International Journal of Minerals, Metallurgy and Materials
Volume 27, Number 1, January 2020, Page 10
https://doi.org/10.1007/s12613-019-1865-y

Effect of particle gradation characteristics on yield stress of cemented paste backfill

Hai-yong Cheng[1,2], *Shun-chuan Wu*[1,2], *Xiao-qiang Zhang*[1], *and Ai-xiang Wu*[2]

1) Faculty of Land Resources Engineering, Kunming University of Science and Technology, Kunming 650093, China
2) Key Laboratory of Ministry of Education of China for Efficient Mining and Safety of Metal Mines, Beijing 100083, China
(Received: 4 April 2019; revised: 12 June 2019; accepted: 18 June 2019)

Abstract: Along with slurry concentration and particle density, particle size distribution (PSD) of tailings also exerts a significant influence on the yield stress of cemented paste, a non-Newtonian fluid. In this work, a paste stability coefficient (PSC) was proposed to characterize paste gradation and better reveal its connection to yield stress. This coefficient was proved beneficial to the construction of a unified rheological model, applicable to different materials in different mines, so as to promote the application of rheology in the pipeline transportation of paste. From the results, yield stress showed an exponential growth with increasing PSC, which reflected the proportion of solid particle concentration to the packing density of granular media in a unit volume of slurry, and could represent the properties of both slurry and granular media. It was found that slurry of low PSC contained extensive pores, generally around 20 μm, encouraging free flow of water, constituting a relatively low yield stress. In contrast, slurry of high PSC had a compact and quite stable honeycomb structure, with pore sizes generally < 5 μm, causing the paste to overcome a higher yield stress to flow.

Keywords: paste backfill; grading theory; yield stress; paste stability coefficient; microscale

1. Introduction

In recent years, safety accidents have frequently happened around tailings ponds [1]. The technology of cemented paste backfill (CPB) provides important assistance in the development of green mines [2–3]. The efficient use of tailings could not only reduce deposits of tailings, which clearly increase the risk of dam breaks [4–5], but also fill in the underground goaf and reduce the hidden danger [6]. CPB is a preferred method for the elimination of environmental pollution and safety hazards, and waste of resources caused by mining [7].

Yield stress is an important index for evaluating the flowability and stability of paste [8–10], specifically in cementitious materials [11], and is of particular significance in quantifying flowability [12–13]. Yield stress, as a good indicator of the magnitude of particle-to-particle forces [14], is defined as the minimum shear stress required to initiate flow [15], for which concentration is an important influencing factor [16–17]. Density differences and gravity can easily result in slurry segregation when at a low concentrations [18–19], although yield stress tends to be stable under comparatively high paste concentrations [20]. However, the paste is a compound made of mixed materials, generally unclassified tailings, cement, and water [21], and has varying size distributions when prepared from different tailings. The fine contents, grain size distributions, and grain shape characteristics are among the major factors affecting the shear strength of tailings [22–23]. Gradation difference is also an important factor in yield stress [24–25]. Fall *et al*. [26] believes that the proportion of fine tailings particles (< 20 μm) significantly affects the porosity of paste backfill. Silva *et al*. [27] showed that yield stress changes with particle size, while Ferraris *et al*. [28] pointed out that with decreasing particle size, yield stress declines after an increase, and reaches a maximum when the mean size of the particles equals 5.7 μm. Guo *et al*. [29] considered that coarser particles need a larger distance to achieve the same yield stress of paste. After analysis, Qi-

Corresponding author: Xiao-qiang Zhang E-mail: zhangxiaoqiang@kmust.edu.cn

an and Kawashima suggested that yield stress of cemented paste originates from the microstructure of particle-particle networks through colloidal interaction or direct contact. The microstructure absorbs the stress before it is broken down and starts to flow, which defines yield stress. Merrill *et al.* [30] analyzed slurry rheology using hyperspectral characterization, and obtained the mixing ratio of some minerals when the viscosity was minimum. Kashani *et al.* [31] analyzed the particle size distribution (PSD) effect on yield stress, and found the relationship between porosity and PSD. Grading refers to the distribution of the size of fine, medium, and coarse particles in a material. Fine particles fill the pores between coarse particles in well-graded materials; this can reduce the porosity of the material. The parabolic maximum density ideal curve was proposed by Fuller, and particles interference theory by Wey-mouth, to solve the maximum packing density of solid particles. However, the yield stress of paste relates to not only the inherent compactness of the solid, but also its water content. Pullum *et al.* [32] believed that yield stress is a function of solids concentration for a number of industrially relevant slurries. It is important to note that yield stress profile is material-specific, being a combined function of mineralogy, processing conditions, PSD, surface chemistry, and shear history. A large number of experiments have proved that the higher the moisture content, the higher the concentration, the greater the yield stress for the same solid material. Also, analysis of the influence of solid or fluid properties on yield stress is often biased. Paste composition materials are diverse, and gradation characteristics differ [33–35]. The volume concentrations of better pastes are different. Yield stress is influenced by the properties of solid and liquid.

The feasibility of estimating the rheological behavior of paste, from the perspectives of gradation character of slurry and granular media, based on the paste stability coefficient (PSC), was studied in this paper. The characteristics of paste pore structure under different gradations were analyzed by environmental scanning electron microscopy (ESEM).

2. Experimental

2.1. Materials

The inert material used was the unclassified tailings of coarse grain size from a nickel mine, of which the density and bulk density were 2.852 t/m^3 and 1.545 t/m^3, respectively. The Jinchuan Nickel Deposit is located in Jinchang City, Gansu province, western China. In 1958, the amount of nickel metal reached 5.57 million tons, accounting for 79% of China's proven reserves，which was the largest copper nickel sulfide deposit in China, and third largest in the world. The deposit is famous for its thick ore body, deep burial, rock fragmentation, and high in-situ stress. It is a large, complex, and difficult underground deposit that is rarely seen at home and abroad.

P.O 42.5 cement was selected as the cementitious material, with a density and bulk density of 3.03 t/m^3 and 1.424 t/m^3, respectively. Grain size compositions of the tailings and cement are shown in Fig. 1. The content of −75 μm particles in the tailings was 60.12%, and the −20 μm particle content was 21.67%. The proportion of −75 μm particles in the cement was 99.9%, and that of −20 μm particles was 70.13%. Tailings were well-graded when the coefficient of uniformity C_u was 5−10, and tailings exhibited good gradation and a high compaction rate when the curvature coefficient C_c was 1−3 [36]. These tailings were well-graded given that their C_u and C_c values were 8.87 and 1.58, respectively. Thus, these tailings benefited the paste preparation process.

Chemical compositions of the materials were analyzed by X-ray diffraction and chemical element calibration, and the results are listed in Table 1. It can be seen that the tailings had a low content of sulfur, only 1.63%, and its overall performance was inert.

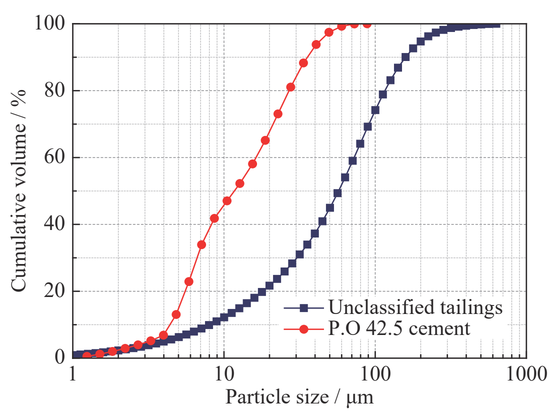

Fig. 1. PSD of the unclassified tailings and P.O 42.5 cement.

Table 1. Chemical compositions of the unclassified tailings and Portland cement at%

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	S	Ni	Cu	Others
Unclassifield tailings	36.41	7.77	9.9	3.09	27.79	1.63	0.28	0.2	12.93
Portland cement	21.5	4.5	2.0	63.5	4.0	2.5	—	—	2.00

2.2. Sample preparation

In order to analyze the works of the grain gradation on yield stress independently, the tailings were prepared under varying 0 s, 2 s, 5 s, 20 s, and 40 s grindings, and were named tailing-1 to tailing-5. Grain size distribution of the tailings was tested using a Malvern Laser 2000, which can measure particle sizes of 0.02 to 2000 μm, with an accuracy of ±1%. PSD curves were obtained as shown in Fig. 2 and Table 2. In many studies, PSD was characterized by the content of −20 μm particles or the average particle size (d_{av}).

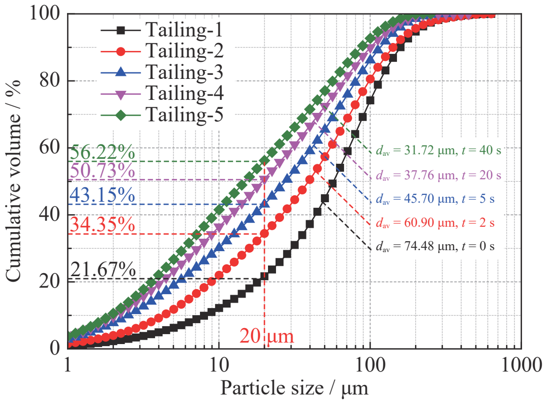

Fig. 2. PSD of the tailings for different grinding times.

2.3. Experimental design

2.3.1. Rheological tests on different grinded tailings

The five types of tailings were mixed with cement at a 12:1 ratio (mass ratio) and ultimately into a volume of 480 mL paste of 69wt%. The slump of this ratio was about 25 cm in previous experiments, which was of good fluidity and stability, and allowed for the study of the effect of gradation on yield stress without the influence generated by material difference factors (solid density, concentration). Mixing time was set at 2 min. The tests were carried out with a R/S plus rheometer using the control shear rate (CSR) method. A constant shear rate of 15 s^{-1} was adopted during the 20 s pre-shear, and then stopped for 10 s before rising from 0 to 180 s^{-1}, and for a shearing duration of 180 s, as shown in Fig. 3. The most commonly used rheological model, the Bingham model, one of several models (i.e., Bingham, modified Bingham, Hershel-Bulkley and Casson models) usually applied to cementitious materials correlating shear stress with shear rate, was adopted for the paste [37]:

$$\tau=\tau_0+\eta\dot{\gamma} \quad (1)$$

where τ is the shear stress, $\dot{\gamma}$ is the shear rate, τ_0 is the Bingham yield stress, and η is the plastic viscosity [38–39].

2.3.2. Gradation and rheological characteristics across different mix proportions

First, we weighed the 5 kg sample and dried it in the oven until it was cooled to room temperature. After cooling, we used a 2 mm aperture sieve to prepare qualified test materials. Second, the emptied bulk density cylinder was put on the electronic scale, filled with the sample through the standard funnel, rammed, and then scraped flat with a ruler. Third, we weighed the mass of sample W_1 in a loose state, and the mass of sample W_2 in a compacted state, in the volume of bulk cylinder V. Thus, the bulk density was obtained by $\gamma = (W_2–W_1)/V$, and tailings compactness was $\varphi = \gamma/\rho$ (ρ is the tailings density). Finally, in order to reduce the random error, the experiment was repeated three times.

The as-received sample (tailing-1), without grinding, was used in the tests under various mix proportions to analyze the rheological properties, and the content was set at levels of 66wt%, 67wt%, 68wt%, 69wt%, 70wt% and 71wt%, and the tailings-cement mass ratios (t/c) at 2:1, 4:1, 6:1, 12:1 and 20:1, respectively.

2.3.3. Microcosmic experiment

ESEM allowed both observation of the micropore and microskeleton structures of the liquid slurry without gold-spraying on the surface, and observation of the microstructure of the paste. In this experiment, the as-received samples (tailing-1 and tailing-5) were used with a tailings-cement ra-

Table 2. PSD of the tailings under different grinding times

Tailing-1		Tailing-2		Tailing-3		Tailing-4		Tailing-5	
Particle size / μm	Cumulative volume / %	Particle size / μm	Cumulative volume / %	Particle size / μm	Cumulative volume / %	Particle size / μm	Cumulative volume / %	Particle size / μm	Cumulative volume / %
7.962	9.89	4.477	10.45	2.518	10.08	2	9.57	2	10.58
17.825	19.79	8.934	20.11	5.637	20.69	3.99	19.35	3.557	19.85
31.698	31.01	15.887	30.05	10.024	30.15	7.096	29.7	5.637	29.14
44.774	40.93	25.178	39.22	17.825	40.85	12.619	41.13	8.934	39.05
56.368	49.35	39.905	50.54	28.251	50.57	20	50.73	15.887	51.35
70.963	59.02	56.368	61.08	39.905	59.08	31.698	60.98	25.178	61.14
89.337	69.22	70.963	68.89	56.368	68.91	44.774	69.47	39.905	71.51
112.468	78.86	100.237	80.55	79.621	79.5	70.963	81.63	56.368	79.87
158.866	90.05	141.589	89.98	112.468	89.17	100.237	89.98	89.337	90.48

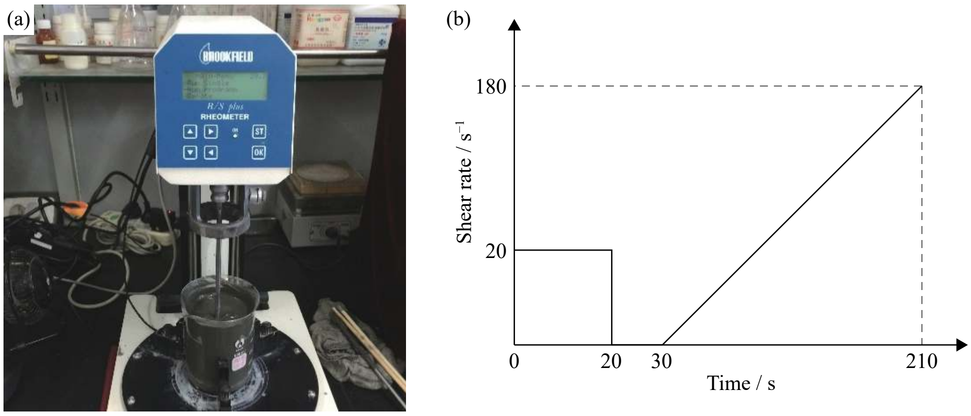

Fig. 3. Rheometer and testing program: (a) rheology testing equipment; (b) program setting.

tio of 12:1 and a content of 70wt% (this facilitated the preparation of microscopic detection samples). ESEM was selected to directly observe the microstructure of the material, as shown in Fig. 4. Superior to traditionally used SEM [40], the ESEM did not require the samples to be covered by a conductive film, thus avoiding the change in hydration and structure of the matrix during the long period of sample processing [41]. Also, magnification of the ESEM could reach 10000×, allowing the microstructure of the paste to be presented more clearly.

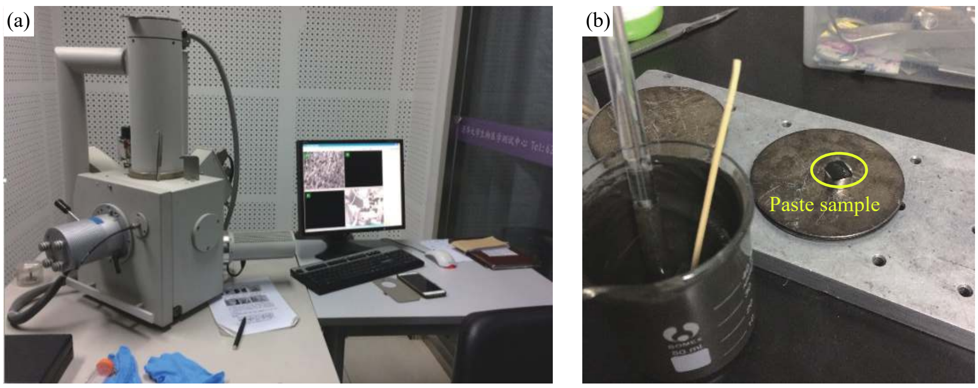

Fig. 4. Quanta 200 ESEM and sample preparation: (a) Quanta 200 ESEM; (b) sample preparation.

3. Results and discussion

3.1. Effect of tailings fineness on the yield stress of fresh CPB

The influence of chemical action should be considered firstly when analyzing the effect of gradation on yield stress due to the existence of binder in the paste slurry [42–43]. The effect of hydration on yield stress is very important under static conditions [44–45]. It is an important factor for strength formation. In filling mining, strengths of 3, 7 and 28 days are generally required [46–47]. In this paper, we emphasize that in the state of continuous stirring, the effect of binder hydration on yield stress within the initial 1 h was almost negligible. Dynamic stirring affected formation of the chemical structure, and yield stress showed little change. This point has been verified by the author in previous experiments.

From the experimental results, it was found that the average particle size tended to be smaller under longer grinding time, and with increasing content of fine particles, yield stress tended to increase, as shown in Fig. 5(a). The increasing trends in yield stress paralleled the increases in the proportions of −20 μm particles, −37 μm particles, and −74 μm particles.

In the experiments under different mixing proportions, the content of fine particles tended to be larger, and the yield stress smaller, when at a lower tailings-cement ratio, in the range of 2:1 to 20:1, as shown in Fig. 5(b), the characteristics of the curves were in contradiction of those in Fig. 5(a). This indicated that the content of fine particles in the paste slurry could not be used as an indicator of the change in yield stress. It is speculated that the change in grain size had caused the alteration of a certain parameter, resulting in the development of the yield stress.

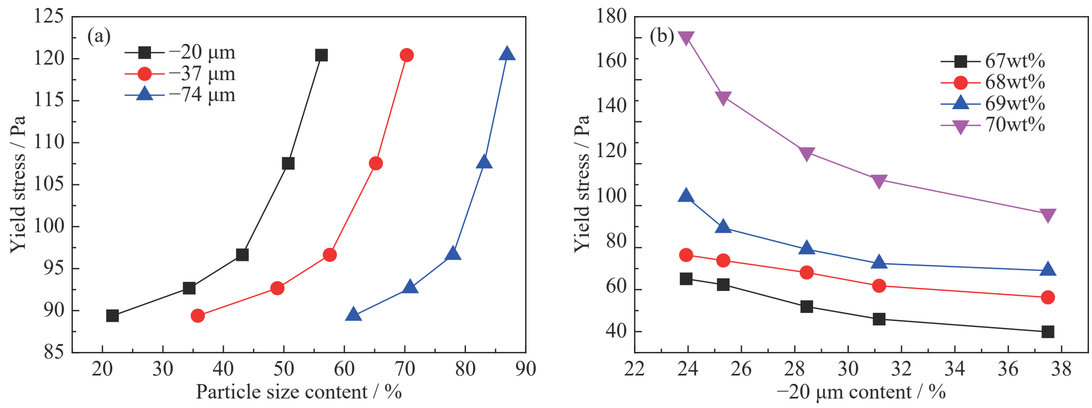

Fig. 5. Curves of the yield stress versus fine particles content: (a) tailings-cement ratio constant; (b) concentration constant.

3.2. Granular media characteristics

Under ideal conditions, packing density is the same between coarse and uniform fine particles [26,48]; this relates only to the gradation state instead of the size of particles [49]. Mixtures were prepared with tailings-cement ratios of 0:1–20:1 to ascertain the proportions of packing density, as seen in Fig. 6. The cement, with a packing density of 0.47, and the unclassified tailings, with a packing density of 0.54, when mixed in different proportions, reached a peak value in packing density. With the polarization extension of coarse particles or fine particles, the packing density will decrease. Before the peak, the density showed a structure dominated by fine particles, while, after that, coarse particles played a leading role. Fig. 7 indicates that the packing density could characterize the gradation of the mixed granular media.

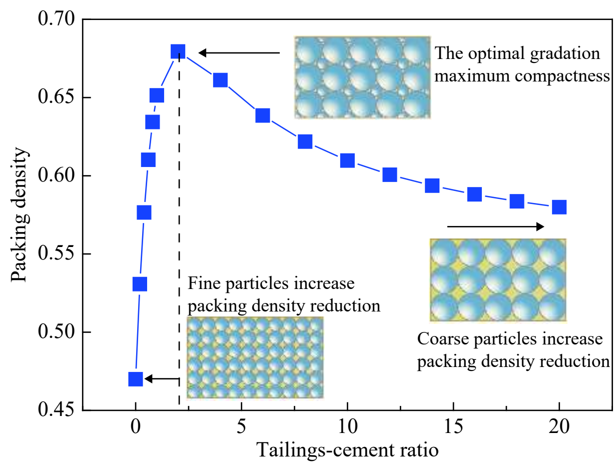

Fig. 6. Packing density at different tailings-cement ratios.

3.3. A new characterization method for paste gradation

The balance between the fluidity and stability of paste is a widely considered issue in paste transportation [50]. They are the combined results of granular media and slurry properties. The differences in granular media could be characterized by the packing density and the characteristics of the paste by volume concentration. In an ideal state, the granular media of a paste is compacted into a container of volume V, with water gradually added until the volume of the water is equal to that of the pores. Currently, packing density (φ) is equal to the volume concentration of slurry C_V:

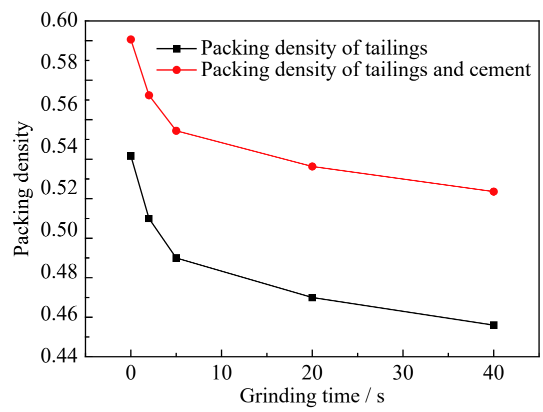

Fig. 7. Variation in packing density with grinding time.

$$\varphi = \frac{m_s/V}{m_s/V_s} = \frac{V_s}{V} = C_V \tag{2}$$

where m_s is the solid mass (kg); V_s is the solid volume (m^3); V is the volume of the container (m^3).

Packing density describes the air medium in a solid, and the volume concentration represents the solid-liquid two-phase flow in water. The two concepts are the same descriptive forms for different media. When $C_V > \varphi$, the slurry is dry and hard, and has no flow capacity; when $C_V = \varphi$, the slurry is in a critical saturation state; when $C_V < \varphi$, the slurry is in a supersaturated state and has a certain fluidity.

According to the composite characteristics of granular media and fluid, the concept of PSC is proposed with the function shown below:

$$k_s = \frac{C_V}{\varphi} \tag{3}$$

where k_s is the PSC, C_V is the volume concentration, and φ is the packing density.

PSC represents the ratio of packing density of solid particles to the maximum packing density in a unit volume of slurry, reflecting the quality of material gradation and the extent to which the current volume concentration reaches this attribute.

As can be seen from Fig. 8, the PSC increased gradually with increasing tailings grinding time, and the yield stress grew exponentially with PSC. Fig. 9 shows the results of the experiments subsequently conducted under various volume concentrations and tailings-cement ratios, which revealed that the PSC reached a minimum of 0.589, and the yield stress reached a minimum value of 32.929 Pa. When the PSC reached a maximum of 0.795, the corresponding yield stress reached a maximum value of 225.353 Pa.

PSC has good applicability to all tailings materials of metal mines, such as copper ore, iron ore, lead, and zinc, among other common tailings. However, its applicability to coal or non-metallic mines has not been verified by experiments.

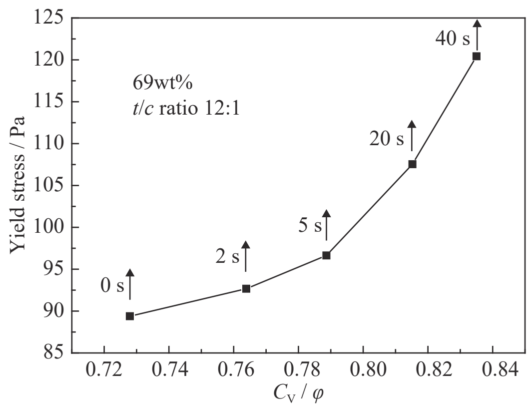

Fig. 8. Variation curves of yield stress with C_V/φ with changes in single tailings gradation.

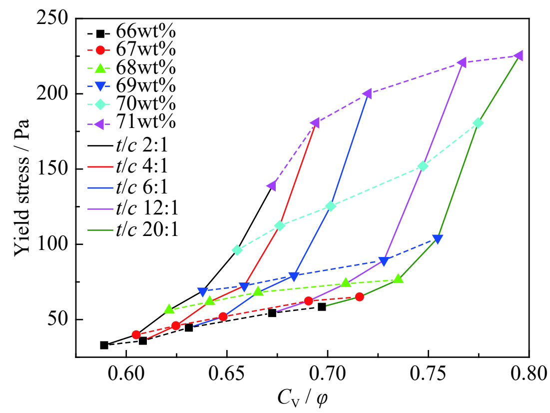

Fig. 9. Variation curve of yield stress with C_V/φ when the mix ratio changed.

3.4. Microstructure of the paste

The microstructure of paste provides an insight into the connectedness of inner water movement channels as well as the stability of the skeleton structure. Fig. 10 shows the microstructure of the paste specimen made of the same tailings of different average diameters. The slurry with a low PSC (average particle size of 74.5 μm) tended to have coarse pores, with diameters of around 20 μm. In contrast, the slurry with a high PSC (average particle size of 31.7 μm) possessed a fine and dense pore structure, with diameters of generally < 5 μm. The distribution of the large number of micropores among the solid particles was like a honeycomb with a uniform and stable structure. These closed pores tended to have a strong water-holding capacity and restricted the free flow of water. It could be concluded that, for a certain material, the slurry flowability declines, and the yield stress increases, with a larger content of fine particles and a more uniform composition.

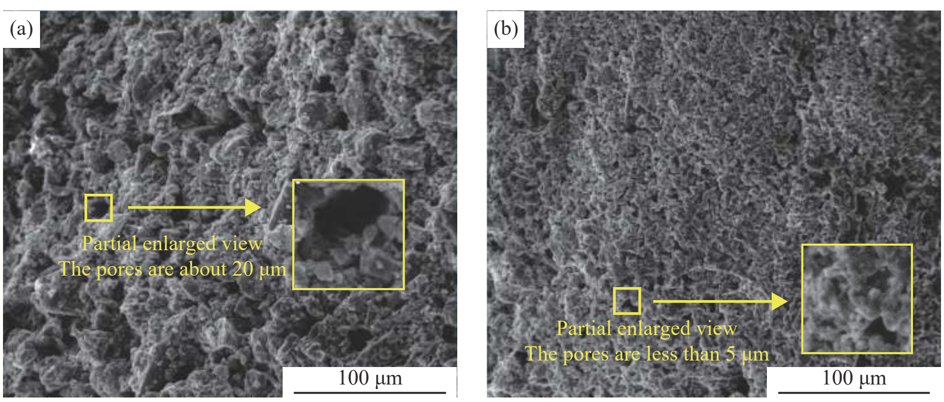

Fig. 10. Micropore structure of paste with different stability coefficients: (a) average particle size of 74.5 μm; (b) average particle size of 31.7 μm.

4. Conclusions

The characterization of paste gradation was put forward through experimental analysis and observation of the microstructure, and it is considered that gradation plays a vital role in the development of yield stress. The main conclusions are as follows:

(1) The gradation of paste affects yield stress from two perspectives: skeleton structure and pore structure. The skeleton determines the strength of internal structure to some extent, and is the foundation of the paste stability.

(2) PSC is proposed for the characterization of particle gradation, which enables the combination of granular medium and fluid medium. Yield stress shows a power exponential growth with increasing paste stability coefficient, both in the tests of grinded tailings and the experiments with paste of different mixing proportions.

(3) The evolution mechanisms of paste structure and yield stress were analyzed from the microscopic scale. It is considered that the yield stress is mainly controlled by gradation. Pastes of high PSC are dense and have a stable honeycomb structure, and the paste needs to overcome a larger yield stress before flowing.

Despite the results obtained, further studies are necessary toward a better understanding of the effect of PSC on the yield stress of paste with the following topics recommended for further work: (i) The adaptability of PSC to waste-tailings paste; (ii) quantitative relationship between PSC and microscopic pore size; (iii) numerical models of influence of PSC on yield stress of paste backfill.

Acknowledgements

This work was financially supported by China Postdoctoral Science Foundation (No. 2019M663576), the National Natural Science Foundation of China (No. 51774020), the Key Laboratory of Ministry of Education of China for Efficient Mining and Safety of Metal Mines (No. ustbmslab201801), the Program for Innovative Research Team (in Science and Technology) in University of Yunnan Province and the Research Start-up Fund for Introduced Talent of Kunming University of Science and Technology (No. KKSY201821024).

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