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Original Research

Characterization and Performance of Sludge Derived Biochar as Conditioner in Textile Wastewater Sludge Dewatering

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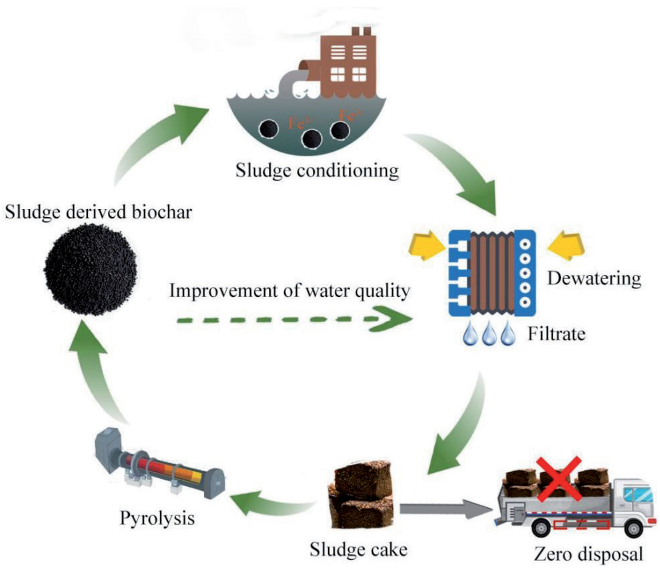
Abstract



Dewatering is a crucial stage in the treatment of sludge since it directly increases capacity and lowers the cost of subsequent handling. Enhanced dewatering of printing and dyeing sludge was experimentally investigated with combined conditioners of sludge-derived biochar and ferric chloride ($FeCl_3$) in this study. Results showed that the optimal conditioner dosages were: $FeCl_3$ of 3% dry sludge (DS) and biochar of 60% DS. Compared with raw sludge, the sludge specific resistance to filtration (SRF) decreased by 77.14%, and the moisture content of sludge decreased from 85.38% to 67.08%. In addition, the chemical oxygen demand (COD), turbidity and chromaticity of supernatant decreased

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by 32.50%, 89.20% and 77.50%, respectively. It was found that the synergistic effect existed between the two conditioners. The possible mechanism was that $FeCl_3$ aggregated small sludge particles to form large flocs and improved the dewatering performance of sludge, while biochar helped to build an incompressible and permeable sludge cake to further improve the sludge dewaterability due to its relatively hydrophobicity and large specific surface area. Therefore, combined addition of $FeCl_3$ and biochar might be a promising conditioning method to enhance textile sludge dewaterability.

Keywords: printing and dyeing sludge, sludge derived biochar, ferric chloride, sludge dewatering, skeleton building

Introduction

Printing and dyeing sludge is an unavoidable by-product present in the wastewater treatment of textile industry. According to the 2018 China Environmental Statistics Yearbook, the total emissions from textile wastewater amount to 1.9 billion tons, whereas the production of printing and dyeing sludge is up to 4.758 million tons (80% water content) [1]. Due to its large amount and high moisture content (MC), the sludge as a typical hazardous waste which contains various persistent organic pollutants will cause secondary pollution to the environment if not properly handled [2-4]. Dewatering is a necessary step in sludge treatment and disposal, which can directly reduce sludge capacity and the cost of subsequent transportation, treatment and disposal [5, 6]. The sludge dewatering supernatant has poor water quality and needs to be returned to the sewage collection tank for treatment, which will increase the pollution load of the biochemical tank and affect the quality of the effluent. Therefore, it is also of importance to have cleaner filtrate quality besides a sludge cake of lower water content.

Generally, sludge dewatering and conditioning method can be divided into chemical and physical method [7]. Chemical conditioning, such as adding ferric chloride ($FeCl_3$) cationic, polyacrylamide and so on to improve sludge dewatering. With the $FeCl_3$ dosage of 13%, the filter cake moisture decreased from 76.90% to 70.112% [8]. $FeCl_3$ has electrical charge neutralization and strong hydrolysis capacity, and biochar can be used as a skeleton builder, with strong adsorption performance [9]. Physical conditioning, such as lignite, slag and biochar were also used for sludge condition and dewatering. With the coal fly ash dosage of 273%, the filter cake moisture decreased from 86.90% to 56.52% [10]. However, chemical flocculant and physical conditioners have their individual limitations. Chemical flocculants cannot achieve deep sludge dewatering. Physical skeleton addition alone can significantly increase sludge dewatering capacity, but too much addition is unsuitable for further processing.

The conversion of sludge into biochar through pyrolysis has become an environmentally friendly and promising method, in line with the concept of "water extraction for water use" [11-14]. Li et al. prepared biochar from paper mill sludge and studied its adsorption performance for methylene blue. The results showed that the maximum adsorption capacity of biochar for methylene blue was 101.01 mg/g at 50ºC, and the adsorption behavior was in accordance with the Langmuir adsorption model [15]. Sohaimi et al. prepared the biochar from the printing and dyeing sludge and studied its effect on the removal of oil in water. Experiments showed that its maximum adsorption capacity for oil to the biochar was 173 mg/g [16]. Rice straw biochar modified by ferric chloride (RSB) was used to enhance sludge dewaterability, when the dosage of the modified RSB was adjusted to 60% dry sludge, SV_{30}%, SRF and MC were decreased to 65.8%, 1.2×10^{12} m/kg and 77.9%, respectively. But the modified biochar preparation process is so complicated [17]. Therefore, the combined methods for sludge conditioning and dewatering would be a promising approach.

However, few of previous studies have investigated the feasibility of combining biochar with chemical flocculant for the sludge conditioning and dewatering. Meanwhile, whether there is a synergistic effect between these two conditioners and what is the possible mechanism of synergistic remained unclear to be explored.

In this study, the feasibility of combined process of biochar derived from textile printing and dyeing sludge and $FeCl_3$ for enhancing the sludge dewaterability was investigated. The dosage $FeCl_3$ and biochar was optimized. And the changes of sludge characteristics and the water quality of the sludge supernatant were explained to analyze the synergistic mechanisms for the sludge conditioning and dewatering.

Materials and Methods

Chemicals and Materials

The printing and dyeing sludge was obtained from wastewater treatment plant in Ningbo, Zhejiang, China. Sludge was stored in a refrigerator at 4ºC to prevent deterioration. $FeCl_3$ were chemical pure, and the concentration stock solution was 100 g/L. Deionized water was used in the experiments for solution preparation. The characteristics of the sludge were given as Table 1.

Table 1. Main characteristics of the printing and dyeing sludge.

Characteristics	Values
pH	6.7-7.1
Total sludge solids (TSS)	11.4-12.1 $g \cdot L^{-1}$
Volatile suspended solids (VSS)	8.99-9.09 $g \cdot L^{-1}$
Specific resistance to filtration (SRF)	2.38-2.53 $\times 10^9$ $s^2 . g^{-1}$
Moisture content (MC)	98.79%-98.88%

Preparation of Biochar

Biochar was produced based on the method reported by Chen et al with slight modifications [18]. Biochar was prepared by oxygen-limited pyrolysis method: dewatered sludge was first dried in an oven at 105ºC for 24 hours to ensure it is dried completely and then placed in a crucible to be compacted and covered after cooling. Sludge was processed thermally at 500ºC for 2 h in an airtight crucible through anaerobic pyrolysis using a muffle furnace. Cooled to room temperature, the biochar was ground and passed through a 100-mesh screen.

Sludge Conditioning and Dewatering

The sludge dewaterability was evaluated with the settled volume after 30 min (SV_{30}%), specific resistance to filtration (SRF) and MC of sludge cake. Biochar was added into 200 ml sludge. After mixing at rapid agitation (120 r/min, 30 s), followed by slow agitation (60 r/min, 5 min), the mixtures were settled for 30 min to measure SV_{30}% in accordance with standard method (APHA, 1998). A standard Buchner funnel test apparatus was used for SRF determination [19]. The sludge cake was harvested after 6 min for dewatering through vacuum filtration, and dried to constant weight at 105°C to measure solid content of sludge cake. The moisture content (MC) of sludge cake was calculated by the gravimetric method. The microstructure of sludge cake was characterized with the scanning electron microscope (SEM). The coefficient of compressibility of sludge cake was measured according to Qi et al [20].

Each average value was obtained from experiments which were repeated three times. And the standard deviations were shown as error bars in the results.

Characterization and Analysis of Biochar

FT-IR infrared spectrometer (Thermo Fisher Scientific, Nicolet 6700) was used to measure the infrared spectra of the biochar and the original sludge particles. The sample was prepared by KBr pressing. The scanning range was 400-4000 cm^{-1}, the resolution was 2.0 cm^{-1}, and the scanning was performed 32 times.. The specific surface area (BET-N_2) of biochar and raw sludge particles needed to be measured with a NOVA-2000E surface analyses (Quanta chrome, NOVA-2000E), and the adsorption of high-purity liquid nitrogen was measured at a liquid nitrogen temperature (77 K). The sample was analyzed after Vacuum degassing for 12 h at 105ºC, multisite BET method was used to calculate the specific surface area; pore volume was calculated using the adsorption amount when relative pressure P/P_0 = 0.99, and pore distribution was calculated according to Barrett-Joyner-Handlebar method for N_2 desorption data.

Other Analyses

Take 100 ml in a beaker, add biochar and $FeCl_3$ according to a certain proportion (measured by sludge dry solid), stir and mix well, transfer to a 100 ml measuring cylinder, and continue to stand for 1 h. The chemical oxygen demand (COD), turbidity and chromatic of the supernatant were determined. The COD was determined to accord to the national standard (GB11914-89) and the chromaticity was determined according to the national standard method (GB11903-89). Turbidity was detected by a turbidimeter (SGZ-200 AS, Shanghai Jingke industrial co., Ltd).

The contact angles of printing and dyeing sludge and sludge derived biochar were measured by standard contact angle measuring instrument and lying drop

Table 2. Basic physicochemical properties of sludge derived biochar and printing and dyeing sludge.

Elemental analysis (wt.%) (Dry basis)	N	C	H	S	O*
Printing and dyeing sludge	4.36	55.7	7.38	4.40	22.51
Sludge derived biochar	4.20	74.5	3.90	1.29	11.36

Table 3. N_2 adsorption performance parameters.

Samples	S_{BET} (m^2/g)	Pore volume (cm^3/g)	Average pore size (nm)
Printing and dyeing sludge	0.1957	0.0014	53.2970
Sludge derived biochar	150.7100	0.1700	23.7000

method. The solid plate to be tested is placed on the sample table, and the drip rate and volume is adjusted and controlled. After the droplet is stabilized, the droplet is contacted the solid to be tested on the sample table. At this time, the dynamic continuous tracking measurement mode of the lying drop type is used to measure the contact angle and analyze its hydrophilic and hydrophobic. Contact angle measuring instrument Contact angle meter (OCA50, Germany).

Elemental analysis of sample was measured by Elementar Unicube, and the O content was calculated by the difference. Where C, H, and O represented weight percentages of carbon, hydrogen, and oxygen in printing and dyeing sludge and sludge derived biochar

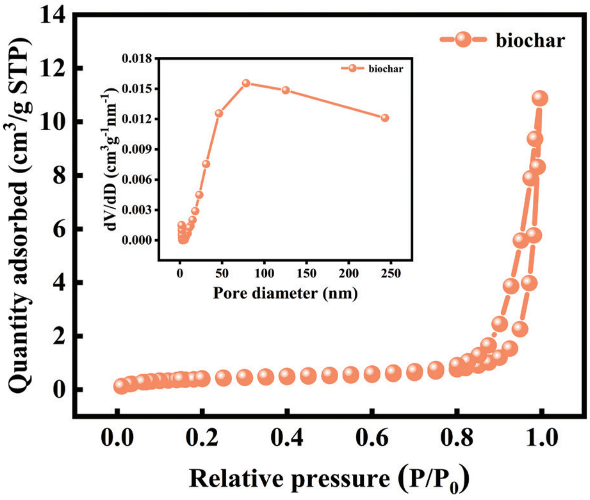

Fig. 1. N_2 adsorption/desorption isotherms of the sludge derived biochar samples.

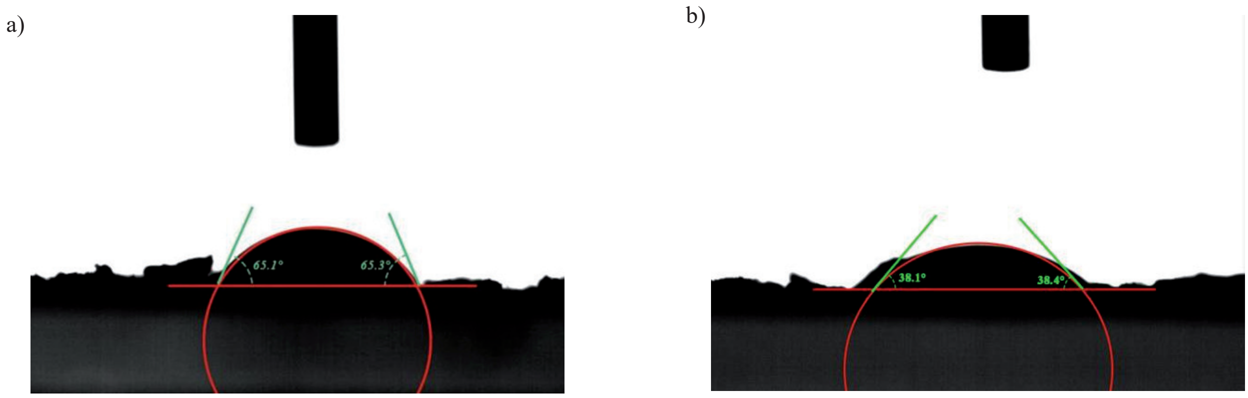

Fig. 2. Contact angle diagram of printing and dyeing sludge and sludge derived biochar a) printing and dyeing sludge b) sludge derived biochar

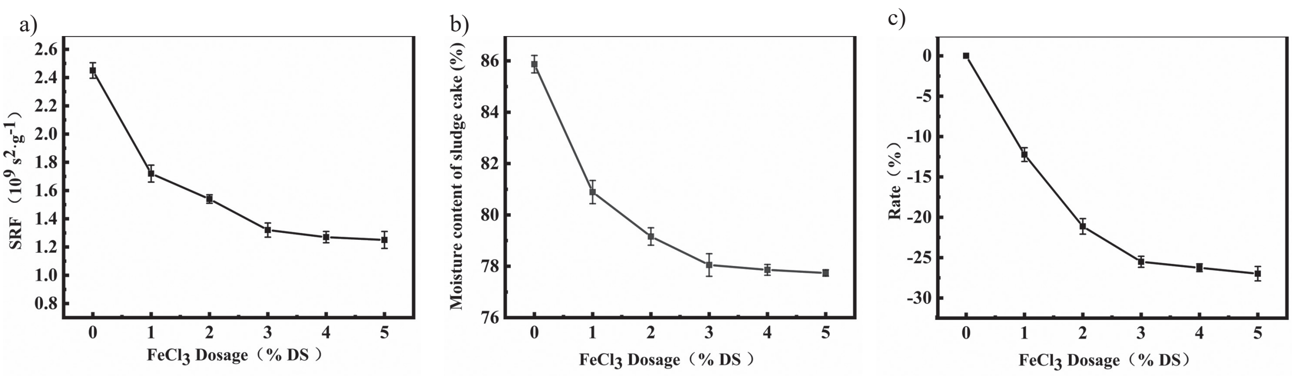

Fig. 3. Selection of the added amount of FeCl3 a) SRF b) MC c) mass change rate

respectively. Ash content of sample was heated at 750ºC for 5 h in a muffle furnace.

Results and Discussion

Physical-Chemical Characteristics of the Biochar

Biochar Pore Volume Distribution

Biochar was analyzed by nitrogen adsorption-desorption meter (BET), and the results are as follows: as shown in Fig. 1, the biochar showed type IV isotherms with H_3 hysteresis loop, indicating that they all have mesoporous structure. The pore size of the sample was mainly concentrated in the range of 2-50 nm, which further revealed the mesoporous properties. Moreover, the pore volume of biochar is 0.170 cm^3/g, and the average pore diameter is 23.700 nm, which on the one hand, the surface biochar has more mesoporous distribution and is easier to adsorb macro-molecular contaminant. On the other hand, the biochar has a distinct pore size distribution, indicating that during the preparation of biochar, the organic matter in the original sludge pyrolysis and volatilized to form pores. Pores of biochar increased which in turn increases the specific surface area of the particles. According to the results of BET-N_2 analysis, the specific surface area of the original sludge particles was 0.1957 m^2/g, and the specific surface area of biochar prepared was increased to 150.71 m^2/g, which was 670 times larger than that of the original sludge particles. Although the specific surface area is smaller compared with other adsorbents such as commercial activated carbon, biochar has more mesoporous distribution and is more likely to adsorb macro-molecular contaminants.

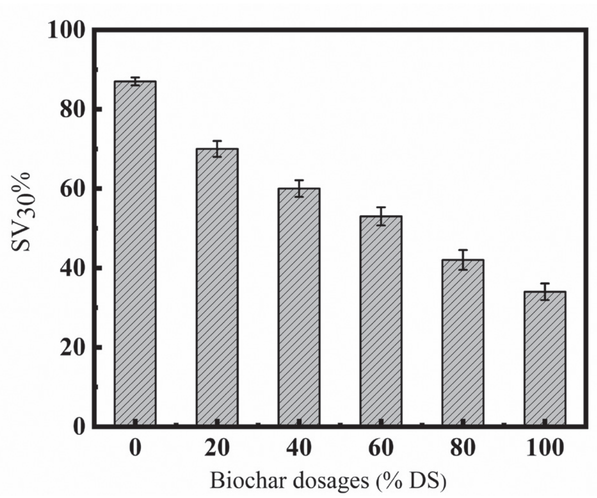

Fig. 4. Effect of biochar dosage on sludge settleability.

The Water Contact Angles of Sludge and Biochar

The smaller water contact angle indicates that the better the spreading effect of the liquid on the biochar surface, and the stronger the hydrophilic. Furthermore, as be seen from Fig. 2, the contact angles of biochar were 65.1°±0.5°, indicating highly hydrophilic surface of the biochar. The excellent hydrophilic of biochar is conducive to the full distribution in the reaction system, thus accelerating the mass transfer rate of the pollutants to the biochar surface [21].

Effect of Biochar on Sludge Dewatering

Selection of the Added Amount of $FeCl_3$

As shown in Fig. 3, The SRF of unconditioned sludge was 2.45×10^9 $s^2\cdot g^{-1}$, and the MC of sludge was 85.87 %. After conditioning sludge with $FeCl_3$, the SRF of sludge and the MC of sludge cakes decreased significantly. When the dosage of $FeCl_3$ was 3% DS, the sludge SRF decreased from 2.45×10^9 $s^2\cdot g^{-1}$ to 1.32×10 $s^2\cdot g^{-1}$, which decreased by 46.12 %. The MC of the sludge cakes decreased from 85.87% to 78.05%, and the dewatering performance was improved. At the same time, the quality of the sludge cakes was reduced by 25.52%, and the quality was significantly reduced, achieving the sludge reduction. Continue to increase the dosage, the sludge SRF and the MC of the sludge cakes did not decrease significantly. It shows that when the dosage of $FeCl_3$ is 3%

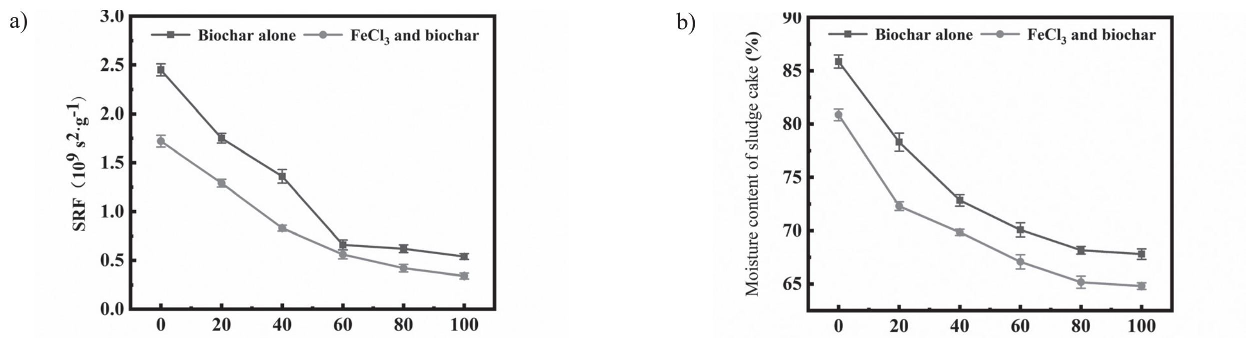

Fig. 5. Effect of biochar dosages on sludge dewatering a) SRF b) MC.

DS, the sludge conditioning effect has reached an ideal effect and was selected in the following experiments.

Effect of Biochar Dosage on Sludge Settleability

As shown in Fig. 4, The SV_{30}% of raw sludge was 87%, which indicated the settleability of raw sludge is poor. When the biochar was used, the sludge SV_{30}% decreased with the increase of biochar dosage. When sludge was conditioned with 20% DS, the SV_{30}% decreased from 87% to 64%, its settleability was enhanced. When the dosage of biochar was 60% DS, the SV_{30}% was reduced to 53%, the settleability was improved significantly. Improved sludge settleability benefits sludge concentration, shorten time of sludge settling.

Effect of Biochar Dosage on Sludge Dewatering

From a practical standpoint, the sludge pH would never be adjusted before dewatering, thus, we investigated the enhancing of sludge dewatering by using the biochar without adjusting sludge pH in this study [9]. The effect of biochar on sludge dewatering was showed in Fig. 5, Sludge SRF and MC of sludge cake decreased with the increase of biochar dosage. When adding 20% DS biochar, the sludge SRF decreased from 2.45×10^9 $s^2\cdot g^{-1}$ to 1.75×109 $s^2\cdot g^{-1}$, and the MC of the sludge cake decreased from 85.87% to 78.31%. Sludge dewaterability was improved. When the dosage was 60% DS, the sludge SRF decreased to 0.66×10^9 $s^2\cdot g^{-1}$, which was 73.06% lower than that of the raw sludge, and the MC of the cake decreased to 70.08%, which was 15.79% lower than the original sludge. When the dosage is 100% DS, the sludge SRF further reduced to 0.54×10^9 $s^2\cdot g^{-1}$.

Sludge is usually conditioned by coagulant prior to dewatering. From Fig. 5, it can be seen that when sludge was conditioned by biochar and 3% DS $FeCl_3$ together. The sludge SRF decreases significantly. When sludge was conditioned with 3% DS $FeCl_3$, the sludge SRF decreased to 1.72×10^9 $s^2\cdot g^{-1}$, which was 29.75% lower than that of the raw sludge. The MC of the sludge cake reduced to 80.99%, which was 4.98% lower than that of the original sludge. When combined conditioned with 60% DS biochar, the sludge SRF decreased to 0.56×10^9 $s^2\cdot g^{-1}$, a decrease of 77.14%, and MC of the sludge cake decreased to 67.08%, which was 18.91% lower than untreated sludge. When the dosage of biochar was 100% DS, the sludge SRF further reduced to 0.34×10^9 $s^2\cdot g^{-1}$, and the MC of sludge cake reduced to 64.78%. The sludge dewaterability conditioned with combined $FeCl_3$ and biochar was superior to that of sludge conditioned with biochar alone.

Effect of Biochar on Supernatant Quality

From Fig. 6, the COD, turbidity and chromaticity of supernatant reduced with increase of biochar dosage. After adding 20%, 40%, 60%, 80%, 100% DS biochar into sludge respectively, when dosage was 40% DS, COD, turbidity and chromaticity decreased by 16.70%, 30.14% and 30.00% respectively. When dosage was 100% DS, COD, turbidity and chromaticity decreased by 32.50%, 65.18% and 82.50% respectively.

Compared with adding biochar alone, when 3% $FeCl_3$ and biochar was added together, the reduction of COD and turbidity was more significant. When adding 40% DS biochar and 3% $FeCl_3$, the COD and turbidity decreased by 22.25% and 82.32% respectively When adding 100% DS biochar and 3% $FeCl_3$, the COD and turbidity decreased by 33.79% and 89.20% respectively. The reduction of chromatically was similar whether combined condition or not. Chromaticity removal effect of $FeCl_3$ and biochar combined conditioning and single biochar conditioning effect difference is not big.

Possible Enhancing Mechanisms of Sludge Dewatering by the Biochar

Skeleton Building

As a physical filter aid, the main function of biochar is to construct the skeleton. It can be explained from the SEM observation. The mechanism of single or combined conditioning of printing and dyeing sludge can be discussed from the following aspects.

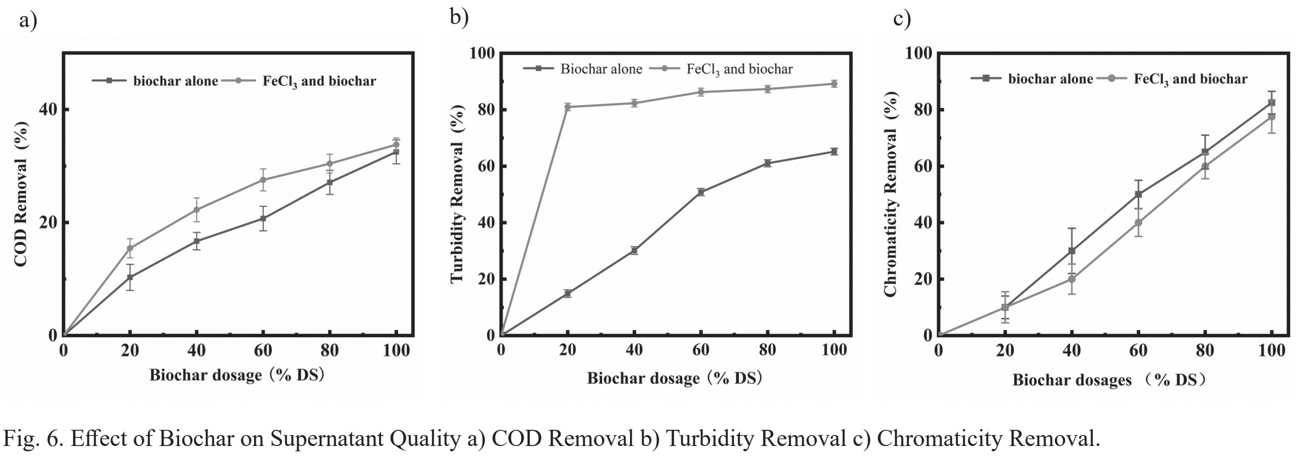

Fig. 6. Effect of Biochar on Supernatant Quality a) COD Removal b) Turbidity Removal c) Chromaticity Removal.

As the water is continuously removed during the dewatering process, the sludge particles are continuously compressed and deformed. A dense sludge cake is gradually forming on the filter medium, then the outlet passage is blocked which make water discharge more difficultly [22]. As be seen from Fig. 7a), sludge cake obtained after the dewatering of the original sludge is dense, with almost no channels. According to the study, the skeleton constructs are mainly to prevent the clogging of sludge cake and filter media, maintain the porosity and permeability of the sludge and ensure the outlet channels, thereby increase the efficiency of

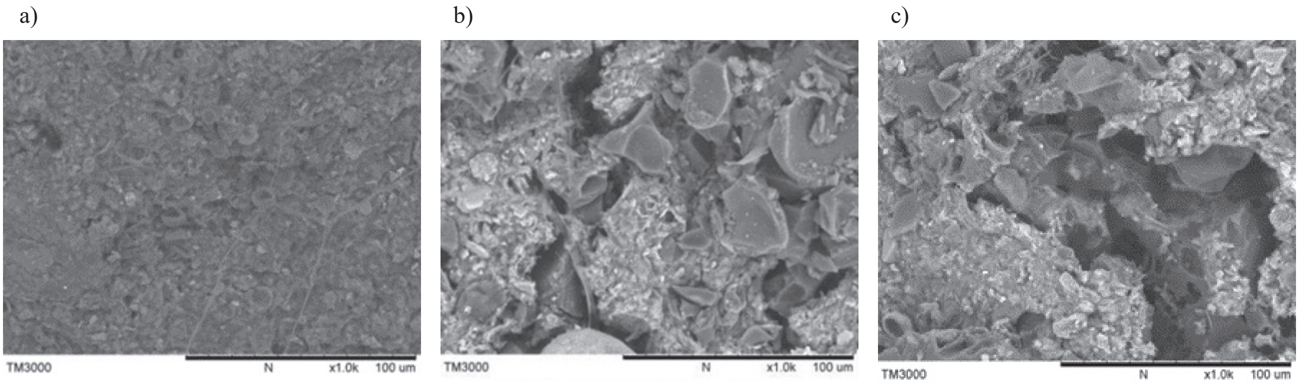

Fig. 7. SEM picture of sludge cake a) Raw sludge; b) Biochar conditioned sludge; c) Biochar and FeCl3 conditioned sludge.

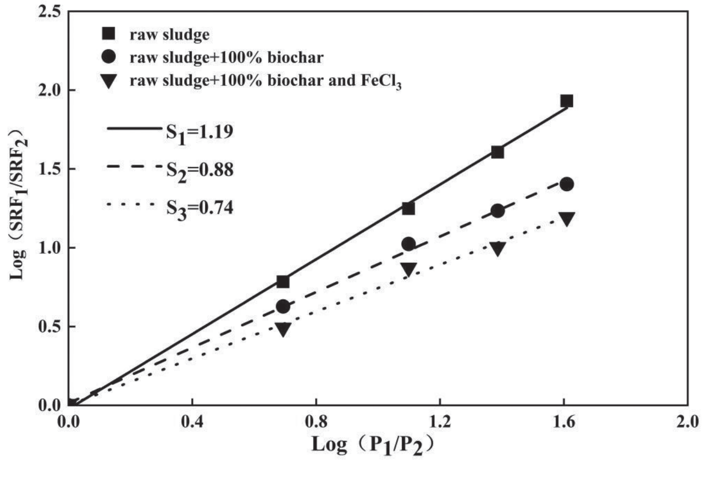

Fig. 8. Effect of biochar on sludge compressibility.

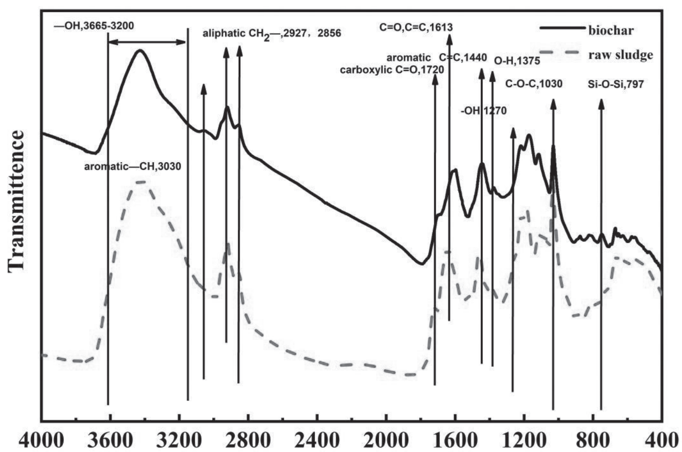

Fig. 9. Infrared spectra of biochar and raw sludge.

dewatering [23, 24]. As see seen from Fig. 7b) and 7c), the biochar conditioned sludge cake is loose, and there are many channels formed in the sludge cake compared to the unconditioned cake. After adding biochar, hard biochar plays a role in skeleton construction in the sludge compression process. As a result, permeability increased and the compressibility decreased after the flocculation sludge was conditioned.

Compressibility of Sludge

When biochar was added into sludge the settleability was improved, because the biochar particle size is small, it can enter the gap between the sludge particles after mechanical stirring. At the same time, the surface of biochar is porous with certain adsorption capacity, and can absorb small and light sludge particles. Because biochar itself has a high density and the sedimentation rate, that will take sludge particles together to solve. At the same time, the sludge particles adhere to each other, thereby accelerating the settling rate of the sludge.

$FeCl_3$ aggregates the sludge particles to form large flocs by compressing the double electron layer, trapping, bridging, etc., and squeezes out the interstitial water among the particles to improve the dewatering performance of the sludge.

The influence of the biochar on the compressibility coefficient was shown in Fig. 8. It was found that the compressibility coefficient decreased when biochar was added. The compressibility coefficient decreased from 1.19 for the raw sludge to 0.88 for the sludge conditioned with biochar dosage of 100% DS. Low compressibility means that the sludge is not easily deformed during the compression process and can maintain the permeability of the filter cake facilitating the drainage of water.

Possible Mechanism of Enhanced Supernatant Quality

The hydrophobicity of the skeleton builder has a great influence on sludge dewatering effect. The hydrophobicity of the particles is closely related to the functional groups on the surface of the particles.

Fig. 9 showed the FT-IR spectra of biochar and raw sludge. There are many functional groups on the surface of biochar, indicating that the biochar surface structure is relatively stable. The broad absorption peak (from 3200 cm^{-1} to 3665 cm^{-1}) was associated with stretching vibration of -OH [25]. That of biochar is relatively weaker than raw sludge, indicating that the hydroxyl of biochar is reduced. The absorption peaks at 3030 cm^{-1} and 1440 cm^{-1} were relatively stronger than those of the original sludge, indicating that the aromatics were enhanced in the sludge pyrolysis process. The C=C and C=O stretching vibration peaks of biochar at 1613 cm^{-1} becomes weaker, indicating that the oxygen-containing group decomposed during low temperature pyrolysis process of sludge. The absorption peak of Si-O-Si at 797 cm^{-1} was enhanced, indicating that the organic matter was volatilized during the preparation process of the sludge, and the inorganic minerals were enriched on the biochar and their components gradually increased. In general, during the preparation of the sludge, the oxygen functional groups decreased, and the aromatic rings gradually increased. The oxygen-containing functional group such as hydroxyl, carboxyl, etc. are all hydrophilic groups, indicating that biochar is relatively hydrophilic and can adsorb organic pollutants.

Fig. 1 showed the specific surface area of biochar is large with ample mesoporous pore. When biochar was applicable to the sludge dewatering, it can adsorb fine particles and dissolved macromolecules in the sludge through surface complexation, electrostatic adsorption, etc. The COD, turbidity and chrominance was reduced in the water. At the same time, the biochar particles are fine and irregular with a rough surface. After being evenly mixed with the sludge, they can intercept the sludge in the filtration process, trap large particles and colloids in the water, and further enhance the quality of filtrate.

Conclusions

Combined conditioning with $FeCl_3$ and biochar was promising to enhance printing and dyeing sludge dewaterability. The sludge particulates were first reconstructed by electrostatic neutralization of $FeCl_3$ (3% DS) to train flocs. The biochar (60% DS) addition helped the flocs to build a sludge cake with incompressible and permeable structure. After combined $FeCl_3$ and biochar conditioning, the sludge SRF and the MC decreased by 77.14% and 18.3%, respectively, compared with raw sludge. Meanwhile, the COD, turbidity and chromaticity of supernatant decreased to 25.60 mg/L, 1.43 NTU and 45, respectively. Sludge can be recycled to prepare low-cost biochar for sludge dewatering process, which provided a reliable way for the reuse of textile printing and dyeing sludge.

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Conflict of Interest

There are no conflicts to declare.

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