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Effect of rice husk flour size on sewage sludge dewaterability during composite conditioning with persulfate

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ABSTRACT

Rice husk flour (RH) was used as a skeleton builder and the effect of its size on sewage sludge dewaterability during composite conditioning with activated persulfate was studied. The results showed that the composite conditioner is effective for the capillary suction time and the specific resistance to filtration was decreased by 95.5% and 92.8%, respectively. Moreover, the water content of sludge cakes were decreased by 26% and the net sludge solid yield was increased by 93%. The RH sizes of <0.075, 0.075–0.15, 0.15–0.25 and <0.25 mm used for sludge conditioning were investigated. Results showed that the best sludge dewaterability was achieved when the RH used as skeleton builder was 0.075–0.15 mm in size. The net sludge yield and compressible coefficient of sludge cake were varied when different skeleton builder sizes were used, indicating that size influenced the sludge dewatering performance. The compressible coefficient decreased after the composite was conditioned, showing that the sludge can form a rigid and permeable lattice structure under high pressure. These features enable the formation of channels where water can flow out, which improved sludge dewaterability. All these findings showed that the net sludge yield and compressible coefficient of sludge cake was the highest and lowest, respectively, when the RH size was 0.075–0.15 mm, corresponding to the best dewatering performance.

Keywords: Rice husk flour; Skeleton builder; Sludge dewaterability; Particle size; Compressible coefficient

1. Introduction

With the wide use of activated sludge process in wastewater treatment, the growing amount of sewage sludge is a critical environmental issue [1,2]. Sewage sludge is high in moisture (>90%) and difficult to dewater, improving its dewaterability is a key point for sludge disposal for economic cost reduction [3,4]. According to the previous research, two ways are available to improve sludge dewatering performance, that is, to destroy sludge stability through chemical conditioning and reduce sludge compressibility by adding a skeleton builder [5,6]. Among chemical conditioning methods, advanced oxidation processes, such as Fenton/Fenton-like treatments [7,8], ozonation [9] and persulfate oxidation, were widely used to improve sludge dewaterability, because these processes can degrade extracellular polymeric substance (EPS) and further release bound water in sludge flocs [10–12]. Gypsum [13], slag [14], biomass and biochar [15], lime [16] and red mud [17] have been used as skeleton builder to improve sludge cake incompressibility and permeability.

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Using skeleton builder solely can remarkably increase excess sludge yield, thus, skeleton builders are always combined with chemical conditioning treatments [18].

Persulfate oxidation technology based on sulfate radical ($SO_4^{\bullet-}$) is an effective method for enhancing the sludge dewaterability [10,19–21]. Compared with hydroxyl radical ($OH^{\bullet-}$, E^0 = 2.7–2.8 V), $SO_4^{\bullet-}$ (E^0 = 2.65–3.1 V) exhibits higher oxidation-reduction potential, more adaptation for a wide range of pH and generate less harmful byproducts [22,23]. $SO_4^{\bullet-}$ should be generated by activated persulfate and Fe^{2+} activated persulfate is a commonly used method because of its relative nontoxicity and low cost [24]. Sludge dewaterability has close correlation with sludge particle size. Zhang et al. [25] used acidification and Fenton treatment to improve sludge dewatering property and investigated the effects of oxidation conditions on average sludge floc size. Results showed that Fenton oxidation can increase the floc size because EPS degradation causes the reduction of electrostatic repulsion between sludge particles. Yu et al. [26] investigated the role of temperature and $CaCl_2$ in activated sludge dewatering under hydrothermal treatment based on the diverse evolution trends between dewaterability and particle size, zeta potential, soluble protein and carbohydrates with different conditions. Results indicated the floc size is closely related with sludge dewaterability.

Rice husk (RH) is a type of biomass that has received extensive attention from many researchers [27,28]. In this study, Fe^{2+} activated persulfate combined RH flour as a novel skeleton builder was used to improve sludge dewaterability. After skeleton builder addition, the sludge floc size must be changed in accordance with many previous studies. Liu et al. [29] reported that sludge flocs are disintegrated into small particles by Fenton oxidation and the skeleton builder strengthened the conditioning efficiency by dispersing small particles. RH is a skeleton builder that effectively improves sludge dewaterability when combined with Fe^{2+}-sodium persulfate oxidation and the sludge floc sizes decrease after RH addition [30]. Skeleton builder can influence sludge dewatering performance with the change of sludge floc size. However, the effect of skeleton builder size on the sludge dewaterability remains unknown. Therefore, to narrow the knowledge gap, this study aimed to analyze the correlation between RH sizes with sludge dewaterability and present a new standing to the role of skeleton builder in sludge conditioning.

2. Materials and methods

2.1. Materials

Raw sludge (RS) was collected after mechanical dewatering from the Luobuzui Wastewater Treatment Plant, Wuhan, China. RS samples were placed in polypropylene containers and stored at 4°C in a refrigerator before the experiments. RH was obtained from Wuhan, Hubei. First, the RH was dried at 105°C until completely dehydrated. Then, the RH was ground using a ball mill (XQM-4L) at 3,500 rpm for 10 min and the RH flour was obtained. Finally, RH was passed through 0.25, 0.15 and 0.075 mm sieves, and RH sizes <0.075, 0.075–0.15, 0.15–0.25 and <0.25 mm were denoted as R1, R2, R3 and R4, respectively. Table 1 shows the main characteristics of RS, which was tested according to standard methods. The main inorganic oxide contents in RS and RH were analyzed using an X-ray fluorescence analyzer (S4 pioneer, Bruker AXS, Germany) and results are shown in Table 2.

Table 1
Main characteristics of RS

Parameter	Value
Moisture (%)	84.5 ± 0.98
pH	7.7 ± 1.56
Organic content (%)	38.9 ± 0.44
CST (s)	155 ± 1.05
SRF (×10^{13} m/kg)	4.66

Sodium persulfate (SPS) ($Na_2S_2O_8$, purity > 99.9 wt.%) and ferrous sulfate ($FeSO_4·7H_2O$, purity > 99.9 wt.%) were analytical reagent grade (Sinopharm Chemical Reagent, China) and were used without further purification. The SPS and Fe^{2+} solutions (solid to liquid ratio = 1:10) were freshly prepared immediately prior to the experiments. Deionized water was used for all the experiments.

2.2. Conditioning procedure

RS was stirred in 500 mL beakers until the sludge particles were evenly mix with the water and the water content of sludge for reaction is 95%. Then 152 mg/g DS (dry solid of sludge) SPS was added and 46 mg/g DS Fe^{2+} solution was then added as chemical conditioner. And both were stirred at 150 rpm for 5 min at 25°C, then 333 mg/g DS RH at different sizes was added and stirred at 150 rpm for 10 min. The sludge conditioned by R1, R2, R3 or R4 composites with Fe^{2+}/SPS was denoted as RH1, RH2, RH3 and RH4, respectively. The sludge conditioned with 333 mg/g DS R4, 152 mg/g DS SPS and 46 mg/g DS Fe^{2+}, 152 mg/g DS SPS, 46 mg/g DS Fe^{2+} and 333 mg/g DS RH are labeled as SR4, SF, SFR, respectively. RS was used as the control. The dosages of conditioner were determined by our previous research [30].

The conditioned sludge mixed liquor was centrifuged at 3,500 rpm for 20 min and the supernatant was filtered

Table 2
Analysis of the main oxides (%) in RS and RH

Sample	RS	RH
SiO_2	48.83	73.50
MgO	2.15	1.00
Fe_2O_3	7.57	0.47
Na_2O	0.78	0.34
CaO	4.38	1.30
Al_2O_3	23.47	13.22
K_2O	2.46	5.58
P_2O_5	6.25	1.79
Cl	0.10	1.06
SO_3	2.61	1.65

through a 0.45 μm membrane filter and collected for investigation. The sediment was used to test the water content of sludge cake.

2.3. CST and SRF

The capillary suction time (CST) and specific resistance to filtration (SRF) were used to evaluate the sludge dewatering performance. The CST was measured by a 304 M CST instrument (Triton, UK). The CST reduction efficiency (Y) was calculated as follows:

$$Y = \frac{\left(\text{CST}_b - \text{CST}_a\right)}{\text{CST}_a} \tag{1}$$

where CST_b – the CST of the RS (s), CST_a – the CST of the sludge after the conditioning (s).

The SRF was tested using a special experimental apparatus. The SRF value was calculated as follows [31]:

$$\text{SRF} = \frac{2PA^2b}{\mu\omega} \tag{2}$$

$$\omega = \frac{1}{\frac{100-\omega_i}{\omega_i} - \frac{100-\omega_f}{\omega_f}} \tag{3}$$

$$\mu = \frac{0.00178}{1 + 0.0337T + 0.000221T^2} \tag{4}$$

where SRF – specific resistance to filtration (m/kg), P – the filtration pressure (N/m^2), A – the filter area (m^2), μ – the viscosity of the filtrate (N/s/m^2), ω – the weight of the cake solids per unit volume of filtrate (kg m^{-3}), b – the slope of filtrate discharge curve (s m^{-6}), ω_i – the dry solid of 100 g raw sludge, ω_f – the dry solid of 100 g sludge cakes.

2.4. Settling property

The time-interface curve was used to evaluate the sludge setting property. Exactly 100 mL of raw and conditioned sludge mixed liquor was placed in a measuring cylinder and the interface position was recorded every 10 min for 90 min.

2.5. Water content of sludge cakes

The water content of sludge cakes was obtained as following procedure: the initial water content of sludge was diluted to 95% for equal treatment. After sludge conditioning, 30 mL of the sludge mixed liquor was transferred to a Buchner funnel for filtration by a vacuum suction filter at a pressure of 0.06 MPa for 10 min, then the filter solids were dried at 105°C for 24 h. Water content can be calculated as follows:

$$\text{Water content} = m_b - m_a / m_b \tag{5}$$

where m_b – the mass of sludge cake after filtration (g), m_a – the mass of sludge cake after drying (g).

2.6. Net sludge yield and compressible coefficient

SRF must only be used to judge conditioner effectiveness at relatively constant sludge solid quantity. Numerous solids are produced by adding a skeleton builder [15]. In this case, the net sludge yield (Y_N) represents the rate of sludge solids filtered per unit area and unit time which was used to evaluate sludge filtration process [32]. Y_N can be calculated as follows [14]:

$$Y_N\left(\text{kg/m}^2\text{h}\right) = F \times \left(\frac{2P\omega}{\mu \times t} \times \frac{1}{\text{SRF}}\right) \tag{6}$$

$$F = \frac{\text{SS}_r}{\text{SS}_r + \text{SS}_c} \tag{7}$$

where F – the correction factor, t – the filtration time (s), SS_r – the solid content of per liter RS (g), SS_c - the conditioner content of per liter RS (g).

The compressible coefficient (c) shows the internal permeability of the sludge cakes, which can be obtained by the following procedure [13,33]:

First, the SRF values were measured under different pressure (0.01, 0.02, 0.03, 0.04, 0.05 MPa) and c, then can be calculated as follows:

$$\frac{\text{SRF}_n}{\text{SRF}_0} = \left(\frac{P_n}{P_0}\right)^c \tag{8}$$

where P_n – the actual pressure (Pa), P_0 – the reference pressure (Pa), SRF_n – the SRF value under actual pressure (m/kg), SRF_0 – the SRF value under reference pressure (m/kg).

The coefficients of compressibility can be obtained from a log-log plot of $\text{SRF}_n/\text{SRF}_0$ against the pressure ratio.

2.7. Particle size and zeta potential

The particle sizes of RS and the conditioned sludges were measured using a BT-9300ST laser particle size analyzer (Bettersize, China).

The zeta potential was tested using a Malvern Zetasizer Nano ZS (Malvern Instruments Ltd., UK).

3. Results and discussion

3.1. Sludge dewatering performance with different conditioners

As shown in Fig. 1a, the CST reduction efficiency for the sludge was 12.6% for the sludge conditioned with only R4, and the CST reduction efficiency increased from 12.6% to 77.2% after sludge conditioning with Fe^{2+}/SPS. R4 addition caused the CST reduction efficiency to further increase from 77.2% to 92.8%. The SRF value decreased from 5.98 × 10^{13} m/kg of RS to 0.32 × 10^{13} m/kg of SFR. Results indicated that RH was a valid skeleton for sludge dewaterability improvement.

Fig. 1b shows that the RS interface position was 94 mL after settling for 90 min. The sludges of SR4, SF and SFR had reduced interface positions to 85, 70.6 and 69 mL, respectively, which illustrated that the SFR composite conditioner can improve the settling property.

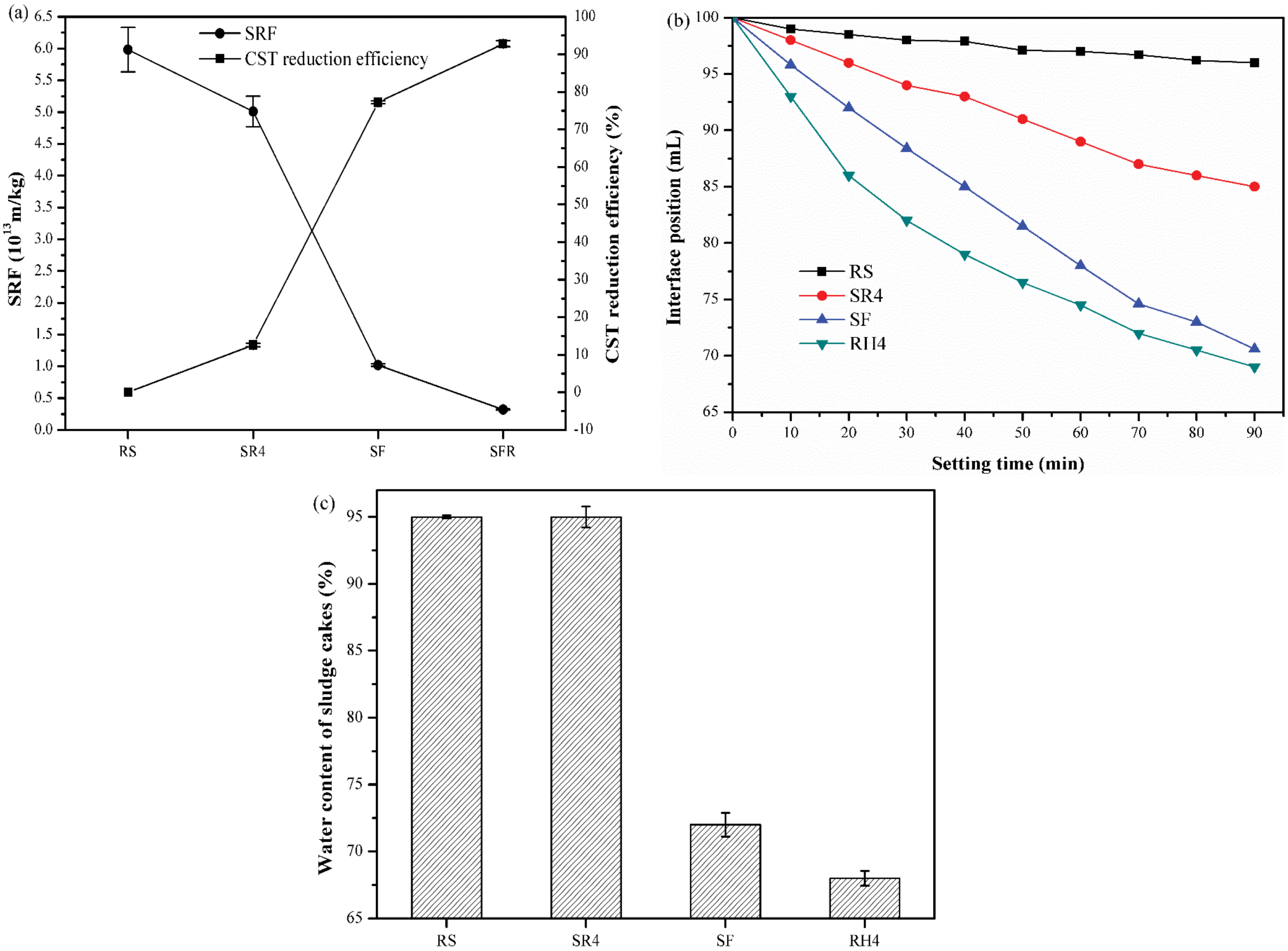

Fig. 1. Sludge dewatering performance: (a) SRF values and CST reduction efficiency, (b) settling property and (c) water content of sludge cakes of RS and sludges conditioned by different conditioners.

Fig. 1c shows that the water content of sludge cakes of RS, SR4, SF, SFR were 95%, 95%, 72% and 68%, respectively. The SFR exhibited the best sludge dewatering performance, which was consistent with the SRF and CST reduction efficiency analysis.

3.2. Sludge dewatering performance with different RH sizes

The SRF values, CST reduction efficiency, settling property and water content of sludge cakes of RS and conditioned sludges are shown in Fig. 2.

Fig. 2a shows that the SRF of RS was 5.98 × 10^{13} m/kg, and the SRF of RH1, RH2, RH3 and RH4 were 0.68, 0.27, 0.39 and 0.32 × 10^{13} m/kg, respectively. Moreover, the SRF reduction and CST reduction efficiency of RH1, RH2, RH3 and RH4 were 88.6%, 95.5%, 93.5% and 94.6%, respectively; 77%, 93%, 87% and 92.8%, respectively.

It can be seen in Fig. 2b that the interface position of RS, RH1, RH2, RH3 and RH4 were 94, 80, 67, 74 and 69 mL after settling for 90 min, respectively.

Fig. 2c shows that the water content of sludge cakes of RS, RH1, RH2, RH3 and RH4 were 94%, 73%, 68%, 70% and 68%, respectively.

According to the sludge dewatering performance analysis, RH particle size between 0.075 and 0.15 mm was good for sludge dewatering, which consistent with the previous literature [34]. This result may be because RH size <0.075 mm can float on the surface of sludge mixed liquor, and the RH size between 0.15 and 0.25 mm can block the channels and pores in sludge structure, which reduces the dewatering property.

3.3. Particle size distribution of different RH

R4 contains R1, R2 and R3, and the mass distribution is shown in Fig. 3. It can be seen from the Fig. 3 that the percentage of R2 exceeded 50%, and the percentage of R1 and R3 was 19.2% and 5.8%, respectively. Based on the sludge dewatering performance, the dewatering property of R2 and R4 were similar, and results were consistent with the mass distribution analysis.

Fig. 4 shows the particle size distributions of different RH. Results showed that the D10 of the samples was determined in the order of R3 > R2 > R1 > R4, which illustrated that the tiny particles in R3 are more than other samples. With increased particle size, the D10, D50, and D90 of R1,

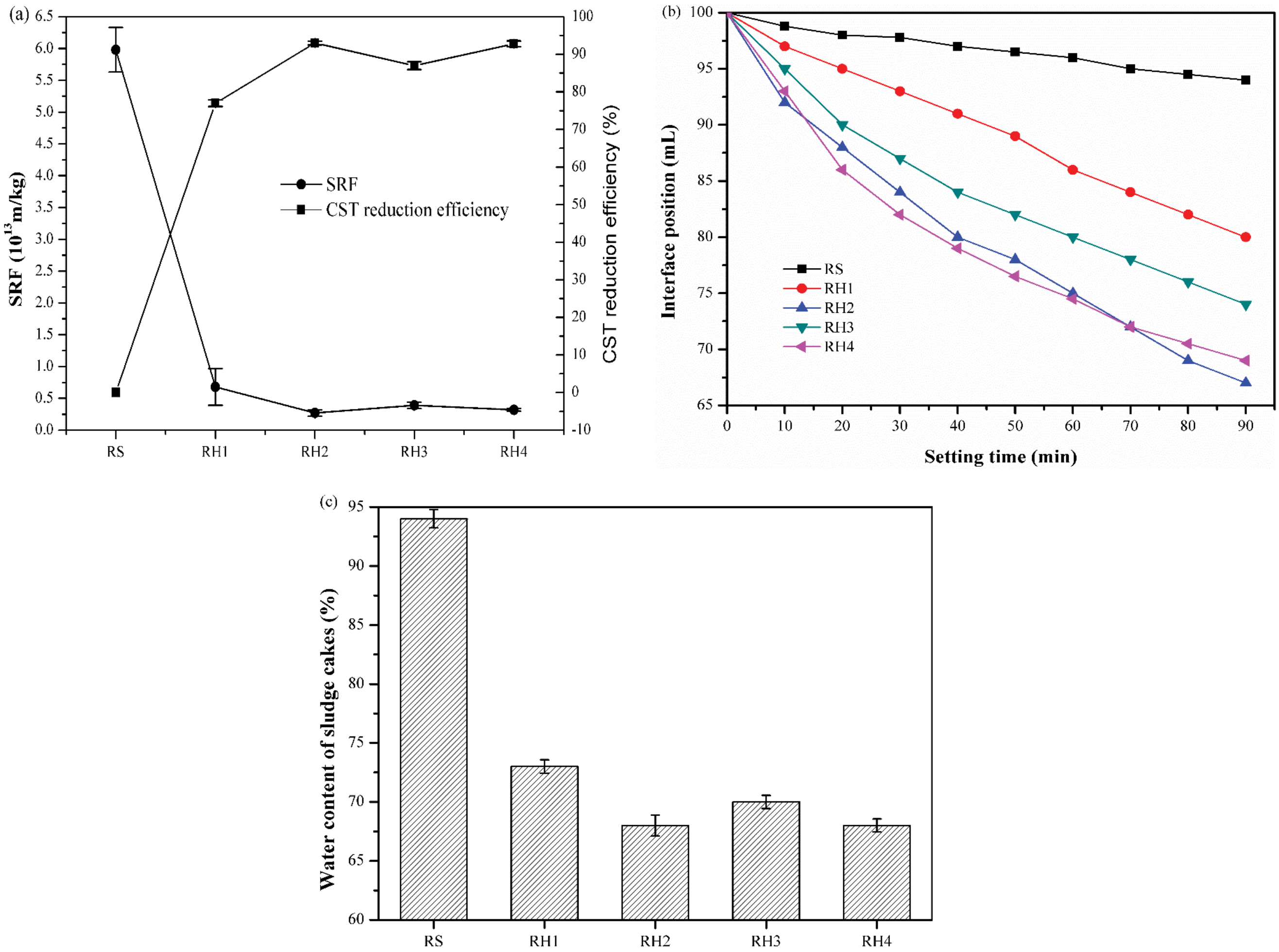

Fig. 2. Sludge dewatering performance: (a) SRF values and CST reduction efficiency, (b) settling property and (c) water content of sludge cakes of RS and sludges conditioned with different RH sizes.

R2 and R3 gradually increased. The average particle size of R4 (82.70 µm) was larger than R1 (41.03 µm) and smaller than R2 (114.7 µm) and R3 (232.7 µm). These results indicated that the matching of different sizes can change the particle size distribution of RH.

3.4. Effect of RH size on Y_N and c

The addition of a conditioner can increase the solid content of sludge consisting of the original sludge and conditioner solids, especially the addition of skeleton builder [6]. Generally, the improvement in sludge dewaterability induced an increase in net sludge yield. Fig. 5a shows the net sludge yield of sludge conditioned by different RH sizes. The result indicated that the net sludge yield of RS was 4.75 kg(m^2/h). The net sludge yield of RH1, RH2, RH3 and RH4 was 6.3, 10, 8.32 and 9.18 kg(m^2/h), respectively, which illustrated that the sludge dewaterability has improved after conditioning. Results were congruent with the sludge dewatering performance. Fig. 5b shows the coefficient of compressibility of RS and different samples. The addition of skeleton builder can decrease sludge compressibility, which indicated that sludge can form a rigid structure and maintain its original structure under high pressure, this feature can improve sludge cake porosity by the formation of pores and channels for water to flow out and thus improving the sludge dewaterability [6]. The coefficient of compressibility of RS was 1.01, which indicated the RS was difficult to dewater at a low sludge floc compaction degree [34]. After conditioning, the compressibility coefficient of RH1, RH2, RH3 and RH4 was 0.78, 0.63, 0.71, and 0.68, respectively. The lower the compressibility the better sludge dewaterability. Results showed that RH2 exhibited the best dewatering property, which consistent with the results above.

3.5. Effect of the RH size on Zeta potential

Fig. 6 shows the zeta potential of the RS and different conditioned sludges. The RS was negatively charged with a –20.9 mV zeta potential, which indicates that the RS was stable for sludge particles in RS has strong electrostatic repulsive interaction [35]. It is known that the decrease of electrostatic repulsive interaction will lead to the sludge flocs aggregate more readily which can improve the sludge

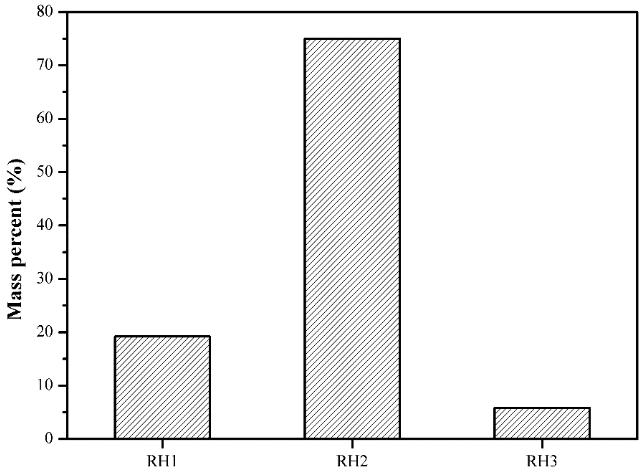

Fig. 3. Mass distribution of different rice husk flour.

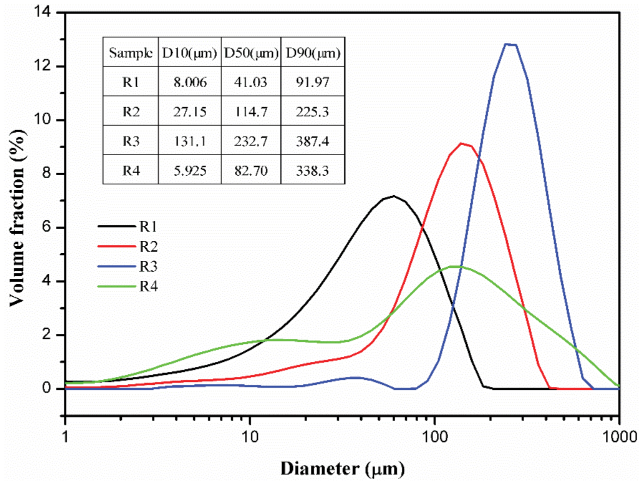

Fig. 4. Particle size distributions of the RH of different size.

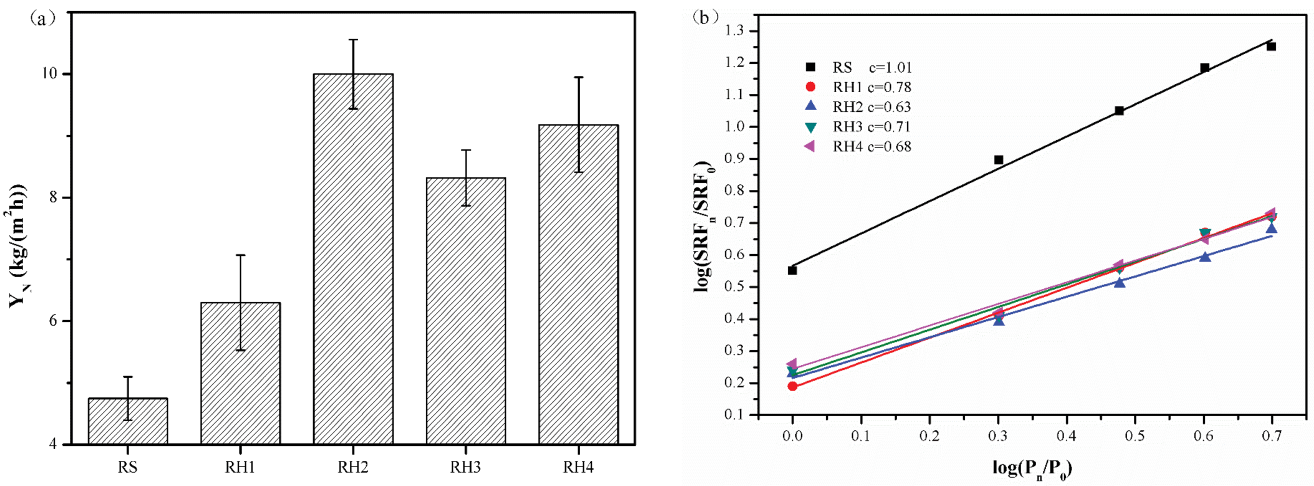

Fig. 5. Effects of different rice husk flour size on (a) Net sludge yield and (b) coefficient of compressibility.

dewaterability [8]. After conditioning, the zeta potential of RH1, RH2, RH3, and RH4 sharply increased to –1.46, –1.05, –1.2, and –2.59 mV, respectively. Results indicated that activated persulfate combined with RH can reduce the negative surface charges of sludge due to electrostatic neutralization and further improve sludge dewaterability. Based on the effect of RH size, minimal influence was applied on the zeta potential.

3.6. Effect of the RH size on the sludge particle size distribution

The particle size distribution of the sludges conditioned with different RH are shown in Fig. 7. The D10, D50, and D90 of RS and sludges conditioned with different sizes of RH are shown in Table 3. It can be seen from Fig. 7 and Table 3 that the sludge particle size gradually increased as the increase of RH size. The D50 and D90 of RH4 was 28.76 and 97.48 μm, respectively, which less than the other samples. According to the dewatering performance, the sludge conditioned by RH2 and RH4 showed better dewaterability that R1 and R3. Sorensen et al [36] reported that small-scale

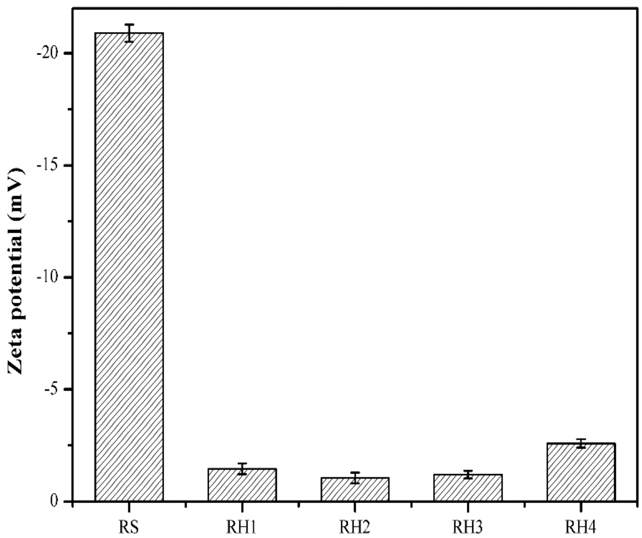

Fig. 6. Zeta potential of the RS and different conditioned sludges.

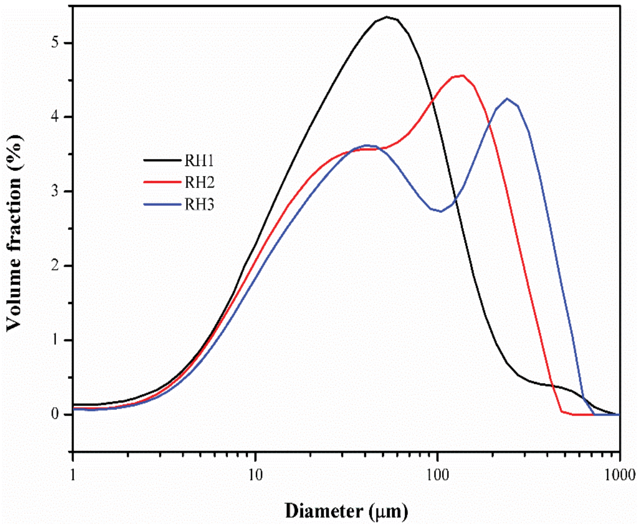

Fig. 7. Particle size distributions of the sludges conditioned with different RH sizes.

Table 3
D10, D50 and D90 of RS and sludges conditioned by different size of RH

Sample	D10 (μm)	D50 (μm)	D90 (μm)
RS	12.65	32.80	124.6
RH1	8.142	36.88	120.8
RH2	8.941	52.13	197.8
RH3	9.859	59.43	301.2
RH4	9.850	28.76	97.48

solids in sludge can influence sludge dewaterability. RH4 was composed of different sizes of RH, which exhibited complementary effect with different sizes in sludge. Therefore, the particle size of skeleton builder must be considered.

4. Conclusions

RH combined with Fe^{2+} activated persulfate was an effective composite conditioner with the highest CST and SRF reduction of 93% and 95.5%, respectively. The water content of sludge cake after filtration is 68%. The RH size affects the sludge dewatering property. When RH2 (0.075–0.15 mm) was used as a skeleton builder, Y_N increased from 4.75 kg(m²/h) of RS to 10 kg(m²/h) and the c decreased from 1.01 of RS to 0.63, and the Zeta potential increased from –20.9 mV of RS to –1.05 mV. The conditioning process can be divided into two phases, the first phase involved the addition of Fe^{2+} activated persulfate to degrade EPS in sludge and release the water in flocs. The second phase involved decreasing compressibility of sludge cake by adding a skeleton builder, which can keep the channel to discharge water. According to the results of this study, 0.075–0.15 mm was the best RH size. In conclusion, the skeleton build size can influence the physical structure of sludge and must be considered.

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