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## Chemical Properties of Bingham Materials in Backfilling

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This paper analyzes the chemical homogeneity properties of backfilling material belonging to Bingham body by rheology experiment, initial start-up experiment and shear experiment, then reveals the reasons for good transport characteristics of bingham material which is composed of high concentration cemented filling slurry with gangue and fly ash. The results show that this bingham material is a homogeneous suspension slurry, which is in the middle stage between structural flow and solid-liquid two-phase flow with good fluidity. This bingham material is divided into a mortar system and a gangue system, and these two systems act on each other in the inner of it. It is considered that the fine particles in the filling material provide adhesive property to the slurry, which is the key reason for the bingham material to present structural flow characteristics. But the gangue particles weaken the structure of floc net and promote the structural flow pattern to change to solid-liquid two-phase transition flow, and stops at the middle stage, which presents good fluidity. So compared with other materials, this bingham material transport resistance is smaller, the transport capacity is larger, the transport distance is longer, which is more suitable for transport during filling mining.

#### 1. Introduction

Longwall mining is the most efficient underground means to recover coal resources and is used widely in United States, Australia, China, and Europe. However, this type of mining method has produced serious surface subsidence problems, which causes the surface structure deformation, groundwater discharge and plants recession due to the formation of the subsidence basin (Lee et al., 2003). Various filling technologies have been invented and implemented to protect the surface structures form damage (Latifi et al., 2015). It helps to decrease or slow down the surface subsidence, and to release the resources of "three-under" covered coal, and to promote ecological harmony in mines (Zhang et al., 2014). There are four main filling mining technologies: fully-mechanized solid filling, high-water material filling, paste filling, and high concentration cemented filling with gangue and fly ash (Feng et al., 2010).

The material of high concentration cemented filling slurry is made of gangue, fly ash, cement and water by mixing them in proportion. Compared with other technologies, the most remarkable feature of high concentration cemented filling is that the gangue particles present good suspending property in the filling slurry, and remain uniform in the slurry all the time. The phenomenon of solid and liquid stratification will not appear. The suspended gangues in the high concentration cemented slurry present two advantages: 1) the gangues suspend and do not sink, and reduce the friction with the transport pipeline in the slurry transport process, and the transport resistance also becomes small; 2) The suspended gangues improve slurry fluidity and reduce the risk of plugging. The transport distance is longer, and the filling system is more reliable when they are under the same pump pressure (Yang et al., 2013). Thus, the long distance transport in filling mining is possible, and the large-scale transport demand can be satisfied because of its good characteristics. In addition, due to the good workable measurements, the gob can be stowed uniform with the slurry, the deformation of overlying strata layers can be effective controlled, and can be controlled in a long term (Bai, 2014).

During the application of high concentration cemented filling mining with gangue and fly ash, it may be found that the suspension state of gangues are not ideal during transport. It is inconvenient for the application of high concentration cemented filling mining method (Kukulka et al., 2018). So author makes some researches on flow characteristics of high concentration cemented slurry with gangue and fly ash, and explores the

homogenization interact mechanism in high concentration slurry. It is hoped that these researches will be useful to guide the configuration of high concentration cemented material, and to improve high concentation cemented filling technology.

#### 2. Parameters and effect verification of high concentration cemented slurry

#### 2.1 High concentration cemented slurry ratio parameters

The composition of high concentration cemented filling materials are shown in Table 1. The size of gangue particles are below 10mm, and 5~10mm particle size gangues account for about 55% of the total gangues, and 2.5~5mm particle size gangues account for about 35% of the total gangues. Fly ash standard is No. II, and the cement standard is PO.425. Figure 1 is the particle size distribution of fly ash and cement, which is used in high concentration cemented filling slurry.

Admixture consists of suspending agent, early strength agent, water reducing agent etc. The role of early strength agent and water reducing agent is to improve the strength and initial setting time of filling slurry. Suspending agent is used to adjust suspension characteristics of coal gangue particles.

Table 1: Composition and component parameters of unit volume (1 m<sup>3</sup>) high concentration cemented filling slurry

Coal Gangu	ie Fly ash	Cement	Water	Admixture	Mass Fraction	n Slump	Expansion	Bleeding Rate
(kg)	(kg)	(kg)	(kg)	(kg)	(%)	(mm)	Radius (mm)	(%)
900	360	180	380-430	16.2	77.00-79.12	240-260	225	4.5

The actual amount of water should to be adjusted to meet the standard of the filling material's slump when slurry out of machine. Water in admixture is 12.96kg.

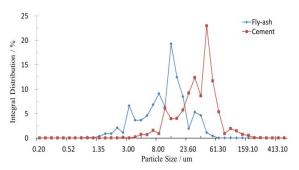




Figure 1: Particles size distribution diagram of fly ash and cement

Figure 2: Two sensors collecting local resistance loss data in bent pipe

#### 2.2 Verification of transport effect of high concentration slurry

Applied in Xinyang coal mine, high concentration slurry shows a very stable saturation, and always keeps a structural flow state without segregation and precipitation particles in filling process. The gangue particles will not sink when the slurry stays within half an hour. If the slurry is cut apart along the horizontal and vertical direction, it can be seen that the gangue particles are evenly distributed in the section, which called homogeneity characteristics. The suspension of gangue particles in slurry is considered the key reason for good transport performance of high concentration cemented slurry with gangue and fly ash. It could better adapt to the requirements of large filling flow and long distance in filling mining.

The length between two sensors of the straight pipeline which are used to collect resistance loss data is 10m. Other two sensors which are used to collect local resistance loss data are arranged in a bent pipe as shown in Figure 2. At this time, the average loss values of on-way resistance and local resistance of pipeline are 2.80KPa/m and 3.11KPa/m respectively, and the maximum values are 3.4KPa/m and 3.78 KPa/m respectively. Because of smaller transport resistance, the slurry can greatly improve the filling flow status.

# 3. Experimental researches on the chemical homogeneity characteristics of high concentration slurry

In order to research the chemical homogeneity characteristics of high concentration slurry with gangue and fly

ash, and to improve the research of the mixed proportion of filling slurry, the following three experiments are carried out.

#### 3.1 Analysis on rheology experiment of high concentration slurry

Slurry with a mass fraction of 78.5% is used to make a rheological experiment according to table 1, which is to determine the rheological properties of the slurry with high concentration. This experiment adopts the Rheolab QC rotational rheometer which is produced by Anton Paar company in Austria, and analyzes with RheoWin software as shown in figure 3.

The rotor is placed in a 500 ml beaker for rheological test, and is stirred for a period of time at the rate of 100n/s, then the shear rate of the rotor is increased linearly from 0 to 300n/s as shown in figure 4. A series of values of shear rate and shear force are obtained, then the curves shown as figure 5 is obtained.



Figure 3: Rheological shear apparatus applied in experiment

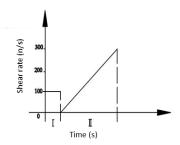


Figure 4: Rheological analysis of high concentration cemented filling

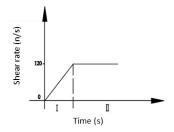


Figure 5: Test program

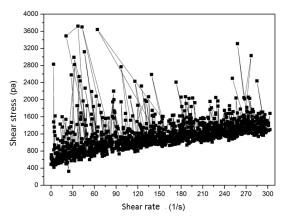
The relation curves between the shear rate and the shear stress is approximately straight line, which accord with the flow pattern characteristics of the Bingham body. The data show that the yield stress of the slurry is 300pa. Figure 5 shows that the flocculent structure exists in slurry, and such structure has certain shear resistance. When the shear stress exceeds the yield stress, suspension begins to flow, otherwise only presents elastic deformation. There is a close relation between the magnitude of yield stress and the content, size of the fine particles.

#### 3.2 Analysis on initial start-up experiment of high concentration slurry

In order to research the characteristics of high concentration slurry further, the following three kinds of slurry as shown in table 2 are made. The fine particles in No. 1 slurry are most (in which the content of -20um particles rate are more than 20%), and this slurry is a typical paste slurry (Xu et al., 2016). The particles of gangue and fly ash distribute evenly in the body of slurry. No. 3 slurry belongs to solid-liquid two-phase fluid, and the solid and liquid stratification is obvious, and the gangues accumulate in the lower part of the slurry. The No. 2 slurry is in the middle stage between No. 1 and No. 3 slurry, and is applied in Xinyang coal Mine. According to test program as shown in figure 6, No. 1,2,3 slurry are tested by rheometer. When the stirring rate is 0-16 / s, selecting the data in stage I of figure 6, the relation between shear rate and shear stress is drawn as figure 7.

There are obvious peaks in No.1 and No.2 slurry, which are due to the existence of flocs inside the slurry. When the rheometer rotor starts, the slurry does not move, Until the shear stress imposed by the rotor of the rheometer is large enough, and it can destroy the block force in slurry to the rotor. The shear stress reaches the peak.

When the slurry is moved, there is a avoidance effect between the slurry and the beaker wall, and an extremely thin aquifer is formed between the wall and the slurry (Dong et al., 2018). The appearance of aquifer greatly reduces the force needed to be overcame by slurry rotation, so the rotor presents a decrease in shear stress and a steady trend. The better the structure of the slurry, the greater the interaction between the particles, the more stable the structure of the floc net, and the greater the blocking force of the slurry. There are many fine grained aggregates in No. 1 slurry, and the interaction force between them is much greater than that in No. 2 slurry, so the peak of No. 1 slurry is much higher than that of No. 2 slurry. The yield stress of No.1 slurry is also higher than that of No.2 slurry. The No. 3 slurry is a solid-liquid two-phase flow with low concentration, obvious granular stratification, and no internal structure among gangues, fly ash and cement. And there is no peak in the whole curve. From the numerical point, No. 2 slurry is sandwiched between No. 1 and No. 3 slurry, and is in the middle and transition stage which is easy to start up before transport. So No. 2 slurry is more suitable for filling mining.



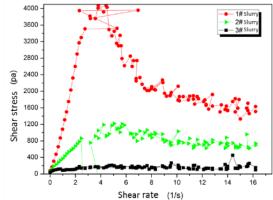


Figure 6: Curves of shear rate and shear force stress of every slurry

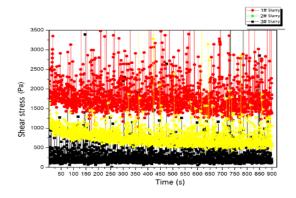
Figure 7: Starting state between shear rate and shear

Table 2: Proportional parameters of unit volume (1 m<sup>3</sup>) slurry

No	. Coal Gangue (kg)	Flyash (kg)	Cement (kg)	Water (kg)	Mass Fraction (%)
1	900	360	180	295	83.0
2	900	360	180	395	78.5
3	900	360	180	533	73.0

The percentage of coal gangue whose size is -2.5mm is 100% in No.1 slurry. The proportion of coal gangue's size of No.2 and No.3 slurry refers to Table 1.

#### 3.3 Analysis on shear experiment of high concentration slurry



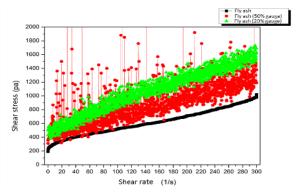


Figure 8: Stress relaxed curves of every slurry stress

Figure 9: Relationship between stirring rate and shear

Selecting data of stage II in figure 6, draw stress relaxed curves of every slurry as shown in figure 8. The large fluctuation range of shear stress value of every slurry is caused by the uneven particle size of gangues, and rheometer rotors shear different sizes of gangues irregularly, but the range of fluctuations is basically within one range. At a constant shear rate of 120 n/s, the shear stress of No. 3 slurry is the most stable. With the passage of time, the shear stress of No. 1 slurry decreases slightly, while that of No. 2 slurry shows an obvious downward trend and stabilizes in later period.

No. 3 slurry is a solid-liquid two-phase flow, and most of the gangue particles have been deposited at the bottom. As the shear continues, the solid-liquid two-phase fluid remains in the initial state without too much change. No.1 and No.2 slurry are structural flow pattern with a floc net structure inside, so the gangue particles are suspended in the slurry. We can see from figure 8 that the yield stress of No.2 slurry is between No.1 and No.3, and it is in the transition state from structural flow to solid-liquid two-phase flow (Bentz et al., 2012). The interact force of No.2 slurry particles is smaller than that of No.1 slurry, so the settling degree of particles is larger than that of No.1 slurry. There is a tendency of large aggregate settlement and stratification when high concentration slurry with gangue and fly ash is static. However, the slurry is in a stirring state at all times under the impact of piston pump in the actual pipeline transport process. The aggregate collides with each other and moves forward, so there is no static condition. When pipeline fails or slurry transport stops, No.2 slurry will be rebooted soon by increasing pump pressure slightly within a certain time. So No. 2 slurry is more suitable for long distance and large scale transport in filling mining.

#### 4. Research on homogenization interact mechanism in high concentration cemented slurry

Baseing on the homogeneity characteristics researches above, the slurry system is a complex system. The size distribution of solid particles ranges from 10 mm to 10 um, together with physical and chemical reactions. In order to research the homogenization interact mechanism of high concentration slurry more clearly, it is assumed that the slurry is mixed after the coal gangue particles are added into mortar system. The mortar system is composed of fly ash, cement and liquid (water admixture), and this system is characterized by small particles and chemical reaction. The coal gangue particles sizes are large, therefore separate the coal gangues, and form gangue system alone (Dudynski, 2018).

#### 4.1 Analysis of mortar system

The mortar system contains a large amount of fine particles, and it can be seen from Figure1 that the grain size of -20um is about 40% of fly ash and cement. These fine particles attract opposite sex particles, and form the charged particles in an electric double layer. Attract each other by the effect of surface electric field, and then gradually overlap to form flocs. With increase of slurry concentration, the particle flocculation develops. The connection among the flocs begin to link and form a loose reticular structure which called flocculation structure, when flocs reaches a certain number (Minne et al., 2018). The floc net has a certain shear capacity and forms initial yield stress. The structure of floc net leads to interaction force among particles, which makes the slurry present structural flow characteristics.

The hydration of cement, fly ash and water in the mortar system will result in hydration reaction to form fibrous or granular calcium silicate hydrate (mCaO·nSiO<sub>2</sub>·xH<sub>2</sub>O, generally abbreviated as C-S-H). C-S-H has strong cementation ability. The existence of C-S-H not only strengthens the fine particle structure of floc net, but also provides a strong guarantee for bonding large gangue particles.

#### 4.2 Analysis of coal gangue system

Coal gangue particle size is large. Flocculent structure will wrap gangue particles, and gangue particles will fill flocculent structure fully. But there is shear effect between gangues and flocculent fine particles, which acts as a hindrance to the relative motion of gangue particles (Zhao et al., 2017).

The addition of gangue particles to the mortar system, breaks the arrangement structure of fine particles in the original mortar system and changes the distance among the particles. According to the proportion in tatble1, the mortar system is configured, and rheological tests are carried out. Then the gangue particles are added, and the rheological tests are carried out again after mixing. The experimental results show the relationship between stirring rate and shear stress as shown in figure 9. From figure 9, mortar system composed of fly ash, cement etc. is a pseudo plastic body. Pseudo plastic presents physical and chemical changes after added gangue particles, and shows Bingham characteristics. Yield stress and plastic viscosity both change too. Gangue particles do not participate in the composition of the flocculation structure, but the flocculation structure encapsulates the gangue particles. Gangue particles cut off the flocculation structure among fine particles, and macroscopically show the weakening of high concentration slurry structure flow and the enhancement of solid-liquid two-phase flow characteristics.

In this paper, author thinks that the homogeneity of high concentration cemented filling material is determined both by the mortar system and gangue system, and they complement each other and also impose on each other. The mortar system provides adhesive force for high concentration slurry, which makes the slurry present structural flow characteristics. The gangue particles weaken the flocculation structure, thus promote the transition state from high concentration slurry to solid-liquid two-phase flow state, which is more suitable for slurry transport during filling mining.

#### 5. Conclusions

- 1) The high concentration cemented filling slurry with gangue and fly ash and is a homogeneous suspension and belongs to Bingham body. It is in the transition and middle stage of structural flow to solid-liquid two-phase flow with good fluidity.
- 2) The mortar system composed of fine particles and admixtures in high concentration cemented filling slurry is the key reason for the performance of structural flow characteristics, and provides adhesion among particles.
- 3) Gangue particles change the arrangement of solid particles in slurry, and cause physicochemical properties changes. Gangue particles weaken the slurry's flow pattern and strengthen characteristics of solid-liquid two-phase flow gradually.

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