

The sedimentation characteristics of low-rank coal slurry with saline wastewater as a coagulant

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Received 13 July 2019; Accepted 22 December 2019

ABSTRACT

The hard-to-treated saline wastewater from the coal chemical industry was investigated as a coagulant in coal slurry sedimentation. In this paper, the effects of saline wastewater as a coagulant on sedimentation characteristics of low-rank coal slurry were studied. Based on the multiple-light-scattering technique, the backscattering (BS), turbiscan stability index (TSI), and supernatant thickness (ST) of the coal slurry system were analyzed at different saline dosages. The ion concentrations in coal slurry were compared with and without saline wastewater. The results showed that saline wastewater was an effective coagulant for coal slurry. Without saline wastewater, the static stability of coal slurry was stable and the BS value almost constant upon the setting time. However, the BS value increased sharply when saline wastewater was added, especially at the upper layer of coal slurry, indicating the coagulation of coal fine particles. With the increase of saline dosage, the floc size increased, resulting in a decrease in the static stability of coal slurry. The TSI value reached the maximum when saline dosage was 50.0 kg/t. Furthermore, the addition of saline wastewater can further enhance the settling rate of coal particles when anionic polyacrylamide was used as a flocculant. The fastest settling rate and minimum turbidity were obtained at the dosages of 12.5 g/t polyacrylamide and 50 kg/t saline. The beneficial effects of saline wastewater in coal slurry coagulation were studied by zeta potential analysis, which indicates that the addition of saline wastewater contributes to depress the electric double layer of coal particles in aqueous suspension, resulting in lower negative surface charges.

Keywords: Coal slurry; Saline wastewater; Low-rank coal; Multiple-light-scattering technology; Sedimentation characteristics

1. Introduction

Thousand tons of wastewater is produced in the coal chemical industry every year [1–3]. It contains a large number of organic matter and inorganic salt that reaches a magnitude of thousand milligrams per liter. When these wastewaters are discharged without proper treatment, they will adversely affect the surface and ground waters [4].

Saline wastewater is conventionally treated by physicochemical processes [5–7]. However, the physicochemical technologies are energy-consuming and the costs are relatively high. Among the physicochemical methods, natural evaporation technology is often used in the treatment of coal chemical wastewater because of its low costs. Nevertheless, the evaporation rate and the efficiency of this method are

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relatively low [8]. Biological treatment of saline wastewater is the most popular treatment method by far [9]. However, high concentrated saline wastewater is difficult to handle by this method due to the toxic effects of salinity on bacteria.

The coal chemical saline wastewater contains lots of Na⁺, Ca²⁺, Mg²⁺, Al³⁺, and other metal cations[10–12]. These cations are similar to the ingredients of coal preparation coagulants, thus could be considered to use as a coagulant in coal slurry sedimentation. Some researchers have studied the effect of cations on the sedimentation effect of coal slurry [13-15], and the results indicated that these metal cations could contribute to reducing the surface charge of coal particles and improving the sedimentation effect of coal slurry treatment. Lin et al. [16] studied the effect of Ca²⁺ on the coagulation and flocculation of coal slime water containing illite, and they found that Ca²⁺ could reduce the surface electric potential, making particles easier to coagulate. Ji et al. [17] studied the effects of solution salinity on coal slurry flocculation. The results show that the increased solution salinity could significantly reduce the electrostatic repulsion among the negatively charged solid particles, lead to more effective polymer bridging interactions, and enhance the settling effect of mineral tailings.

However, the effect of saline wastewater on the sedimentation performances of coal slurry was rarely investigated. In this paper, saline wastewater was used as a coagulant of coal slurry, and the effects of saline wastewater on the sedimentation characteristics of low-rank coal were studied. By using a multiple-light-scattering analyzer, the changes in backscattering (BS), turbiscan stability index (TSI), and supernatant thickness (ST) values of the coal slurry system with different saline dosages were studied. The effects of saline dosage on the changes in pH value, zeta potential, and ion concentration were also analyzed.

2. Experimental section

2.1. Materials

Non-caking coal sample collected in slurry from a coal preparation plant (Erdos, Inner Mongolia province) was used in this study. After being filtered and dried at 60°C, the sample was stored in a container sealed for further analysis and experiments. The particle size distribution of the coal sample was measured by a BT-9300Z laser particle size analyzer (Bettersize Instruments Ltd., China), and the main mineral composition was analyzed by X-ray diffraction (XRD).

High-concentration saline wastewater was collected from a coal chemical plant in Inner Mongolia. Crystalline salt was extracted by evaporating the high-concentration saline wastewater sample to quantify the saline dosage. Then a 5 g crystalline salt sample was added to a 100 mL volumetric flask, and a saline solution with a 50 g/L total dissolved solids was prepared before each sedimentation test. The cation concentration in the solution was analyzed by an inductively-coupled plasma mass spectrometry (ICAPQ, Thermo, Waltham, USA), while the anion concentration was analyzed by an ion chromatography system (ICS-1100, Thermo Dionex).

2.2. Methods

2.2.1. Multiple-light-scattering stability analysis

A 2-gram coal sample was added with 50 mL deionized water into a graduated cylinder equipped with a cylinder stopper. After the saline solution was added to the slurry, the cylinder was turned upside down 10 times. Then, 20 mL prepared coal slurry was transferred to a transparent glass bottle (height 55 mm, volume 30 mL) for agglomeration and sedimentation analyses by a Turbiscan Lab (Turbiscan Lab, Formulaction, France). The backscatter light detector and the infrared light source were swept from the bottom of the sample to the top. The scanning process of the sample was set in three stages. The first stage was 2 min each time, scanning 4 times. The second stage was 5 min each time, scanning 5 times and the third stage was 30 min each time, scanning 7 times, thus the entire scanning process last 4 h. Then the changes in BS, TSI, and ST values of the sample at different regions over time due to the spectrum change were recorded. In order to analyze the variation of the sedimentation process at different heights, the sample bottle was divided into three areas: top area (34-41 mm), middle area (8–34 mm), and the bottom area (0–8 mm).

2.2.2. Flocculation and sedimentation experiments

The flocculation and sedimentation experiments were carried out in a 500 mL graduated cylinder with a stopper. For each test, 18.0 g coal sample and deionized water were added to the cylinder to prepare 450 ml coal slurry with a concentration of 40 g/L. Then a certain volume of saline solution was added into the coal slurry. After turning the cylinder up and down 10 times, the flocculant, anionic poly-acrylamide (PAM) with 10 million molecular weight was added. The PAM was prepared for a solution with a mass concentration of 0.1% before each test and the PAM dosage was fixed at 12.5 g/t (dry coal). After turning the cylinder up and down for 10 times once again, the coal slurry was allowed to settle, and then the supernatant layer height over time was recorded.

2.2.3. Zeta potential

The zeta potentials were measured by a zeta potential analyzer (Zetasizer Nano ZS90, Malvern Instruments Ltd., UK).

2.2.4. Supernatant turbidity

For turbidity measurement, the suspension was allowed to settle for 8 min after the saline solution and PAM was added at each experiment. The supernatant was pipetted under 5 cm from the upper level from the cylinder into a measuring cell. The turbidity value was measured by a turbidimeter (WGZ-800, Shanghai precision instruments Co. Ltd., China).

2.2.5. Particle size distribution analysis

The particle size distribution of the sample at different saline dosages was measured by a laser particle size analyzer (BT-9300Z, Bettersize Instruments Co. Ltd., China).

2.2.6. Ion test

The cations in the solution were analyzed by an ICAPQ (ICAPQ, Thermo, Waltham, USA), while the anions were analyzed by an ion chromatography system (ICS-1100, Thermo Dionex).

2.3. Principle of Turbiscan Lab measurement

The static stability of the coal slurry was characterized by a Turbiscan Lab which could measure the static stability of samples without influence the stability of the slurry samples [18]. The backscattered light along the axis of the measuring cylinder in the turbiscan could be measured as a function of time by a synchronous optical sensor receiving light backscattered [19]. The embedded technology is based on the Multiple-light-scattering technique. BS and TSI values were used to analyze the effects of saline dosage on the coagulation and sedimentation of coal particles.

The relationship between BS value and photon transmission mean free path l^* , dispersed phase volume fraction ϕ , and particle average diameter *d* is shown in Eq. (1) [20,21].

$$BS = \frac{1}{\sqrt{l^*}} = \sqrt{\frac{3\emptyset(1-g)Q_s}{2d}}$$
(1)

where *g* is the asymmetric factor, Q_s is the extinction section divided by the geometrical cross-section. It can be seen from Eq. (1) that the BS value is inversely proportional to the square root of the *l** and particle average diameter *d*.

The TSI value can evaluate the stability of the slurry by measuring light intensity variations of a sample from the bottom to the top [22]. The determination for TSI is shown in Eq. (2).

$$TSI = \sum_{i=1}^{n} \frac{\sum h \left| \operatorname{scan}_{i}(h) - \operatorname{scan}_{i-1}(h) \right|}{H}$$
(2)

where *H* is the total height of the sample, *h* is the scanning point height, $scan_i(h)$ and $scan_{i-1}(h)$ are the light intensity values measured at *i* and *i*-1 times, respectively.

3. Results and discussion

3.1. Characteristics of the coal sample and saline wastewater

The particle size distribution of the coal sample is shown in Fig. 1. The D_{50} of the sample is 9.43 µm and more than 50% of the particles are less than 10 µm. Fig. 2 shows the XRD patterns of the coal sample. And the results indicate the presence of kaolinite, quartz, feldspar, illite, and calcite in the coal sample. Due to the negatively charged surface, repulsive interactions exist among the clay mineral particles, which makes them difficult to aggregate [23,24].

The water quality index of the prepared saline solution is shown in Table 1. The results show that the main ions in the prepared saline solution are Cl⁻, Na⁺ and SO₄^{2–}. The concentrations of Cl⁻, Na⁺ and SO₄^{2–} in saline solution are 22,652.97, 19,854.10, and 2,413.32 ppm, respectively. Compared with these three ions, the concentrations of other ion species are relatively low.



Fig. 1. Particle size distribution of the coal sample.



Fig. 2. XRD analysis of the coal sample.

Table 1 Water quality index of the saline solution

pН	9.26
Total alkalinity (mg/L)	237.51
Total hardness (mmol/L)	34.40
Chroma (PCU)	1,243.00
Conductivity (µm/cm)	86,200.00
Ion content (ppm)	
Cl-	22,652.97
SO ₄ ²⁻	2,413.32
NO ₃	186.71
PO_4^{3-}	21.4
Na⁺	19,854.10
K ⁺	26.04
Ca ²⁺	8.55
Mg^{2+}	100.64



Fig. 3. Effect of saline dosage on BS value. Saline dosages: (a) 0 kg/t; (b)12.5 kg/t; (c)25 kg/t; (d)37.5 kg/t; (e)50.0 kg/t; and(f)62.5 kg/t.

3.2. Effect of saline dosage on BS value

The variation of BS value in the coal slurry as a function of settling time at different saline dosages is shown in Fig. 3. When no saline wastewater was added, as shown in Fig. 3a, the BS values of the coal slurry almost had no changes with the increase of settling time, which indicated the coal slurry system was stable and the particles were well dispersed. However, the BS value increased sharply when saline wastewater was added, especially at the upper layer (height 34–41 mm) of coal slurry. The peak values of the BS were located at the height of 39.1, 38.5, 37.1, 37.0 and 38.0 mm, respectively, when the saline dosages were 12.5, 25.0, 37.5, 50.0 and 62.5 kg/t. The position where the BS peak was located corresponding to the phase segregation interface of the sedimentation system, so the shift of the BS peak from the top to the middle area indicates that more precipitates were formed with the increase of saline dosage. Therefore, the coal slurry system tends to be unstable with the increase of saline dosage.

3.3. Effect of saline dosage on ST value

When selected the top area (height 34–41 mm) to investigate the relationships between ST value changes with the saline dosages at a different setting time, the results are shown in Fig. 4. To take the temporal distribution of readings in an account, the settling time was divided into two periods: an initial 33 min and 33 to 240 min (4 h). When no saline wastewater was added, there was nearly no phase segregation on the top of the coal slurry after settling for 28 min. The supernatant was observed after 30 min, and the ST value increased very slowly with the increasing of settling time. It was apparent to observe the segregation phenomena on the top area of the coal slurry with the saline dosages of 37.5, 50.0, and 62.5 kg/t (with a ST value of 5.00, 6.03, and 5.55 mm, respectively, after 4 h). Besides, the ST value increased obviously with the increase of saline dosage. When 50.0 kg/t saline was added to the coal slurry, the ST value reached the maximum, and the further increase of saline dosage led to a slight decrease in ST value.

3.4. Effect of saline dosage on TSI value

The changes in TSI values were calculated by turbiscan software. When Δ TSI values are greater than 0.4, the samples are regarded as significantly different. The effect of saline dosage on TSI value is shown in Fig. 5. During the first settling period (settling time from 0 to 33 min), the TSI values for all the samples had slight changes and the samples with larger changes in TSI value at a short time were those with larger saline dosages. During the second setting period



Fig. 4. Relationship between ST value and saline dosage (a) setting time from 0 to 33 min and (b) setting time from 33 to 240 min.



Fig. 5. Effect of saline dosage on TSI value (a) settling time from 0 to 33 min and (b) settling time from 33 min to 240 min.

(settling time from 33 to 240 min), the TSI values increased significantly when the saline dosage was greater than 37.5 kg/t. Those samples also showed significant changes in BS values (Fig. 3), therefore, the static stability of the coal slurry decreases with the increase of saline dosage. The TSI value reached the maximum after 4 h when the saline dosage was 50.0 kg/t.

3.5. Effect of saline dosage on sedimentation of the coal slurry

The effects of saline dosage on sedimentation of the coal slurry are shown in Fig. 6. PAM was used as a flocculant and its dosage was fixed at 12.5 kg/t for dry coal sample. Without saline added, the setting rate of the coal particles was rather slow, however, it increased gradually with the increase of saline dosage. The changes in the settling rate showed the same trend as the changes in TSI values when saline was added to the coal slurry. The settling rate value reached the maximum at the saline dosage of 50.0 kg/t and a further increase of saline dosage led to a slight decrease in the settling rate. The results indicated that the addition of saline wastewater can further enhance the settling rate of coal particles when PAM was used as a flocculant. Therefore, the saline wastewater could be used as an effective coagulant for the sedimentation of coal slurry.

As an important index for evaluation of the sedimentation effect, the supernatant turbidities of the coal slurry were measured when different saline dosages were added, and the results are shown in Fig. 7. When no saline was added, the supernatant turbidity was 324 NTU. However, this value gradually decreased with the increase of saline dosage. When the saline dosage was increased to 50.0 kg/t, the turbidity value reached the minimum, which was 173 NTU.

3.6. Effect of saline dosage on the zeta potential of particles

The zeta potential has a significant influence on particle agglomeration in coal slurry. Fig. 8 shows the relationships between the saline dosage and zeta potential on particle



Fig. 6. Sedimentation curve with different saline dosages.

surfaces. With the increase of saline dosage, the zeta potential on particle surfaces increased and then tended to be stable. Researches showed that cations could compress the electric double layer and reduce the surface potential of coal particles, which could make particles aggregate [15,25,26]. The results show that when the concentration of cations in the solution reaches a certain value, the surface adsorption of the particles will reach a saturated state, and the zeta potential will tend to be stable.

3.7. Effect of saline dosage on pH value of the coal slurry

The relationships between the pH value of the coal slurry and saline dosage are shown in Fig. 9. As can be seen from Table 1, the pH value of the saline solution was 9.26,



Fig. 7. Effect of saline dosage on supernatant turbidity of the coal slurry.



Fig. 8. Relationship between saline dosage and the zeta potential value of coal particles.



Fig. 9. Relationship between the pH value of the coal slurry and saline dosage.

so the pH value of the coal slurry increased gradually with the increase of saline dosage. When the saline dosage was less than 37.5 kg/t, the coal slurry was weakly acidic. When 50.0 kg/t saline was added, the pH value was about 7.0.

3.8. Effect of saline dosage on particle size distribution

Fig. 10 shows the particle size distribution of the coal particles in slurry under different saline dosages. It can be seen from Fig. 10 that larger size flocs were obtained when saline was added. The floc size increased with the increase of saline dosage. When no saline was added, the $d_{20'}$, $d_{50'}$, d_{90} values of the flocs were 3.30, 9.43 and 45.57 µm, respectively. When the saline dosage increased to 50.0 kg/t, the $d_{20'}$, $d_{50'}$, d_{90} values increased to 3.81, 13.75 and 68.45 µm respectively, indicating that the addition of saline caused aggregation among the particles in coal slurry.

3.9. Variation of ion concentration in the coal slurry

Table 2 shows the effect of saline dosage on the ion concentration of the coal slurry. Without saline added, the coal slurry contains only a small number of ions dissolved from the coal and gangue minerals. The main species of cations

Table 2 Effect of saline dosage on the ion concentration of the coal slurry



Fig. 10. Effect of saline dosage on particle size distribution.

are Mg²⁺, Ca²⁺, Na⁺ and K⁺ and the concentrations of Mg²⁺, Ca²⁺ and Na⁺ are all about 5 ppm, while the concentration of K⁺ is relatively low, which is less than 0.5 ppm. The main species of anions are Cl⁻, SO₄²⁻, PO₄³⁻ and NO₃⁻. According to the results, the concentration of SO₄²⁻ is 53.6 ppm, and the concentrations of the other three anions are basically the same, which is about 5 ppm. When saline was added to the coal slurry and the dosage increased from 12.5 to 62.5 kg/t, the concentrations of Na⁺ and Cl⁻ are significantly increased from 526.42 to 3,726.84 ppm and 921.74 to 4,920.65 ppm, respectively. The concentrations of other ions only have slight changes with the increase of saline dosage, which may due to their low concentrations in coal slurry.

In order to analyze the changes in ion concentration of the coal slurry before and after coagulation, the concentration difference values were calculated by the following Eq. (3).

$$C = C_{s1} + C_{c1} - C_{c2} \tag{3}$$

where *C* is the ion concentration difference before and after coagulation in the coal slurry; C_{s1} is the ion concentration in the saline solution before coagulation; C_{c1} is the ion

Saline dosage (kg/t)				Ion concen	tration (ppm)			
	Na ⁺	K^{*}	Ca ²⁺	Mg ²⁺	Cl⁻	NO_3^-	SO_{4}^{2-}	PO ₄ ³⁻
0	5.38	0.34	4.23	6.57	4.31	2.91	53.6	0.71
12.5	526.42	3.05	16.15	16.15	921.74	3.96	116.36	0.86
25.0	1,303.17	3.49	18.90	18.90	1,849.63	5.16	239.52	1.47
37.5	2,060.72	4.02	20.27	20.27	2,820.81	9.73	325.33	2.19
50.0	2,791.91	4.73	20.98	20.98	3,853.40	13.68	463.89	3.42
62.5	3,726.84	5.32	26.14	26.14	4,920.65	26.99	571.49	4.28

Table 3 Changes in ion concentration of the coal slurry

Saline dosage (kg/t)			Io	n concentratio	n difference(ppr	n)		
	Na ⁺	K*	Ca ²⁺	Mg^{2+}	Cl⁻	NO_3^-	SO_{4}^{2-}	PO ₄ ^{3–}
12.5	273.13	-2.01	-11.58	-5.76	-11.31	3.51	-2.35	-0.20
25.0	290.54	-1.41	-13.99	-6.27	-33.08	9.78	-7.15	-0.40
37.5	327.16	-0.89	-15.01	-6.69	-98.14	12.67	-15.92	-0.30
50.0	390.13	-0.56	-15.38	-7.31	-224.61	16.19	-24.16	-0.30
62.5	249.36	-0.11	-20.20	-9.75	-385.75	10.35	-35.23	-0.30



Fig. 11. Schematic diagram of coal particles coagulated by sodium ions (a) coal particles dispersed in the water; (b) coagulation between coal particles after saline added; and (c) flocs formed after PAM added.

concentration in the coal slurry before coagulation; C_{c2} is the ion concentration in the coal slurry after coagulation.

The changes in ion concentration of the coal slurry are shown in Table 3. The concentration difference of Na⁺ is positive, indicating that the concentration of Na⁺ in the solution reduced after coagulation. Besides, the concentration difference of Na⁺ increased with the increase of saline dosage. As shown in Table 3, when saline dosage increased from 12.5 to 50.0 kg/t, the concentration difference of Na⁺ increased from 273.13 to 390.13 ppm. However, this value decreased to 249.36 ppm with a further increase of saline dosage to 62.5 kg/t. The variation of Na⁺ concentration is in good agreement with the turbiscan lab measurement results and the sedimentation tests. The schematic diagram of how sodium ions influence the coagulation of coal particles is shown in Fig. 11. When there was no saline wastewater added, the coal particles were dispersed in the slurry due to their high electronegativity. After the addition of saline wastewater, the double electric layer would be compresses and the zeta potential would decrease due to the adsorption of Na⁺ on the particle surface. Then flocs would be formed by coagulation of coal particles. Thus, the sedimentation effect of the coal slurry increased with the increase of Na⁺ concentration difference. When saline dosage exceeded 50.0 kg/t, the adsorption of Na⁺on the surface of the particles tends to be saturated.

4. Conclusions

The effects of saline wastewater on the sedimentation characteristics of low-rank coal were studied when saline wastewater was used as a coagulant. The BS, TSI, and ST values of the slurry system at different saline dosages were measured by a multiple-light-scattering stability analyzer, and the concentration changes of different ions were analyzed. The following conclusions could be made:

- The particle size distribution of the non-caking coal sample indicated that more than 50% of the particle was less than 10 μm, which may affect the sedimentation behaviors of the particles in coal slurry.
- Without saline wastewater, the BS values of the coal slurry almost had no changes with the increase of settling time, and there was nearly no phase segregation on the top region of the coal slurry. The BS value increased sharply when saline wastewater was added, especially at the upper layer of coal slurry, indicating the coagulation of coal fine particles.

- The static stability of the coal slurry decreased with the increase of saline dosage. When the saline dosage was greater than 37.5 kg/t, the TSI values increased significantly, and the TSI value reached the maximum at the saline dosage of 50.0 kg/t.
- The addition of saline caused aggregation among particles in coal slurry. With the increase of saline dosage, the floc size of the particles in coal slurry increased and the zeta potential on the particle surfaces increased greatly.
- Under the same PAM dosage, the settling rate of the coal slurry increased gradually with the increase of saline dosage. The fastest settling rate and minimum turbidity were obtained at the saline dosage of 50.0 kg/t. The results show that the saline wastewater could be used as an effective coagulant for the sedimentation of coal slurry.
- The concentration of Na⁺ in the coal slurry reduced after the coagulation process. The concentration difference of Na⁺ increased with the increase of saline dosage from 12.5 to 50.0 kg/t, which is in good agreement with the Turbiscan Lab measurement and the sedimentation results.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant no.51504262, 51604280), the Open Fund of State Key Laboratory of Water Resource Protection and Utilization in Coal Mining (Grant No. SHJT-17-42.2) and the China Scholarship Council.

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