution of pyrolysis products under different conditions of temperature, pyrolysis time, and shielding gas flow rate for dried OS was examined through tube furnace experiments. The pyrolysis oil yield of dried OS reached a maximum value of 34.77% at 550°C , beyond which it decreased with increasing temperature. Complete pyrolysis was achieved within 30 min, and the shielding gas flow rate exhibited negligible influence on the pyrolysis product distribution.
- (2) Thermogravimetric analysis revealed that the pyrolysis of both original and dried OS proceeded through five distinct stages, with stages 2, 3, and 4 being the predominant mass-loss phases. Kinetic analysis further indicated that the activation energies of dried OS in these key stages (56.032 , 152.166 , and $211.462\text{ kJ}\cdot\text{mol}^{-1}$) were lower than those of the original OS (81.481 , 235.933 , and $214.194\text{ kJ}\cdot\text{mol}^{-1}$). The associated mechanism functions shifted from $\{[1/(1-\alpha)]^{1/3}-1\}^2$ and $(1-\alpha)^{-1}-1$ (dried OS) to $-\ln(1-\alpha)$, $(1-\alpha)^{-1}-1$ and $1-(1-\alpha)^{1/4}$ (original OS), suggesting an



alteration in the pyrolysis reaction pathway due to drying pretreatment. This result implies that removing light components via drying reduces the energy barrier for pyrolysis, thereby enhancing the reactivity of OS. Consequently, in practical applications, such pretreatment could significantly promote pyrolysis efficiency at a given temperature.

- (3) IR and TGA analysis showed that the main gaseous products of dried OS pyrolysis were H₂, CO₂, CO, CH₄, and organics. The formation of harmful substances like benzene first increased then decreased with temperature, while high temperatures inhibited CH₄ generation. To maximize combustible gas recovery, the pyrolysis temperature should be kept below 724.61 °C, where CO and CO₂ yields peak.
- (4) The pyrolysis oil obtained at 600 °C demonstrated superior quality for fuel recycling, as GC-MS and char analysis showed a higher hydrocarbon content and a lower proportion of nitrogen-containing compounds compared to the oil produced at 500 °C.

CRedit Authorship Contribution Statement

Siduo Song: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation. Zhishan Bai: Writing – review & editing, Supervision, Project administration, Funding acquisition. Weijie Zhang: Visualization, Investigation, Formal analysis. Likun Ma: Writing – review & editing, Supervision, Resources, Funding acquisition. Xuedong Liu: Writing – review & editing, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Nomenclature

A	pre-exponential factor of the Arrhenius equation, min ⁻¹
E_a	apparent activation energy, kJ·mol ⁻¹
M	mass of the sample at given temperature, g
m_f	mass of the sample at final temperature, g
m_0	mass of the sample at initial temperature, g
R^2	linear correlation
$-R_m$	the maximum rate of mass loss at any stage, %·min ⁻¹
R_u	universal gas constant, 8.314×10 ⁻³ kJ·mol ⁻¹ ·K ⁻¹
T	absolute temperature, K
T_e	the final pyrolysis temperature of any stage, °C
T_0	the initial pyrolysis temperature of any stage, °C

T_p	the peak temperature corresponding to the maximum mass loss rate at any stage, °C
WL	the mass loss in any stage, %
α	conversion degree, %
β	heating rate, K·min ⁻¹

Supplementary Material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cjche.2025.12.036>.

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