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RESEARCH PAPER

Study on the stability of coal water slurry using dispersion-stability analyzer

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Abstract: Effect of modified lignin series and naphthalene series dispersants on the stability of coal water slurry (CWS) and sedimentation behavior of coal particles were investigated using Turbiscan Lab dispersion-stability analyzer. The results indicate that the sedimentation behavior of coal particles of CWS belongs to differential sedimentation and there is a conglobation between coal particles in CWS preparation. Stability of CWS prepared with lignin series dispersants is better than that prepared with naphthalene series, and the height and mean sedimentation rate of clarifying zone is about 68% of that of FDN when the dosage of additives is 1.0%. The Turbiscan Lab dispersion-stability analyzer can analyze the stability of CWS and also can be useful to investigate the stability mechanism of CWS.

Key Words: dispersants; coal water slurry (CWS); stability; dispersion-stability analyser

Coal water slurry (CWS) is a type of novel fuel prepared using a physical method to make the liquefaction of coal, and it is a highly-loaded suspension of coal particles in water, which contains about 60–70% coal, 30–40% water and a small amount of dispersant. CWS fuels are attractive as an alternative to fuel oil due to its lower cost and similarity to oil with respect to convenience in transporting and handling, and have received worldwide attention[1]. However, CWS is a solid−liquid suspension system and belongs to unstable thermodynamics so that it sometimes leads to solid-liquid separation related to its stability[2]. CWS with bad stability cannot meet the requirements of pumping and spraying in application. Therefore, research and evaluation of the stability of CWS are important for the applications of CWS.

Currently, four methods are mainly used in determining the stability of CWS: the standing observation method[3,4], the rod dropping method[5–7], the differential concentration method[8–9] and the residue mass percentile method[10]. The standing observation method assesses the stability of CWS by measuring the percentage of the upper bleeding water volume of CWS out of the total volume of CWS and judging whether hard sedimentation of slurry occurs. The larger water bleeding ratio is, the more hard sedimentation is, the worse stability of slurry is. In The rod dropping method, a glass rod is placed into the CWS and measures the depth of rod inside or the time required for the rod to go through the whole CWS so as to evaluate the stability of CWS. The two methods mentioned above require only simple equipments and have a wide range of applications. But some subjective factors during the measurement lead to no quantitative description and bad reproducibility of results. In the differential concentration method, the CWS sample is placed into a special vessel with a sampling place, keep it for a certain time, and then measures the differential concentration at different heights of CWS in the vessel to investigate the stability of CWS. It can be used to compare the stability of CWS quantitatively and the resulting data are relatively accurate and reliable. The disadvantage of this method is that it is hard to assess CWS with bad stability, because hard sedimentation makes the measurement of concentration rather difficult. The residue mass percentile method is mainly used to assess the static and dynamic stability of CWS and it measures the weight percentage of the residue of the CWS sample in inversion after storing and oscillating for a certain time. The method of operation is very simple in this study, and the data are accurate and steady. But it cannot investigate the sedimentation behavior of coal particles in the coal sedimentation process.

The Turbiscan Lab dispersion-stability analyzer is an optical instrument with a transmission detector and a

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backscattering detector. The migratory rate of particle, the width of sedimentation and emulsification phase, the mean particle size and the volume concentration of the highly-loaded dispersion system can be calculated directly using the multiple light reflex technology[11–13]. In this article, the effect of modified lignin series and naphthalene series dispersants on the stability of CWS is studied using the Turbis can Lab dispersion-stability analyzer. It provides references for the preparation and application of CWS.

1 Experimental

1.1 Measurement principle of dispersion-stability analyzer

When a light beam sends through the sample with low transparency, the backscattered flux BS measured by the dispersion-stability analyzer can be linked to λ^*(as a first approximation, BS is inversely proportional to the square root of λ^*):

$$BS \approx \frac{1}{\sqrt{\lambda^*}}, \quad \text{with} \quad \lambda^* = \frac{2d}{3\phi(1-g)Q_s}$$

where λ^* is the transport mean free path of photons in the dispersion system, ϕ is the volume fraction and d is the mean diameter of particle. Q_s and g are the optical parameters given by Mie theory.

When a light beam was sent through the transparent sample, the Lambert–beer law gives an analytical expression of the transmitted flux T measured by the dispersion-stability analyzer as a function of the photon mean free path λ:

$$T = T_0 e^{-\frac{2r_i}{\lambda}}, \text{with} \quad \lambda = \frac{2d}{3\phi Q_s}$$

Where r_i is the internal radius of the measurement cell, and T_0 denotes the transmittance of the continuous phase.

Therefore, the stability of CWS can be evaluated by analyzing the variation of the particle volume fraction ϕ and the particle mean diameter d in the suspension, which was reflected by the variation rate of backscattered flux BS and transmitted flux T.

1.2 Materials

Shenhua coal taken from China was used in this investigation. The proximate and ultimate analyses of coal sample are listed in Table 1 and the chemical composition of coal ash sample is given in Table 2. The coal was initially dried under vacuum at 105°C for 24 h, and then grinded by a $\phi 20\times L23$ mill to obtain two coal samples, a coarse coal and a fine coal. The coarse coal was the one comminuted for 40 min and screened through a 70-mesh screener, and the fine one was comminuted for 3 h and screened through a 100-mesh screener. The coal sample used in experiments was prepared by mixing two coal samples in the proportion of 1:1. Its particle size distribution is measured by the Eyetech-Laser particle size analyzer (Co. Ankersmid, Holland), and it is presented in Table 3.

Dispersants used in this study are naphthalene series dispersants (FDN) and modified lignin series dispersants (GCL3S)[6]. The former is supplied by the Zhanjiang Additives Factory of Guangdong Province (China) and its molecular weight is 8200. The latter with molecular weight of 15000 is prepared by our laboratory.

1.3 Preparation of CWS

The coal powders were slowly mixed in a pot containing known quantities of dispersants and deionized water. The contents were continuously stirred using a mixing device during the addition of coal, and the stirring of slurry was continued for another 10 min at 1200 r/min to ensure the homogenization of the CWS. The slurry so prepared was left for the study of its characteristics. To investigate the performance of the dispersants, no stabilizer was added to all the CWS.

Table 1 Proximate and ultimate analyses of coal sample on air dried basis

Coal sample	Proximate analysis w_{ad}/%				Ultimate analysis w_{ad}/%				HGI
	M	*A*	*V*	FC	H	O	N	S	
Shenhua coal	5.52	5.27	30.68	72.30	3.86	11.89	0.94	0.22	52~56

Table 2 Results of ash chemical composition of coal sample

Sample	Ash composition *w*/%								
	SiO_2	Al_2O_3	TiO_2	SO_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O
Shenhua coal	41.03	15.42	0.96	10.31	7.05	20.43	2.16	1.92	1.54

Table 3 Particle size distribution of coal sample

Particle size *d*/μm	<2	<5	<10	<30	<80	<100	<300	D(4, 3)
Shenhua coal	11.03	25.81	43.77	88.48	99.97	100	100	16.739

1.4 Measurements of water-bleeding ratio of CWS

The water-bleeding ratio of CWS was measured as follows. 50 mL of CWS sample was dropped into a ground glass cylinder. The permeate water characteristic was determined with the dewatering ratio of the water volume permeating from the slurries to the total slurry volume. The stability tends to decrease with the increase in dewatering ratio.

1.5 Stability measurements of CWS using dispersion-stability analyzer

The coal loading of CWS was fixed at 50% and the stability of CWS was measured using the dispersion-stability analyzer. The stability tests were carried out at 25°C in cylindrical glass tubes with a sample height of 40 mm(±0.5 mm). It was ensured that there was no samples adhered to the tube wall. The scanning procedure was divided into three steps, with a total of 80 min. First, second and last step were performed for 10, 20 and 60 min with 2, 5, and 10 min interval respectively.

Figure 1 is the scanning spectrum consisting of two parts: (a) transmission spectrum and (b) backscattering spectrum. The transmission spectrum was mainly used to investigate samples changing from transparent to opaque. CWS is black and opaque, and it can be seen from Fig. 1 (a) that two peaks occur at the top and bottom of the sample cell and the transmission flux is zero in the other zone. Therefore, the transmission spectrum was used to determine the boundary layer of CWS samples.

The backscattering spectrum was mainly used to investigate opacity samples.

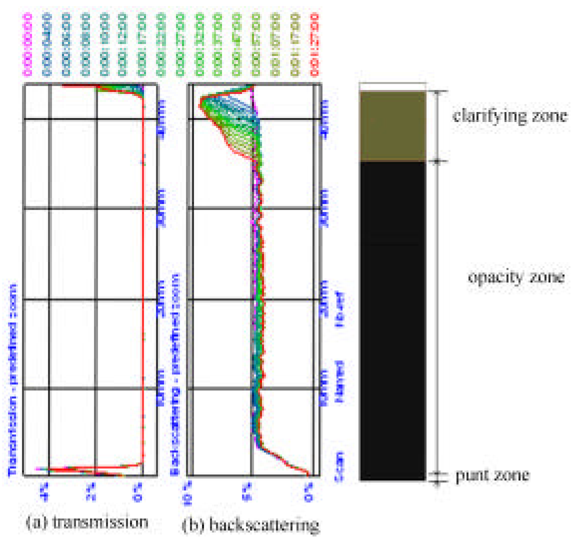

Fig. 1 Scanning of CWS

In the stability measurement, it was used to investigate the variation of clarifying zone and the sedimentation behavior of coal particles of CWS. The peak of the backscattering spectrum at the bottom was introduced by the bottom of sample cell and was defined as punt zone. It can be seen from Fig. 1 that with the increase in storing time, there is a progressive increase in the backscattering light, indicating that clarifying solution occurs at the top of the CWS sample, which was defined as the clarifying zone. Consequently, the region between the clarifying zone and the punt zone was defined as the opacity zone. If the backscattering light in the opacity zone has no change with the progress in time, it means that the appearance of the opacity zone and the clarifying zone is just the result of descending of the whole CWS, which was defined as hindered sedimentation. If the backscattering light has an asymmetric distribution and increases or decreases from above to below, it implies that there is sedimentation of coal particles, which will bring a graded distribution by degrees of the loading of CWS and defined as differential sedimentation[12]. As can be seen from Fig.1, the backscattering intensity in the opacity zone varies with the time. Thus, the sedimentation process of CWS is differential sedimentation. The relative width D_r of the opacity zone and the clarifying zone (obtained from equation below) and the variation of light intensity can be used to reflect the stability of CWS.

$$D_{\mathrm{r}} = \frac{\text{the relative width of the different zones}}{\text{the height of CWS samples}} \times 100\%$$

The variation of the coal particle diameter can be calculated by using the TLAb EXPERT software from the region chosen from the opacity zone. The average sedimentation rate of the coal particle can be computed by TLAb EXPERT from the whole clarifying zone. And the stability index of CWS can be calculated by the EASYSOFT. The stability index is related with the variation rate of transmission and backscattering intensity of CWS relative to the original. When the stability index is lower, the transmission and backscattering intensity show less variation, and when the time changes, the stability of CWS is improved. Therefore, the stability index can be used to characterize the global stability.

2 Results and discussion

2.1 Influence of dispersants on the clarifying zone of CWS

CWS is a type of solid–liquid suspension system. As the storage time increases, the coal particles begin to settle. CWS was prepared at 50% coal loading.

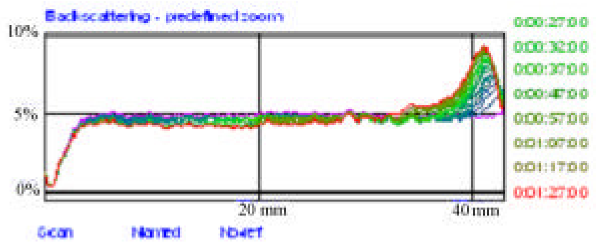

Fig. 2 Scanning of CWS adding GCL3S

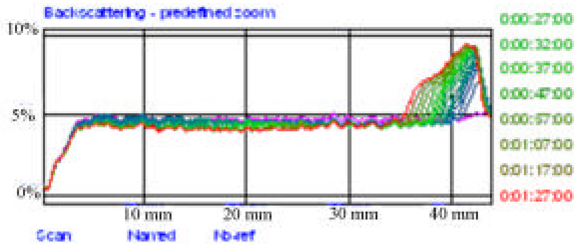

Fig. 3 Scanning of CWS adding FDN

Table 4 Effect of dispersants on the clarifying zone of CWS

Sample	Dosage w/%	Sedimentation-starting time t /min	Opacity zone D_r/%	Clarifying zone D_r/%	Sedimentation type	Mean sedimentation rate of clarifying zone /μm·min^{-1}
FDN	0.6	3.07	87.89	10.02	differential sedimentation	53.25
	0.8	2.04	85.58	12.33	differential sedimentation	65.50
	1.0	2.06	85.25	12.66	differential sedimentation	67.25
	1.2	3.5	85.53	12.38	differential sedimentation	65.75
	1.4	2.03	86.85	11.06	differential sedimentation	58.75
GCL3S	0.6	4.27	91.04	6.87	differential sedimentation	35.63
	0.8	8.92	90.85	7.06	differential sedimentation	36.63
	1.0	6.11	89.26	8.65	differential sedimentation	44.88
	1.2	5.99	89.38	8.53	differential sedimentation	44.25
	1.4	6.88	91.98	5.93	differential sedimentation	30.75

The clarifying zone of slurry after standing for 80 min was chosen as the research subject to investigate the effect of modified lignin series and naphthalene series dispersants on the clarifying zone of CWS. Figs. 2 and 3 show the scanning spectrum of CWS prepared using FDN and GCL3S, respectively. It is found that the backscattering intensity of the opacity zone of CWS using FDN and GCL3S varies with time, which indicates that the sedimentation process of CWS is differential sedimentation. Obviously, the clarifying zone of CWS prepared with FDN is larger than that of GCL3S, and the relative width of regions and the mean sedimentation rate of coal particles in the clarifying zone are listed in Table 4.

As for CWS, the later the sedimentation time begins, the narrower the width D_r of the clarifying zone is, and the more stable the CWS is. It can be seen from Table 4 that the sedimentation starting time of coal particles of CWS with added FDN is earlier than that of GCL3S. With the increase in the dosage of FDN, the height and the mean sedimentation rate of the clarifying zone increase at the beginning. And when the dosage of dispersant exceeds a certain value, both begin to decrease. Under the same condition, the height and mean sedimentation rate of clarifying zone of CWS with added GCL3S become significantly lower than that of FDN. And when the dosage of dispersants is 1.0%, the height and mean sedimentation rate of clarifying zone of CWS with GCL3S is about 68% of that of FDN, indicating that the stability of CWS prepared with GCL3S is better than that of FDN.

2.2 Influence of dispersants on the particle size in the opacity zone of CWS

The coal loading was held constant at 50%. The part (20.0–20.1 mm) of the opacity zone of CWS was taken to investigate the influence of store time on coal particle size and the results are shown in Figs. 4 and 5. It can be seen that the coal particle size of CWS using FDN is initially 44.08 μm. After storing 60 min, the coal particle size changes to 51.54–53.16 μm, increasing by 20.05%. However, the initial coal particle size of CWS using GCL3S is 43.21 μm and after storing 60 min, the coal particle size changes to 47.41–51.54 μm, increasing by 19.4%. Compared with the mean coal particle size 16.74 μm measured by the laser particle size analyzer shown in Table 3, the coal particle size of CWS is 2–4 times larger. It is evident from the data that the agglomeration occurs among the coal particles during the slurry preparation. Moreover, the particle size progressively increases with the increase in store time, but the particle size does not change when the time exceeds 40 min.

2.3 Influence of dispersant on stability of CWS

The coal loading was held constant at 50%. The effects of the dispersants on the stability of CWS were investigated using the traditional water bleeding ratio method and the stability index method measured by the dispersion-stability analyzer. The results are shown in Figs. 6 and 7.

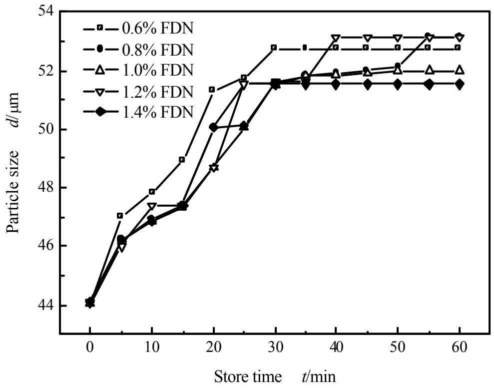

Fig. 4 Effect of FDN on the coal particle size of CWS

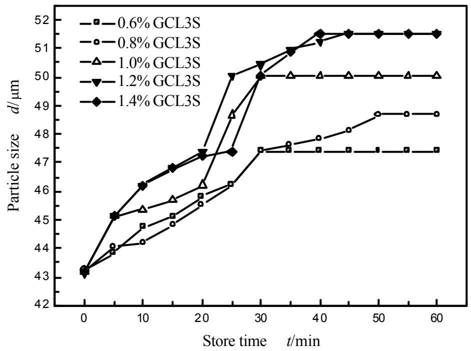

Fig. 5 Effect of GCL3S on the coal particle size of CWS

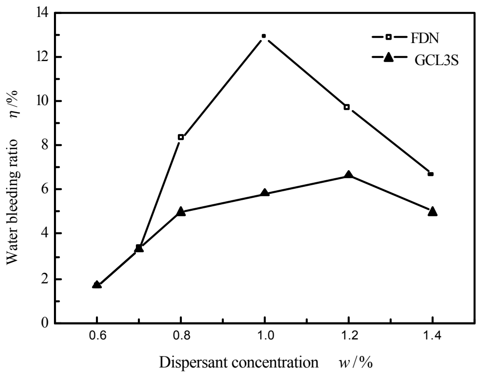

Fig. 6 Effect of dispersants amount on the water bleeding ratio of CWS

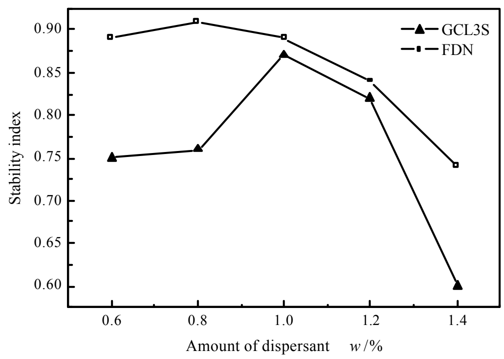

Fig. 7 Effect of dispersants amount on the stability index of CWS

It can be seen from Fig. 6 that the standing water-bleeding ratio increases with the increase in the dosage of FDN at the beginning and decreases when the dosage of FDN exceeds 1.0%. Under the same condition, the water bleeding ratio of slurry with the dispersant GCL3S is less than that of FDN. Especially, the water-bleeding ratio of slurry with the dispersant GCL3S is only 45.3% compared with FDN when the dosage of dispersants is 1.0%.

It can be found from Fig. 7 that the stability index decreases when the dosage of FDN increases, indicating the increase in the stability of CWS. However, with the increase in the dosage of GCL3S, the stability index increases at the beginning and then decreases. And there is a peak of the stability index when the dosage of GCL3S is 1.0%. It indicates that the stability of CWS increases first and then decreases as the dosage of additive increases. The stability of CWS with added GCL3S is better than that with FDN under the same condition, which is basically in accordance with the results from the traditional water bleeding ratio.

The stability index method considers not only the top clarifying zone (the bleeding zone) of CWS but also the variation of the coal particle size in the opacity zone. Therefore, the experiment data of CWS measured using the stability index method is more reasonable.

In conclusion, the effect of modified lignin series and naphthalene series dispersants on the width of the clarifying zone, the particle sedimentation rate, the particle size and the stability index of CWS were studied using the dispersion-stability analyser. The results indicate that the stability of CWS with GCL3S is better than that with FDN, which is related to the molecular weight and the molecular configuration in aqueous solution of the dispersant. The research[14] shows that GCL3S is a macromolecular compound with three-dimensional network structure and its molecular weight is higher than that of FDN. Because of the characteristics of long chain macromolecule, flexibility and several polar groups of GCL3S, a network structure can be formed by cross-linking in solution medium and the pores of the network structure are filled with water. Thus, CWS with added GCL3S does not bleed water easily. Furthermore, the network structure can also be formed by the molecular attraction and partial hydrogen bond, which has the feature of thixotropy. Therefore, the network structure can be destroyed under the external force and reformed after storing to enhance the whole stability of CWS. However, FDN is a linear polymer, therefore, it is hard to form a network structure in aqueous solution, therefore weakly affects the stability of CWS.

3 Conclusions

In this article, the influence of modified lignin series and naphthalene series dispersants on the stability of CWS has been investigated. The results indicate that the sedimentation behavior of coal particles of CWS belongs to differential sedimentation and there is a conglobation among the coal particles in CWS preparation. The height of clarifying zone and the mean sedimentation rate of CWS prepared with GCL3S is about 68% of that of FDN, and the performance of GCL3S in CWS preparation is better than that of FDN.

The Turbiscan Lab dispersion-stability analyzer can not only characterize the stability of CWS but also observe the changes in clarifying zone and opacity zone. According to the changes in transmitted and backscattering light, it can be used objectively to reflect changes of slurry system such as agglomeration and sedimentation, which is helpful to investigate the stability mechanism of CWS. Compared with the traditional methods, the method using the dispersion-stability analyzer presents more accurate testing data and provides wider applications.

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