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Agglomeration and leaching behaviors of copper oxides with different chemical binders

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Abstract: The chemical binder is one of the critical factors affecting ore agglomeration behavior and leaching efficiency. In this study, we investigated the effect of the type of binder and mass fraction of the H_2SO_4 solution used on the curing, soaking, and leaching behavior of agglomerations. The results revealed that Portland cement ($3CaO·SiO_2$, $2CaO·SiO_2$, and $3CaO·Al_2O_3$) was the optimal binder for obtaining a well-shaped, stable agglomeration structure. A higher extraction rate was achieved when using Portland cement than that obtained using sodium silicate, gypsum, or acid-proof cement. An excessive geometric mean size is not conducive to obtaining well-shaped agglomerations and desirable porosity. Using computed tomography (CT) and MATLAB, the porosity of two-dimensional CT images in sample concentrations L1–L3 was observed to increase at least 4.5vol% after acid leaching. Ore agglomerations began to be heavily destroyed and even to disintegrate when the sulfuric acid solution concentration was higher than 30 g/L, which was caused by the excessive accumulation of reaction products and residuals.

Keywords: agglomeration; binder; acid leaching; copper oxide; sulfuric acid solution

1. Introduction

Compared with traditional pyrometallurgy practices, heap leaching is a more environmentally friendly, lower-cost, and effective method in the hydrometallurgical operations involving copper, gold, and uranium [1–3] in the United States, Australia, South Africa, China, India, and Chile [4–5].

However, an undesirable particle-size distribution (PSD) of the ore and a highly concentrated clay content can lead to segregation/stratification [6–8] and preferential flow [9] in non-agglomerated heaps [10–11]. This is mainly because adjacent wet and fine particles rapidly adsorb onto each other to form liquid bridges, which eventually lead to compaction of the heap by microscopically driven forces, e.g., van der Waals forces [12–13]. These undesirable absorptions and compactions result in poor pore structures, especially in non-agglomerated heaps. Thus, the leaching solution flows and diffuses non-uniformly in segregated heaps, resulting in liquid holdup such that valuable minerals cannot be efficiently extracted [14–15]. To better control the fluid flow behavior and copper recovery, ore agglomeration was proposed and gradually became utilized in the leaching of laterite-nickel and copper oxide minerals [16–19]. In this process, the quality of ore agglomeration is controlled by key operational parameters, including the feed PSD, ore mineralogy, chemical binders, curing time, and drum setup [20]. According to Lewandowski and Kawatra, the agglomeration compressive strength, Cu extraction, and hydraulic conductivity of the heap obviously increase if suitable chemical binders are added during the ore agglomeration preparation [21–22]. In general, the chemical binders added during the agglomeration process can be divided into inorganic and organic chemical reagents, e.g., sodium silicate, gypsum, polyacrylamide, stucco, and ionic liquid [23–24]. Of these, the curing process of agglomeration assisted by sulfuric acid binders is commonly used in industrial operations. Polyacrylamide can increase the Cu extraction rate but is toxic and threatens the health of environments around mining operation areas [25]. Current studies indicate that the agglomeration efficiency and optimum additive concentrations of binders obviously differ among actual heap operations. The effect of different binders on the agglomeration molding and leaching efficiency re-

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main uncertain pending further study. With the development of high-resolution X-ray computed tomography (X-ray CT) scanning, the effect of the agglomeration condition on the pore structure becomes observable at the microscale [26–28], which enables investigation of the effect of the binder parameters on the porosity evolution.

In this paper, to gain a deeper understanding of the potential effect of different binders on the shape stability, leaching behavior, and porosity evolution of agglomerations, we conducted comparative soaking experiments of agglomerations bonded by chemical binders. Five chemical binders were carefully considered, including sodium silicate (Na_2SiO_3), gypsum ($CaSO_4{\cdot}2H_2O$), Portland cement, acid-proof cement, and sulfuric acid solution (H_2SO_4). We determined the optimal binder and its mass fraction in the sulfuric acid solution via CT and MATLAB.

2. Experimental

2.1. Ore samples

The ore samples for this study were collected from the Yangla Copper Mine from a shallow stratum of earth with a high clay content caused by intense weathering. In the sample, the mass fraction of copper was 1.473% (44.98% free copper oxides, 18.85% combined copper oxides) and the other metal mineral was iron (11.28wt%). The gangues consisted of SiO_2 (33.75wt%) and CaO (10.68wt%). As shown in Table 1, the major copper minerals were malachite and limonite.

2.2. Binders

We considered five types of chemical binders, including sodium silicate (Na_2SiO_3, purity 99%), gypsum ($CaSO_4{\cdot}2H_2O$, purity 98%), Portland cement ($3CaO{\cdot}SiO_2$, $2CaO{\cdot}SiO_2$, $3CaO{\cdot}Al_2O_3$, purity 99.5%), acid-proof cement ($Na_2O{\cdot}n SiO_2$, Na_2SiF, purity 95%), and sulfuric acid (H_2SO_4, 3 mol/L), which were all provided by the Alibaba Group. The oxide ores (−0.075+0 mm) were uniformly mixed with the binders. To promote bonding reactions and agglomeration strength, the agglomerates were placed in a stable environment (moisturizing 35% relative humidity, natural aeration, 27 ± 2°C) and cured for 48 h. The ore agglomerations comprising different binders were soaked in sulfuric acid solution for 5 d to determine the optimal binder.

2.3. Experimental scheme and setup

The experiments conducted in this study involved ore agglomeration preparation, selection of the optimal bonding conditions, and acidic soaking and leaching with different sulfuric acid solutions. Fig. 1 shows a schematic of the experimental procedure.

Details of the experimental procedure are as follows: (1) A 5-d soaking test was conducted to identify the optimal binders and analyze the agglomeration features. (2) As shown in Table 2, ten sets of experiments were conducted with different PSDs to confirm the optimal proportion and leaching behavior of the agglomerations. (3) Five tests were conducted on the acidic leaching (L1–L5) of agglomerations using sulfuric acid solutions of different concentrations (10, 15, 20, 30, and 40 g/L). The effects of the sulfuric acid concentration on the Cu extraction rate and porosity evolution were then evaluated. The initial volume of the sulfuric acid solution was 150 mL in samples L1–L5. The ambient temperature was 27 ± 2°C. The acidity of the leaching media was adjusted using a 3 mol/L sulfuric acid solution.

Table 1. Mineral composition of experimental copper oxides wt%

Natural Cu	Chalcopyrite	Malachite	Silicated-malachite	Limonite	Magnetite
0.001	0.160	1.282	0.425	41.307	1.044
Quartz	**Garnet**	**Chlorite**	**Kaolin**	**Calcite**	**Others**
13.650	5.362	3.656	24.706	7.807	0.006

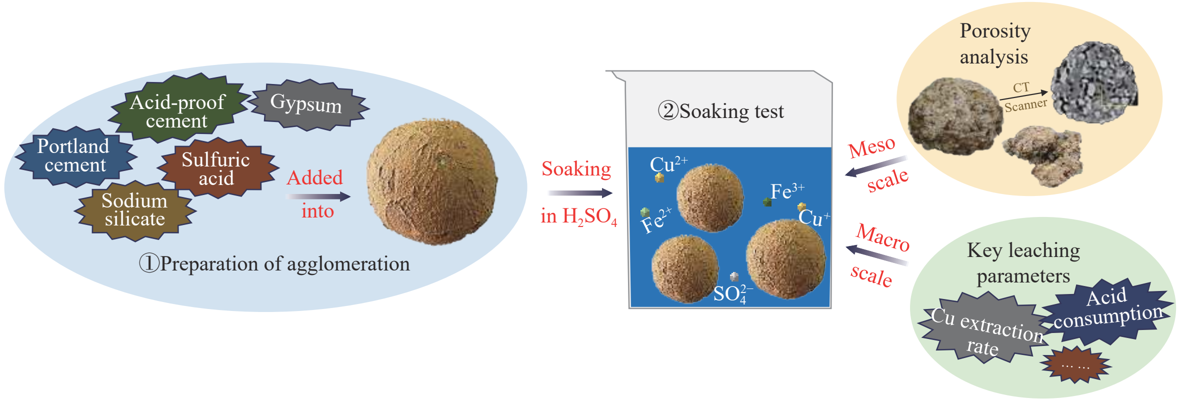

Fig. 1. Schematic of experimental agglomeration and acidic leaching.

Table 2. Particle-size distributions of the ore after agglomeration preparation in acid leaching experiment

Experimental group	Particle size distribution / wt%					Geometric mean size / mm	Size variance (σ^2)
	−5+3 mm	−3+1 mm	−1+0.45 mm	−0.45+0.2 mm	−0.2+0 mm		
S1	30.0	30.0	25.0	10.0	5.0	2.705	2.744
S2	25.0	30.0	30.0	10.0	5.0	2.505	2.519
S3	20.0	30.0	30.0	12.5	7.5	2.271	2.397
S4	20.0	28.0	36.0	8.0	8.0	2.252	2.234
S5	20.0	36.0	28.0	8.0	8.0	2.412	2.162
S6	20.0	34.0	30.0	8.0	6.0	2.404	2.16
S7	20.0	34.0	34.0	8.0	4.0	2.404	2.158
S8	19.0	27.0	27.0	8.0	19.0	2.104	2.113
S9	35.0	35.0	20.0	5.0	5.0	3.033	2.108
S10	27.5	42.5	20.0	5.0	5.0	2.883	2.072

Using an X-ray CT scanner (Siemens Ag X, Germany), we obtained two-dimensional (2D) CT images of the agglomerations after 0 and 20 d of acid leaching. Then, to determine the porosity evolution during acid leaching, we used an algorithm in MATLAB 2016 to binarize and process the 2D CT images to obtain the sectional porosities.

2.4. Analysis methods and equipment

The raw ores were processed in turn by a jaw crusher (PX/PEF60 × 100, China), a roll crusher (XPZ-200 × 125, China), and a screener (GB/T 6003.1–1997, China). The pH value was determined using a pH meter (pHS-3C, Leici, China). The cupric concentration in the leaching solution was determined using an atomic emission spectrometer (ICPE-9810, Shimadzu, Japan), and the copper mineral phase was analyzed using atomic absorption spectroscopy (AAS, AA900 PerkinElmer, the United States). We analyzed the data using Origin Pro 2016 software (OriginLab, United States).

3. Results and discussion

3.1. Effect of different binders on agglomeration curing and soaking features

After acid soaking, Table 3 shows the agglomeration curing results and an analysis of the structural stability obtained when using different binders. To better evaluate the agglomeration behavior of fine ore particles, two conceptual parameters were defined: agglomeration evaluation (AE) and structural stability evaluation (SSE). The AE indicates how well the ore agglomerations are shaped and the SSE indicates whether the agglomerations have sufficient strength. If the fine particles have bonded to form well-shaped agglomerations, the AE is considered to have been successful (S), otherwise it failed (F). Similarly, the SSE was marked with S if the ore agglomerations had not disintegrated, otherwise it was marked F.

Considering the results presented in Fig. 2 and Table 3, it is clear that well-shaped agglomerations were obtained using acid-proof cement, sulfuric acid, and Portland cement. The SSE of the agglomerations was good, especially for the Portland and acid-proof cements. This indicates that the cement composition was effective in obtaining a stable agglomeration pore structure. Except for any van der Waals forces and liquid bridges that formed, this structure occurs when sulfuric acid reacts with the oxides to produce complexes such as $CaSO_4$, which bond adjacent particles and improve the strength of agglomeration [29–30]. No well-shaped agglomerations were obtained when using sodium silicate or gypsum

Table 3. Agglomeration and acid leaching experiments on ore samples with different binders

Sulfuric acid (3 mol/L)		Sodium silicate		Gypsum		Portland cement		Acid-proof cement	
Content / wt%	AE/SSE	Content / wt%	AE/SSE	Content / wt%	AE/SSE	Content / wt%	AE/SSE	Content / wt%	AE/SSE
10	F/F	5	F/F	4	F/F	5	F/F	6	S/F
15	S/F	6	F/F	5	F/F	6	F/F	7	S/F
17	S/F	8	F/F	6	F/F	11	S/S	11	S/F
18	S/F	9	F/F	8	F/F	14	S/S	14	S/F
18	S/F	11	F/F	11	F/F	16	S/S	18	S/S
—	—	—	—	—	—	18	S/S	20	S/S
—	—	—	—	—	—	20	S/S	22	S/S
—	—	—	—	—	—	24	S/S	24	S/S

Note: “F” indicates that agglomeration failed and “S” that agglomeration succeeded.

binders. Judging by the surface glossiness of the agglomerations bonded by the Portland and acid-proof cement binders [31], the moisture contents of the agglomerations were nearly saturated. As shown in Fig. 2(a), the agglomerations bonded by sulfuric acid were destroyed and crumbled into blocks with the dissolution of the minerals. On the contrary, Portland and acid-proof cements both exhibited good bonding performance in that the agglomerations maintained good shape even after 5 d of soaking. The agglomeration bonded by cement would not crumble if the mass fraction was higher than 18% (Table 3). Fig. 2(b) shows the curve of the copper extraction rate, in which we can see that the agglomeration bonded by sulfuric acid cracked after 1 d, resulting in an increase of the reaction interface and eventual copper recovery. However, cracks are known to easily clog the flow paths in actual heaps [32]. The agglomerations bonded by Portland cement showed better leachable potential in that the peak Cu extraction increased to 18.4% in 5 d, which is slightly lower than 19.1% (bonded by sulfuric acid) and much higher than 12.2% (bonded by acid-proof cement). In short, based on the results of this study, Portland cement was determined to be the optimal chemical binder.

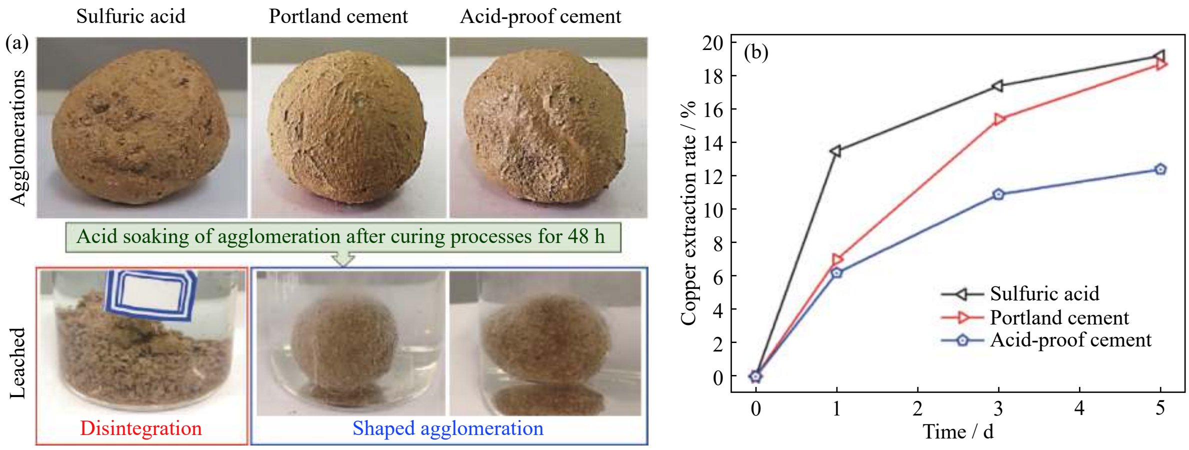

Fig. 2. Ore agglomeration experiment using different binders and the resulting copper extraction characteristics: (a) disintegration and shaped agglomerations and after acidic soaking; (b) copper extraction rate with acid soaking time.

3.2. Effect of PSD on the size of agglomeration

The PSD in agglomerations is an essential parameter that strongly affects the bonding behavior and extraction efficiency [33]. In this study, we used the variance of the particle size to indicate the effect of the PSD. The disintegration period is indicated by the leaching date on which the agglomeration cracks. Fig. 3 shows the relationship between the particle-size variance and the disintegration time. Roughly, in this saturated acidic leaching condition, the agglomeration disintegration period was negatively correlated to some extent with the variance of particle size. In other words, the agglomerations bonded by fine particles (e.g., S5, S6, and S7) tended to be more stable, whereas those bonded by coarse particle (e.g., S1 and S2) could be easily disintegrated after just a short leaching period. However, we note that an excessively high proportion of fine particles (i.e., S8) or an excessively high proportion of coarse particles (i.e., S9) both easily led to the rapid disintegration of the agglomerations. The proportion used in S5, i.e., −5+3 mm (20wt%), −3+1 mm (36wt%), −1+0.45 mm (28wt%), −0.45+0.2 mm (8wt%), and −0.2+0 mm (8wt%), was regarded as the optimal proportion in the subsequent acid leaching.

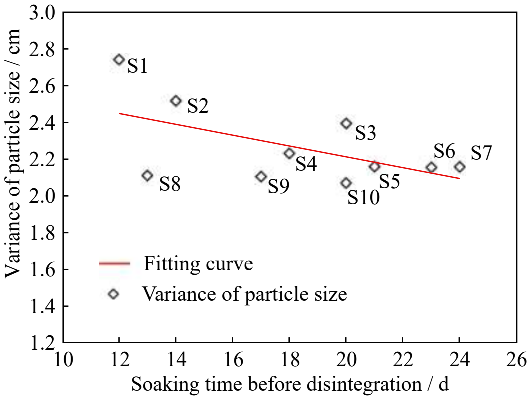

Fig. 3. Relationship between particle-size variance and soaking time before disintegration.

3.3. Acid leaching of agglomerations with different mass fractions of sulfuric acid

3.3.1. Copper extraction rate

Fig. 4 shows the relationship between the copper extraction rate and the acid leaching time. As shown in Fig. 3, the optimal copper extraction rate of sample L5 (40 g/L H_2SO_4) reached 60.6% in 11 d, which was obviously higher than those of the other four groups. This is because the agglomerations disintegrated in the 40-g/L H_2SO_4 media at 12 d, thus exposing the potential reaction interface and increasing the

Cu extraction rate. The formation of calcium sulfate and other passivation materials could be the potential reason that the copper recovery of the 40-g/L H_2SO_4 increased more slowly after 14 d [34], much like the calcium sulfates and calcium silicates. The L4 sample (30 g/L H_2SO_4) exhibited a good potential after 15 d and its copper extraction rate reached 67.2% at 20 d. As shown in Fig. 4(b), the excessively high mass fraction of the sulfuric acid solution not only led to a lower peak extraction rate but also an earlier disintegration. In the acidic leaching process, sulfuric acid reacts with copper oxides (e.g., malachite, $Cu_2[OH]_2CO_3$) and collects targeted cupric ions. At the same time, the reaction products accumulate in void spaces and can eventually cause the agglomerations to disintegrate.

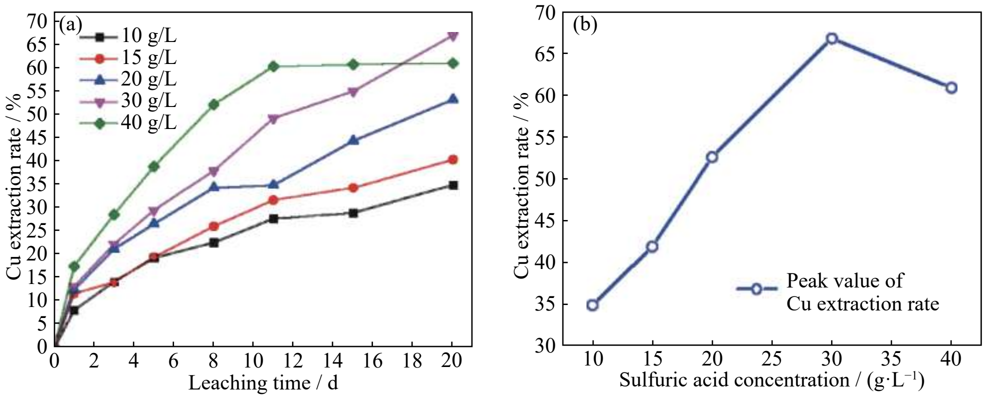

Fig. 4. Key parameters and acid consumptions during leaching process: (a) relationship between Cu extraction rate and leaching time; (b) relationship between Cu extraction rate and H_2SO_4 concentration.

3.3.2. Consumption of sulfuric acid

Fig. 5 shows the consumption of sulfuric acid in samples L1–L5. The results indicate that the sulfuric acid was consumed quickly in the first 10 d and then gradually the consumption rate became consistent after 16 d when the valuable metal attached to the oxide minerals had been rapidly extracted. We can speculate that the leaching reaction was controlled by liquid diffusion in the later leaching stage. With dissolution of minerals, some insoluble substances such as calcium sulfate, silicon dioxides, and magnesium sulfates and complexes formed, the formulas for which are shown in Eqs. (1) and (2). The formula for the leaching reaction of the copper oxides ($Cu_2[OH]_2CO_3$) in sulfuric acid media is shown in Eq. (3). The acid consumption in samples L4 and L5 tended to become consistent after 10 d, whereas it leaped to a higher rate after 12 d. This sudden increase in acid consumption was closely related to the disintegrating agglomerations. This disintegration provided new reaction interfaces and promoted further solute diffusion.

$$CaO\cdot SiO_2 + H_2SO_4 \rightarrow CaSO_4 + SiO_2 + H_2O \tag{1}$$

$$CaO\cdot Al_2O_3 + 4H_2SO_4 \rightarrow Al_2(SO_4)_3 + CaSO_4 + 4H_2O \tag{2}$$

$$Cu_2(OH)_2CO_3 + 2H_2SO_4 \rightarrow 2CuSO_4 + CO_2 + 3H_2O \tag{3}$$

Fig. 6 shows the relationship between mass fraction and the total consumptions of sulfuric acid, in which we can see that the acid consumption was proportional to the mass fraction of the sulfuric acid. Moreover, the acid-consuming reaction tended to be stronger at a higher mass fraction of sulfuric acid (>30 g/L H_2SO_4). Alkaline gangue minerals and oxide substances consume sulfuric acid [35], which explains

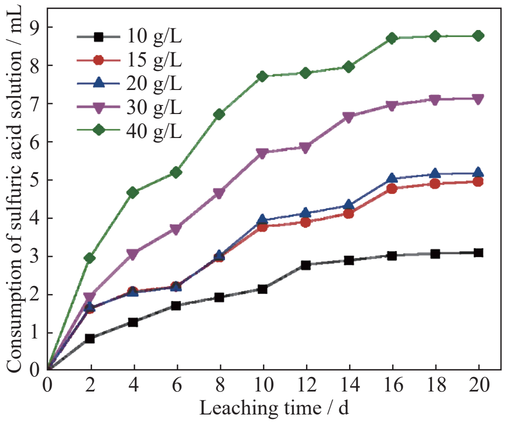

Fig. 5. Relationship between sulfuric acid consumption and leaching time.

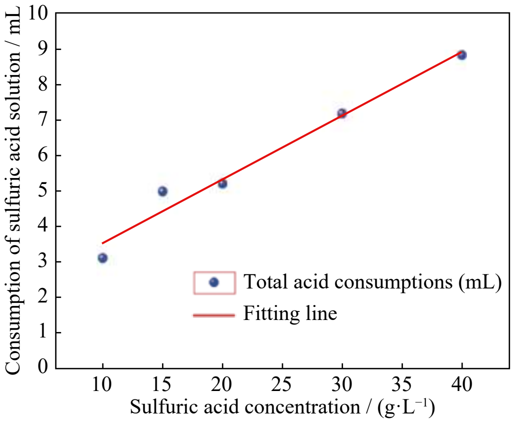

Fig. 6. Relationship between mass fraction and total consumption of sulfuric acid.

why a lower mass fraction would not be suitable for metal extraction, i.e., due to the presence of excessive gangues. Thus, the total acid consumption is mainly determined by the initial mass fraction of sulfuric acid and by the disintegration features.

3.4. Disintegration behavior of agglomerations with different mass fractions of sulfuric acid

Fig. 7(a) shows the agglomeration disintegration with different mass fractions of sulfuric acid. Based on visual observation, the color of the acid solution changed from white to light green to blackish green as the concentrations of cupric and ferric ions increased in the acidic media. Porous, loose exfoliations, and reaction residues including unreacted substances, i.e., the calcium and magnesium compounds, were observed on the agglomeration surfaces especially in samples L4 and L5 (>30 g/L H_2SO_4), which indicate structural destruction and disintegration. The leaching reaction not only

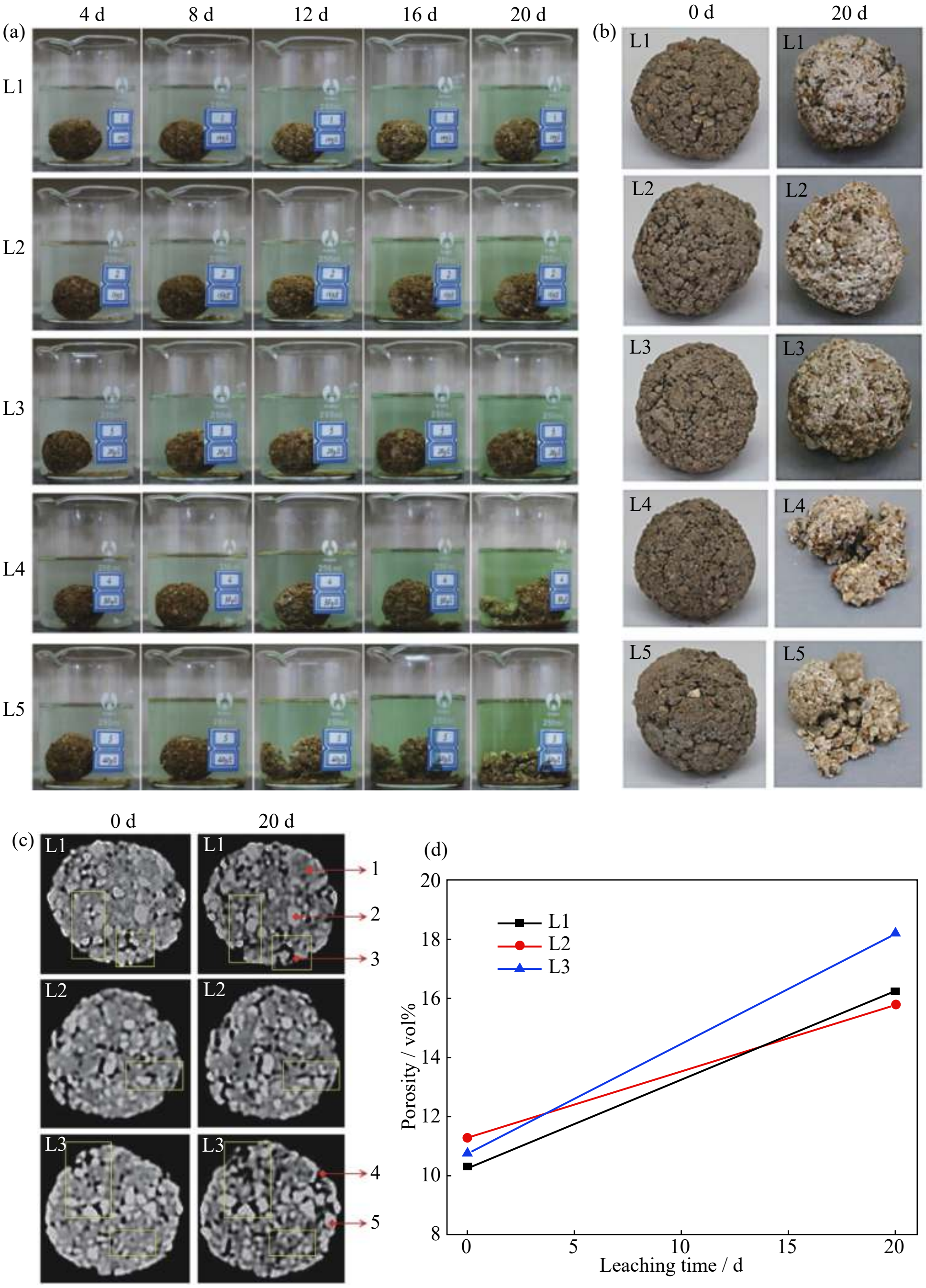

Fig. 7. Agglomeration disintegration and porosity evolution during acid leaching: (a) agglomeration dissolution with soaking time; (b) agglomeration before/after soaking; (c) 2D image at 0 and 20 d. 1—Fine ores, 2—Coarse ores, 3—Neonatal pores, 4—Pore expansion, 5—Surfacial bonding layer; (d) porosity changes with leaching time.

occurs on the surface of the ore agglomerations, but also inside the agglomerations, controlled by solute diffusion [36]. Fig. 7(b) shows the substances precipitated on the agglomeration surface, which are predicted to be either calcium sulfate dehydrate ($CaSO_4 \cdot 2H_2O$) or aluminum sulfate octadecahydrate ($Al_2[SO_4]_3 \cdot 18H_2O$) based on the results of previous studies [37]. For the agglomerations without ruptures, the reaction intensity decreased from the surface to the core of the agglomerations depending on the solute diffusion paths [38]. Even though a higher copper extraction rate could be obtained, such extensive disintegrations were not beneficial to the industrial heaps because the expansion of these crystalline hydrates are known to clog the connective pores and voids [39] and accelerate further compactions and clogging in industrial heaps. If we compare samples L3 (20 g/L), L4 (30 g/L), and L5 (40 g/L), it is also evident that excessive sulfuric acid promotes agglomeration disintegration.

3.5. Pore evolution of agglomeration

Unlike non-agglomerated particles, the pore structure of agglomerations is well-developed, which can lead to a better liquid saturation and leaching condition [40–41]. To examine the agglomeration pore structure before and after acid leaching, Figs. 7(c) and 7(d) show 2D CT images of the non-disintegrated condition (L1, L2, and L3) and that with porosity, respectively. By comparing their meso-morphological differences, we can see that the original potential paths developed and the path “dead ends” were re-connected. This is because after the dissolution and detachment of valuable minerals and impurities, the intra-particle pores developed inside the agglomerations, which is a key factor leading to agglomeration disintegration and a higher Cu extraction rate. As shown in Fig. 7(d), the porosity of samples L1–L3 increased after 20 d of leaching. The net increase in porosity was 6.0vol% (L1), 4.5vol% (L2), and 7.4vol% (L3). The optimal porosity of 18.1vol% was obtained in L3 (20 g/L H_2SO_4), which positively corresponded to the highest Cu extraction rate in the agglomeration (L1–L3). However, as noted in the above discussion of the leaching behavior, the development of intra-particle pores inside the agglomerations is a remote cause of agglomeration disintegration. Thus, in industrial heaps, the optimal binders and their mass fraction must be carefully considered to obtain the desired leaching efficiency and prevent unexpected disintegration of ore agglomerations.

4. Conclusions

In this paper, we used soaking tests and X-ray CT scanning to investigate the leaching behavior and pore evolution of copper oxide agglomerations bonded with different chemical binders. The results are as follows.

(1) A single ore agglomeration particle consists of an internal core and a porous shell. This porous shell promotes the development of intra-particle porosity, which results in a significant increase in the peak Cu extraction rate.

(2) After acidic soaking, ore agglomerations bonded by Portland cement were found to have a better shape and structural strength than those bonded by acid-proof cement and sulfuric acid media.

(3) With the dissolution of copper oxides and gangue minerals, agglomerations composed of excessively high contents of fine or coarse particles easily cracked and were even completely destroyed during the early period of acidic leaching.

(4) Although highly concentrated sulfuric acid (40 g/L) can obviously promote copper recovery and porosity, it can also lead to the undesirable disintegration of agglomerations.

In short, parameters closely related to the leach ability and structural stability of agglomerations should be carefully considered in industrial operations involving agglomerated heaps.

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