Chemosphere 117 (2014) 559–566

Contents lists available at ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

Combined effects of Fenton peroxidation and CaO conditioning on sewage sludge thermal drying

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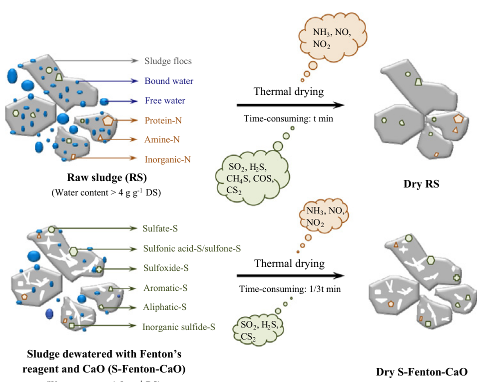
HIGHLIGHTS

- Joint application of Fenton's reagent and CaO improves sludge drying performance.
- Conditioners reduce the amounts of both free and bound water in dewatered sludge.
- Conditioners create porous structure and efficiently promote sludge heat transfer.
- Emissions of S- and N-containing gases during sludge drying are greatly suppressed.
- Decreased odor emissions are related to the variations in sludge-S and -N species.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 27 June 2014
Received in revised form 4 September 2014
Accepted 4 September 2014
Available online 4 October 2014

Handling Editor: O. Hao

Keywords:
Sewage sludge
Drying
Fenton's reagent
CaO
Sulfur transformation
Nitrogen transformation

ABSTRACT

Joint application of Fenton's reagent and CaO can dramatically enhance sludge dewaterability, thus are also likely to affect subsequent thermal drying process. This study investigated the synergistic effects of the two conditioners on the thermal drying behavior of sewage sludge and the emission characteristics of main sulfur-/nitrogen-containing gases. According to the results, Fenton peroxidation combined with CaO conditioning efficiently promoted sludge heat transfer, reduced the amounts of both free and bound water, and created porous structure in solids to provide evaporation channels, thus producing significant positive effects on sludge drying performance. In this case, the required time for drying was shortened to one-third. Additionally, joint usage of Fenton's reagent and CaO did not increase the losses of organic matter during sludge drying process. Meanwhile, they facilitated the formation of sulfate and sulfonic acid/sulfone, leading to sulfur retention in dried sludge. Both of Fenton peroxidation and CaO conditioning promoted the oxidation, decomposition, and/or dissolution of protein and inorganic nitrogen in sludge pre-treatment. As a consequence, the emissions of sulfurous and nitrogenous gases from dewatered sludge drying were greatly suppressed. These indicate that combining Fenton peroxidation with CaO conditioning is a promising strategy to improve drying efficiency of sewage sludge and to control sulfur and nitrogen contaminants during sludge thermal drying process.

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http://dx.doi.org/10.1016/j.chemosphere.2014.09.038
0045-6535/

1. Introduction

Wastewater treatment process generates large amounts of sewage sludge, which is a complex mixture of moisture, inorganic compounds, microorganism and certain undigested substances (Manara and Zabaniotou, 2012; Tyagi and Lo, 2013). Without proper treatment and disposal, this byproduct will cause serious environmental pollution. It is generally acknowledged that thermal drying represents an essential intermediate stage (Bennamoun et al., 2013). Through this process, the water content of mechanical dewatered sludge was further lowered, so as to meet the requirements of final disposal (Hassebrauck and Ermel, 1996; Vaxelaire et al., 2000). Simultaneously, costs for storage, transport and operation were also reduced sharply (Bennamoun, 2012; Bennamoun et al., 2013). Many researchers have tried to improve sludge drying efficiency by upgrading equipment or optimizing parameters. For example, Chun and Lee (2004) obtained satisfactory results through combining a contact dryer and a fluidized bed dryer together. Arlabosse et al. (2004) developed an experimental methodology to design efficient paddle dryers with vertical agitator. Yan et al. (2009) and Deng et al. (2009a) investigated the influences of system pressure, stirrer speed as well as dryer load both experimentally and theoretically. Zhu et al. (2012) examined thermal drying efficiency of sludge with various shapes at different temperatures.

Actually, by comparison to these external factors, sludge characteristics, e.g. the contents and types of water, thermal conductivity and textural properties of solid substances, are even more important to its drying behavior (Dewil et al., 2005; Léonard et al., 2003, 2004; Peeters et al., 2013). In traditional wastewater treatment plant, sludge was usually subjected to polyelectrolyte conditioning and belt press or centrifugal dewatering. After this procedure, bound water and partial free water are still tied to solid residual, and the water content decreases from 93–99.5% to 75–80% (Neyens et al., 2003; Tyagi and Lo, 2013). Subsequently, a lot of energy needs to compensate for latent heat of moisture evaporation during sludge drying process. A good way to solve this problem is to reduce initial water content of dewatered sludge (Peeters et al., 2013). Our previous studies (Liu et al., 2012b,2013b,2014) have found that joint application of Fenton's reagent (Fe^{2+}/H_2O_2) and lime (mainly presented as CaO) could significantly enhance sludge dewaterability. Deep dewatering (water content $\leqslant 1.5$ g g^{-1} DS, 60%) can then be realized directly by using filter press, which will contribute to low energy consumption in followed thermal drying. Furthermore, these conditioners may also change solids properties, and play other role in this process. By calculating, Dewil et al. (2005) demonstrated that Fenton peroxidation is effective in improving sludge thermal conductivity. Huron et al. (2010) deduced that CaO conditioning exerts a positive influence on heat and mass transfer. However, the mechanism of Fenton peroxidation and CaO addition affecting sludge drying is still enigmatic, and little research has touched upon the synergies of Fenton's reagent and CaO on thermal drying behavior of sewage sludge.

Besides, it should not be ignored that the emissions of odorous compounds, especially nitrogen- and sulfur-containing ones, pose great challenges to environment and public health, which will hinder the practical application of sludge thermal drying. According to our previous work (Liu et al., 2012a), Fenton oxidation facilitates the generation of H_2S, SO_2 and COS in conditioning process. CaO treatment reduces the release amount of each sulfurous gas, but increases that of NH_3. Unfortunately, there is a lack of relevant literatures having illustrated the individual or combined effects of the two types of conditioners on odor emission from sludge drying.

Considering the problems mentioned above, this study aims to (1) clarify the mechanism of Fenton peroxidation and CaO conditioning influence sludge thermal drying behavior; (2) elucidate the synergistic effects of composite conditioner on emissions of main S- and N-containing gases by speciation analysis; and (3) propose possible strategies for reducing energy consumption as well as polluting gases formation during sludge thermal drying.

2. Experimental

2.1. Materials

Raw sludge (RS) was collected after mechanical dewatering with cationic polymeric flocculants as conditioner from a municipal wastewater treatment plant in Wuhan, China. To ensure sample comparability, partial of this sludge was subjected to mixing with water, and the sludge slurry was treated by one or both of Fenton's reagent (Fe^{2+} 40 mg g^{-1} DS, H_2O_2 32 mg g^{-1} DS) and CaO (0.3 g g^{-1} DS) followed by filter press dewatering. Three obtained sludge were named as S–Fenton, S–CaO, and S–Fenton–CaO, respectively. The detailed sample preparation procedures and specific dosages of conditioners have been described in our earlier reports (Liu et al., 2012b, 2013a, 2014).

It can be seen from Table 1 that, Fenton treatment reduced the content of fixed carbon and increased that of volatile matter, since strong oxidation was able to destroy many stable organics (Neyens and Baeyens, 2003; Tony et al., 2008). Moreover, H_2SO_4 and $FeSO_4$ were used for adjusting pH and providing Fe^{2+}, thus S–Fenton comprised of a little more sulfur than RS. CaO addition enhanced the relative ratio of ash from 40.8% to 53.3%, resulting in reductions of 7.5%, 2.5%, and 0.2% in carbon, nitrogen, and sulfur content. It is not difficult to find that the absolute loss of carbon is only about 1%, probably due to the dissolution of some organics in alkaline condition. For the case of S–Fenton–CaO, the results of proximate and ultimate analysis were very close to that for S–CaO.

2.2. Sludge drying procedure

Experiments were performed in a specific horizontal quartz reactor (500 mm length, 36 mm i.d.) which is similar to that in our previous study (Liu et al., 2014). A commonly used drying temperature, 473 K (Zhu et al., 2012), was selected in this study. Prior to each test, the reactor was electrically heated to the set value with 2 NL min^{-1} high purity N_2 passing through. When the system had stabilized, a quartz boat carrying 2 g of sample (based on DS)

Table 1
Characteristics of different sludge samples.

Materials	Proximate analysis (wt%) (dry basis)			Ultimate analysis (wt%) (dry basis)					Water content (g g^{-1} DS)			LHV (kJ kg^{-1}) (dry ash-free basis)	
	Volatile matter	Ash	Fixed carbon	C	H	O[a]	N	S	Total	Free	Bound	Before drying[b]	After drying[c]
RS	51.4	40.8	7.8	29.2	5.0	18.3	5.7	1.0	4.4	3.6	0.8	19206	19038
S–Fenton	57.6	40.8	1.6	27.3	4.7	20.3	5.3	1.6	1.9	1.5	0.4	18250	18198
S–CaO	46.5	53.3	0.2	21.8	4.1	16.2	3.8	0.8	2.1	1.6	0.5	17111	16963
S–Fenton–CaO	47.0	52.7	0.3	20.1	4.0	17.9	3.5	1.8	1.1	0.7	0.5	15245	15178

[a] Calculated by difference.
[b] Freeze-dried sample.
[c] Sample collected from the drying experiments.

was placed in the water-cooled section of reactor, guaranteeing that sludge moisture does not evaporate in a short time. After inert atmosphere was reestablished, thermal drying started by quickly moving the quartz boat to the reaction zone. Steam and some volatiles derived from sludge were carried out by N_2 and were measured online (recorded every 1 s) by FTIR gas analyzer (Gasmet DX4000) or a hydrogen sulfide monitor (FIX550-H2S-A-G). In order to prevent condensation of water vapor, the tube between reactor outlet and analyzer inlet was maintained at 453 K. When release amount of steam approached zero, drying process had been completed. At this time, the quartz boat was rapidly pulled back to the water-cooled section and stayed for several minutes until dry solid was cooled down. Duplicated experiments were conducted to guarantee the reproducibility and validity of the results.

2.3. Sample analysis

The amount of free water was determined through differential scanning calorimetry (DSC) method (Lee and Hsu, 1995; Lee and Lee, 1995). The bound water content was calculated by the difference between the total amount of water (measured by drying at 378 K to constant weight) and the amount of free water (Vaxelaire and Cézac, 2004). To reveal the changes in solid phase, dewatered sludge were freeze-dried for representing the solids before drying, while the residuals collected from the above experiments were on behalf of ones after drying. The evolution of the surface morphology was investigated by scanning electron microscope (SEM, FEI Quanta 450 FEG). Further analysis regarding nitrogen and sulfur were carried out with elemental analyzer (Elementar, Vario Microcube) as well as X-ray photoelectron spectroscopy (XPS, VG Multilab 2000).

3. Results and discussion

3.1. Effects of Fenton peroxidation and CaO conditioning on sludge drying characteristics

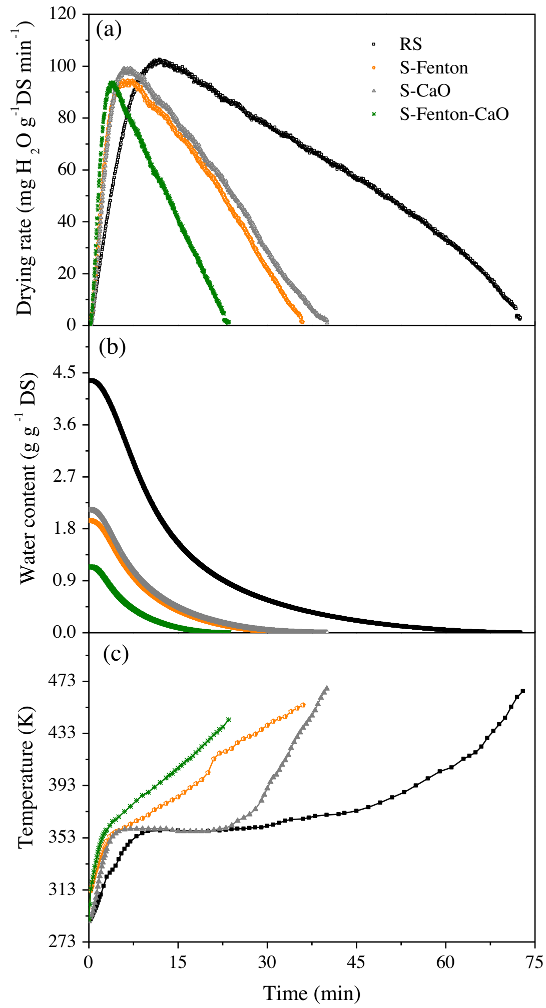

Fig. 1. Drying characteristics of different sludge samples: (a) drying rate vs. drying time, (b) water content vs. drying time, (c) the actual temperature of different sludge samples.

Fig. 1a and b describes the drying curves, and the rate as well as water content is expressed on dry base. Overall, thermal drying of each sludge sample comprised of an increasing rate stage and a decreasing rate stage. The process as to RS lasted for about 73 min, with the peak rate of 103 mg g^{-1} DS min^{-1} being detected in 11 min. When sludge was conditioned with Fenton's reagent, the water content of dewatered sludge reduced sharply from 4.4 g g^{-1} DS to 1.9 g g^{-1} DS, resulting in a shorter drying time of only 36 min long. Compared with Fenton oxidation, the effects of CaO conditioning was slightly weaker, and S–CaO drying processed 40 min long for 2.1 g g^{-1} DS of water evaporation. It is reasonable to conclude that separate application of the two conditioners was capable of improving sludge characteristics, which is in satisfactory agreement with the findings in literatures (Dewil et al., 2005; Huron et al., 2010). More excitingly, the required time (23.5 min) for S–Fenton–CaO drying was significantly shortened, proving the great advantages of combined usage of Fenton's reagent and CaO. In this case, the total amount of water in sludge

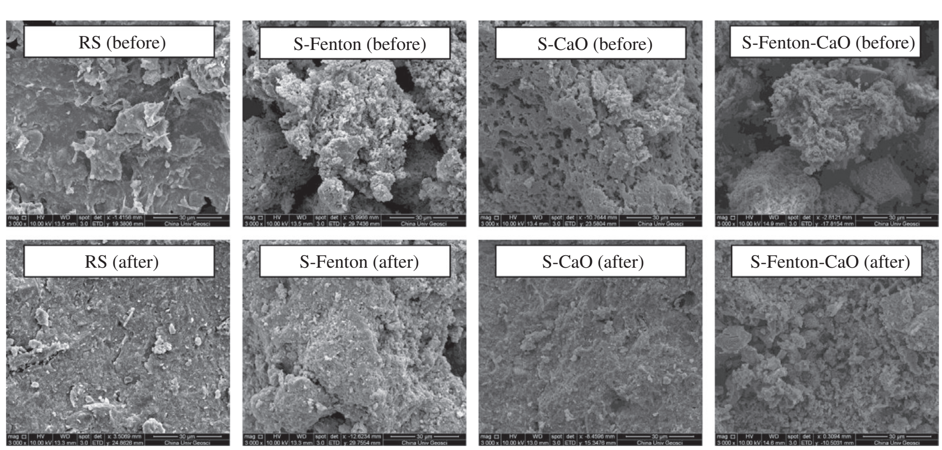

Fig. 2. SEM photographs of sludge samples before and after drying.

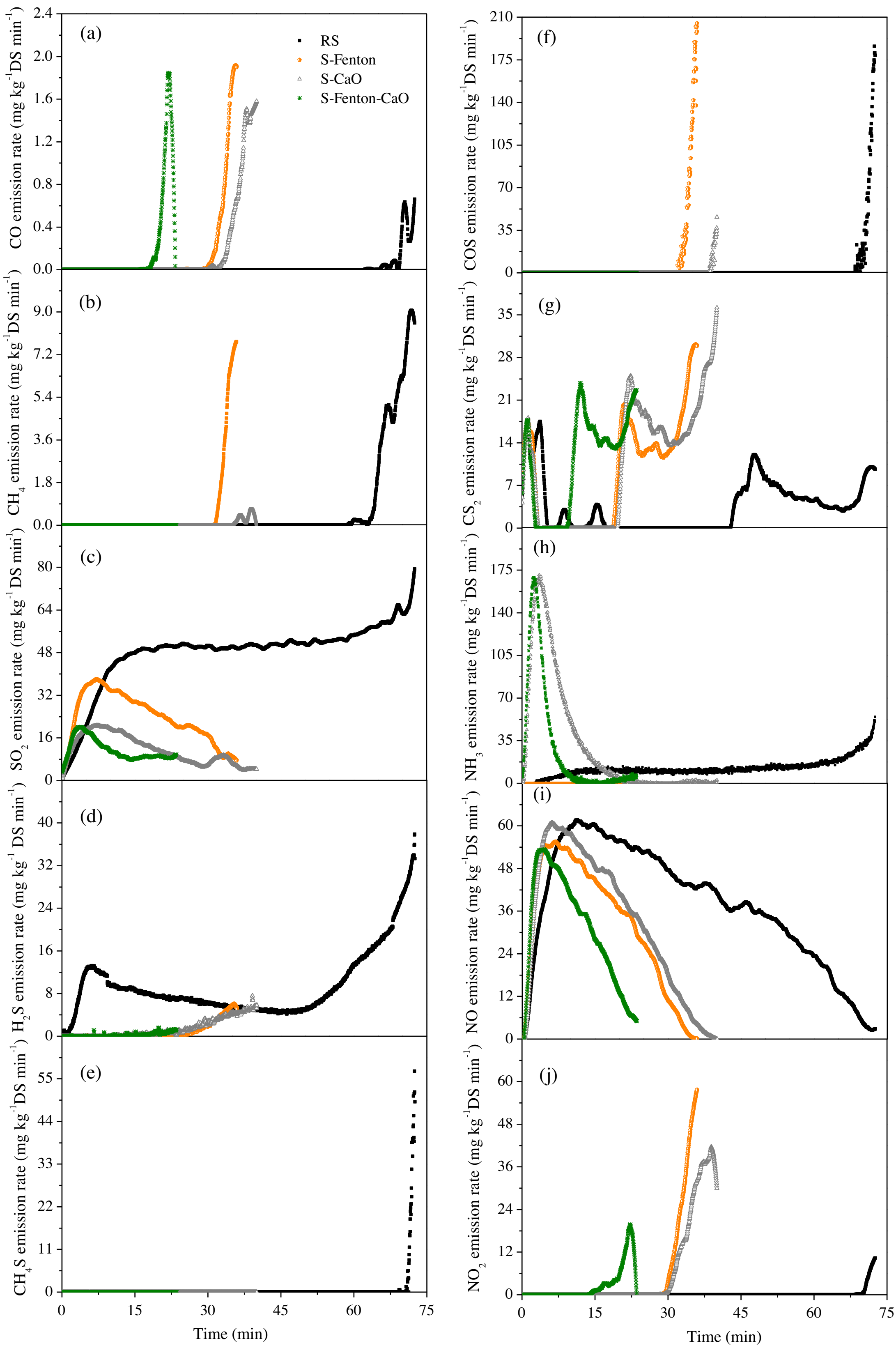

Fig. 3. Emissions of (a) CO, (b) CH_4, (c) SO_2, (d) H_2S, (e) CH_4S, (f) COS, (g) CS_2, (h) NH_3, (i) NO, and (j) NO_2 during sludge drying process.

was only 1.1 g g^{-1} DS, and the maximum drying rate of 93.8 mg g^{-1} DS min^{-1} was obtained earlier.

It is well known that, water evaporation is closely related to sludge temperature. To clarify the mechanisms involved, the actual temperature of sludge particles under the same experimental conditions were first monitored with a high sensitivity thermocouple embedded in samples. As shown in Fig. 1c, three stages were found in RS drying: first, a preheating stage during which sludge temperature increased and reached progressively its stable value of 353 K, secondly, a plateau stage during which most of moisture evaporated at a gradually reduced rate (see Fig. 1a and b), finally, a secondary heating stage during which solid temperature rose again to the highest value. In the case of S–Fenton, there was no plateau stage, indicating that oxidized sludge had better thermal conductivity. For S–CaO, the entire temperature curve is almost equal to compressed RS curve along the time axis. This means that conditioner CaO functioned through shortening each heating stage. When sludge was treated by both Fenton's reagent and CaO, effects of them on sludge temperature during drying process were superposed. As a consequence, sludge was most likely to be heated.

Additionally, comparison of Fig. 1a–c demonstrated that, increasing rate stage and preheating stage for each sludge sample were almost fully synchronized. In other words, one reason for Fenton peroxidation and CaO conditioning speeding up drying process is that they promote the heating efficiency of sewage sludge.

On further examination, changes in sludge properties were detected. Results summarized in Table 1 shows that, the amount of both free and bound water in sludge was reduced after conditioning. As Herwijn (1996) proposed, the water is chemically bound if its binding energy exceeds 1 kJ kg^{-1}, otherwise it is free. Therefore, sludge containing less bound water is more easily dried. This probably explains why S–Fenton has a slightly better drying performance than S–CaO. For S–Fenton–CaO, the decomposition of extracellular polymeric substances and the formation of rigid skeleton structure occurred simultaneously in sludge conditioning process (Liu et al., 2012b, 2013b), producing dewatered sludge with least moisture and maximum drying efficiency.

There is no denying that channels for vapor escape are also crucial to water removal. Fig. 2 shows the SEM photographs of sludge samples before and after drying. RS flocs consist of many fine layers, which will form an enclosed structure trapping the residual water. Therefore, it takes long time and more energy for steam releasing from RS. After Fenton oxidation, sludge solids turned to loose flocs composed of many small particles linked together, creating evaporation channels for moisture. After CaO conditioning, sludge flocs was somewhat like a sponge with a lot of pores therein, also providing a way out for water. Joint usage of Fenton's reagent and CaO combined the two effects together. Although thermal drying caused disappearing of some pores (see Fig. 2), the structures of S–Fenton, S–CaO and S–Fenton–CaO after drying were still more loosely than that of RS, demonstrating the existence of channels throughout the process.

Besides improving sludge drying performance, addition of these conditioners did not increase the losses of organic matter. It can be seen from Fig. 3a, CO was generated from RS in the 65th min, at which moment sludge temperature was about 423 K (Fig. 1c). This is entirely consistent with Ogada and Werther's point of view that volatiles (e.g. CO, CO_2, C_xH_y) generate when drying temperature exceeds 423 K (Ogada and Werther, 1996). Fig. 3a also demonstrated that Fenton oxidation and CaO conditioning slightly increased CO emission rate, and CaO addition slightly lowered the onset temperature of CO formation. As to CH_4, opposite results were obtained (Fig. 3b). When sludge was treated by composite conditioner, no CH_4 was detected in the whole drying process.

The results listed in Table 1 show that the differences in low heating values (LHV) of each sludge sample before and after drying were less than 200 kJ kg^{-1} (dry ash-free basis), indicating that separated or combined use of Fenton's reagent and CaO did not lead to variations in LHV reduction during sludge thermal drying.

3.2. Effects of Fenton peroxidation and CaO conditioning on S- and N-containing gas

Five types of sulfurous gases were found in thermal drying of sewage sludge. As illustrated in Fig. 3c–e, the emission rate of SO_2, H_2S, and CH_4S released from other three samples were smaller than that from RS, and the minimum value was received in S–Fenton–CaO drying. Moreover, Fenton's reagent did not affect the COS formation, while CaO addition prevented COS from generating (Fig. 3f). According to Fig. 3g, the curves of CS_2 emission rate during treated sludge drying was shaped like that during RS drying, suggesting the conditioners did not change the source of CS_2. The advanced and elevated peaks might be interrelated with elevated sludge temperatures caused by Fenton oxidation and CaO conditioning.

Three types of N-containing gases with different releasing rules were detected by FTIR analyzer in sludge drying process. As depicted in Fig. 3h, the emission rate of NH_3 derived from RS passed through three zones, including rising rate, constant rate and secondary increasing rate states. In the study conducted by Deng et al. (2009b), the third zone is decreasing rate stage other than increasing one, which is most likely associated with lower operating temperature (433 K) they used. When sludge was conditioned by Fenton's reagent, nitrogenous substances were oxidized, and thus no reducing NH_3 emitted. On the contrary, NH_3 emission was strengthened by Ca addition in first 21 min. For composite conditioned sludge, NH_3 releasing peak with same height appeared within 10 min, probably since Fenton's reagent counteracted the effects of CaO to some extent. Results shown in Fig. 3i reveal that compared with RS, NO was produced with a declining rate during treated sludge drying, especially for S–Fenton–CaO. In contrast, as illustrated in Fig. 3j, NO_2 emission was accelerated by both Fenton oxidation and CaO addition, which might be connected with the oxygen supplied by the two conditioners.

The total amount of each sulfurous and nitrogenous gas was estimated by integrating its release rate over drying time, as summarized in Table 2. Obviously, individual applying of Fenton's reagent remarkably reduced the total emissions of SO_2, H_2S, CH_4S, NH_3, and NO by 2.63, 0.65, 0.04, 0.82, and 1.58 g kg^{-1} DS, respectively. Under the same conditions, a little more CS_2, COS and NO_2 were generated. On the other hand, after single use of CaO, emissions of SO_2, H_2S, CH_4S, COS, and NO were limited, accompanied by which the formation of CS_2, NH_3, and NO_2 were slightly encouraged. If joint the two conditioners together, just the emissions of NO_2 respectively increased by 0.05 g kg^{-1} DS, the release amounts of all the rest S- and N-containing gases (except for CS_2) were reduced sharply.

To clarify the underlying causes of these polluting gases emission, the determination of sulfur and nitrogen forms in sludge samples before and after drying were conducted with XPS, and the detailed method of peak resolution and matching criteria can be found in our previous work (Liu et al., 2012a). It can be seen from Fig. 4a that, six sulfur functionalities, including sulfate, sulfonic acid/sulfone, sulfoxide, aromatic, aliphatic and inorganic sulfide, existed in RS. After Fenton oxidation and/or CaO conditioning, variations in S species primarily connected with sulfate and sulfonic acid/sulfone. As Fig. 4b presents protein, inorganic nitrogen (including ammonia and nitrates/nitrites) and amine were the dominant nitrogen species in sludge. Conditioning and dewatering process changed the relative ratio of nitrogen functionalities rather than types of them.

Statistics analysis of XPS data was then performed to provide more specific information, and semi-quantitative results are described in Fig. 5. According to Fig. 5a, after thermal drying of RS, the amounts of sulfate, sulfonic acid/sulfone, and inorganic sulfide were decreased, and this may attribute to sulfurous gases

Table 2
Total emissions of nitrogenous and sulfurous gases.

Samples	Total emissions of sulfurous gases ($g\ kg^{-1}$ DS)					Total emissions of nitrogenous gases ($g\ kg^{-1}$ DS)		
	SO_2	H_2S	CH_4S	COS	CS_2	NH_3	NO	NO_2
RS	3.48	0.68	0.04	0.23	0.25	0.82	2.74	0.01
S–Fenton	0.85	0.03	0	0.29	0.31	0	1.16	0.18
S–CaO	0.46	0.06	0	0.03	0.42	1.24	1.33	0.24
S–Fenton–CaO	0.27	0.01	0	0	0.25	0.69	0.73	0.06

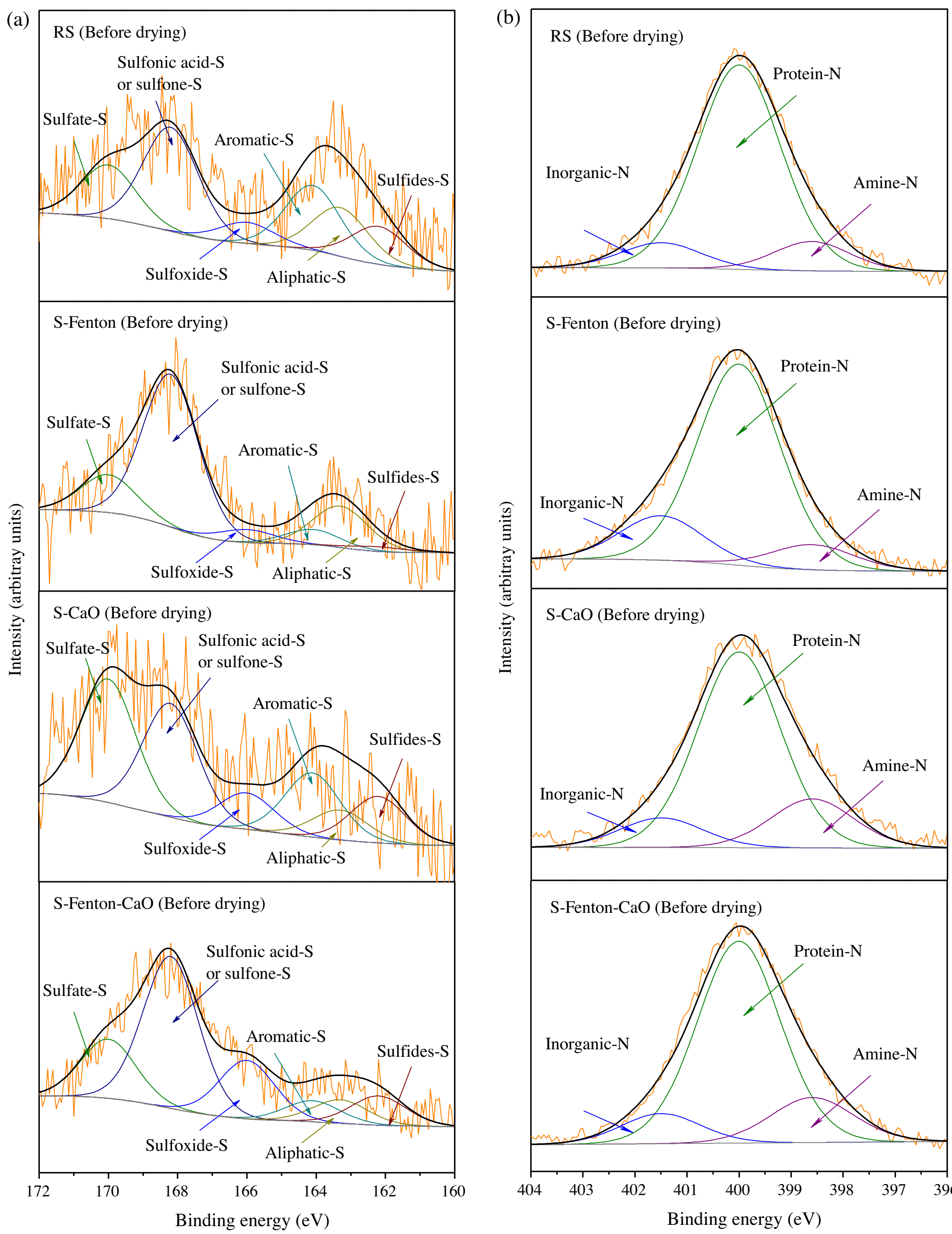

Fig. 4. (a) S 2p XPS spectra of sludge samples before drying; (b) N 1s XPS spectra of sludge samples before drying.

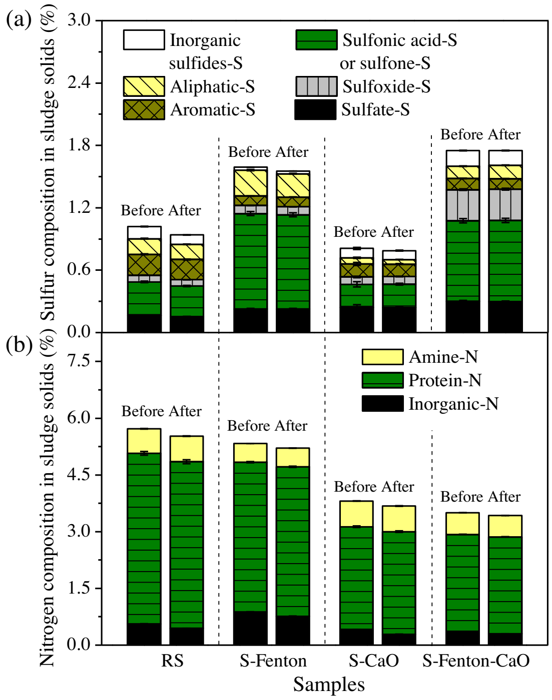

Fig. 5. (a) Sulfur, and (b) nitrogen composition of different sludge samples before and after drying.

emissions, especially for SO_2, CS_2, and H_2S. Fenton's reagent introduced some ferrous sulfate to sludge. Meanwhile, it can facilitate the sulfonation of organic compounds and the decomposition of sulfur proteins and other organic substances, resulting in increased content of sulfonic acid/sulfone and aliphatic as well as decreased content of aromatic. In addition, inorganic sulfides also turned to sulfate through oxidation reaction. Generally, sulfur retained in S–Fenton was relative more stable, and only a little reduction in aliphatic was detected after S–Fenton drying, which may need to be responsible for the enhancement in CS_2 generation. In the case of S–CaO, strong alkaline conditions promoted the solubilization of all organic sulfur, accompanied by which more sulfate (mainly refers to calcium sulfate) was formed. After S–CaO drying, a little reduction amount of aliphatic were found, confirming that increased CS_2 was indeed from aliphatic. The most exciting result is that the amount of sulfur in each species was basically unchanged during sludge thermal drying when integrating Fenton oxidation with CaO conditioning. And this is why S-containing gases emissions were effectively suppressed (see Fig. 3c–g).

Compared with sulfur, nitrogen compositions in sludge samples are simpler, as illustrated in Fig. 5b. It is not difficult to find that, after RS drying, both the content of protein and inorganic nitrogen were declined. That is to say NH_3, NO, and NO_2 formed in RS drying probably through the decomposition of protein and nitrates/nitrites, as well as the desorption of ammonia. For S–Fenton and S–CaO, partial of these unstable nitrogen species were oxidized, decomposed to form gas in conditioning process (Liu et al., 2012a), or dissolved in filtrate during mechanical dewatering procedure, leaving relative more stable nitrogen compounds in sludge. Hence, only a small reduction in inorganic nitrogen content was observed, which just accords with the less emissions of nitrogenous gases. When Fenton's reagent and CaO coexisted in dewatered sludge, less than 0.1% of nitrogen was released in drying process. Consequently, the minimum amount of N-containing gas was received in the case of S–Fenton–CaO.

4. Conclusions

Fenton's reagent and CaO were shown to positively influence the drying performance of sewage sludge, especially when they are used in combination. Fenton peroxidation and CaO conditioning significantly increased the sludge drying rate and shortened the drying time through improving its thermal conductivity, reducing the amounts of free and bound water, and creating porous structure in sludge to provide evaporation passages for moisture. Nonetheless, the two conditioners did not increase the losses of organic matter during sludge drying process. Joint usage of Fenton's reagent and CaO effectively reduced the emissions of SO_2, H_2S, CH_4S, COS, NH_3, and NO in sludge thermal drying. On the one hand, they facilitated the formation of sulfur in more stable species (e.g. sulfate, sulfonic acid/sulfone), leading to the retention of sulfur in dried sludge. On the other hand, Fenton peroxidation and CaO conditioning promoted the oxidation, decomposition, and/or dissolution of protein and inorganic nitrogen in pre-treatment process. Consequently, less nitrogenous gases were generated from dewatered sludge drying. It is a promising method to improve drying efficiency of sewage sludge with releasing very little sulfur and nitrogen contaminants during sludge thermal drying process by combining Fenton peroxidation with CaO conditioning.

Acknowledgments

This study is financially supported by Key Project of Chinese National Programs for Fundamental Research and Development (2011CB201505), National Natural Science Foundation of China (51161140330), and the National High-tech R&D Program of China (2011AA050106). Testing instruments was provided by Analytical and Testing Center of Huazhong University of Science and Technology.

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