CHAPTER 10.1

Agglomeration Pretreatment

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Pretreatment for hydrometallurgy is a very broad subject, but within this chapter, it is limited to ore agglomeration for the benefit of heap leaching. The reason for narrowing the scope stems from the injustice that this chapter would do to other forms of pretreatment, complex and widespread, that merit their own coverage. Such is the case with crushing, grinding, classification, screening, separation of all types, flotation, and pyrometallurgical pretreatments, which are all covered in other chapters of this handbook.

Other pretreatments for hydrometallurgy are worth noting here but will not be covered in more detail, mostly because of their small-scale experimental nature. Such pretreatments include biooxidation, ammonia leaching (e.g., Feng and Van Deventer 2011), alkaline leaching (e.g., Celep et al. 2011), mycohydrometallurgy (e.g., Ofori-Sarpong et al. 2011, 2013), and microwave use (e.g., Olubambi et al. 2007; Olubambi 2009).

A further restriction to the scope of this chapter is the type of ores discussed; specifically the exclusion of coal and iron ore, both good candidates for agglomeration. Iron and coal are not discussed in this chapter because their post-agglomeration treatment is pyrometallurgical in nature.

From 2005 to 2015, four review-type articles were published on the subject of agglomeration as a pretreatment for hydrometallurgy (Bouffard 2005; Lewandowski and Kawatra 2009a; Moats and Janwong 2008; and Dhawan et al. 2013). Yet, during this decade, there were few research-type articles published on this subject, and those published were predominantly academic in nature, reporting on small-scale testing. In previous decades, more publications of large-scale testing, at times in operations themselves, were reported. This chapter reviews published works on agglomeration for hydrometallurgy, beginning with the benefits of agglomeration, followed by equipment design, quality control, and concluding with commodity-specific observations and recommendations.

BENEFITS OF AGGLOMERATION

The work of T.C. Scrutton in 1905 (as cited in Dorr and Bosqui 1950) is one of the earliest references to agglomeration. Scrutton agglomerated ore by rolling it down a chute inclined at 60° and stacked the agglomerates in a vat. The initial work on iron ore pelletization began a few years later in 1911 and quickly expanded to manganese, fluorspars, and phosphate. The greatest advances in agglomeration for hydrometallurgy, more precisely for heap leaching, occurred in the late 1970s at the U.S. Bureau of Mines (USBM) in Reno, Nevada (Potter 1983). In the 1960s, this organization developed cyanide heap leaching for recovering precious metals from low-grade ores. Some commercial heaps commissioned prior to the USBM agglomeration studies had poor solution permeability, resulting in low gold and silver recovery over the leach cycle. The wide range of particle sizes in the heap, the size segregation during stacking, and the ore mineralogy (e.g., clays) were the ultimate culprits of low permeabilities. The agglomeration work of pioneers, such as Gene McClelland and Paul Chamberlin, contributed to the rapid commercialization of cyanide heap leaching. The USBM determined the optimum practical requirements (moisture level, binder type, binder dosage, curing time, agglomeration equipment, residence time) for agglomeration of precious metal ores and tailings.

In 1979, the first pilot-scale heap leach test was performed on crushed and agglomerated ore in eastern Nevada. The first commercial agglomeration heap leach began operation in 1980. According to Gomes (1983), there were 36 commercial operations in the western United States that agglomerated ore in 1983. Gold or silver extraction from agglomerated crushed ore was as high as 90% in sometimes as little as 10 days. Agglomeration at an Arizona silver heap leach increased recovery from 37%–90%, and the leaching time dropped from 90 to only 7 days. Significant improvements were also obtained at a northern Nevada gold heap leach operation where the gold extraction increased by 60%, and the leach time was reduced by half from 50 to 20–30 days. The Alligator Ridge mine in northeastern Nevada reported a reduction of the cyanide heap leach time from 60–90 days down to 30–40 days, with gold recoveries of 70% (DeMull and Womack 1984; Strachan and Van Zyl 1987). Agglomeration was a technical and economical breakthrough technology for heap leaching of clayey ore and ores containing high fines content. Up to 80% of the metal value of tailings could be extracted in 20–70 days.

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In the copper industry, the thin layer leaching concept developed in the 1980s by Sociedad Minera Pudahuel, originally for copper oxides and then applied to copper sulfides, contributed to the phenomenal expansion of heap leaching, solvent extraction, and electrowinning in South America. In general, the ore, crushed to <12–16 mm, was agglomerated with concentrated sulfuric acid, stacked in heaps typically 6–8 m tall, and cured for a couple of days prior to irrigation with raffinate or intermediate pregnant leach solution.

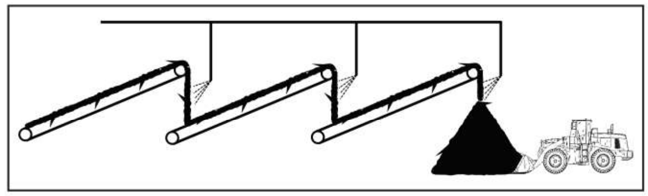
The ultimate benefits of agglomeration are higher and/or faster metal extraction and recovery from heaps, notwithstanding lower reagent consumption and faster rinsing of the heap with potentially lower volumes of wash water used at closure. The benefits of agglomeration are summarized as follows:

- From the perspective of the leach chemistry, agglomeration reduces the travel time of reagents and increases the initial recovery rate. These benefits arise because of the faster contact between the sulfide minerals and the reagents contained in the agglomeration solution, although undesirable chemical reactions between gangue minerals and reagents are also possible (e.g., sulfuric acid and carbonates in copper sulfide ores).
- From the perspective of stacking, agglomeration helps minimize or avoid size segregation (typically larger particles at the bottom) and fines migration, and generally helps form a more structurally stable heap (less slumping), capable of bearing its weight and those of the lifts above the base level. Agglomeration does not prevent the expansion of swelling clays on contact with water but avoids the formation of impermeable zones by distributing clay particles more evenly into the heap. According to Kappes (2002), higher, slower or staged water additions in agglomeration allow clays to swell in the drum.
- From the perspective of hydrodynamics, agglomeration increases the heap permeability, which translates into less solution retained overall, less stagnant solution, and a higher volume of pores filled with air for greater oxygen transfer, with the expectation that such parameters remain relatively constant over the long leach cycle. Agglomeration helps minimize solution ponding, channeling, and potentially slope failure.

AGGLOMERATION EQUIPMENT

Agglomeration begins with wetting ore particles and evolves through agglomerate nucleation, growth, consolidation, and breakage. Growth by layering does not change the number of agglomerates but increases their mass. The coalescence process (two agglomerates forming one) has the opposite effect. Breakage produces more uniformly sized agglomerates by splitting large, poorly consolidated agglomerates into smaller ones. During and after agglomeration, solid bridges (such as crystals or interlocking fibers) and liquid bridges are formed.

According to 24 of the 43 responding mining companies that crushed ore before heap leaching (Kappes 2002), 8 did not agglomerate, 5 turned to belt agglomeration, and 11 used rotating drums. Belt conveyor and drum agglomeration are the two principal pieces of industrial-scale agglomeration equipment employed. Some references to rotating disc agglomerators and pug mills are included.



Adapted from Chamberlin 1986

Figure 1 Belt conveyor agglomeration

Belt Agglomeration

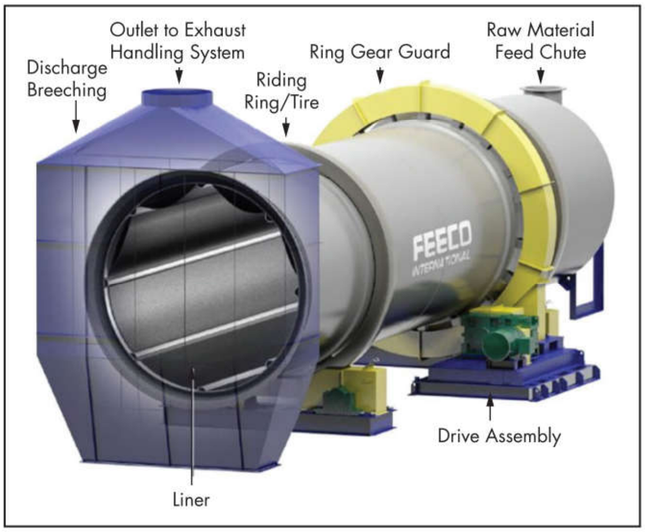
Conveyor belts are well suited to agglomerate ore typically containing less than 15% of –104 µm (150 mesh size) fines (Figure 1). Belt conveyors can be operated in two ways:

1. When all belt conveyors are inclined at approximately the same angle (about 15°) and are moving in the same direction, agglomeration occurs when particles touch each other at the transfer point between belts or when they bounce on the belt upon landing. Dispersion bars hanging at the discharge of a belt improve the mixing of the ore in its free fall of 1.2–1.8 m.
2. When the ore falls from a low-angle conveyor moving relatively slowly onto a high-angle (35°–55°) conveyor moving rapidly in the opposite direction, agglomeration occurs primarily on the high-angle belt. Too high of a belt angle can cause the agglomerates to slide down the belt rather than roll.

A solution can be sprayed at the transfer points or along the belts. LeHoux (1997) recommends staging the points of solution addition at the end of the conveyor trains to avoid buildup of slimes underneath the equipment and solution running down the belt. Too little solution results in excessive dusting at the transfer points. Too much solution results in spillage at the transfer points, more frequent shutdown for cleanup, solution running down the belt, as well as compaction of agglomerates on landing on the pile (rather than rolling down the pile).

Drum Agglomeration

An agglomeration drum (also called a rotary drum, balling drum, or agglomerator) is well suited for ores containing high clays or a large fines content. Chamberlin (1986) prefers using an agglomeration drum rather than a belt conveyor when a binder must be added or when an application combines a chemical reaction during agglomeration. Drum agglomeration consists of injecting ore into a cylindrical, inclined drum that rotates to impart rolling, cascading, and tumbling of the ore particles (Figure 2). Two manufacturers of agglomeration drums are FEECO International and Sepro Mineral Systems, to name a few. According to industrial drum design data, the length of the drum is typically three times longer than the drum diameter (Bouffard 2005). The drum length rarely exceeds 15 m. Sepro Mineral Systems offer drums from 1.8 m in diameter by 5 m long with one 45-kW motor to 3.6 m in diameter by 10 m long with four 60-kW motors. The motors power either chain and sprocket drives (low power), friction drives (low power), gear and pinion drives (high power), pneumatic tire drives, or direct drive assemblies (specialty applications). Drums can be equipped with spray systems;



Courtesy of FEECO International
Figure 2 Drum agglomeration equipment

various liners; screw conveyor feeders; scrapers; gear lubrication systems; variable speeds (9–17 rpm), slopes (5°–8°), and frequency drives; rotary control consoles; tire pressure monitoring systems; and safety limit switches.

Regular adjustment of drum bearings prevents serious wear and damage to the drum. Normal wear and tear causes the drum to fall out of alignment, resulting in excessive chatter and drive component vibration, formation of grooves and gouges on the face of the tires, and wear and damage to the thrust rollers, pinions, and girth gear. Regular inspection and realignment of a drum are important preventive maintenance tasks having the greatest impact on costs, downtime, and longevity.

The interior of the drum may be rubber-lined to prevent corrosion and equipped with loose chains or rubber strips to prevent ore from sticking. The solution is pumped through nozzles or perforated pipes preferentially located along the first two-thirds of the drum length. In rare applications, a drum agglomerator may be equipped with screens on the discharge end to separate oversize from undersize agglomerates. Recycle ratios between 2:1 and 5:1 are common in iron ore pelletization and fertilizer granulation circuits. Recycling affects the moisture content and the agglomerate size distribution.

Miller (2010) published a comprehensive study on drum agglomeration design. He developed a mathematical model for predicting power draw and optimum fill as a function of the ore feed rate. The model considers drum geometry, total static and operating volumes, solids residence time based on operating volume, and critical speed. Miller verified the model predictions with the performance of industrial drums.

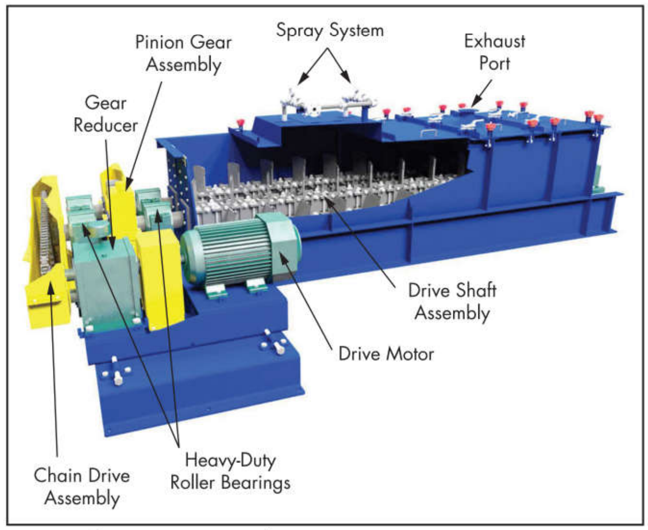
Bouffard (2005) developed an empirical relationship calculating the drum's ore throughput as a function of drum diameter, drum length, and rotation speed. This author demonstrated that the rotation speed of laboratory and commercial drum agglomerators used in heap leaching, in the fertilizer and iron ore industries, lies between 30% and 50% of the critical speed. The rotation speed translates into a residence time that Chamberlin (1986) suggested to be at least 60 seconds for coarse ore and 240 seconds for fines.

Abbaszadeh et al. (2013) built a 1-m-wide by 3-m-long drum agglomerator to agglomerate a gold/copper ore. The position of the spray nozzles in the drum, the load in the drum, the uniformity of the ore on entry into the drum, and the residence time were important factors.

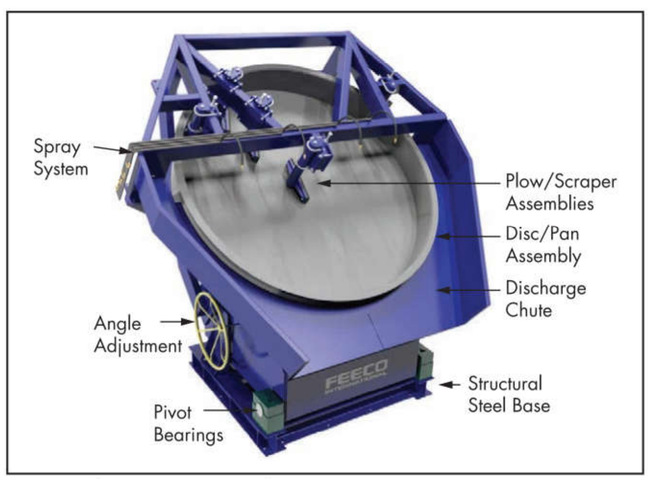
Other Agglomeration Equipment

Ores containing low clay content and fairly coarse particles can also be agglomerated by simpler means, such as spraying the agglomeration solution on the sides of the heap or onto the ore as it discharges the stacking conveyor as stacking progresses. As the ore is rolling down the slopes, fines will stick to coarse particles. This is referred to as stockpile agglomeration.

Pug mills or paddle mixers (Figure 3) and disc agglomerators (Figure 4) are rarely used in heap leaching (Kappes 2002). A pug mill is a device equipped with a horizontal trough in which a central shaft slowly rotates, to which is attached mixing blades, bars, rods, or paddles. This creates a kneading and folding-over motion that thoroughly blends the



Courtesy of FEECO International
Figure 3 Pug mill



Courtesy of FEECO International
Figure 4 Disc agglomerator

materials. A disc agglomerator is a rotating, tilted disc or pan with a rim, primarily used in the iron ore, agricultural, and chemical industries. Disc agglomerators have lower throughputs than drum agglomerators but produce a product in a narrower range size and have fewer maintenance requirements (FEECO International 2017).

OPERATING CONDITIONS AND QUALITY CONTROL

Heap Stacking, Irrigation, and Operation

The beginning of this chapter cited the benefits of greater permeability possible with agglomeration. To achieve good permeability, ore handling and irrigation practices are just as important as agglomeration.

There are at least three types of stacking systems: (1) a haul truck for dump leaching, (2) a mobile conveyor unit (or grasshopper) combined with radial stacker for heaps, and (3) a spreader conveyor employed primarily for dynamic heap leach pads of constant width and height. Because spreader conveyors travel across the entire width of the pile, there is less segregation across the length of the pile. Radial stackers tend to create discontinuities at the intersection between ridges and fingers (Scheffel 2002) and were the cause for the size gradation measured across the 9-m height of a heap (Kinard and Schweizer 1987).

Miller (2003) demonstrated a proportional relationship between heap height and its bulk density. The heap bulk density increases with heap height, almost in a linear fashion. A lower bulk density should help minimize the retention of solution and keep more pore spaces filled with air.

Scheffel (2002) recommends ripping the heap surface two to four times in crisscross direction and to rip the lower lift before stacking the next lift. Uhrie and Koons (2001) have shown that truck traffic areas should be ripped to a 2.4-m depth before irrigation.

There are three common methods of irrigation: drip emitters, wobble sprinklers, and reciprocating sprinklers. Drip emitters have the gentlest impact on the heap surface but are susceptible to hole plugging. Rainbird sprinklers are undesirable for heaps stacked with agglomerates. The jets cause disintegration of the agglomerates, unless the heap surface is covered by coarse particles to dampen the impact of the droplets, as practiced by the Cerro Rico operation of Compañia Minera del Sur S.A., Bolivia. Heavy rainfall in tropical climates and stalled sprinklers are responsible for agglomerate disintegration, which can be prevented with laying shadecloth or similar material on the heap.

Like ripping, remining the heap (ore turnover by hydraulic excavators), pioneered at the Australian Bullabulling gold heap leach operation and then the Girilambone Copper Company, may increase recovery (e.g., copper recovery up by 2%–10% during the same leach time).

The following subsections review methods for determining agglomerate quality: moisture content, size distribution, attrition, flowability, crush strength, and green/wet strength. Many of these direct methods can be automated and used in operations. Other methods indirectly determine the quality of agglomerates by testing for bulk density, slump, compression, porosity, permeability, and leachability of agglomerates stacked in a bed (typically a column). A lower bulk density, a lower slump, a higher porosity, and so forth, are the benefits of proper agglomeration. These indirect methods are not specifically reviewed in this section but are cited elsewhere in this chapter. Care should be exercised when scaling up parameters from columns to heaps, as the column walls are a significant influence on bulk density and permeability (Bouffard 2008).

Moisture Quality Control

There is no specific guideline for the appropriate moisture addition during agglomeration; it depends on the particle size distribution, the initial moisture content of the ore, the ore mineralogy, and the use of binders. Here are some rules of thumb for determining the moisture content before the agglomeration begins or during the operation.

1. Use this empirical equation relating the density of solid and liquid (before agglomeration):

$$\omega = \frac{1}{1 + 2.17\frac{\rho_s}{\rho_l}} \text{ for particle size} > 30\ \mu\text{m} \qquad \textbf{(EQ 1)}$$

where ω is the moisture content in kg liquid/kg dry ore, ρ_s is the solid density, and ρ_l is the density of the liquid having similar properties as water. For a solid density of 2.8 g/cm^3 and a water density of 1.0 g/cm^3, the moisture content is approximated to be 14 wt %. This equation would be unsuitable for tailings, which contain a greater proportion of fines, thus typically requiring twice as much moisture as crushed ore (15–30 wt % vs. 5–15 wt %).

2. Perform a soak test (before agglomeration). Vethosodsakda et al. (2013) suggested that the optimum moisture content of agglomerates prepared in batch can be determined from the volume of water soaked up in 1 hour by the non-agglomerated ore placed in a column whose bottom is submerged in water. The amount of ore, and more precisely the height of the ore in the column, was found to be very influential in this soak test. A height of 10–18 cm for a column diameter of 15 cm was preferred.

3. Use the filter cake test (before agglomeration). Chamberlin (1986) estimated that the moisture content of the agglomerates is 1%–3% less than the moisture content of a dewatered filter cake.

4: Perform a sheen test (during agglomeration). According to the sheen test, there should be no moisture glistening on the surface of agglomerates.

5. Use squeeze and drop tests (during agglomeration). Squeeze or drop shatter tests have been used to control the amount of acid and moisture added in batch or continuous agglomeration.

6. Check for electrical conductivity (during agglomeration). Fernández (2003) and Velarde (2005) introduced the use of electrical conductivity to evaluate the proper amount of moisture for agglomerating a copper ore. The electrical conductivity increases exponentially with the moisture added, with the largest signal detected when a liquid film forms around the agglomerates. Fernández (2003) related conductivity readings to the optimum moisture content of 4% (no clay in ore), 6.5% (medium clayey ore), and 10% (high clayey ore).

Some additional recommendations concerning moisture addition are discussed in the following paragraphs.

If the material to be agglomerated is too wet, it should be dried, because excessive moisture does not produce individual agglomerates but rather clumps of agglomerates that do not roll down the slopes of the heap. A starting material that is too wet also limits the quantity of soluble reagents that can be mixed with the ore during agglomeration.

Tibbals (1987) suggested that the amount of input energy for agglomeration of crushed ore was more important than the method of solution addition. However, when the ore particle size is smaller than the droplet size, as for tailings, some authors (Tibbals 1987; Eisele and Pool 1987) agree that the moisture should be added as droplets rather than as an atomizing spray. Fines adhere to the droplet to create a nucleus.

Some researchers incorrectly equate agglomerate drying and agglomerate curing. Agglomerates should remain moist at all times to avoid disintegration and other negative effects on leaching (Connelly and West 2009). Cement-based agglomerates that were allowed to dry out immediately after agglomeration disintegrated when less than 50 kg/t of cement was added.

Size Distribution Quality Control

There is no denying that agglomerating particles, particularly fines smaller than 104 µm, has been beneficial; the question is rather how wide the agglomerate size distribution should be. Views are mixed on this subject. On the one hand, Dhawan et al. (2012) postulate that a wider distribution is preferable to mono-sized agglomerates. On the other hand, Lipiec and Bautista (1998) recommend uniformly sized agglomerates. Bouffard (2008) also recommends a narrow size distribution, hypothesizing that mono-sized agglomerates have fewer points of contact between them when stacked in a bed. Bouffard and West-Sells (2009) later proved that the agglomerate size distribution affects the bed moisture content. With fewer points of contact, there is less of a tendency for the solution to accumulate between surfaces, thereby reducing the length of diffusion channels and increasing the gas–liquid surfaces.

Bouffard (2008) controlled the size distribution of agglomerates using the following methods:

- Reduce the amount of fines in the ore size distribution, thereby avoiding the production of agglomerates that are too small. Screening the ore can reduce the amount of fines.
- Ensure a narrow initial size distribution of the ore to produce a narrow agglomerate size distribution. This could mean crushing run-of-mine ore to eliminate boulders or, at other times, adding coarse particles (Serrano 2003).
- Recycle undersized agglomerates to skew the feed size distribution and avoid too wide an agglomerate size distribution.
- Control the moisture added at just the right level to produce a narrow size distribution of agglomerates (too little or too much moisture added produces a wide size distribution).
- Extend the time of agglomeration to enable growth and breakage phenomena to form better-shaped agglomerates. This can be done with more conveyor transfer points, a longer drum operated at lower speed and set at a lower angle, or with recycling agglomerates.

The agglomerate size distribution can be measured with online cameras and digital imaging, or screening freeze-dried, air-dried, or wet agglomerates in a less automated way. To understand the growth and breakage phenomena occurring during agglomeration, agglomerates can be wet-screened to literally split the agglomerates into their constituent particles.

Strength Quality Control

Here are a range of methods for measuring agglomerate strength. Some are simple but subjective, suitable for in-field measurement, and others require laboratory testing.

- **Squeeze test.** The interpretation of this very subjective method is mixed. Some argue that after squeezing a bunch of agglomerates in one's hand, the clump should hold if it is poked; others believe the clump should fall apart.
- **Drop test.** Good agglomerates should roll on the ground after they have been thrown up in the air. A variant on this test is the measurement of the highest height of the drop that leads to complete disintegration. Another variant is the number of falls that an agglomerate can sustain when dropped from a fixed height.
- **Attrition test.** Herkenhoff (1987) proposed to tumble dry agglomerates and measure the proportion of abraded fines.
- **Soak test.** There are multiple variations on this theme. Chamberlin (1986) suggested that good agglomerates submerged in water should not disintegrate for many hours. Rather than dipping pellets, the author placed them in a burette and covered the top with glass wool. He then applied water at increasing flow rates and measured the fines content in the discharge solution. Milligan and Engelhardt (1984) measured the amount of fines produced when dipping pellets in water 10 times. The pellets must be previously cured for 6 hours at 90°C and cooled before dipping. Milligan and Engelhardt (1984) proposed a more general soak test in which a known amount of agglomerates between 9.5 mm and 12.7 mm in size are placed onto a 10-mesh screen, which is then submerged in water for 24 hours. After this period, the ore remaining on the screen is weighed.
- **Drainage test.** The USBM (Heinen et al. 1979; McClelland et al. 1985; Eisele and Pool 1987) rated agglomerates to be of high strength if a column filled with agglomerates and flooded with water drained rapidly.
- **Percolation test.** Known informally as the *Kappes test* (Pyper et al. 2015), this percolation method determines the optimum binder requirements for clayey ores. The test involves soaking cured agglomerates in a column for a set time and then tapping the column walls. The test records bed slump, flow rate, agglomerate disintegration, bubble formation, and fines release as indicators of agglomerate strength.
- **Chemical resistance test.** Yijun et al. (2002) measured the number of intact agglomerates remaining in a column subjected to sequential irrigation with water, 20–30 g/L sulfuric acid, and finally 50–100 g/L sulfuric acid. The number of unbroken agglomerates relative to the original number of agglomerates was a proxy for agglomerate strength.

CAPITAL AND OPERATING COSTS

Estimates proposed by Rose et al. (1990) and Kappes (2002) indicate that agglomeration and stacking account for 6%–10% of the total capital costs (Table 1). For cyanide heap leach operations, agglomeration and stacking account for 1%–2% of total operating costs; cement, in a dose of 10 kg/t, for 9%–15%; and other reagents (e.g., cyanide, lime) for 3%–5% (Kappes

Table 1 Heap leaching operation scale influences agglomeration/stacking unit capital, operating, and total costs

Production	400 t/d (Rose et al. 1990)	1,000 t/d (Rose et al. 1990)	3,000 t/d (Kappes 2002)	15,000 t/d (Kappes 2002)
Capital costs				
Agglomeration/stacking, thousand $	460	N/A*	1,000	3,500
Total capital cost, thousand $	4,600	N/A	14,000	53,600
Percentage for agglomeration	10%	N/A	7%	6.5%
Operating costs				
Agglomeration/stacking, $/t	N/A	1.15	0.20	0.10
Reagent for agglomeration, $/t	N/A	N/A	1.00	1.00
Total operating costs, $/t	N/A	7.20	11.50	8.30
Percentage for agglomeration	N/A	16%	10%	13%

Note: All figures are in U.S. dollars.
*N/A = not available

2002). By comparison, a typical Nevada cyanide heap leach operation of 30,000 t/d of coarse-crushed, non-agglomerated ore would have operating costs about 30% less than a similar cyanide heap leach operation agglomerating 15,000 t/d of fine-crushed ore (Kappes 2002).

CONDITIONS SPECIFIC TO CERTAIN COMMODITIES

The following subsections describe agglomeration conditions and results specific to gold, copper, and nickel.

Gold

Agglomeration of gold ores commonly utilizes cement and lime as binders and typically a cyanide solution as an agglomeration solution.

A dosage of 1 kg of sodium cyanide (NaCN)/t of ore is fairly typical for agglomeration. The use of a strong cyanide solution can be a safety hazard for hydrogen cyanide (HCN) emanation at too low solution pH. To control alkalinity and hence minimize cyanide losses as HCN at pH less than 9, 1.5–25 kg lime/t ore is added during agglomeration. Agglomeration with a cyanide solution does not affect the chemistry of the agglomerates. The benefits of adding cyanide during agglomeration on the rate of metal recovery from a heap are not conclusive (Chamberlin 1983 vs. O'Brien 1982). According to Kappes (2002), some operations have stopped adding cyanide during agglomeration because the cyanide code requires containment of spillage of agglomerates in transport from the agglomeration equipment to the heap.

Cement is comprised of 50%–70% tricalcium silicate ($3CaO \times SiO_2$), 15%–30% dicalcium silicate ($2CaO \times SiO_2$), 5%–10% tricalcium aluminate ($3CaO \times Al_2O_3$), 5%–15% tetracalcium aluminoferrite ($4CaO \times Al_2O_3 \times Fe_2O_3$), and various hydrated forms of gypsum ($CaO \times SO_3 \times H_2O$) (Kosmatka et al. 2002). Curing refers to hydration reactions between calcium silicates and water to form calcium hydroxide and calcium silicate hydrate. The chemical composition of calcium silicate hydrate varies, but it typically contains three parts lime to two parts silicate. The calcium silicate hydrate forms dense bonds between particles. Electron microscopy showed calcium to be uniformly distributed throughout agglomerates of clayey particles, even though it typically accounts for less than 1% of the material agglomerated (Heinen et al. 1979).

Cement does not cure by drying. If the moisture is too low, cement stops gaining strength. Hydration resumes after resaturation; the strength increases again. Approximately 40 kg of water per 100 kg of cement is necessary for curing. The strength continues to increase provided that unhydrated cement is still present and the temperature remains favorable. Although 28 days of curing is the standard in the concrete industry, 8–24 hours sufficed in previous agglomeration studies of crushed ore (Chamberlin 1986; McClelland 1986a, 1986b; Eisele and Pool 1987; Zárate and Guzmán 1987). Seventy-two hours was preferable for tailings (Eisele and Pool 1987). The installation of solution header lines and emitters on top of the heap typically takes more than 3 days, and thus agglomerates have sufficient time to cure before the cyanide solution is applied.

Some recommendations on the dosage of cement to use are as follows:

- There are five types of cement under the Canadian Standards Council Standard A5 (CAN/CSA-A5-98), and eight types of cement under the ASTM C150 standard. Cement types I, III, and I/II performed similarly at equivalent dosages added. The handling of cement-based agglomerates after agglomeration is more important to the agglomerate strength than the types of cement used.
- Eisele and Pool (1987) recommend a dosage of 7.5 kg cement/t ore and the same dosage of lime for crushed ore or tailings. Zárate and Guzmán (1987) recommend as much of each binder, that is, 8–12 kg cement/t crushed ore and 8–12 kg lime/t crushed ore. McClelland (1986a, 1986b) and Heinen et al. (1979), however, recommend 2.8–5.5 kg cement/t ore, without or with 3.9 kg of lime/t ore. In operations, the cement dosage ranges from 2.5 to 5 kg/t for crushed ore and 5 to 17 kg/t for tailings.
- Zárate and Guzmán (1987) found that the quality of cement/lime-based agglomerates was optimum at a moisture of 17 wt % but declined at higher water dosages. All three methods employed in these authors' work to measure the agglomerate quality (dip, flooding, compaction) recommended the same moisture content.
- Amaratunga and Hmidi (1997) used gypsum alone and in combination with cement to agglomerate tailings. Agglomerates were stronger with the combination of cement and gypsum.

Following are some positive and negative experiences using cement-agglomerated ore:

- At Little Bald Mountain in Nevada, cement agglomeration reduced slumping from 24% to 8% (Tibbals 1987).

- At the Alligator Ridge mine in northeastern Nevada, agglomerating gold ores with cyanide reduced the overall cyanide consumption and increased the initial recovery (DeMull and Womack 1984). However, adding more than 50 g NaCN/t of ore did not improve the gold extraction and increased the overall cyanide consumption.
- Although a very clayey ore had been well agglomerated with 5 kg lime/t ore and 4 kg cement/t ore, trenches dug across the 9-m height of the heap showed uneven material size gradation (Kinard and Schweizer 1987).
- Keuhey and Coughlin (1984) reported a rare instance of lower gold extraction from cement-based agglomerates. Eight percent less gold was recovered after 32 days from a column containing agglomerates prepared with 10 kg cement type II/t ore.

Some recommendations about lime-agglomerated ore and heap leaches are as follows:

- At the Alligator Ridge mine in northeastern Nevada, agglomeration with 50 g NaCN/t ore and 1.5–5 kg lime/t ore increased gold recovery and shortened the leach cycle by half (DeMull and Womack 1984; Strachan and Van Zyl 1987).
- Litz (1993) reported on the development and patent of a modified lime (Walker and Oliphant 1992) for use as a binder.
- Walker and Oliphant (1992) also patented a mixture comprised of 10%–80% calcareous component, such as quick lime, 5%–50% siliceous-calcareous component, and 10%–80% sulfated component, such as gypsum. The mixture should be added in the amount of up to 2 wt % to precious metal ores for cyanide heap leaching.

Here are some experiences with other polymeric binders for agglomeration of gold ores:

- Milligan was unsuccessful with both wood fibers and molasses but was slightly successful with 1–2 kg/t carboxymethylcellulose/t ore.
- At the USBM, Heinen et al. (1979) observed faster drainage with 0.05 kg polyethylene oxide/t ore mixed with lime and added to the agglomerates.
- Nivens and Given (1993) used polymer aids for agglomeration of gold ores. Newmont gold mines (Carlin, Nevada) and Philex Mining's Sibutad gold project in the Philippines also employed Nalco binders and cement to agglomerate gold ores.
- Brewer Gold found that column gold recovery was superior with the use of 125 g/t Nalco Extract-Ore 9760/t ore and 2.5 kg cement/t ore compared to an ore agglomerated with 7.5 kg cement/t ore (Pautler et al. 1990). In the field, the dual mixture of cement and Nalco 9760 reduced channeling. In addition, the bulk density of the heap with the dual mixture was 3% lower than the heap with cement alone.
- The Betz Dearborn Company developed binder HL 9121 for agglomeration of gold ores. HL 9121 is a cross-linked, borated polyvinyl alcohol added in a dose of 50 g/t ore to 3 kg lime/t ore and 3 kg cement/t ore. In columns, it improved the gold extraction by 5% under simulated heavy rainfall conditions (Polizzotti et al. 1997) and increased the drainage rate by five times (Polizzotti 1993).

Copper

Key issues for the agglomeration of copper ores include the amount of sulfuric acid added, the binder added, and the curing time.

Copper heap leaching operations agglomerate with water and sulfuric acid. On average, 15–25 kg sulfuric acid/t ore is added to 60–100 kg water/t ore. The final moisture content ranges from 7.5 to 12.5 wt %, which is a typical moisture content for agglomerated crushed ore. Sulfuric acid is thought to react with gangue minerals (e.g., kaolinite), to render them amorphous and to inhibit silica dissolution (Cruz et al. 1980; Farias et al. 1995). Curing times with sulfuric acid have been reported from 14 to 336 hours (Lu et al. 2007). Holle (1996) found that more acid added during agglomeration increased the copper recovery by 30%. Morenci mine-for-leach operation also recovered 15% more copper with the addition of 5 kg sulfuric acid/t ore. The increase in copper recovery was proportional to the acid dosage (Uhrie et al. 2003).

Cement is not a suitable binder for ores leached with sulfuric acid: Calcium, sodium, and magnesium sulfates attack calcium aluminate hydrates and calcium hydroxide. A search for a suitable binder to agglomerate copper ores has been underway, with the following findings to date:

- The Cuban Research Center for the Mining-Metallurgical Industry has developed a proprietary binder termed *Additive 1* to agglomerate clayey copper ores (Serrano 2003). The author claims that Additive 1 is low cost, resistant to acid solution, and forms porous agglomerates with good mechanical resistance.
- Nalco binders were utilized by the Toquepala/Caujone copper mine in Chile and the Ray mines in Arizona to agglomerate copper ores. Nifty Copper operation in Washington (United States) has used two other products commercialized by Nalco, Extract-Ore 9560 being one of them, used in a dose of 1 kg/t ore (Efthymiou et al. 1998). Extract-Ore is a medium-molecular-weight latex copolymer of moderate anionic charge.
- Kodali (2010) and Kodali et al. (2011) used stucco (calcium sulfate hemihydrate) in combination with a sulfuric acid solution to agglomerate copper ore prior to leaching the agglomerates in columns. Greater bed permeability and larger copper leaching after 110 days were obtained when using the combination of stucco and acid for agglomeration.
- Hill (2013) tested the effects of polymeric binders and sulfuric acid for agglomeration of a copper ore. The author measured the hydraulic conductivity, the permeability in columns, the agglomerate size distribution, and the column bulk density.
- Kawatra and Lewandowski published extensively on the testing of binders for copper ores. Lewandowski et al. (2006) identified five nonionic polymers as the most promising binders for agglomerating copper ores. The screening was performed with an adapted soak-test method. Agglomerates prepared with these five binders were then subjected to an adapted percolation test. Lewandowski and Kawatra (2009b) preferred cationic polyacrylamide binders over nonionic polyacrylamide binders for agglomeration of copper ore using a sulfuric acid solution of pH 2. Cationic binders created more stable

agglomerates. Using surface charge analysis, the authors found the cationic binders neutralized the negative surface charge of the ore. Hydrogen bonding occurred when the hydrogen bond donor sites on the polyacrylamide chains were attracted to the silanol group hydrogen bond acceptor sites in the ore.
- Following from the earlier works of West-Sells et al. (2007) and Bouffard (2009) on the agglomeration of copper ores with sulfuric acid, elemental sulfur, and lignosulfonate, De Oliveira et al. (2014) mixed a copper ore with fine elemental sulfur (up to 13.3 kg sulfur/t ore) and an inoculum before placing the agglomerates in columns for sulfuric acid leaching. Compared to the control column, more copper was leached from the ore agglomerated with sulfur and microorganisms.
- Barriga Vilca (2013) found it unnecessary to add ferric sulfate to the sulfuric acid solution used for agglomeration of a secondary copper ore: There was no difference in copper recovery after curing.

Nickel

Several researchers, based at the University of Queensland, at the Ian Wark Research Institute, and at the Commonwealth Scientific and Industrial Research Organisation (CSIRO), have published 11 articles on the agglomeration of nickel laterite ores from 2011 to 2014. Following is a summary of their published findings, from the earliest to the latest:

- By increasing the water-to-sulfuric-acid ratio, Nosrati et al. (2011) observed faster nucleation and granule growth of nickel laterite ore particles crushed to less than 2 mm, forming agglomerates 5–40 mm in size. The faster growth of agglomerates was not necessarily ideal to achieve high compressive strength.
- Liu (2012) used a pellet press to agglomerate nickel laterite fines with water or sulfuric acid, and later allowed some to dry and others to remain moist. The pellets were stronger if agglomerated with water and if dried. In these experiments, the conditions tested were not typical of heap leaching.
- Liu et al. (2012) postulated that the agglomeration of nickel laterite ore begins with a kernel that subsequently grows by coalescence. The population balance model that was developed clearly predicted the agglomerate size distribution under the one condition tested.
- Nosrati et al. (2012a) found that an acidic solution of high concentration hindered the growth of agglomerates, producing smaller agglomerates.
- Nosrati et al. (2012b) used micro-tomography to reveal the agglomerate structure and pore volume.
- Addai-Mensah et al. (2013) found that the compressive strength of agglomerates was higher with the increasing ratio of fines to coarse particles. It took as long as 8–14 minutes in batch mode to agglomerate the ore into agglomerates 5–40 mm in size.
- Nosrati et al. (2013) hypothesized about the formation of solid bridges between particles agglomerated with acid. Solid bridges provide structural stability to the dry agglomerates, but on contact with an acidic solution, the acid-soluble bridges dissolve and the structural stability is greatly diminished.
- Quast et al. (2013) crushed nickel laterite ore to two different particle sizes (–38 µm and 2–15 mm) and agglomerated each batch with a sulfuric acid solution before leaching in columns. After 100 days of leaching, agglomerates made of –38 µm particles had leached only 10% more nickel than those made of coarser particles, and it is difficult to envision heaps made entirely of agglomerates made of –38 µm particles.
- Xu et al. (2013) recommended mixing goethitic laterite ore particles with clays during agglomeration and recommended allowing agglomerates to dry out moderately to accelerate leaching.
- Nosrati et al. (2014) measured the agglomerate properties using agglomerate size, compressive strength, three-dimensional microstructure analysis, and laboratory columns leaching tests. Of interest in this study were the effects of ore mineralogy and particle size on agglomeration.
- Quaicoe et al. (2014) performed the soak test (dipping agglomerates in a sulfuric acid solution) as a proxy for the compressive strength of the agglomerates, which were prepared by mixing ore particles smaller than 2 mm with 30 wt % sulfuric acid for 15 minutes. In this work, the authors compared the compressive strength of agglomerates made of saprolitic and geothitic ores. The latter had higher compressive strength.

Other works on the pretreatment of laterite ores are assigned to Guo et al. (2011) for their use of sodium carbonate (Na_2CO_3) roasting to break the mineral lattice and Janwong (2012) for his observation of two types of laterite agglomerates (layered and coalesced) using scanning electron microscopy and X-ray spectroscopy.

Other Commodities

There are few published works on the agglomeration of other types of oxide- or sulfide-bearing ores. Four articles refer to the agglomeration of uranium ores:

1. Scheffel (1982) reported on a 544,000-t heap stacked with agglomerates made of uranium ore mixed with 45–68 kg sulfuric acid/t ore. Some prior success had been obtained using Polyox WSR301 and sulfuric acid, but its addition was considered unnecessary.
2. Videau and Roche (1990) reported on the uniform leaching of a 1,000-t heap stacked with agglomerates made of uranium ore mixed with sodium silicate, concentrated sulfuric acid, and water. The success of the leach was attributed to the silicates added during agglomeration, as solution ponding and channeling were evident in another 1,000-t heap stacked with ore agglomerated without silicates.
3. Jianhua et al. (2004) used N_6O_2 and sulfuric acid to agglomerate uranium ore prior to acid leaching in columns.
4. Sinclair (2010) performed small and large column tests with agglomerated uranium ore.

Following are a few references for sulfide ores:

- Lastra and Chase (1984) used gypsum to agglomerate sulfide ores leached with acid.
- Perez et al. (1990) agglomerated with a sodium or calcium hypochlorite solution and cement to destroy, modify, or passivate sulfide minerals. After curing, the agglomerates were rinsed with water and then cyanide-leached.

Ahmadiantehrani et al. (1991) also used hypochlorite and cement to agglomerate sulfidic gold ores and achieved 80% gold recovery.

- Misra et al. (1996) found the combination of fly ash and cement to be superior to either material alone with regard to the strength of agglomerates made of pyrite tailings.
- According to the sulfide bioheap model developed at the University of British Columbia, sulfide heaps can also benefit from agglomerating the ore with the leaching solution (Bouffard 2003).
- Nalco Holding Company has developed polymeric binders (9704, CX-2131, CX-2134, CX-2185, CX-2194, 98DF108, and 97DF125) that would not inhibit the microorganisms present and would not be consumed by microorganisms (Nalco Water 2017).
- Saririchi et al. (2012) tested the bioleaching in small columns of low-grade zinc sulfide ores agglomerated with an inoculum.

CONCLUSION

Agglomeration for gold and silver ores is a mature process with no recent significant development. For copper, researchers have concluded that there is a need for an agglomeration binder to be added to the sulfuric acid solution; research in the last decade has focused on polymeric binders. For nickel, many recent articles have been published, limited to laboratory tests under conditions, which are at times atypical of heap leach environments.

While agglomeration was a breakthrough technology for heap leaching gold and silver ores of high fines and clay content, the benefit of agglomeration for copper ores is not as well substantiated. The slow (>300 days), difficult-to-control, and ever larger heap may overwrite the imperfections of agglomerates. The benefits of increased recovery and shorter leach cycles must be weighed against the 5%–10% extra capital costs for the agglomeration equipment and instrumentation, as well as the higher operating costs for labor, energy, and agglomeration medium and binder. In-line instrumentation is displacing the rather subjective manual methods for measuring agglomerate moisture content and size distribution, but no in-line method is yet available for measuring agglomerate strength. Agglomeration may also require adapting the crushing and screening processes to produce the right ore size distribution, configuring stacking equipment to place the agglomerates more gently, and modifying irrigation practices to distribute the leaching solution more evenly.

With the broader adoption of high-pressure grinding rolls (HPGRs), one must question whether this technology could be a substitute to agglomeration for non-clayey ores, delivering a similar gain in recovery without the additional cost of agglomeration, and whether the combination of agglomeration and HPGR, known to generate large amounts of fines, could deliver greater recovery than would agglomeration alone.

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