Novel conditioner for efficient dewaterability and modification of oily sludge with high water content

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Abstract
The oily sludge with high water content (OS) was dewatered, modified, and converted into solid fuel by a novel chemical conditioner (OSO-101). The effect of OSO-101 dosage on the dewaterability of OS was studied, showing that OSO-101 dosage of 15% (wt.) could achieve the best dewaterability efficiency of OS (98.18%). Meanwhile, compared with some conventional conditioners, OSO-101 developed by our team was more effective in improving OS dewaterability efficiency. And OSO-101 may have free radical reaction, polar reaction, and redox reaction with petroleum hydrocarbons in OS, thereby polymerizing and forming condensed solid structures. The calorific value change of OS after conditioning, heavy metal content, and dioxin content of fly ash leached from incinerated product were measured for resource analysis and environmental assessment. Results showed that the resultant OS fuel blocks had extremely low content of heavy metals, dioxins, and other toxic and hazardous substances leached from fly ash. And this process did not require secondary treatment and fully met environmental protection emission standards. Additionally, OSO-101 had certain economic rationality and could effectively recover the calorific value contained in OS. This research is expected to provide new insights for efficient dewaterability and modification of OS, as well as subsequent resource utilization and harmless treatment, bringing potential environmental and economic benefits.

Keywords Oily sludge · Conditioner · Dewatering · Harmless treatment · Resource recovery

Introduction
With the rapid development of global economy and the deterioration of crude oil, the amount of oily sludge with high water content (OS) is ascending year by year (Ren et al. 2020). OS is composed of water, organic matter, and solid residue, forming a multiphase mixed system. The water content, oil content, and solid content of OS are generally 80~99%, 0.5~50%, and 0.2~40%, respectively (Ningbo et al. 2021). The water in OS does not exist alone, most of which is combined with other ingredients (e.g., petroleum substances) to form a fairly stable emulsified structure (Zhang et al. 2017a, b). Organics in OS include crude oil, asphalt, alkanes, and some benzene series. Thus, OS has the characteristics of serious emulsification, high viscosity, and difficulty in sedimentation and dewaterability (Zhang and Liu 2013). Additionally, OS also contains toxic, harmful, and refractory organics such as benzene, phenols, and anthracene as well as heavy metal salts (copper, chromium, mercury, etc.) (Zheng et al. 2017). It will endanger ecological environment and human health if directly stacked or improperly handled (AL-Doury 2019). According to “Solid Waste Environmental Pollution Prevention Law” and “Hazardous Waste List,” OS needs to be harmlessly treated and used as resource.

At present, main treatment methods of OS include conditioning-mechanical separation (Hou et al. 2013), solvent extraction (Mohit et al. 2020), freezing/thawing (Ju et al. 2012), pyrolysis (Liu et al. 2021), incineration (Dal Mas et al. 2021), solidification method (Zhang et al. 2018), etc. Among them, the incineration method is to treat OS with
the characteristics of low oil content, a certain calorific value (Tunçal and Uslu 2014). The technical advantages of incineration are rapid operation, thorough treatment, high sterilization rate, and great reduction. After treatment, the reduction rate of OS can reach 95%. Organics in OS will be completely oxidized and discharged in the form of fly ash, flue gas, slag, etc. (Xiehong et al. 2017). However, problems such as secondary pollution, high cost, and energy consumption still exist. In particular, leaching amount of heavy metals and dioxins is relatively high after incineration (Hu et al. 2013). Hence, it is urgent to transform OS into solid fuel by conditioning. Briefly, OS is firstly dehydrated and modified and then converted into solid fuel. Harmless treatment of OS can be reached, and residual value of OS can be recycled to realize resource utilization to the greatest extent.

Based on this, we developed a novel OS conditioner (OSO-101). On one hand, the reduction, harmlessness, and stabilization of OS processing were realized. On the other hand, the resource utilization of OS was completed. Free radical reaction, polar reaction, and redox reaction were possibly involved between OSO-101 and petroleum hydrocarbons; thereby, OS could be polymerized and converted into condensed solid structures. Moreover, the mixed solid was stable and irreversible and would not cause secondary sludge (Qinghua et al. 2021). Simultaneously, OSO-101 made long-chain nonflammable components (e.g., asphaltenes and gums) polymerize, and the water adsorbed in OS was converted into free water and released (Zhang et al. 2014). Under normal temperature and pressure, OS was finally transformed into solid fuel after curing in natural conditions or reacting by mechanical drying, which had great incineration performance. The whole treatment process would not produce wastewater.

Herein, the effect of OSO-101 dosage on the dewaterability of OS was studied. Then, the effects of conditioning and incinerating on the solid-phase morphology, element composition, and surface functional groups of OS were analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD), and some other characterization methods. Finally, the calorific value change of OS after conditioning, heavy metal content, and dioxin content of fly ash leached from incinerated product were measured for resource analysis and environmental assessment.

Materials and methods

Materials

OS used in this work was taken from Shanghai Huarun Dadong Hazardous Waste Disposal Center (Shanghai, China), and the conditioner OSO-101 was produced by Zhongze Green Link Energy Environmental Technology Co., Ltd (our group). OSO-101 mainly contains calcium oxide (CaO), calcium sulfate (CaSO₄), calcium carbonate (CaCO₃), etc., in which the auxiliary materials were agricultural organic waste or industrial solid waste. For comparison, alum, lime, gypsum, and fly ash, which were all purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China), were also used in this work. In the experiment, deionized (DI) water was used for solution preparation. All chemicals were purchased in analytical grade or higher purity and can be used directly without further purification.

Characterization of oily sludge

The oil content of OS samples was evaluated by Soxhlet extraction method. According to crude oil water content determination method (GB/T 8929–2006), the water content of OS was determined. Then, the solid content was the total amount minus water content and oil content. The elemental composition of OS was determined by XRF (Model Var10EL-III, Elementar, Germany). Fourier transform infrared spectroscopy (FT-IR, Nicolet 6700, Thermo Fisher Scientific, USA) was used to analyze the surface functional groups of OS. SEM (S-3400 II, Hitachi, Japan) was used to scan OS after spraying gold, and the scanning voltage was 2.0 kV, and the magnifications were 5000, 10,000, 20,000, and 30,000 times. The composition and crystal phase of OS were analyzed by XRD (X’TRA, ARL, Switzerland) (40 kV and 40 mA). X-ray photoelectron spectroscopy (XPS, PHI 5000 VersaProbe, Japan) was performed to investigate surface chemistry of OS after dewatering (OSAD) and after incineration (OSAI).

Conditioning process

The effect of OSO-101 dosage on the dewaterability of OS was studied to explore the optimum dosage of conditioner. A certain amount of OS was weighed and placed in ceramic dishes, in which 5%, 10%, 15%, and 20% (wt.) OSO-101 was added respectively and stirred evenly. OSAD was obtained, weighed at intervals after drying at 20 °C, and compared with dewaterability of alum, lime, gypsum, and fly ash. Then, OSAD was incinerated at 900 °C to obtain OSAI. The dewatering rate (Dw) and capillary suction time (CST) were used to measure dewaterability of OS. Dw was calculated by Eq. (1) (Zhang et al. 2014). CST was determined by using a TRITON Type 304 M CST-meter.

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D_w = \frac{m_c - m_s}{m_s} \times 100\% \tag{1}
\]

where \(m_c\) (g) is the mass of water in OS (g) and \(m_s\) (g) is the reduced water mass of OS after adding OSO-101.
Analytical methods

In order to explore the resource utilization potential of OS after conditioning, industrial analysis and calorific value measurement were carried out on OSAD. The calorific value of OSAD refers to “Coal Calorific Value Measurement Method” (GB/T 2013–2008), and the industrial analysis of OSAD refers to “Coal Industrial Analysis Method” (GB/T 2012–2008). The fly ash after incineration was leached in accordance with standard method “Solid waste leaching toxicity leaching method, sulfuric acid and nitric acid method” (HJ/T 299–2007). The inductively coupled plasma optical emission spectrometer (ICP-MS, PerkinElmer, Optima 8000, USA) was used to quantitatively detect heavy metal content and chloride ion concentration of fly ash leached from OSAD. Gas chromatographic-mass spectrometry (GC–MS) (7890AGC/597D, Agilent Technologies Co., Ltd, USA) was used to analyze the content of dioxins.

Results and discussion

Characterization of OS

As presented in Fig. 1a, it is experimentally found that the water content, oil content, and solid content of OS are 79.30%, 11.31%, and 9.39%, respectively, in which the water content in OS is relatively high. The main constituent elements in OS are C and O (Fig. 1b). In addition, the generation of S element may be attributed to concentrated sulfuric acid added during demulsification and degreasing (Jin et al. 2021). Al element may originate from corrosion and leaching of Al coating on oily tank bottom. Moisture (Mad), ash (Aad), volatile content (Vad), and fixed carbon (FCad) of OS are 79.30%, 8.74%, 10.39%, and 1.57%, respectively (Table S1). OS has the characteristics of high Mad, low Aad, low Vad, and low FCad. High water content made OS disposal extremely energy-consuming by incineration and released excess offgas simultaneously. Therefore, efficient dehydration of OS is imperative for resource utilization.

Effect of OSO-101 dosage on dewatering rate

After adding varying dosage of OSO-101, the results of \( D_w \) are shown in Fig. 1c. \( D_w \) of OS could reach 79.52% after the addition of 5% OSO-101, and its effect on dewaterability of OS was obvious. When the dosage of OSO-101 reached 15%, \( D_w \) could further increase to 98.18%, showing that the rise of OSO-101 dosage could significantly improve oil–water separation efficiency of OS. However, \( D_w \) of OS was reduced to 95.99%, while OSO-101 dosage reached 20%. This may be because excessive OSO-101 reacted with organic matters in OS through free radical reaction, polar reaction, and redox reaction, which made particle sizes of OS smaller and affected its dewaterability (Guo et al. 2016). The drying images and incineration images of OS after adding different OSO-101 dosage also proved the above
conclusions (Fig. 2). The appearance of OSAD gradually changed from black to light yellow, and the appearance of OSAI also gradually changed from dark red to light yellow, which demonstrated efficient dewaterability and modification of OSO-101 on OS (Yang et al. 2005). In summary, it can be preliminarily determined that 15% OSO-101 could achieve the best dewaterability efficiency for OS studied in this experiment.

**Effect of different conditioners on CST**

Some conventional modifiers such as alum, lime, gypsum, and fly ash were used in the comparative study of OS dewaterability and conditioning (Fig. 3). After conditioning OS with alum, lime, gypsum, and fly ash, CST decreased from 2000 to 758 s, 1232 s, 1362 s, and 1426 s, respectively, while CST dropped from 2000 to 342 s with the addition of OSO-101. High CST reveals that OS is difficult to dehydrate. The significant reduction on CST indicated that OSO-101 had the advantage of high-efficiency dehydration for OS, which could greatly reduce volume of OS and reduce cost of subsequent incineration treatment. Furthermore, the changing trend of CST was basically similar for different conditioners (Fig. 3). As the dosage of conditioners increasing, CST firstly dropped to the lowest value and then showed an upward trend. As reported in the literature, although both alum and lime are the most commonly used conditioners in OS treatment, alum has better dewaterability efficiency and chemical conditioning properties for OS. It is well known
that lime will increase solid content and pH value of final products (Buyukkamaci and Kucukselek 2007). Gypsum (CaSO₄·2H₂O) is another material used for conditioning OS, which is usually precipitated from water with high salinity. The addition of gypsum obviously enhanced release of water and established a more rigid lattice structure. Fly ash is the residual waste in incineration process. Although it can also be used as a conditioner for zero-cost material, the use of fly ash will possess serious risks of toxic heavy metal release (Chen et al. 2018). In view of these discussions above, OSO-101 developed by our team was more effective in improving OS dewaterability.

**Effect of solid-phase morphology and composition on dewatering process**

SEM images and drying images of OSAD at optimal OSO-101 dosage are shown in Figs. 4 and 5, respectively. The microscopic morphology of OSAD under different magnifications were observed. OSAD was mainly composed of spherical particles, and there were some rod-shaped particles attached to the surface (Fig. 4). This may be ascribed to successful conditioning and modification of OS by OSO-101, which significantly promoted efficient dewaterability. It was found that \( D_w \) increased from 0 to 98.18% during the 72-h conditioning and drying experiment (Fig. 1c). OSAD gradually changed from black viscous solids to yellow–brown solid powder (Fig. 5). As demonstrated in Fig. 6a from XRD results, OSAD contained CaSO₄ (JCPDS File No. 01–0385), CaCO₃ (JCPDS File No. 24–0027), SiO₂ (JCPDS File No. 30–1127), and other substances. There may be some minor minerals, such as MgCO₃ (JCPDS File No. 08–0479), BaMg(CO₃)₂ (JCPDS File No. 12–0530), and CaAl₂SiO₈ (JCPDS File No. 41–1486). OSAD was incinerated to form OASI, and characteristic diffraction peaks of CaSO₄ and CaCO₃ disappeared due to high-temperature decomposition. Additionally, the intensity of characteristic diffraction peaks of CaO at 32°, 38°, and 39° (JCPDS File No. 74–1226) increased, and the peak shape became sharper, indicating that crystallinity was enhanced (K and A 2008).

FT-IR spectra of OSAD and OSAI were compared in Fig. 6b. It can be found that the vibration peak of OSAD at 3420 cm⁻¹ represented the vibration of O–H. This finding was due to the carboxyl and hydroxyl structures in OSAD (Girault et al. 2015). A vibration peak of C = O appeared at 1660 cm⁻¹, illustrating that there may be carboxyl groups in OSAD. The characteristic peak at 1461 cm⁻¹ corresponded to the asymmetric stretching vibration of C-H in -CH₂ and -CH₃. There was a characteristic peak at 1116 cm⁻¹, resulting from the existence of C-O bond. It can be inferred that OSAD should be dominated via alcohols, phenols, and oxygen-containing heterocyclic compounds (Guo et al. 2011). However, compared with FT-IR spectra of OSAI, there was no vibration peak at 1660 cm⁻¹ and 1461 cm⁻¹, indicating

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**Fig. 4** SEM images of OSAD at optimum dosage of OSO-101 under different magnifications: a 30,000 times, b 20,000 times, c 10,000 times, d 5000 times
that there was no carboxyl group in OSAI. OSAI had a characteristic peak of C-O bond at 1157 cm⁻¹, demonstrating that there may be ester compounds in OSAI. It was speculated that main chemical functional group should be C≡C and C=O in OSAI. Therefore, OSAI mainly contained ketones, esters, and unsaturated hydrocarbons. As a result of that, chemical conversion reactions occurred in the petroleum compounds during incineration process, mainly from alcohols and phenolic compounds to ketones. C–C bond and C-O bond may react to form ketones and olefins through dehydrogenation condensation reaction (Zhao et al. 2018).

The specific existence forms of C, O, and S elements in OS and OSAD are determined by XPS. Figures 7a, b1 and c1 are XPS peak fitting diagrams of C 1s, demonstrating that there were three main forms of C elements in OS and OSAD, namely sp²-C, C-O, and C=O (Mu et al. 2021). The specific parameters of each C element form are shown in Table S2. The sp²-C had a maximum of 72.36%, followed by C-O (26.41%), and C=O bond had a minimum of 1.23% in OS (Table S2), while sp²-C had a maximum of 70.65%, followed by C-O (27.54%), and C=O bond had a minimum of 1.82% in OSAD. Thus, C in OS and OSAD mainly existed in the form of sp²-C, which may come from the polycyclic aromatic hydrocarbon coke on OS and OSAD (Long et al. 2013). It is worth mentioning that after OS was conditioned via OSO-101, the content of sp²-C decreased from 72.36 to 70.65%, which meant that OSO-101 may have a free radical reaction, polar reaction, and redox reaction with petroleum hydrocarbons in OS, thereby polymerizing and forming condensed solid structures (Kuang et al. 2011). Figures 7b2 and c2 are the O 1s high-resolution analytical spectra of OS and OSAD. There were two main forms of O elements in OS and OSAD. Most of O elements in OS and OSAD existed in the form of quinone groups. Notably, after being conditioned by OSO-101, the content dropped from 68.68 to 57.63% (Table S3). Similarly, the content of phenolic-OH and etheric C–O–C rose from 31.32 to 42.37%, which confirmed that OSO-101 may have an oxidation–reduction reaction with OS. Figures 7b3 and c3 are the XPS spectra of S 2p. There were six main forms of S element in OS and OSAD, which were sulfate sulfur, sulfonic sulfur (sulfone), sulfide (sulfoxide), aromatic sulfur (thiophene), aliphatic sulfur, and mercaptan (Zhen et al. 2019). In OS, aliphatic sulfur existed at most 27.03% and sulfate sulfur existed at least 3.14% (Table S4). In contrast, aliphatic sulfur was at most 23.61% and sulfide (sulfoxide) was at least 9.70% in OSAD. Therefore, S in OS and OSAD was commonly present in the form of aliphatic sulfur, and the total content of organic sulfur was relatively higher. Additionally, XPS is used to determine element content on the surface of OS and OSAD (15% OSO-101), indicating that the content of C, O, and Cl decreased slightly after conditioning and the content of N and S elements rose (Table S5). XPS characterization proved successful chemical conditioning of OSO-101 on OS.
Resource recovery analysis

Analysis of calorific value change

Generally, calorific value of materials is the basic condition for fuel utilization. To realize fuel treatment of OS after dehydration and conditioning, its calorific value needs to be analyzed. After adding different OSO-101 dosages, the changes of OSAD calorific value are shown in Fig. 8. Calorific value of OSAD without OSO-101 was basically the same as that after adding fly ash, which were 10,285.13 kJ/kg and 10,354.56 kJ/kg, respectively. When the dosage of OSO-101 increased from 5 to 15%, calorific value of OSAD increased from 12,842.14 to 14,564.45 kJ/kg, which is an increase by 11.82%. As OSO-101 dosage was increasing, calorific value of OSAD slightly dropped to 14,337.15 kJ/kg. It shows that OSO-101 can not only strengthen efficient dewaterability of OS, but also increase calorific value of OS and improve feasibility of fuel utilization. Actually, the treatment process of OS was a natural curing and drying process, and its purpose was to further reduce the water content and increase calorific value through evaporation. Basically, the heat released by incineration cannot be increased by simple natural curing and drying. But the latent heat loss of vaporization was reduced due to reduction of water in OS, thereby improving incineration efficiency of OS (Zubaidy and Abouelnasr 2010). As a result, it is economically reasonable to use OSO-101 to strengthen efficient dewaterability and modification of OS, and it can effectively recover the calorific value contained in OS (Leena and Kurian 2021).

Analysis of fly ash leaching

Dioxins are composed of two groups of 210 chlorinated aromatic compounds, which are carcinogenic and teratogenic to humans. The formation of dioxins was due to that the organics containing chlorine decomposed or burned (Zhang et al. 2017a, b). Thus, chlorine is a necessary factor for the generation of dioxins, and chloride ions in OS should be reduced during OS incineration process. Heavy metal content and dioxin content of fly ash leached from incinerated products were measured (Table 1 and Table 2). The results suggested that the fly ash leached from OSAD had extremely low content of toxic heavy metals. Among them, the concentrations of beryllium (Be), total chromium (Cr), and Cr$^{6+}$ were all below detection limit. Moreover, the dioxin content in fly ash produced after OSAD incineration was reduced from 3.61 to 0.051 μgTEQ/kg. The chloride ion concentration dropped from 170.80 to 35.18 ppm which is a decrease of 79.4%, so OSAD can be fabricated to an OS fuel block with better overall performance. Additionally, the content of heavy metals and dioxin produced in fly ash after incineration can be further reduced without affecting fuel performance. The content of dioxins in fly ash after incineration was in full compliance with the “Air Pollutant Emission Standards” (GB3095-2012). OSO-101 conditioner can completely realize the harmlessness and cleaning of OS and has high environmental benefits (K and A 2007).
and economic benefits: (1) simple equipment, convenient operation, low energy consumption, no secondary sludge treatment; (2) The solid fuel after polymerization has good thermal stability and better incineration performance (14,564.45 kJ/kg). Thus, OSO-101 conditioner can completely realize harmless, clean and resource treatment.
Conclusions

After analyzing the physical and chemical properties of OS through X-ray fluorescence (XRF) and other methods, it was found that the water content in OS was relatively high (79.30%). The effects of different dosages of OSO-101 on the dewaterability of OS were experimentally studied. The results suggested that the dosage of OSO-101 to achieve the best dewaterability for OS studied in this experiment was 15% (wt.%) and $D_w$ can reach as high as 98.18%. Meanwhile, some conventional conditioning agents such as alum, lime, gypsum, and fly ash were used in comparative studies of OS conditioning, indicating that OSO-101 developed by our team was more effective in improving OS dewaterability. OSO-101 may have a free radical reaction, polar reaction, and redox reaction with petroleum hydrocarbons in OS, thereby polymerizing and forming condensed solid structures through SEM, XRD, FT-IR, and XPS characterization. Resource recovery analysis showed that oily sludge fuel blocks made of OS after OSO-101 conditioning had extremely low content of heavy metals, dioxins, and other toxic and hazardous substances leached from fly ash, not requiring secondary treatment and fully meeting environmental protection emission standards. What’s more, adding OSO-101 as a conditioner to OS can also can increase calorific value of OS and improve feasibility for fuel utilization, realizing environmental protection and energy saving of oily sludge disposal with prevalent application prospects.

Supplementary Information

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Data availability Data are available within the article or its supplementary materials. The authors confirm that the data supporting the findings of this study are available within the article and/or its supplementary materials.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication This manuscript does not contain data from any individual person, so it is not applicable in this section.

Competing interests The authors declare no competing interests.
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