CHAPTER 10.6

Pressure Leaching and Oxidation

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Pressure leaching and pressure oxidation are hydrometallurgical processes applied to the extraction of many metals. The processes occur above atmospheric boiling temperature, require a sealed reactor vessel, and often operate in highly corrosive and oxidizing environments. Pressure leaching and pressure oxidation have been applied to a broad range of processes over a wide range of conditions and include similar features such as

- Slurry feed: Chemical conditioning, preheat, and high-pressure pumps;
- Reactor vessel: Digester and leach or oxidation autoclave;
- Pressure letdown: Flash vessel;
- Off-gas handling: Atmospheric condenser and scrubber; and
- Ancillary systems: Acid, oxygen, air, steam, and water injection systems, and an agitator seal system.

Pressure leaching is primarily applied when the goal is to solubilize desirable elements, recover the solution fraction, and reject the residual solids. The process can operate under alkaline or acidic conditions and may include the use of oxygen gas (or an alternative oxidant such as hydrogen peroxide) to oxidize and leach base metal sulfide minerals. The most common alkaline pressure leach processes are applied in the extraction of alumina (e.g., Bayer process), tungsten, uranium, and rare earth elements, as well as nickel, cobalt, and copper sulfide minerals. The most common acidic processes are applied in the extraction of platinum group metals (PGMs), nickel–cobalt laterites, and uranium, as well as copper, nickel, cobalt, molybdenum, and zinc sulfides. Acidic pressure leaching of iron from an ilmenite slag to produce synthetic rutile is a process in which the solid fraction contains the product and the impurities leached into solution are rejected (solution is recycled).

Pressure oxidation is applied when the goal is to decompose sulfide minerals for the recovery of desirable elements from the solids fraction and the rejection of the solution fraction. Pressure oxidation is mostly applied to refractory gold-bearing ore or concentrates where the gold is locked in the sulfide minerals and recovery using conventional cyanide leaching is not effective. The process is mostly operated under acidic conditions; however, alkaline pressure oxidation is also practiced.

Variations of pressure leaching and pressure oxidation processes have been developed over time and have become known by different terms. For the sake of simplicity and clarity, the aforementioned fundamental process definitions have been applied consistently in this chapter unless otherwise identified.

This chapter provides a review of pressure leaching and pressure oxidation unit process systems, including discussions of process design, metallurgical test work, key economic drivers for operating and capital costs, and typical practices for equipment selection and design. Readers are directed to the respective commodity sections elsewhere in this handbook for more detailed discussions of process metallurgy fundamentals.

HISTORICAL DEVELOPMENT

Pressure leaching and pressure oxidation processes have developed sporadically over the last 120 years, driven by mineralogical complexity, fluctuation in metal demand, commercial opportunity, and the technological progression of process equipment. These key drivers continue to influence development and application of new technologies in the industry today.

Pressurized extractive metallurgy began with the alkaline leaching of alumina from bauxite ores using the Bayer process, named after its inventor, Karl Josef Bayer. The process, which was patented in 1888 and first applied in St. Petersburg, Russia, in 1892, continues to be applied in aluminum production to this day (Habashi 1995).

Many leaching and recovery processes were postulated at the turn of the 20th century (Habashi 2004). Those that progressed into commercial application at the time were supported

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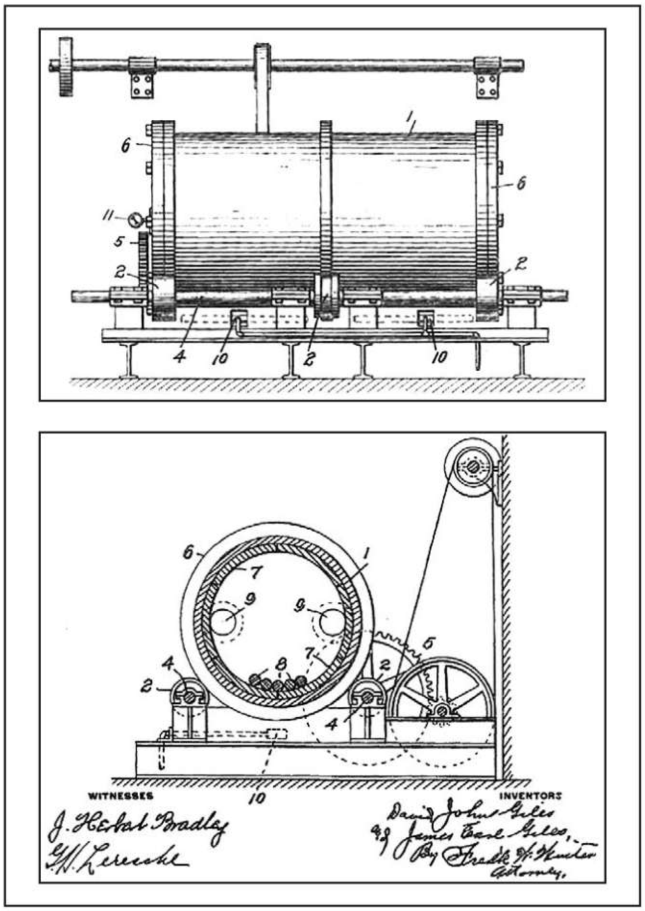
by testing and understanding of the process chemistry for the complete extraction and recovery process, the economical availability of reagents, and suitable process equipment technology. The 1919 U.S. patent for the alkaline digestion and extraction of tungsten (Giles and Giles 1919) provides insight into the pressure leaching equipment applied at the time. As shown in Figure 1, the patent illustrates the use of batchwise process equipment that combines pressure leaching and grinding to liberate minerals and promote the alkaline leaching of tungsten minerals.

The mid-20th century was a period when pressure leaching technology expanded to a range of metals. This century also witnessed the development of high-capacity process equipment. Pressure leaching was applied to alkaline and acidic uranium extraction (Fraser and Thomas 2010); the extraction of cobalt from an arsenic cobalt sulfide concentrate in acidic oxidizing conditions (Mitchell 1956); the extraction of cobalt, nickel, and copper from a mixed sulfide concentrate in alkaline conditions using ammonia; and the acidic pressure leaching of nickel–cobalt laterites (Chalkley et al. 2004). The extraction of tungsten also evolved to produce a high-purity ammonium paratungstate for industrial consumption (Kurtak 1998).

Pressure hydrometallurgy was first extended to uranium at Eldorado Nuclear's Beaverlodge Mill (Saskatchewan, Canada) in 1953 (Fraser and Thomas 2010). The oxidative process included a 96-hour alkali–carbonate Pachuca leach circuit consisting of twenty-four 255-m^3 vessels sparged with steam for temperature control. Pachuca technology was later extended to the first application of pressure hydrometallurgy for nickel laterite at Moa Bay, Cuba, which included five trains of four vessels 3 m in diameter and 15 m tall, agitated by the injection of superheated steam to an internal draft tube (Mason and Gulyas 1999).

Importantly, during the mid-20th century, the technology for pressure leach equipment evolved from small-scale batch processes to the use of continuous multicompartment horizontal autoclaves. This evolution of autoclave technology supported an increase in treatment rate and improvement in economies of scale. One of the first horizontal autoclaves was introduced for pressure oxidative leaching of an arsenic-rich cobalt sulfide concentrate at the Garfield Refinery in Utah (United States). The process, designed to operate at 190°C and 3,400 kPa, experienced many corrosion and erosion challenges during the first few years of operation (Mitchell 1956). The challenges and subsequent development resulted in the introduction of specialized materials, including titanium for agitator components and slurry discharge and ceramics for pressure letdown control. Undoubtedly, the lessons learned from the Garfield Refinery and subsequent commercial production of titanium and ceramic components supported the design of pressure oxidation and pressure leach circuits in the years to follow.

After many years of testing and development, pressure leaching was applied to the extraction of zinc from sulfide concentrates. The first commercial zinc pressure leach plant was introduced in 1981 as an expansion of the Cominco Trail operations in Canada (Weir 1985) and was later introduced to other plants in Canada and Germany. The process, operating at 150°C, was the first to target partial oxidation of sulfide to elemental sulfur (instead of complete oxidation to sulfate) and to overcome challenges with the formation and agglomeration of molten elemental sulfur in the autoclave. The goal of promoting elemental sulfur formation rather than sulfate is to lower the operating costs associated with oxygen consumption and acid neutralization while still achieving high zinc extraction.



Source: Giles and Giles 1919

Figure 1 Historical tungsten extraction "grinding" autoclave

In March 1985, gold was produced from the McLaughlin pressure oxidation plant in California (United States). The project was the first to pretreat sulfidic refractory gold ore and built upon lessons learned from Anglo American Corporation's 1977–1981 Western Deeps/Vaal Reefs uranium pressure leach demonstration plant in South Africa. The São Bento pressure oxidation plant in Brazil followed in 1986, and another seven operations were constructed between 1986 and 1993, including the Goldstrike and Twin Creeks operations in Nevada (United States), the Campbell mine in Canada, and the Porgera mine in Papua New Guinea. The first commercial alkaline pressure oxidation plant, Mercur (Utah), was commissioned in 1988 (St. Louis and Edgecombe 1990). Subsequent refractory gold projects have included the Lihir operation in Papua New Guinea, Macraes in New Zealand, and the Pueblo Viejo operation in the Dominican Republic.

The 1990s witnessed rapid development in the acidic pressure leaching of nickel and cobalt from laterites, with a series of plants constructed in Western Australia. The projects were initiated as a result of an increase in nickel and cobalt prices; advantages in project location; acid availability; low mining complexity; and advances in the design of solvent extraction, electrowinning, thickening, and autoclave processes (Kyle 1996). Although aspects from early pressure leach plants were incorporated into the designs, the project start-ups were prolonged and had many technical challenges (Nice 2004). Of the three projects constructed—Bulong, Cawse, and Murrin Murrin—only Murrin Murrin has continued to operate and achieve nameplate throughput. Lessons learned from these first-generation projects have since supported the development of a second generation of laterite projects, including Coral Bay in the Philippines; Ravensthorpe in Western Australia; and Goro Nickel in New Caledonia; and, more recently, a third generation, which includes the Ambatovy project in Madagascar and Ramu in Papua New Guinea.

In the mid- to late 1990s, various interests turned to the use of pressure leaching of copper concentrates as an alternative to conventional smelting (King et al. 1993; Defreyne et al. 2006; Marsden et al. 2007). The CESL (Cominco Engineering Services Ltd.) process was one of the first methods developed and progressed to a demonstration plant (Defreyne et al. 2008). The process, which was developed by operators of the first zinc leach plant at Trail, applies similar process conditions to the zinc pressure leach, but with the addition of chloride to promote copper sulfide oxidation and elemental sulfur formation.

Medium- and high-temperature pressure leaching of copper concentrate was also under development in the United States around the same time. The processes, which are well documented by Marsden et al. (2007), include fine grinding of copper concentrate to promote oxidation to elemental sulfur under medium-temperature conditions and direct electrowinning of copper from the leach solution. Under high-temperature conditions, the oxidation reaction progresses to sulfate and sulfuric acid, and the acid can be recovered for use elsewhere in the process (e.g., heap leach operations, as in the case for the demonstration plant at the Bagdad mine and the commercial operation at the Morenci mine in Arizona, United States).

The expansion of pressure leaching and oxidation from the 1950s and the associated development in process equipment have firmly cemented the processes as mature metallurgical technologies. Many of the challenges encountered during the evolution of the processes have led to new techniques or processes that provide the modern metallurgical engineer with extensive research data and project references to work with. Key developments in pressure leaching and pressure oxidation are illustrated in Figure 2.

APPLICATION AND SELECTION

The selection of pressure leaching or pressure oxidation for the recovery of metals depends on many factors particular to the material to be treated (e.g., mineralogy and grade, deposit size, geographic location). In some cases, the selection may be subject to evaluation against other treatment technologies. Pressure leaching and oxidation processes maintain the key advantages of occurring in aqueous process conditions, which often have advantages for metal recovery and environmental emissions control while providing fast reaction rates. These advantages become more apparent when the material or ore source has specific challenges and other processes, such as atmospheric leaching, are not as effective or appropriate.

For example, the increase in the oxidation kinetics associated with the pressure oxidation of gold-bearing refractory sulfides provides a significant advantage over biological oxidation for high-sulfide throughput. A typical metric used for biological oxidation is 7 kg of sulfide sulfur oxidized per cubic meter of reactor process volume per day (7 kg $S^{2-}/m^3/d$), whereas a typical metric for pressure oxidation is 700 kg $S^{2-}/m^3/d$. Similarly, biological oxidation typically occurs over 4 to 5 days (96–120 hours), whereas a 60- to 90-minute retention time is typical for pressure oxidation. This order-of-magnitude unit capacity difference provides an advantage in equipment size and plant layout for pressure oxidation when a comparatively high-sulfide-sulfur throughput is required. An early economic assessment of applying pressure oxidation for the pretreatment of a sulfidic refractory gold ore body is described in the study by Singh et al. (2013).

Select commercial pressure leach and oxidation process applications are summarized in Table 1 and illustrated relative to a temperature–pressure-saturated steam curve in Figure 3. In general, the processes can be categorized into low to medium temperature (130°–160°C) and high temperature (200°–260°C). In low- to medium-temperature conditions, the saturated steam pressure is less than 1,000 kPa(g), whereas at high-temperature conditions, the pressure is significantly higher at 2,000–4,500 kPa(g). The high-temperature processes require special consideration to the circuit design and materials of construction, especially those operating in oxidizing or acidic process conditions.

A key differentiator between sulfide and nonsulfide processes is the energy balance. The oxidation of sulfide sulfur is exothermic (i.e., heat-generating); therefore, the pressure oxidation process does not require energy input as long as the feed grade is sufficiently high (approximately >5 wt % sulfide sulfur). The process is said to be autothermal when no heat energy input is required to sustain the operation. Nonsulfide leaching processes often require significant heat energy input to sustain the target operating temperature. Samples of pressure leaching and pressure oxidation chemical reactions and heats of reaction are provided in Table 2 (Outotec 2016).

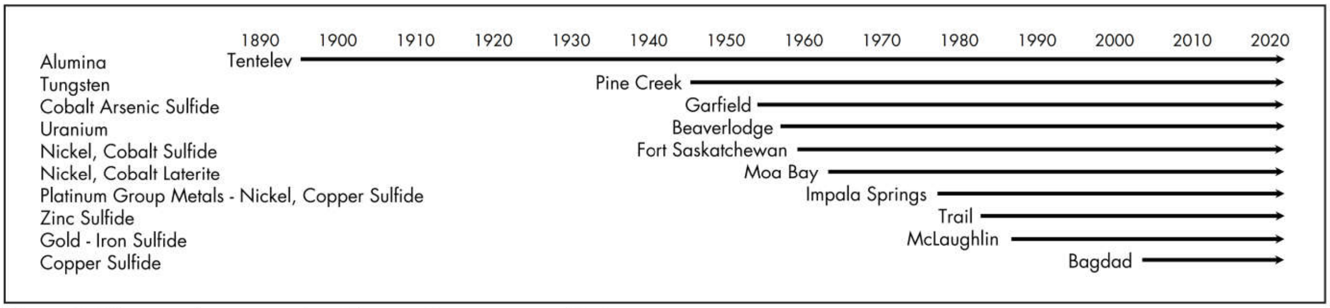


Figure 2 History of pressure leaching and pressure oxidation

Table 1 Pressure leach and oxidation process comparison

Aspect	Pressure Leach									Pressure Oxidation
Application	Aluminum (Bayer)	Tungsten	Nickel, cobalt, copper (sulfide)	Uranium	Nickel–cobalt (laterites)	Zinc (sulfides)	Copper/molybdenum (sulfides)	Platinum group metals	Titanium	Gold/silver (refractory in sulfides)
Process objective	Solubilization	Solubilization	Solubilization	Solubilization	Solubilization	Solubilization	Solubilization	Solubilization/ solids enrichment	Solids enrichment	Liberation
Feed material	Ore (bauxite)	Flotation concentrate	Flotation concentrate	Ore	Ore (upgraded)	Flotation concentrate	Flotation concentrate	Matte	Slag	Ore/flotation concentrate
Common minerals	Gibbsite, boehmite	Scheelite, wolframite	Pentlandite	Uraninite, brannerite, coffinite	Goethite, garnierite, asbolite	Sphalerite	Chalcopyrite/ molybdenite	Digenite, covellite, polydymite, millerite	Ilmenite	Pyrite, arsenopyrite, realgar, orpiment
pH	Alkaline	Alkaline	Alkaline	Alkaline/acid	Acid	Acid	Acid	Acid	Acid	Acid/alkaline
Operating temperature, °C	140–270	175–250	110–120	70–180	230–270	130–150	140–225	140	145–165	190–230
Operating pressure, kPa	400–5,500	1,000–4,000	800–900	300–1,700	3,000–5,700	1,100–1,400	1,000–3,200	600	350–500	1,600–3,350
Reagent	Sodium hydroxide	Sodium carbonate/ sodium hydroxide	Ammonia	Sodium hydroxide/sulfuric air/oxygen	Sulfuric acid	Oxygen	Oxygen/acid	Oxygen	Hydrochloric acid	Oxygen
Vessel configuration	Multiple vertical	Vertical/ horizontal	Horizontal	Multiple vertical/ horizontal	Horizontal	Horizontal	Horizontal	Horizontal	Vertical	Horizontal
Batch/ continuous	Continuous	Batch/ continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Batch	Continuous
Heat recovery	3 to 12 Stages	—	1 Stage	0 to 3 Stages	3 to 4 Stages	—	—	—	—	0 to 3 Stages
Project examples	Pinjarra, Australia		Fort Saskatchewan, Canada	Key Lake, Canada	Murrin Murrin, Australia	Trail, Canada	Morenci, United States	Impala Platinum, South Africa	Quebec Iron and Titanium, Canada	Goldstrike, United States
	Alumar, Belem, Brazil	Pine Creek, United States	Kwinana, Australia	McClean Lake, Canada	Ravensthorpe, Australia Goro Nickel, New Caledonia	Kidd Creek, Canada	Bagdad, United States	Lonmin, South Africa		Twin Creeks, United States
	Weiqiao, China			Dominion Uranium, South Africa	Ambatovy, Madagascar	Ruhr Zinc, Germany				Pueblo Viejo, Dominican Republic

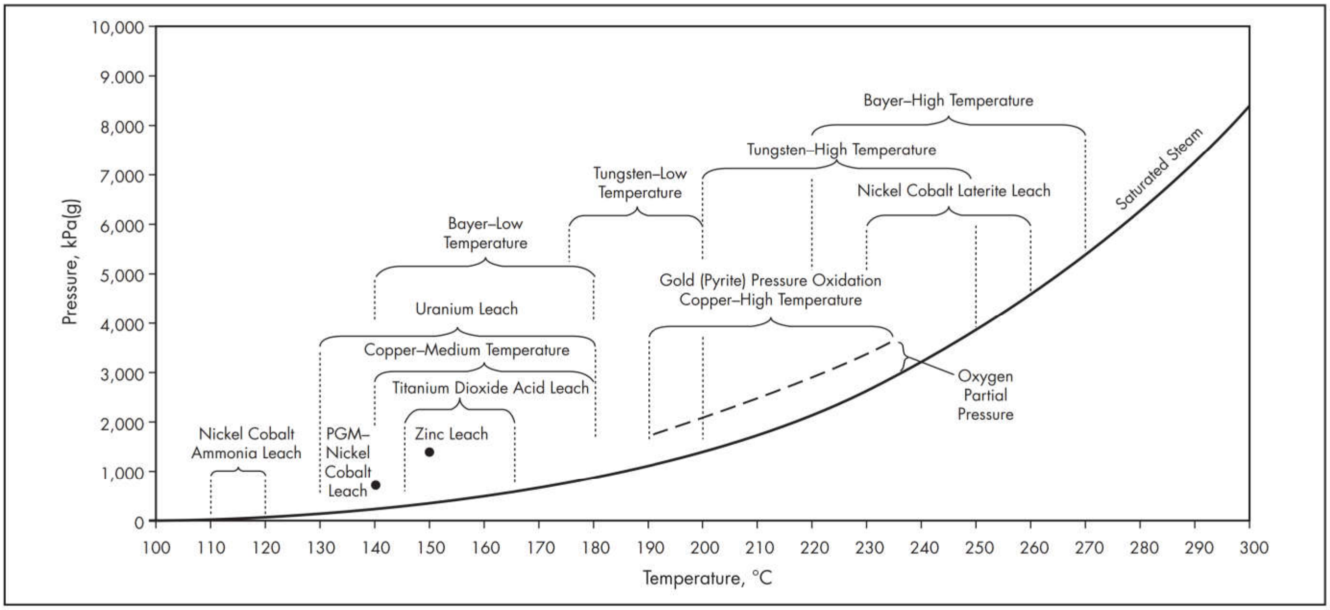


Figure 3 Pressure leach and pressure oxidation pressure–temperature conditions

Table 2 Pressure leach and pressure oxidation heats of reaction

Mineral	Chemical Formula	Heat of Reaction, mJ/kg Mineral
Uraninite (acid)	$UO_2 + Fe_2(SO_4)_3 \rightarrow UO_2SO_4 + 2FeSO_4$	–0.5
Beohmite (Bayer)	$AlO(OH) + NaOH + H_2O \rightarrow NaAl(OH)_4$	–0.3
Wolframite	$(Fe, Mn)WO_4 + 2NaOH \rightarrow NaWO_4 + (Fe, Mn)(OH)_2$	–0.1
Uraninite (alkaline)	$UO_3 + Na_2CO_3 + H_2O \rightarrow Na_2UO_2(CO_3)_3 + 2NaOH$	0.0
Goethite (Ni bearing)	$Ni(OH)_2 + H_2SO_4 \rightarrow NiSO_4 + 2H_2O$	1.0
Geothite (Co bearing)	$Co(OH)_2 + H_2SO_4 \rightarrow CoSO_4 + 2H_2O$	2.0
Goethite (leach)	$2FeOOH + 3H_2SO_4 \rightarrow Fe_2(SO_4)_3 + 4H_2O$	2.3
Arsenopyrite	$2FeAsS + 7O_2 + 2H_2O \rightarrow 2FeAsO_4 + 2H_2SO_4$	8.9
Sphalerite	$ZnS + O_2 \rightarrow ZnSO_4$	8.9
Chalcopyrite	$CuFeS_2 + 4O_2 \rightarrow CuSO_4 + FeSO_4$	9.0
Metal sulfide (NH_3 leach)	$MS + 20_2 + 4NH_3 \rightarrow M(NH_3)_2 + (NH_3)_2SO_4$ (M = Ni, Co, Cu)	10.0 (Ni)
Pyrite	$2FeS_2 + 7O_2 + 2H_2O \rightarrow 2FeSO_4 + 2H_2SO_4$	12.1

Source: Outotec 2016

The need for heat input to the process strongly influences the application of heat recovery and recycle from the reactor discharge to the reactor feed. Nonsulfide pressure leach processes that require significant heat input use multiple stages of pressure letdown and heat transfer to maximize energy recovery. Acid leach processes may also receive 30°–40°C of supplementary heating from acid dilution. Pressure leach or whole-ore pressure oxidation projects with low sulfide sulfur grades (3.0%–4.5%) typically include two stages of letdown and heat recovery and the ability to directly add steam to the reactor to maintain the operating temperature. Ores with sulfide sulfur grades between 4.5% and 6% typically include one stage of letdown and heat recovery to ensure that autothermal operation is maintained. The Mercur alkaline process was designed for 1.7% sulfide sulfur and uses three stages of preheating to achieve an operating temperature of 225°C (Mason 1990). Whole-ore or concentrate feeds with sulfide sulfur feed grades greater than 6% usually have excess heat, requiring energy dissipation instead of energy input.

General flow-sheet configurations for the Bayer pressure leach process, the nickel laterite pressure leach process, and pressure oxidation are shown in Figures 4–6. Key points of variation between the three example flow sheets include the following:

- **Bayer leach:** Vertical plug flow digesters; extensive multistage letdown and indirect-contact heat recovery system
- **Nickel laterite leach:** Horizontal multicompartment autoclave with mixing agitators and steam, acid, and air addition; multistage letdown and direct-contact heat recovery system
- **Pressure oxidation:** Horizontal multicompartment autoclave with gas dispersion agitators and oxygen and cooling water addition; one stage of letdown and potential direct-contact heat recovery to allow operation at lower sulfide sulfur grades

The selected process conditions for pressure leach or pressure oxidation processes (e.g., operating temperature, pressure, retention time, and reagent addition) are highly

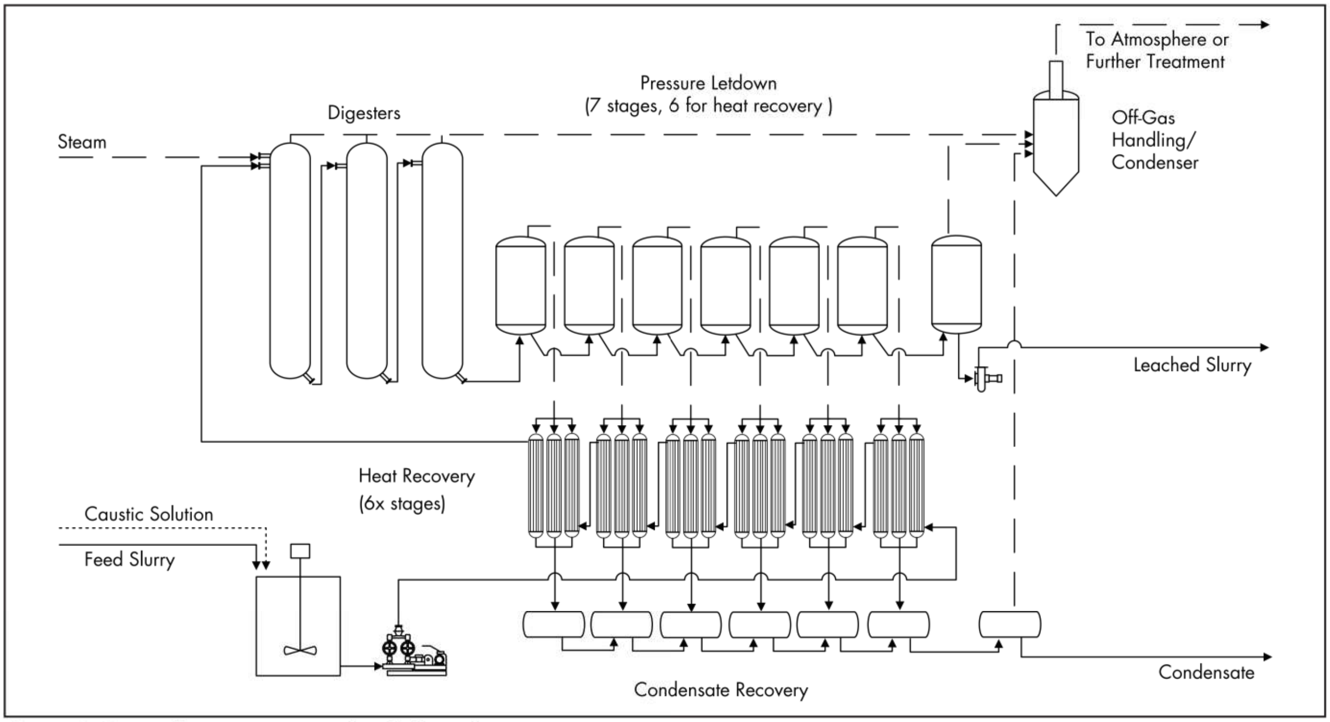


Figure 4 General Bayer pressure leach flow sheet

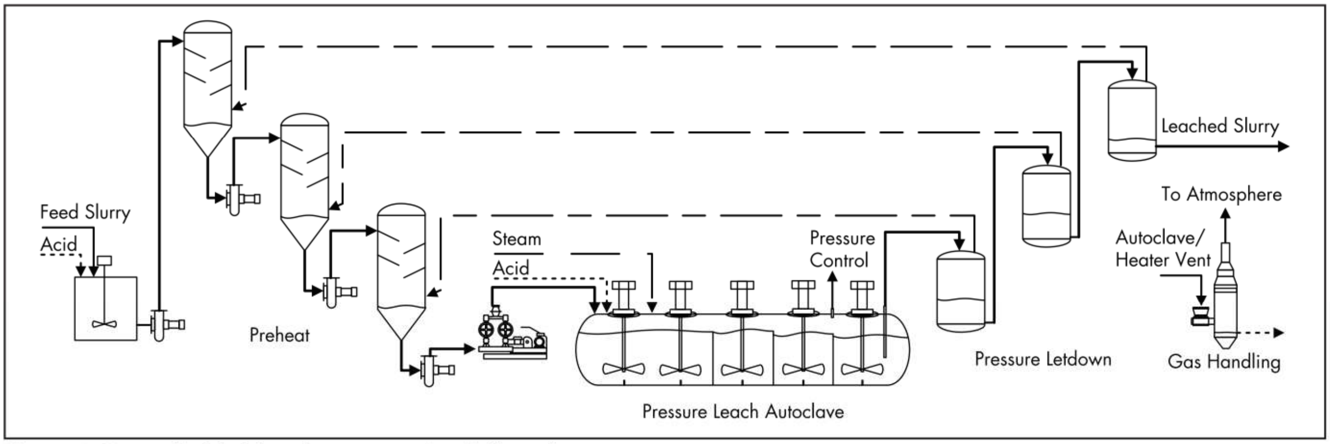


Figure 5 General nickel laterite pressure leach flow sheet

dependent on the specific mineralogical composition of the ore or concentrate and the goal of the process. As a result, the pressure leach or oxidation circuit is designed specifically for a type of feed material at an economical mass throughput. The treatment of substantially different feed material or throughput is often not possible without significant modification to the circuit.

Where applicable, concentrating the ore using flotation may be an effective way to improve the process so that additional equipment or continuous energy addition may not be required. The benefit of concentrating an ore is dependent on the sulfide sulfur and pay metal recovery, the achievable concentrate sulfide sulfur grade, and the rejection of undesirable minerals that affect the process (e.g., carbonates). Concentration is often selected under the belief that the autoclave size will be smaller due to the decrease in the mass to be processed; however, this is often not the case, as the autoclave size is more dependent on sulfide sulfur throughput than mass throughput. A comparison of whole-ore and concentrate refractory gold pressure oxidation facility configurations is provided in Table 3. Some pressure oxidation circuits actively treat a combination of whole ore and concentrate to achieve autothermal operation or to maximize pay metal production.

An assessment of process conditions and the potential benefit of alternate circuit configurations, such as mineral concentration, are important to process flow-sheet development. Completing the necessary supporting metallurgical test work is also a critical phase of project development.

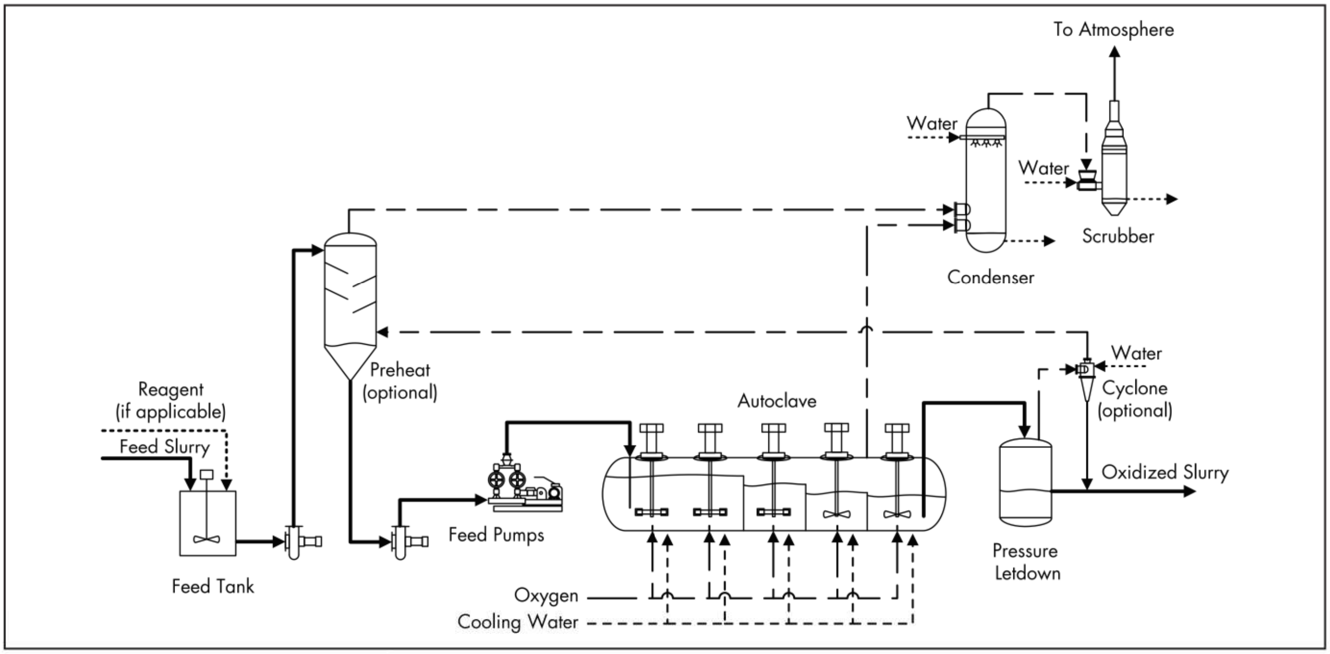


Figure 6 General pressure oxidation flow sheet

Table 3 Refractory gold pressure oxidation facility process configurations

Whole Ore	Concentrate
Getchell	Amursk
Goldstrike	Campbell
Lihir*	Con mine
Lone Tree	Kittila
McLaughlin	Macraes
Mercur	Porgera
Pueblo Viejo	São Bento
Twin Creeks*	

*Operations that treat whole ore and concentrate.

Metallurgical Test Work and Process Flow-Sheet Development

The success of a commercial operation is directly affected by the degree of test work performed during its development, and this is especially true for more complex high-pressure processes (McNulty 1998). Therefore, the correct balance must be obtained between the extent of test work performed and the risk tolerance in reaching commercial design capacity in a timely and cost-effective manner following commissioning. The goals of a test-work program should be to

- Define feed characteristics, such as ore mineralogy, including potential variability;
- Confirm process parameters and operating conditions;
- Understand the complete process chemistry, including materials of construction requirements;
- Pilot-test any process operations with higher degrees of risk/unknowns; and/or
- Establish criteria for engineering design and equipment sizing.

Table 4 Sample economic comparison of test program impacts

Description	Limited Test Program	Extensive Test Program
Total process test-work cost	\$1 Million	\$5 Million
Process commissioning delay	0 Months	3 Months
Process capacity after 36 months	81%	94%
Revenue at 100% process capacity*	\$10 Million per month	
Net present value of first 36 months†	\$85 Million	\$85 Million

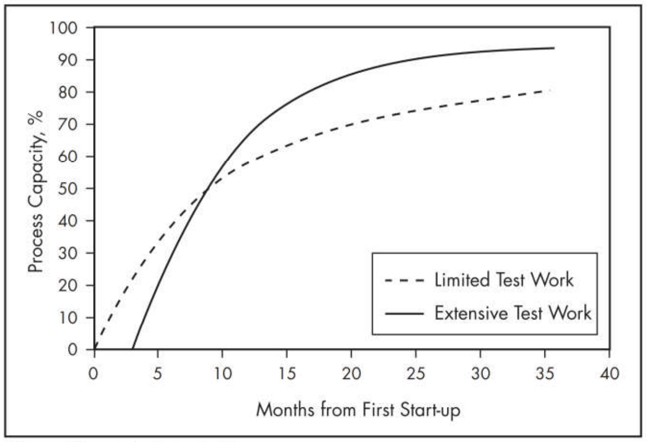
*Revenue is assumed to scale proportionally to process capacity.
†Includes cost of test work and impact of commissioning delay at 5% discount rate.

For process design, it is imperative that test work be performed on samples that are representative of the overall material. This is particularly important to an economic assessment of the process in terms of operating costs and engineering design requirements. When a high degree of variability is expected in the feed, additional test work may be required to establish appropriate design boundaries. The presence of potentially troublesome components (e.g., As, Cl, CO_3, F, Hg, Sb) should be determined in the initial analysis. If present, the test program should then be expanded to assess their impact.

An example of balancing test work with the timeliness of implementation is provided in Table 4. The example is based on an analysis of plant data for complex processes with different degrees of test work and the process start-up production curves provided in Figure 7 (McNulty 1998). The example shows that additional test work at the incremental cost of \$4 million with a 3-month delay has a negligible bearing on the economics for the first 36 months of operation, and that additional test work establishes better performance (and greater value) for the following years. Increased unit operating costs that would be expected at lower production capacities have been ignored, which would further favor the extensive test program scenario.

Test work is performed batchwise and continuously (pilot), depending on a project's stage of development. Batch work is typically performed in 2-L vessels to initially indicate whether the material is amenable to the proposed process and can be used as a comparison to alternative processes. Further batch tests can later be performed to establish operating conditions and assess ore variability to form a basis for scoping or prefeasibility studies. Typically, mass and energy balances are carried out with the tests to support the understanding of process chemistry and process cooling or heating requirements.

Continuous pilot tests support feasibility studies with a greater level of detail and provide proof-of-concept assurance to the previously completed batch work. Shorter duration campaigns (e.g., 12–24 hours, compared to 60–100 hours for a full feasibility campaign) may be completed during the prefeasibility stages of a project to validate process configurations. Continuous pilot tests represent commercial operation more closely than batch tests, provide more detailed inputs to engineering design (e.g., scale formation, corrosion, off-gas composition), and demonstrate the impact of recycle streams within the process. Continuous test work (and expected commercial operation performance) can differ in performance from batch test work (e.g., required retention time, process chemistry, extraction extents) and is therefore critical to a project's success. Pilot autoclaves usually operate at a capacity of 20–70 L, resulting in a scale-up ratio for commercial operations ranging between 8,000 and 20,000 (Adams et al. 2004).



Adapted from McNulty 1998

Figure 7 Sample comparison of test program impact on start-up process capacity

A general process flow for test work is demonstrated in Figure 8. A basic sample test program specific to a pressure oxidation process is provided in Table 5, which excludes all feed analysis (e.g., mineralogy), downstream processing (e.g., solid–liquid separation), or supplementary work (e.g., environmental). Material estimates are provided on a whole-ore basis; that is, the total mass required would be significantly greater if a concentrate is required for the test program (e.g., a concentrate with a mass recovery of 20% would require 5 times the mass input to the program). The sample pilot program only considers a single ore feed and does not include additional run time for campaigns of differing feed material. The overall costs are indicative for a similar test program for an acidic pressure leach or pressure oxidative leach process. The total cost would be higher for additional feed material campaigns and variability testing or for testing various process configurations.

EQUIPMENT SIZING, SELECTION, AND MATERIALS OF CONSTRUCTION

An early understanding of the overall plant throughput and the key drivers determining the selected throughput needs to be developed from the perspective of the leach or oxidation process. It is not uncommon during early project development for mine planning to limit its focus to the pay metals and neglect the variability of minerals that may affect the leaching or oxidation process. For example, the mine and ore delivery plan for a refractory gold deposit may be initially optimized to gold production and neglect the impact on the sulfide sulfur feedrate variability to the pressure oxidation circuit, potentially resulting in an incorrectly sized autoclave vessel and oxygen plant. Depending on the complexity of the ore and the number of pay metals, it is typical for the mine plan, ore blending, and delivery strategy, as well as the plant production profile, to undergo several iterations before the optimum throughput is selected. At this stage, attention should be paid to understanding the ore mineralogy, metal deportment, and gangue mineralogy that may interfere with the pressure leach or pressure oxidation process.

In many cases, the pressure leach or oxidation circuit and its ancillary equipment are the most capital-intensive process areas in the plant, and maximizing their utilization is important to optimizing operating economics. To support this philosophy, it is prudent to ensure that the upstream ore preparation circuit is designed to maintain the required feed to the leach or

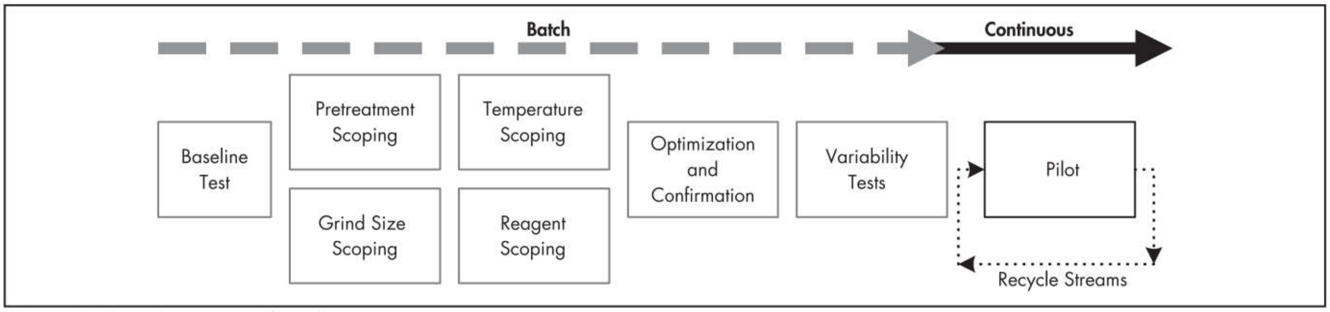


Figure 8 Sample process flow for test program

Table 5 Sample pressure oxidation test-work program

Description	Quantity	Total Mass Required	Total Approximate Cost*, US$
Batch autoclave tests, 2 L			
Baseline gold recovery test	2	1 kg	1,000–3,000
Process conditions scoping (four variables at three levels)	4 × 3 = 12	6 kg	30,000–40,000
Process conditions optimization (two variables at three levels)	2 × 3 = 6	3 kg	15,000–25,000
Ore variability tests	12	6 kg	30,000–40,000
Gold recovery (one per test)	30	NA†	25,000–35,000
Batch test total	**–**	**16 kg**	**100,000–150,000**
Continuous autoclave pilot, 72 hours		5–15 kg/h‡	
Setup, management, takedown	1	–	55,000–65,000
Operator labor	72 hours	–	110,000–140,000
Shift (12-hour) assays (feed and discharge for seven shifts)	7 × 2 = 14	–	5,000–15,000
Autoclave profile assays (8 six-compartment assays)	8 × 6 = 48	–	25,000–35,000
Gold recovery (one per assay)	14 + 48 = 62	–	60,000–90,000
Discharge mineralogy (X-ray diffraction, QEMSCAN)	2	–	5,000–15,000
Corrosion testing	1	–	5,000–15,000
Scale characterization	1	–	3,000–6,000
Gas sampling	1	–	10,000–20,000
Continuous autoclave pilot total		**350–1,000 kg‡**	**275,000–400,000**
Total for pressure oxidation test-work program		**355–1,015 kg‡**	**375,000–550,000**

*Quarter 4, 2015.
†NA = Not applicable; tests performed on material allocated to other tests.
‡Mass dependent on ore sulfur content, apparatus oxidation capacity, and retention time.

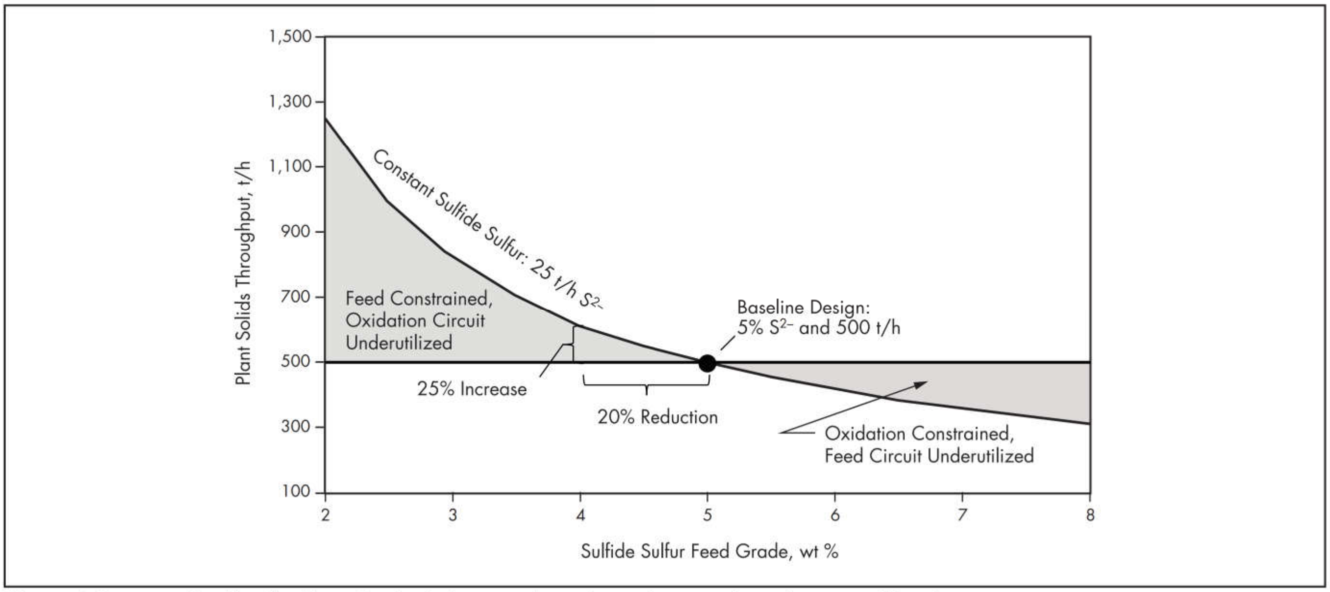


Figure 9 Impact of sulfur feed grade deviation on plant throughput and equipment utilization

oxidation circuit, with consideration of the anticipated range in feed grades, ore hardness, or other physical characteristics. For example, the capacity of a sulfide flotation concentrator for a pressure oxidation circuit should be determined by the lowest anticipated sulfide feed grade and sulfide recovery to ensure that the sulfide sulfur throughput is maintained to the pressure leach or oxidation circuit. In this way, by design, the plant should mostly be limited by the pressure leach circuit and not by the upstream ore preparation circuit. The design of a concentrator for a pressure leach circuit may be constrained by other factors, including transport or pregnant solution concentrations; however, a similar philosophy should be considered where applicable.

Figure 9 demonstrates the required concentrator throughput for changes in the sulfide sulfur feed grade from a baseline case. As shown in the figure, a 20% decrease in the sulfide sulfur feed grade from the baseline case of 5 wt % sulfide sulfur at 500 t/h requires a 25% increase in the ore preparation circuit capacity to maintain a constant feed sulfide tonnage to a pressure oxidation circuit. The increase is independent of any design requirements associated with variation in the feed, such as hardness. Sizing the ore preparation circuit from the perspective of the pressure leach or oxidation circuit often results in a revision of the ore stockpiling and plant feed blending strategy to minimize sulfide feed grade variation and the potential need for a larger circuit.

Feed Systems—Heating

Heat recovery is essential to the operation of a process in which the net exothermic heat of reaction is insufficient to raise the feed slurry temperature to the desired operating temperature. In pressure leaching operations in which the net heat of reaction is endothermic, efficient recovery of heat from the autoclave discharge stream is essential to minimize the usage of boiler steam. In pressure oxidation circuits in which the autoclave feed sulfide sulfur grade is insufficient to achieve the desired operating temperature, the operating temperature can be achieved by using single- or multistage heat recovery stages to preheat the feed slurry. The exact sulfide grade required to achieve a selected operating temperature is dependent on several factors, including mineralogy, feed density, oxidation reaction rate, retention time in the first autoclave compartment, rate of gas vent from the autoclave, and heat loss from the vessel (Mason 1990). An example is provided in Figure 10, where the autothermal sulfide grade is lowered for each additional stage of preheating (i.e., higher feed slurry temperature).

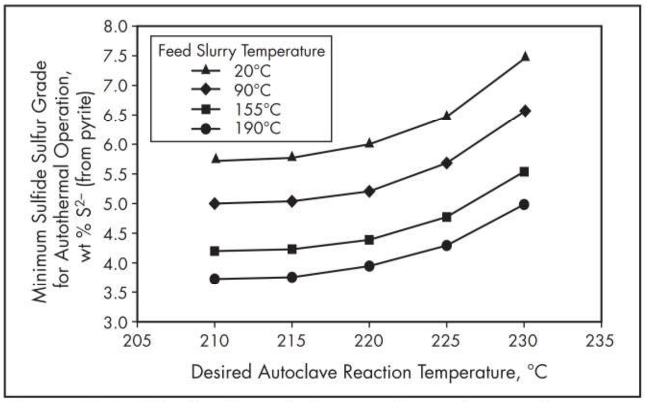


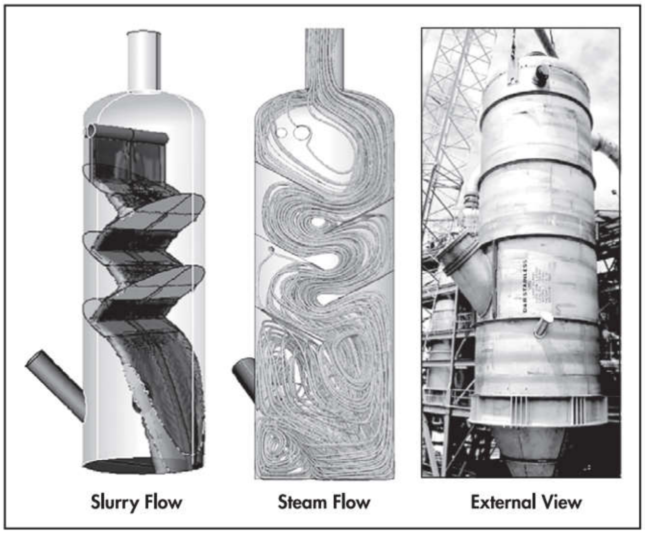
Figure 10 Sulfide feed grade for autothermal operation

As of 2017, feed slurry pumping temperature is limited by the feed pump check valve elastomers to approximately 200°C. For applications in which the incoming slurry temperature is higher than the elastomeric material's rated working temperature (e.g., after preheating), water-cooled "dead legs" are used to protect the elastomeric materials. The water-cooled slurry legs allow the diaphragms to displace hotter process slurry at a distance, thus allowing for a higher operating temperature. The temperature of steam to the first stage of preheating aligns with the coincident pressure letdown vessel from which steam is generated and is usually slightly above the ambient boiling temperature. Intermediate heating stage set points are typically determined through process modeling and balancing volumetric steam flows from each source, with consideration of process requirements.

Direct-Contact Condensation

One of the most widely used and successful unit operations for preheating of slurries is a form of baffled direct-contact condenser (or slurry preheater vessel), as shown in Figure 11. Feed slurry enters the vessel through a distributor pipe at the top and flows downward by gravity while flash steam enters at the bottom of the vessel and flows countercurrently upward. Multiple angled baffles within the vessel redirect the slurry back and forth across the heater, decelerating the steam and forcing it to contact curtains of slurry. Slurry preheaters are thermally efficient and can achieve approach temperatures of <10°C—and sometimes <5°C—provided that sufficient energy is available in the steam to heat the slurry and the presence of noncondensable gas is negligible (where the *approach temperature* is defined as the difference in temperature between the incoming steam and the final heated slurry).

A rule of thumb for heating slurries is that every 10 kg of steam condensed will raise the temperature of 1 t of slurry by approximately 6.5°C, based on the latent heat of steam and a slurry specific heat capacity equal to 70% that of water.


Slurry Flow **Steam Flow** **External View**

External View: Courtesy of Newcrest Mining Limited

Figure 11 Direct-contact slurry heater with segmented baffles

The mechanical design of slurry heaters generally follows international codes and standards for the design of unfired pressure vessels, particularly the American Society of Mechanical Engineers' standard, *ASME Boiler and Pressure Vessel Code* (BPVC), Section VIII, Division 1 (ASME 2015a); Australian National Standard AS 1210; or British Standard BS PD 5500. The vessel diameter and baffle spacing are dictated by the volumetric flow rate of the flash steam, whereas the number of baffles and baffle geometry are determined by the slurry properties. The performance of a baffled heat exchanger is highly dependent on slurry fluid properties (e.g., yield stress, apparent viscosity) as well as its thermal properties (e.g., conductivity, specific heat capacity).

Segmented baffle design has been applied successfully in many refractory gold slurry heaters since its original development by Anglo American Corporation in 1977 (Fraser and Thomas 2010). Heater vessel sizing generally follows the design principles established by James Fair (1993) for the design of baffle tray fractionation columns for the petroleum

Courtesy of Ma'aden Bauxite and Alumina Company

Figure 12 Jacketed pipe indirect heat exchangers for slurry heating in tube digester

refining industry. These principles include the number of baffles, gas or vapor rise velocity, and expected heat/mass transfer rate (in nephelometric turbidity units) as a function of the liquid-to-gas ratio. Several other baffle designs have also been tried with varying degrees of success, such as lathe grids, disks and donuts, and cups and cones.

A disadvantage of direct-contact heating is the dilution of the process stream. For multistage heating from 40° to 250°C, direct-contact heating adds approximately 370 kg of condensate per metric ton of autoclave feed slurry. If feed dilution affects the process (e.g., increased heating requirements), there may be an economic advantage to apply indirect slurry heating.

Indirect Slurry Heating

Indirect heating is accomplished without dilution of the process slurry, resulting in a lower volumetric flow rate through the circuit, and hence, smaller equipment and piping for the same ore treatment capacity can be selected. The Bayer process requires a high degree of heat recovery and minimal process liquor dilution for economic treatment of low-grade bauxite. Indirect heating has been used extensively in alumina plants since the mid-1960s. Modern alumina tube digester plants use jacketed pipe heat exchangers to preheat bauxite slurry prior to entering the tube digester. Examples include Rio Tinto's Yarwun alumina refinery in Australia; Ma'aden alumina refinery in Ras Al-Khair, Saudi Arabia; and the Emirates alumina refinery in the United Arab Emirates. Figure 12 shows a photo of a high-pressure tube digester plant constructed at the Ma'aden Bauxite and Alumina Company refinery.

Over the past 30 years, there has been substantial research conducted on indirect heat exchange, including the construction and operation of a 1/1,000-scale integrated pilot plant that used indirect heating of nickel laterite slurry from 100° to 250°C using shell-and-tube heat exchangers by AMAX and Inco Technical Services Ltd. in 1999, as well as research by Anglo American and Anaconda Nickel. The authors designed, constructed, and operated an indirect slurry heating demonstration plant in 1998 for the Murrin Murrin nickel laterite facility, as shown in Figure 13.

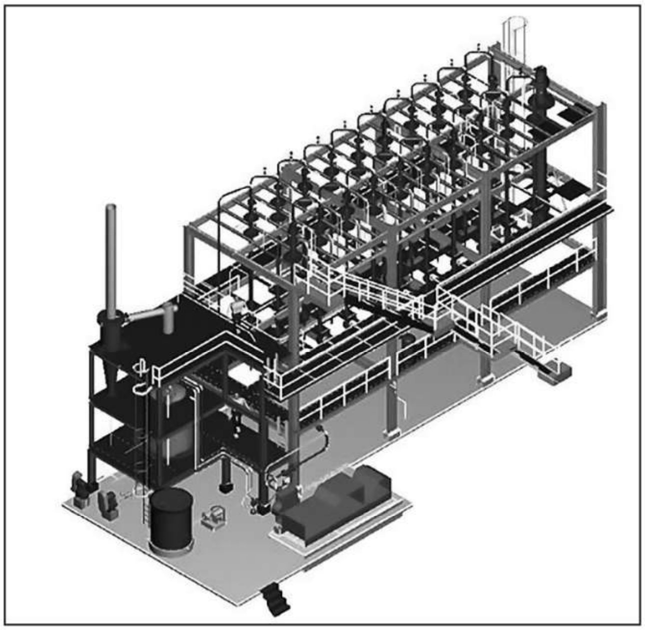
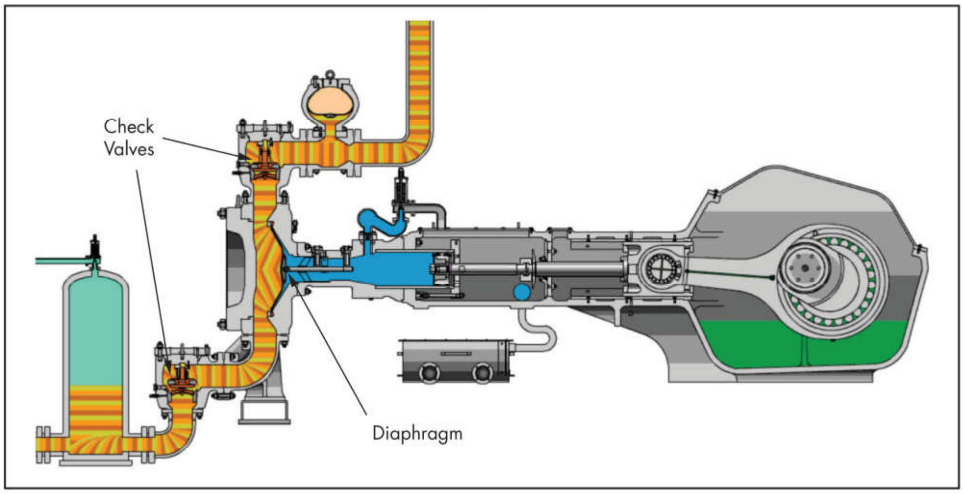


Figure 13 Murrin Murrin indirect heating demonstration plant

The challenges associated with the indirect heating of slurries are primarily associated with non-Newtonian rheology and hindered settling, where the interaction of fine particles results in rheopectic (shear thinning) behavior coupled with a high yield stress. A high apparent viscosity makes it difficult to achieve turbulent flow (i.e., a Reynolds number greater than 2,000), and heat transfer is dominated by conduction and diffusion rather than convection. Under a high apparent viscosity scenario, the effective heat transfer surface area needed to achieve the specified heating duty is substantial, and multiple heat exchanger units are required for each stage of heat recovery. For example, in nickel–cobalt laterite pressure leaching, heat transfer coefficients up to 600 $W/m^2 \cdot °C$ have been observed for a 1:1 blend of saprolite and limonite.



Courtesy of Weir Minerals

Figure 14 Positive-displacement slurry feed pump configuration

However, it is not uncommon to see values as low as 300 W/$m^2 \cdot °C$ for very fine limonite slurries.

An additional challenge of indirect heat exchange is maintaining the heat transfer rate in the presence of heat exchanger tube fouling, which is often a result of precipitating solids on hot surfaces. For example, the insulating effect of 1-mm-thick hematite scale on the inner surface of a 25-mm-diameter heat exchanger tube can reduce the overall heat transfer coefficient by 50%. For commercial operations, a means of tube cleaning has been developed using automated water-jet lancing to address this issue; however, the heat exchanger unit operation still needs to be taken off-line for cleaning duty.

Indirect heat exchangers are more susceptible to erosion and channeling when applied to ore slurries, leading to issues such as cross-contamination of exchange media due to wear-through of heat exchange boundaries or plugging as a result of fouling or solids accumulation. Therefore, more maintenance is usually required for this configuration, and redundant equipment may be required to maintain production rates during maintenance activities.

Pumping

The reactor vessel feed pump is typically a positive-displacement pump, most commonly a piston diaphragm configuration, where one or more hydraulically operated elastomeric diaphragms are situated between two check valves. The rated service temperature for feed pump diaphragm materials (Buna-N, HBN, EPDM) is 60°–90°C, but in a high-temperature service (greater than 100°C), the diaphragms are protected by a water-cooled “dead leg” or static section of slurry that allows the diaphragms to displace the higher-temperature slurry at a distance of 1.5–3.0 m from the diaphragm housing. A feed accumulator and discharge dampener are typically installed on the suction side and discharge side of the pump, respectively, to reduce pressure variation from the pulsating nature of the pump and to reduce energy losses associated with slurry acceleration. The pump is usually equipped with a variable-speed drive to control the pump stroke rate, and the associated mass flow is determined using in-line volumetric flow rate and density measurements. Figure 14 demonstrates a crankshaft-driven, single-acting, high-pressure piston diaphragm pump with feed accumulator and discharge dampener (Weir Group PLC 2016).

The reactor vessel feed pumps generally require a thorough preventive maintenance program to ensure reliable operating time. The highest frequency of wear occurs on the check valves and diaphragms, which account for about 30%–50% of the routine maintenance downtime. It is common practice to operate multiple feed pumps in parallel at low stroke rates to reduce the wear rate of wetted parts and to minimize process feed interruption from unplanned downtime. Figure 15 shows an example of the impact on stroke rate for typical check valve operating life. Parallel feed pumps are often sized to operate at 66%–75% of their design throughput to reduce the overall impact when one pump is off-line. Depending on project economics, it may be advantageous to select pumps that are each capable of delivering the full process design throughput for complete redundancy. At the Twin Creeks pressure oxidation facility, a third feed line was eventually added to each autoclave not only to expand capacity but also to improve pump run time and reduce the impact of pump maintenance (Krumins et al. 2014). It is common to have a centrifugal “charge” pump preceding the autoclave feed pump to provide adequate net positive suction head, but this is not always required.

A basket-style slurry strainer is typically used in advance of the reactor vessel feed pump to prevent oversize material from entering the pump and causing premature failure of the check valves or diaphragm. Quick change-out strainers or self-cleaning strainers should be used where excess oversize material is anticipated. More recent refractory gold plants such as Pueblo Viejo and alumina tube digesters at Yarwun, Ma’aden, and Emirates Alumina use self-cleaning strainers where oversize material can be discharged while the process remains online.

Multistage centrifugal pumps have been proposed as an alternative to positive-displacement pumps; however, they pose the risk of catastrophic failure of the pump casing due to reverse impeller rotation, as centrifugal pumps permit reverse flow when their motors are de-energized. Pump failure can be minimized with the installation of antireverse devices such as dynamic clutches; however, reverse process flow is still possible. For most pressure vessel operators, the safety risk and

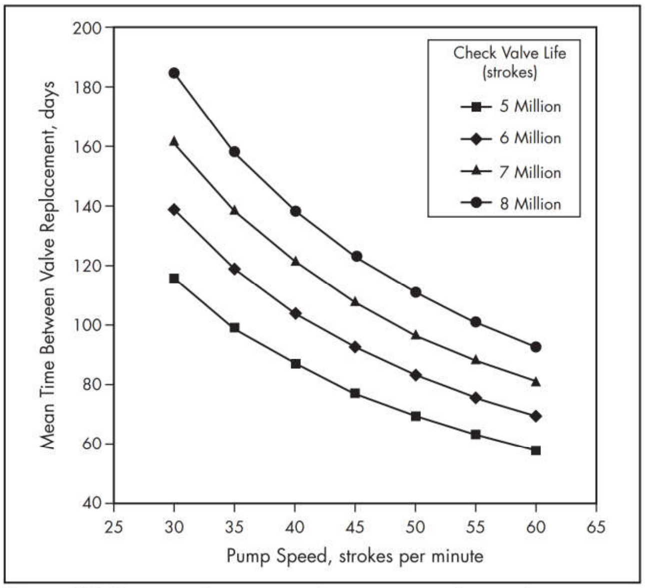
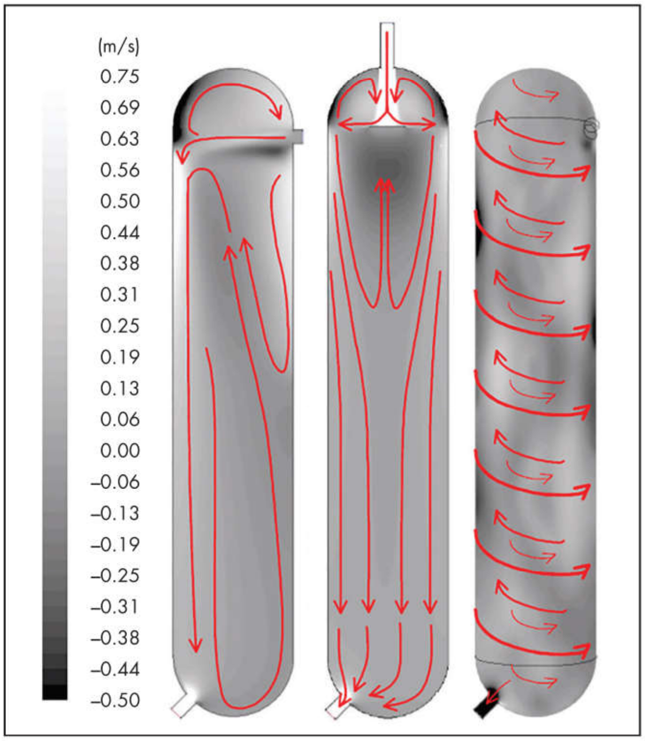


Figure 15 Impact of pump stroke rate on valve replacement requirement



Source: Woloshyn et al. 2006. Copyright 2006 by The Minerals, Metals & Materials Society. Used with permission.

Figure 16 Comparison of axial velocity and the characteristic flow patterns in digesters with normal, central, and tangential slurry inlets

the cost of pump seal maintenance outweigh any potential savings in capital cost; therefore, positive-displacement piston diaphragm pumps are selected for most feed applications to autoclaves and high-pressure reactors.

Reactor Vessel

Vessel type and configuration. Reactor vessels are most often one of three configurations, depending on the application and project constraints:

1. Vertical digesters
2. Horizontal compartmentalized and mixed vessels
3. One or more vertically oriented vessels, mixed or lightly agitated by draft tubes

Vertical digesters, as used in alumina processing, are typically used as a series of agitated or unagitated vessels that either use direct steam injection to maintain temperature or are indirectly heated using internal steam coils or external heaters. The vessels are commonly 3–4.5 m in diameter and up to 30 m tall to promote an ideal residence time distribution for the chemical reaction (Hill and Sehnke 2006). Digester agitators may divide the vessel into a series of multiple compartments with staged baffle plates to prevent short-circuiting within the vessel.

Developments in digester technology have led to the use of plug flow tube digesters, which have been implemented in recently constructed facilities and have been able to reduce plant capital and operating costs (Gorst et al. 2013). A simulation of fluid flow through various tube digester configurations is provided in Figure 16 (Woloshyn et al. 2006).

Pressure acid leach and pressure oxidation autoclaves are typically configured as horizontal vessels with four to eight stages of agitation (process dependent), separated by internal compartment walls. The compartments act as a series of continuous stirred-tank reactors and provide a residence time distribution that ensures that the feed material is given sufficient opportunity to react. In oxidative processes, the initial stages (typically the first 25%–50% of the vessel) contain the most intensive oxidation, driven by oxygen mass transfer rates into the slurry, whereas the latter stages are required to satisfy kinetic limitations. A typical arrangement is shown in Figure 17.

Many autoclaves are designed with combined initial stages (i.e., an enlarged first compartment), which benefits the process in three ways:

1. For rapid reactions, the concentration of reaction products is distributed over a greater volume, reducing the potential for local precipitation and scale deposition, which can result in increased maintenance requirements.
2. Back-mixing of slurry allows overall compartment temperature moderation. Heat generated in the second stage can back-mix and heat the first stage, or cooling water introduced in the second stage can moderate the temperature in the first and/or third stages.
3. Acid generated by pressure oxidation has a greater concentration in the first stage (i.e., the acid produced in the second stage can mix back into the first stage), promoting soluble iron and increased oxidation rates or overcoming carbonate neutralization, if present.

Slurry cascades over the internal compartment walls, similar to a series of overflow-configured tanks. Alternatively, each wall may have an underflow port, allowing slurry flow through the bottom of the wall. An underflow configuration facilitates movement of coarse reaction product solids to the discharge end of the vessel with the intent of improved slurry level control and improved ease of cleanout.

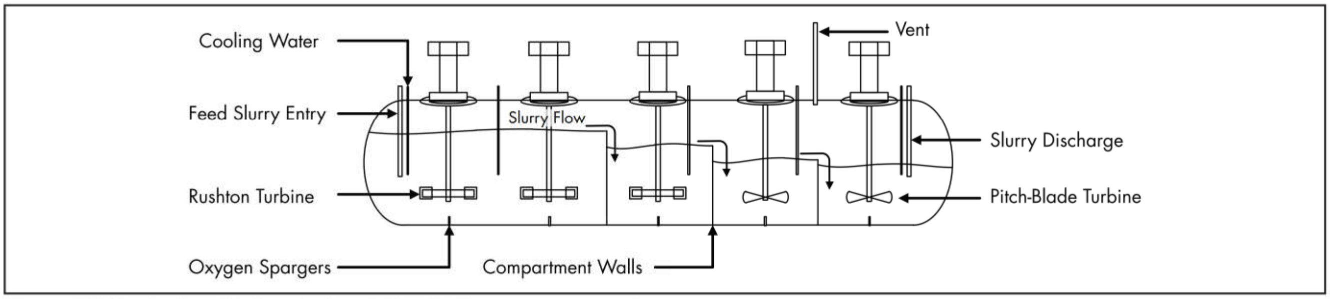


Figure 17 Typical oxidative horizontal autoclave arrangement

In oxidative processes, oxygen is delivered to each stage, usually below the agitator impeller and proportional to the expected oxidation profile through the vessel. High-pressure cooling water is directly injected into the slurry at a well-mixed location in each stage to achieve the target process temperature. The water addition rate is based on local temperature measurement and is directly indicative of the sulfur oxidation in each stage.

In some cases, multiple horizontal autoclaves can be used in series at staged conditions to optimize operations. Such is the case at Hudbay Minerals' zinc pressure leach plant in Canada, which was designed to leach 75% of the zinc in a low-acid leach autoclave, followed by pressure letdown, thickening, and re-leaching in a high-acid leach autoclave to recover the remaining zinc (Krysa 1995).

Autoclave vessel process diameters range from 1.0 to 6.0 m, and tan–tan lengths range from 6 to 45 m for demonstration-size vessels and commercial-size vessels, respectively. The corresponding process volumes can be as low as 4 m^3 for demonstration units and as high as 865 m^3 for commercial units. The largest autoclave (by volume) currently in operation is at Lihir Gold, Papua New Guinea, and is approximately 47.7 m long and 5.6 m in diameter (Collins et al. 2011). A comparison of vessel sizes for select operations is provided in Figure 18.

It may not be economically feasible to fabricate and transport large equipment to certain mine sites; therefore, alternative options such as on-site fabrication—or a series of vessels (e.g., vertical pots)—need to be considered. A series of vertically oriented vessels performs similarly to a combined horizontal vessel, with each stage segregated to its own piece of equipment. In this configuration, there is a loss of synergy with respect to the reduction of construction materials, piping, and instrumentation. However, this configuration may be advantageous if there are major restrictions on vessel fabrication and transportation and if the process is not prone to scale formation. The comparative cost implications are lessened for processes that do not require expensive materials of construction. Furthermore, a series of vessels may allow for bypasses around a vessel to continue operation during maintenance or descaling if this is not prohibited by piping and valve costs. An example of this configuration is the five-stage pressure oxidative leach for the recovery of cobalt from slag and copper production in Zambia (Munnik et al. 2003).

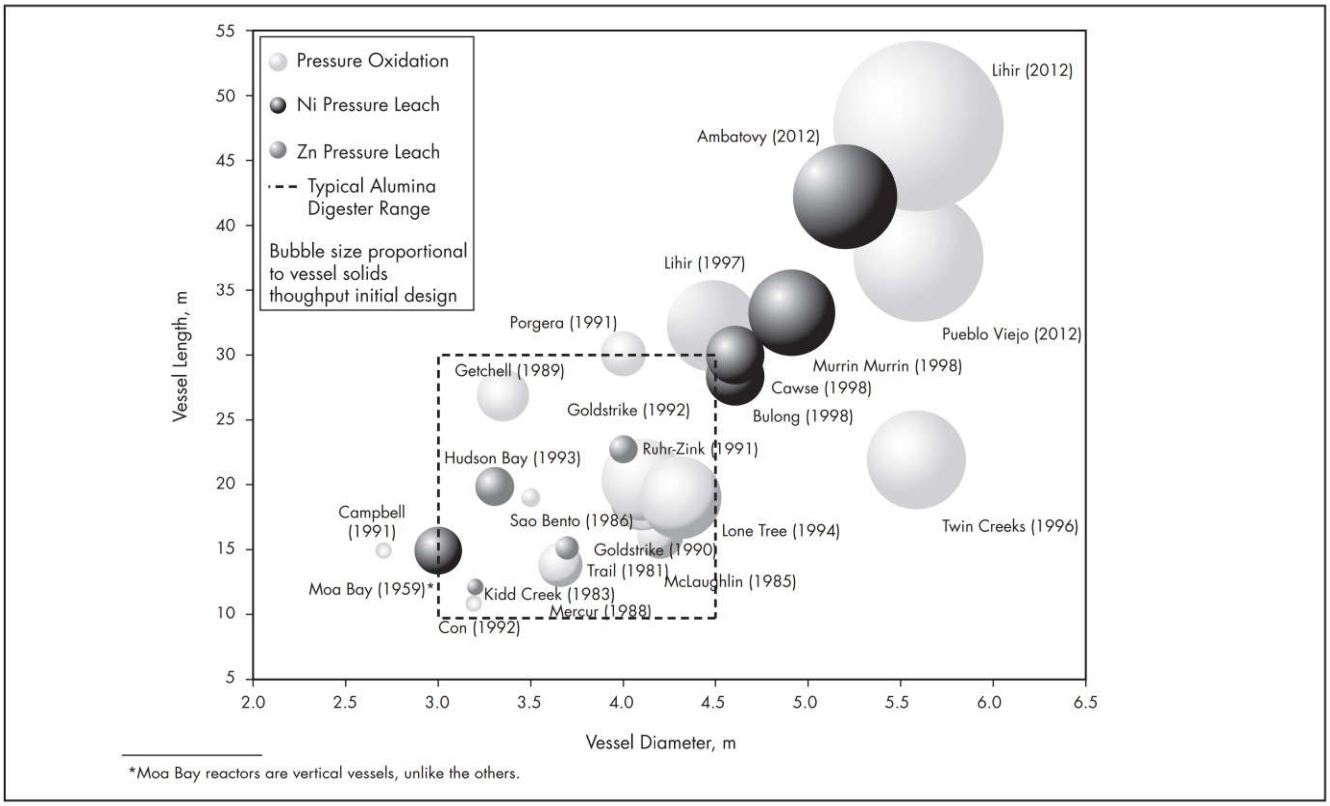
A vertical vessel is also appropriate if only one reaction stage is required. This is more often the case for processes that operate batchwise. For example, two 15-m^3 batch brick-lined autoclaves were commissioned at Xstrata Copper Canada's Canadian Copper Refinery in 2006 (now Glencore) and were designed to operate at a rate of 14 batches per week. The autoclaves operate in a two-stage batch process, first leaching nickel at 160°C in the absence of oxygen and then, in the same vessel, dissolving copper and tellurium at 110°–140°C through pressure oxidation (Doucet and Stafiej 2007).

Initial reactor vessel sizing. Sizing and selection for reactor vessels require careful consideration of many aspects of a project. One important aspect is the overall plant throughput and anticipated range in feed composition and circuit throughput, as discussed in the introduction to this section. Once the circuit feed throughput and variability are defined, the reactor vessel sizing can progress with consideration of typical process condition parameters: temperature, pressure, particle size distribution, retention time, reagent addition, and a leach recovery or oxidation extent target.

The approach to reactor vessel sizing depends on the phase of project development. In the preliminary phases, vessel sizing may be based on benchmarking or theoretical process modeling. As the project develops, the vessel sizing is revised with data obtained from a batch metallurgical testwork program and is updated during the feasibility and design project phases with pilot metallurgical test work, depending on the process. Projects with challenging mineralogy and oxidation processes require pilot test work to support the vessel design, whereas a noncomplex ore and widely used technology may use batch test work and benchmarking to support the vessel design (e.g., alumina digesters).

Initial pressure leach or pressure oxidation vessel sizing can be established with theoretical process modeling and industry benchmarking using operations with similar expected feed composition and operating conditions. Theoretical process modeling, as described by Papangelakis and Demopolous (1992) and Crundwell and Bryson (1992), can be used for initial vessel sizing and assessment of initial compartment configurations; however, the models do require fundamental assumptions of the oxidation or leach rate and an initial particle size distribution. Use of the methodology is iterative with adjustment to the vessel configuration and size until the target reaction extent is achieved. Industry benchmarking can be used to support the theoretical model assumptions or used in a simpler way with the comparison of leach rate or oxidation capacity metrics. A value of 0.7 sulfide sulfur metric tons oxidized per cubic meter of process volume per day ($0.7 \text{ t } S^{2-}/m^3 \cdot d$) is a typical vessel oxidation capacity for a whole-ore pressure oxidation operation with a pyritic feed sulfide sulfur grade between 6% and 8%.

Consideration of transportation restrictions between the vessel fabrication facility and the project site is important for the sizing, selection, and design of pressure vessel equipment.



*Moa Bay reactors are vertical vessels, unlike the others.

Data from Mason and Gulyas 1999; Collins et al. 2011

Figure 18 Comparison of reactor vessel sizes for various pressure hydrometallurgy processes

Courtesy of Mammoet USA North Inc.

Figure 19 Autoclave vessel transportation for the Pueblo Viejo Project, Dominican Republic

Transport restrictions include axle loading, load width and height, clearance requirements for overpasses and signage, road gradients, and turning radii. The cost of specialized transport for vessels over 100 t can be as much as the cost of fabrication, and delays in the delivery of key equipment can result in costly route traffic disruption and construction delay claims. Figure 19 shows the transport of a 780-t autoclave that is 37.6 m long and 5.6 m in diameter (Hatch Ltd. 2016).

Alternatively, the use of prefabricated assemblies enables conventional transport equipment but requires more assembly/fabrication at the site. A transport and associated site assembly/fabrication study should be conducted in the early stages of a project to determine which approach will be most cost-effective and least disruptive to communities along the transport route.

Preliminary reactor vessel sizing. Key process conditions are tested and selected during the batch test-work phase. The results from batch test work can be used to develop a preliminary mass and energy balance of the proposed commercial process to estimate the volumetric flow rates by compartment. These flow rates are then used to estimate a preliminary process volume and vessel size based on retention time. The mass and energy balance can be developed from first principles or by using process modeling software such as METSIM and supplementing batch test-work data with simulated values, where data are limited in the early stages of project development. The preliminary size can then provide information for an assessment of the mechanical constraints (primarily the vessel transportation requirements and fabrication).

Reactor vessel scale-up from pilot test work. Although batch test work is important to establish process conditions, pilot test work is required to verify the conditions and obtain data to support the vessel design, particularly for an oxidation process. Oxidation kinetics change from batch operation (static) to continuous (dynamic) operation, the latter of which emulates a commercial process and allows compartment profile sampling and composition analysis. Continuous piloting also provides a valuable opportunity to test different autoclave compartment configurations and ore blends at the target conditions and to identify challenges that may not have been apparent in the batch test work (Simmons 1995).

The key information obtained during pilot-plant test work is a leach or oxidation profile consistent with the selected vessel compartment configuration (e.g., one-, two-, or three-stage first compartment). Figure 20 provides an example of cumulative sulfide sulfur oxidation profiles for both a single- and dual-stage first compartment of the same ore type. The significant difference in the oxidation profile between the one- and two-stage first compartments is mostly associated with the acidification of carbonate minerals from the recycling of acid generated during the oxidation reaction. In this case, the change in compartment configuration enables a reduction in the cost to pre-acidify the autoclave feed.

It is not unusual for the oxidation profile information to indicate a range of oxidation rates with different ore blends or conditions. A design oxidation rate needs to be established based on a statistical analysis of all data and the selection of a defining ore blend. Once established, the oxidation profile information sourced from the pilot test work is used to update the mass and energy balance, and the vessel process volume is recalculated based on the cumulative retention time of all reactor compartments. Some important considerations for final vessel sizing include the following:

1. The density of water decreases as the water temperature increases; however, dissolved metals and acid concentration increase the solution density. The use of process modeling software accounts for both of these effects.
2. An allowance must be made for gas holdup for reactor designs with gas addition. The allowance needs to be tempered with consideration to any gas generated during the process, introduced inert gases, and gas solubility.
3. Final consideration must be given for any mechanical or physical constraints associated with fabrication and transportation to a site.

Design standards. Pressure vessels such as autoclaves impose a potential risk to health and safety if they are not designed, fabricated, installed, operated, and/or maintained in accordance with pressure vessel code requirements. The ASME BPVC is globally recognized and widely accepted for the design and fabrication of unfired pressure vessels and satisfies the regulatory requirements of authorities in many countries.

Within the ASME BPVC, a vessel can be designed using Section VIII Division 1 or Division 2 (ASME 2015a, 2015b). Division 1 is commonly referred to as "design by rule" in which the component sizes and material thicknesses are governed by preset rules, equations, and conservative values of allowable material stresses that have been developed by materials researchers and adopted by the ASME Code Committee. The most commonly used pressure vessel code tends to be Division 1, where a design-by-rule method is implemented to achieve a code-compliant design.

Within Division 2, a vessel (or components of a vessel) may be designed using either *Part 4: Design by Rule* or *Part 5: Design by Analysis*. Typically, design by analysis is used for nonstandard components that are not addressed through design by rule. Division 2 is considered a more sophisticated approach because it permits increased utilization of material strength (i.e., higher allowable stress) and generally results in thinner, lighter vessel wall components. The greater reliance on the material properties of Division 2 is accompanied by additional—and more stringent—quality requirements for materials and fabrication compared to those of Division 1. Division 2 is typically only used for a vessel when the additional design effort is compensated by material cost savings or weight restrictions for transport or when this is required by the end user.

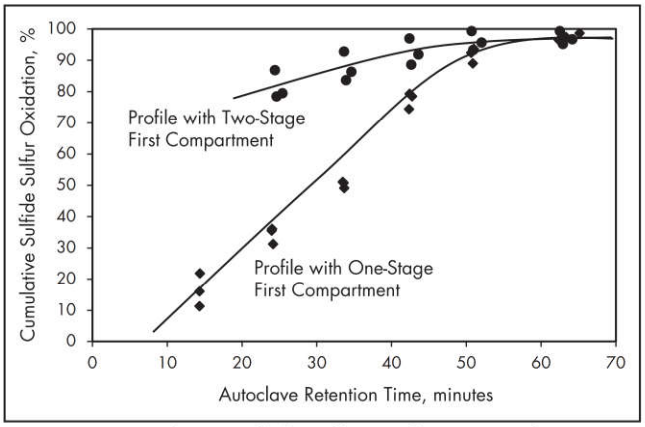


Figure 20 Cumulative sulfide sulfur oxidation as a function of cumulative retention time

The use of Section VIII Division 1 is sufficient for most autoclave applications. Where design rules are not provided to address a particular aspect of the design, then the use of Division 2 is considered acceptable to qualify that aspect. Examples of this provision are the assessment of fatigue in agitator nozzles and thermal displacement stresses at support locations for metal-clad autoclaves. However, in this case the design is still conducted using Division 1 allowable stresses in such applications because the basic vessel design is still based on Division 1 requirements.

Pressure vessels conforming to ASME requirements receive a U stamp in addition to other markings that may be required to identify registration or compliance with a local regulatory body. In the United States, some jurisdictions require National Board registration identified with an NB mark. In Canada, a pressure vessel must be registered in the province or territory of its end user and be stamped with a Canadian Registration Number certifying federal approval of the vessel for legal operation. If the vessel is built outside of Canada for Canadian use, it must also be registered with the National Board. For an ASME pressure vessel to be used within the European Union (EU), the vessel must demonstrate full compliance with the Pressure Equipment Directive (PED; EU Directive 97/23/EC). Within the EU, the PED is a legal requirement in which compliance is identified by CE marking, indicating that the marked vessel is permitted for use anywhere in the EU. The PED dictates essential safety requirements governing the design and fabrication of pressure vessels. Instead of providing a design methodology for unfired pressure vessels, the PED references the harmonized standard BS EN 13445 that has demonstrated PED conformity. The use of BS EN 13445 is not mandatory, and the ASME BPVC can be used pending completion of a PED conformity

Courtesy of Patrick Lauzon, Hatch

Figure 21 Pressure vessel refractory lining installation in progress

assessment. ASME Section VIII Division 2 is comparable to BS EN 13445, making compliance with PED easier than with ASME Section VIII Division 1. PED compliance may be achieved by following a guide published by ASME that outlines the additional requirements to meet the PED based on ASME Section VIII Division 1. Australian vessels typically comply with Australian National Standard AS 1210.

For autoclaves, internal refractory lining design requirements are not addressed by pressure vessel codes. Within the design codes, an internal refractory lining is considered a dead load. A vessel designed in accordance with a pressure vessel code will have adequate strength, allowing the vessel to deform under specified operating or design conditions without concern. However, an internal refractory lining responds poorly to vessel deformation, which can induce excessive compressive stresses in the refractory face course and tensile stresses in the refractory back course. Both are detrimental to lining stability, potentially resulting in brick spalling and/or complete lining failure. Because a refractory lining has a very low resistance to induced bending stress, additional calculations and finite element analyses (FEAs) are commonly completed to investigate vessel deformation and the interaction between the refractory lining and the vessel. Vessel deformations are investigated and reinforcement options are developed to minimize deformation gradients in areas that could affect lining stability. Because there is no specific standard addressing these design practices, the combination of experience and the criteria of BS EN 14879, Annex D (formerly DIN 28060) are potential resources to develop a basis for determining acceptable deformation gradients in a refractory-lined vessel (Donohue et al. 2012).

Areas of concern for excessive deformation gradients tend to be discontinuities inherent to vessel designs (e.g., nozzles, re-pads, supports, stiffeners, and heads). A hemispherical head would be preferred for lining stability; however, a semielliptical 2:1 head is common in autoclave applications to suit process requirements. The transition from vessel cylinder end to the dished portion of the head is typically a region of concern. The suggested best practice to reduce vessel deformation gradients in an autoclave is simply increasing the shell thickness because the addition of stiffeners or re-pads creates additional discontinuities with the potential to induce bending stresses in the refractory lining. Depending on the design pressure of the autoclave and the shell thickness required by code, it may be determined that the additional shell thickness for lining stability is minimal in comparison. As additional shell thickness benefits refractory lining stability, it is advantageous to design the vessel using ASME BPVC Section VIII Division 1 (ASME 2015a) because the code generally results in a thicker vessel shell compared to using Section VIII Division 2 (ASME 2015b) or BS EN 13445.

Lining systems. Many reactor vessels are made of C-Mn-Si steel and are fire clay refractory lined, with an acid-resistant protective membrane between the shell and thermally insulating refractory lining. Figure 21 shows the installation of a four-course refractory lining within an autoclave. Designing the lining system with respect to stresses is critical to its performance, maintenance requirements, and longevity. A balance must be obtained between keeping the brick under compression (due to low tensile strengths) without imposing so much compression that bricks are spalled or crushed. Therefore, many considerations must be made with respect to the vessel size, operating conditions, and selection of materials. Furthermore, operating procedures should be established to limit the rate of pressurization to approximately 350 kPa/h and rate of temperature change to approximately 10°C/h (or as recommended by the brick lining supplier) to minimize transient stresses on the lining. Sufficient brick must be installed to maintain a membrane temperature less than the maximum working temperature of the selected membrane material, which is usually between 85° and 120°C.

Historically, chemically pure (>99.95% w/w Pb) lead has been used as the protective membrane for pressure oxidation and zinc pressure leach processes; however, many lining suppliers have developed alternatives (e.g., elastomeric, polymeric, bituminous mastic) to meet the needs of various vessel designs and user preferences. As of 2010, chemical lead membranes were still in service after 54 years in the Moa Bay leach reactors. Similarly, a bituminous mastic membrane had provided more than 15 years of service with no failures or repairs at the Twin Creeks pressure oxidation facility (Wei et al. 2010).

One of the greatest challenges is the design of the lining system around vessel nozzles. Nozzles are troublesome, as their geometry and proximity to one another promote hotter surface temperatures (McMullen 2014), which can lead to membrane creep and failure (Donohue et al. 2013). In such cases, the membrane may be replaced with high-nickel alloy weld overlay (e.g., Inconel 625, Hastelloy C2000, Alloy 59), which has a greater resistance to temperature creep, although at a higher cost (Bristowe et al. 2004). Alloy nozzle seal rings are typically required, with a spiral-wound gasket seal to ensure that the autoclave process fluids cannot contact the steel shell where the lining ends. In smaller-diameter nozzles, access does not permit the installation of refractory brick for thermal protection. In these cases, either fire clay or thermoplastic insulating inserts are used (Fraser et al. 2008). Detailed finite element analysis is typically performed during detailed engineering to determine the optimal nozzle lining design.

For processes operating above 235°C, such as pressure acid leaching (250°–270°C), the design and installation of structurally stable refractory linings become more difficult and costly with additional lining thickness (Donohue et al. 2012). For such applications, the industry has adopted

Table 6 Comparison of refractory and titanium-clad autoclave linings

Argument	Refractory Lining	Titanium-Clad Lining
Advantages	Excellent corrosion resistance in both oxidizing and reducing sulfuric acid environments Excellent abrasion resistance Excellent resistance to oxidation and ignition (pyrophoricity)	Excellent corrosion resistance in oxidizing environments Higher service temperature (up to 315°C) is permitted by pressure vessel codes Can be in direct contact with process media, resulting in a smaller and lighter vessel for a given process volume
Disadvantages	Lower service temperature limits (<250°C) due to stability limitations in lining thickness Higher maintenance costs associated with face course refractory relines Requires larger vessel to accommodate lining thickness for a given process volume	Susceptible to pitting and/or crevice corrosion in reducing environments Reduced abrasion resistance, especially to siliceous ore slurries Potential for ignition in enriched oxygen environments (pyrophoricity) Weld repair of corroded cladding is difficult and expensive

Source: Pearson et al. 2010

metal-clad construction—specifically explosion welding—as a means of bonding corrosion-resistant metal such as titanium to a C-Mn-Si steel substrate. This has been shown to be a cost-effective alternative to refractory lining at vessel sizes of 300 t or more (Pearson et al. 2010). Although it varies with project specifics (e.g., vessel sizes, materials, fabricator shop loading), a typical delivery period for a large, horizontal, titanium-clad autoclave is in the range of 80 weeks, whereas that of an equivalently sized brick-lined vessel is approximately 60 weeks, including 8 weeks for on-site brick installation (Zunti and Pearson 2004).

The titanium layer is considered a corrosive barrier and is not included in the design for pressure containment (e.g., wall thickness). The explosion-bonded material is created in flat sheets, rolled or formed into the sections that make up the pressure vessel, and sealed using a batten strap welding technique. A comparison of the two autoclave lining options is provided in Table 6.

Mixing and agitation. Mixing and solids suspension are typically required for most slurry leaching applications in which reagents (e.g., acid) are added to produce a chemical reaction, and maintaining solids suspension is important. Depending on slurry solid content and viscosity, mixing and solids suspension duty can be performed using hydrofoil or pitch-blade, turbine-style axial flow impellers. For low agitation requirements, pneumatic or steam-based agitation is possible in specifically designed vessels (e.g., Pachucas) to reduce wear components and has been successful for uranium and alumina leaching applications.

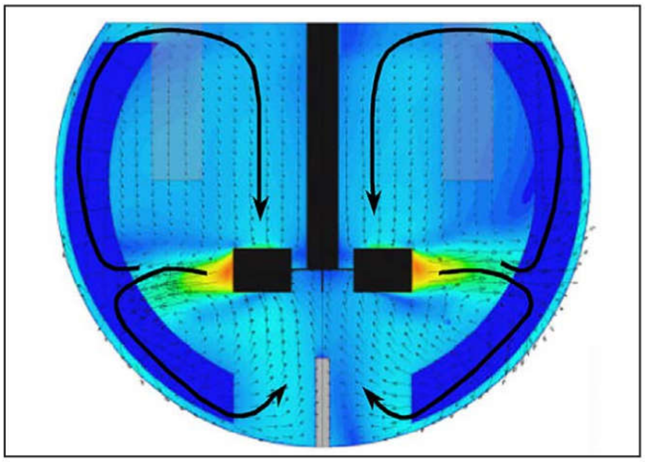
Agitator impeller tip speeds are typically maintained below 6 m/s during normal operation to limit abrasive wear from the slurry on both the agitator and the vessel lining. Some operations use protective coatings (e.g., titanium dioxide, chromium oxide) to reduce agitator wear and extend the service life; however, regular maintenance, refurbishment, or replacement is still expected. Agitator materials are typically selected to be similar to vessel internals (e.g., titanium), according to process conditions. In the case of titanium agitators in high-oxygen concentration environments, the upper shaft is fabricated from an ignition-resistant material (e.g., nickel alloy) as a safety measure against breach of containment, should the lower titanium agitator shaft ignite. Many agitators are designed with impellers that must be assembled within the vessel itself to reduce requirements for larger vessel nozzles, which can be problematic in terms of thermal lining design, gasket seal tightness, and long-term maintenance of the nozzle.

The primary design intent of pressure oxidation autoclave agitators is oxygen gas dispersion, typically achieved through the use of high-shear radial turbines, such as Rushton turbines. These agitators typically achieve power-per-unit volume inputs of 1 to 3 kW/m^3 (Pieterse 2004). The theoretical unit-power requirement is based on several variables, including oxygen partial pressure, oxygen solubility, and operating temperature, which determine the system oxygen mass transfer coefficient ($k_L a$) to suit the oxidation reaction. A simulation of radial fluid flow for gas dispersion is provided in Figure 22.

For the latter stages of pressure oxidation, when the oxidation reaction is typically kinetics limited, the agitation duty changes from gas dispersion to solids suspension. At this point, oxygen addition and dispersion are typically minimal, as most of the oxidation reaction has already occurred in the initial high-power mixing stages. In this regime, pitch-blade or hydrofoil impellers can be used and typically require substantially lower power input (approximately 0.5–1.0 kW/m^3) to achieve the required solids suspension.

Positive isolation requirements. Pressure leaching and pressure oxidation processes have many inherent hazards; therefore, effective isolation or disconnection of equipment from every energy and process source is essential for safe operational and maintenance practices. Table 7 lists common hazards of pressure hydrometallurgical processes that require positive isolation.

The autoclave piping design needs to consider the appropriate isolation requirement for the service and isolation under different scenarios (e.g., full shutdown vs. hot standby). Many of the risks from hazardous conditions cannot be mitigated using single-block (on/off) valves; rather, single-block valves with bleed streams can only be considered sufficient until positive isolation is provided by other acceptable means such as a process shutdown. Double-block-and-bleed valves (i.e., a pair of block valves fitted into a line with a bleed valve in the space between the block valves) are advisable for all systems containing hazardous fluids to provide effective isolation, as long as the bleed line can be verified as being clear and routed to a safe destination.



Source: Nicolle et al. 2009

Figure 22 Radial slurry flow within an autoclave compartment (axial vessel view)

Table 7 Hazardous conditions typical for pressure hydrometallurgy

Hazard	Description
Elevated temperature	Above 100°C
Elevated pressure	Above 1.0 MPa
Corrosive materials	Highly acidic or alkaline slurries
Oxygen-enriched environment	Oxygen concentration greater than 35 vol %
Asphyxiation	Abnormal oxygen levels
Flammable materials	Flash point <60°C
Pyrophoric materials	Potential for spontaneous ignition in air
Explosive	Potential to exceed lower explosive limits
Radiation	>1 mSv per annum dosage rates
Reduced temperature	Less than –5°C
Toxic materials	For example, lead mortar

Isolation valves for hazardous fluids typically subjected to physically demanding conditions, such as corrosive and abrasive slurry flow, are designated as "severe service." Examples of severe service valves include autoclave inlet and outlet (slurry and vent) valves, acid injection block valves, oxygen block valves, and steam injection block valves. Only 50–100 valve cycles are typically expected for slurry duty prior to failure (leakage) due to the demanding nature of the service. Severe service valve performance has a direct impact on the mechanical availability of the processes, as failure to achieve positive isolation results in protracted, unscheduled shutdowns (Lauzon et al. 2004) if the valves fail on demand. Key features of severe service valves include the use of bidirectional seating and ceramic coatings in the design of balls and seats. Both the design and selection of severe service valves are critical to extend valve life, as their sizes, construction materials, and specialized design typically result in significant maintenance or replacement costs. Valve repair or replacement costs can represent up to 85% of the annual maintenance cost of the piping system (Caruana et al. 2010).

Pressure Letdown

Dedicated process equipment is required to return the pressurized reactor products to ambient or near-ambient conditions. This is typically accomplished through the use of a refractory-lined phase separator (flash vessel) in which slurry is subjected to a controlled pressure letdown and the slurry and steam phases are separated. In this process, steam rapidly evolves from the solution phase (flashing) as the slurry temperature and enthalpy decrease to maintain thermodynamic equilibrium through the pressure reduction. The result is a highly abrasive, high-velocity flow field that requires the use of ceramic materials to achieve equipment service life.

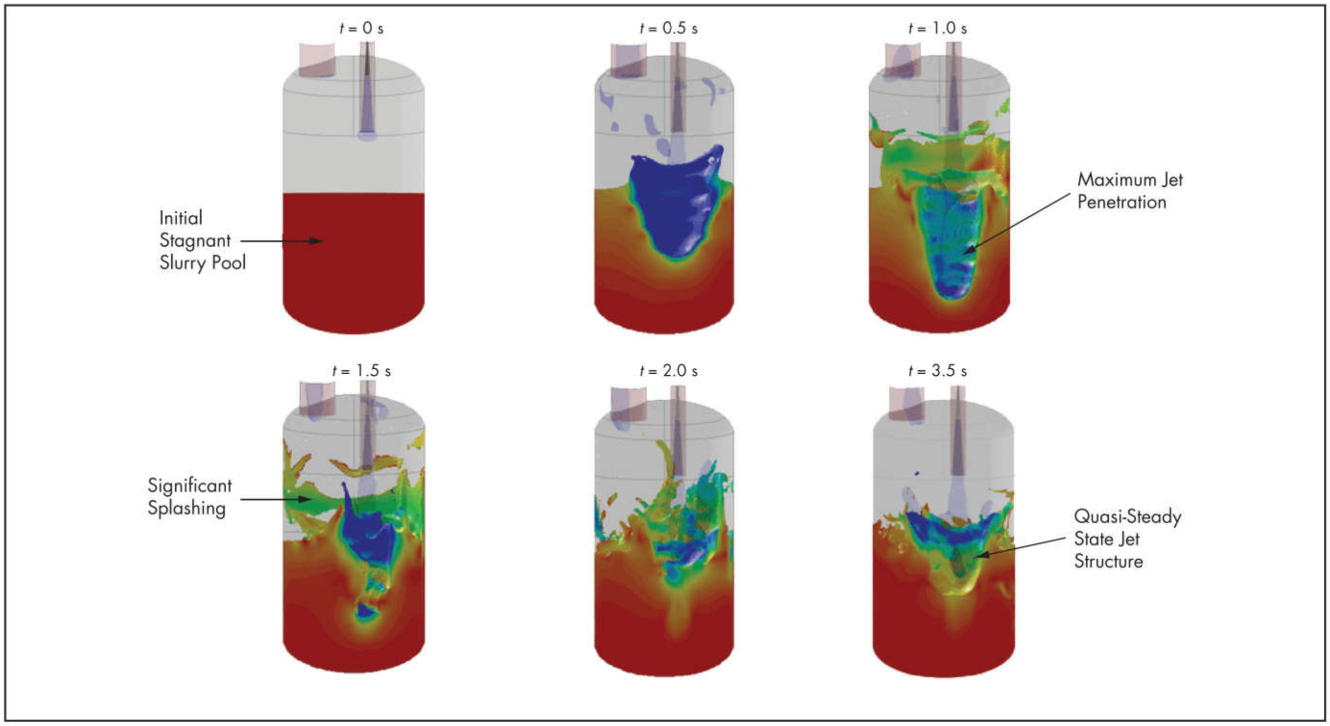
The letdown arrangement can be a single- or two-stage configuration. In a single-stage configuration, a partial pressure reduction occurs through the autoclave level control/letdown valve and the final pressure reduction occurs as the combined steam and slurry enter the flash vessel through a ceramic-lined blast tube. In a two-stage configuration, the pressure reduction through the level control valve is maintained above the saturated steam pressure (to avoid flashing), and the final pressure reduction occurs across a fixed orifice (choke) located within the vessel at approximately midheight. Bottom-entry flash vessel arrangements have been used in alumina digester processes to dissipate energy into the slurry volume. In this design, a recirculating draft tube prevents slurry from flashing as it enters the vessel.

Water may be added to the incoming slurry to modify the flash point of the slurry and defer flashing from the level control valve to the choke outlet and to reduce the total amount of flash steam produced. This can be an effective technique for increasing slurry flow through a capacity-limited system to the physical limits of the piping and choke size.

Energy dissipation is critical to flash vessel design. Slurry typically enters the vessel at or above sonic velocity as a result of choked flow (Blackmore et al. 2014). Therefore, consideration of the overall slurry momentum balance is critical to understanding how the high-energy stream is dissipated within the vessel. Furthermore, the depth to which the slurry jet penetrates the vessel operating volume (slurry pool) should be determined, such that the risk of excessive wear and potential breach of containment can be prevented through selection of adequate slurry pool depth. The inclusion of a sacrificial silicon–carbide impingement block is often installed at the base of the flash vessel to minimize the risk of vessel wear. Figure 23 demonstrates the modeled variation of top-entry slurry penetration depth during start-up (Blackmore et al. 2014).

The steam evolved from the flash vessel is typically near the ambient boiling point of water (100°C) and has the potential to be used as a low-grade heat source in other parts of the process, such as preheating slurry to the reactor vessel. Should higher-temperature steam be required for process heating, the opportunity exists to let down the system pressure in stages (i.e., a series of flash vessels) to generate steam at an intermediate temperature and pressure. An example of such practice is the Twin Creeks pressure oxidation facility, where two stages of pressure letdown supply flash steam to two stages of slurry preheating, allowing for operation at lower sulfide grades (Yernberg 1996).

For alumina processes, the number of flash vessels is an important economic consideration. Low-temperature plants that process largely gibbsitic bauxite may require three of four stages of pressure letdown, whereas high-temperature digestion for boehmitic or diasporic bauxite may require 8–12 stages (Haneman 2012).



Source: Blackmore et al. 2014

Figure 23 Slurry jet penetration for top-entry flash vessel operation

The diameter of the flash vessel is typically governed by a steam mass flux (kg/s·m^2) to allow for sufficient separation from the slurry and minimization of carryover. For large-capacity processes, the vessel diameter may be limited by the stability of the refractory lining in the domed upper head of the vessel or by transport restrictions. In this case, the duty may be split between two or more vessels in parallel. The mechanical design of pressure letdown vessels generally follows international codes and standards for the design of unfired pressure vessels, particularly ASME BPVC Section VIII, Division 1 (ASME 2015a); Australian National Standard AS 1210; or British Standard BS PD 5500.

Off-Gas Handling

Cyclone systems. A cyclone may be used as an initial measure to remove entrained slurry and recover solids material from the flash vessel steam stream, particularly where there could be an impact on the downstream process or if it is important to recover the solids or solution streams. As shown in Figure 24, the cyclone feed gas is fed tangentially into the top of the cyclone, where the rotational inertia of the gas and gravity promote separation of entrained material. The gas exits the cyclone through a vertical dip pipe (vortex finder) in the top center of the vessel. The cyclone geometry defines the recovery efficiency and size of the entrained slurry or solution droplets that are removed. A differential pressure reduction of 10–40 kPa is typical for cyclone operation; however, the differential will vary depending on the operation of the downstream off-gas system.

The cyclone typically experiences high velocity with variation in solids content and therefore abrasion-resistant materials are required to reduce the maintenance frequency and to achieve the required equipment service life. Refractory or engineered ceramic linings allow for resurfacing of wear areas within the cyclone. The internal surface of the cyclone is commonly wetted with a small flow of water to promote the transport of captured material and to reduce solids buildup on internal surfaces.

Condenser systems. Water-based atmospheric condenser (quench) systems are frequently used to reduce the volume and temperature of waste steam from the process, to lessen the load on downstream gas cleaning processes, and to reduce environmental emissions. The condenser system also provides an opportunity to recover low-grade waste heat from the process. As shown in Figure 25, water is typically sprayed in direct contact with the combined process vent gas (flash and autoclave vents) to promote the condensation of steam. Spray nozzles are selected to atomize the water and increase surface contact area between it and the gas for improved performance. The condenser does not typically include any packing if fouling and scale formation (e.g., the condensation of elemental sulfur) will lead to significant maintenance requirements and increased process downtime. Sufficient gas contact time of 2–5 seconds must be included to achieve sufficient condensation. Water should be added at a rate such that the operation is condensation limited rather than heat transfer limited, if possible. Depending on its temperature, the water addition rate typically varies between 6 and 12 times the mass flow rate of the gas (i.e., the liquid-to-gas mass ratio).

Alternative quench media, such as process or tailings slurry, may be considered where the removal of elemental sulfur is desired prior to gas scrubbing. If slurry is used, cascading baffles are typically used as opposed to spray nozzles (similar to slurry heater vessels). In all cases, any chemical interaction between both streams must be considered, as well

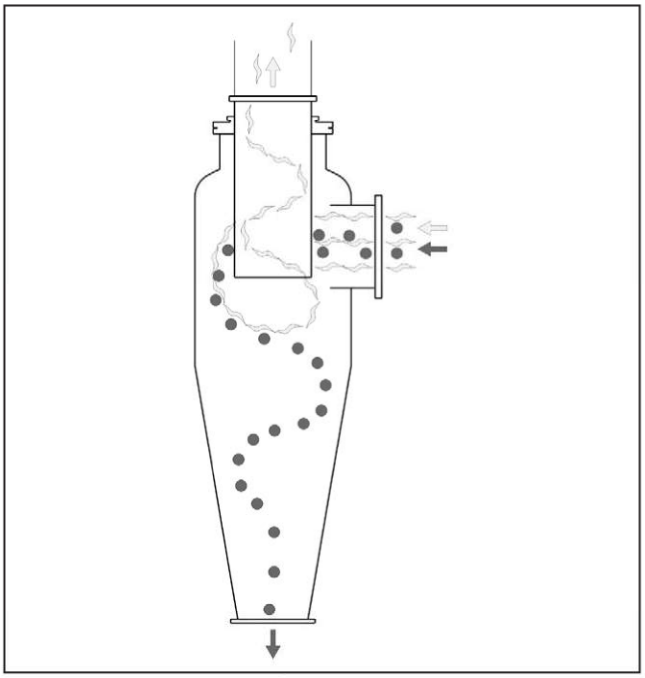


Figure 24 Gas cyclone configuration

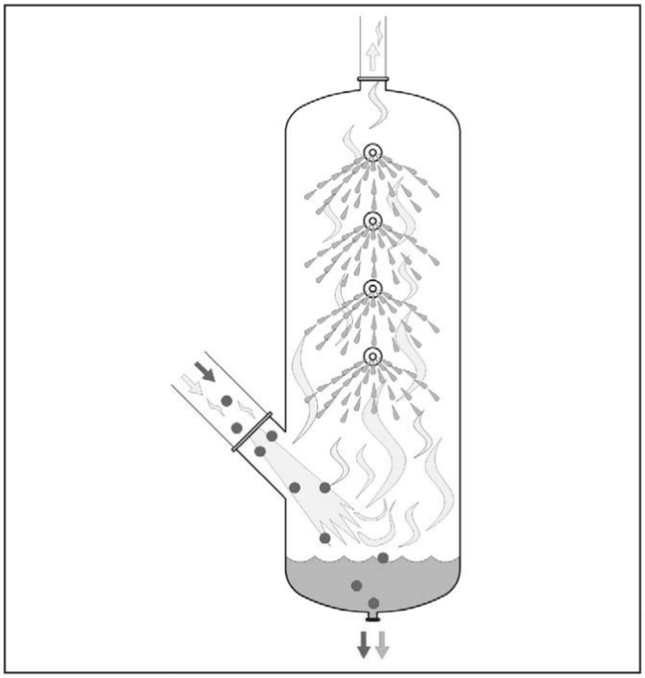


Figure 25 Condenser vessel example

as the potential for any component of the quench media to be stripped by the gas and emitted from the process.

Scrubber systems. High-energy wet scrubbers typically provide a final stage of vent gas cleaning (>99% particulate removal) prior to the gas being discharged to the atmosphere. In the wet scrubber, the remaining gas from the condenser system comes into contact with water, typically at a rate of 1.3 L/m^3 of gas, and is accelerated through a venturi and a 90° bend to promote the removal of particulate matter (see Figure 26). A pressure differential reduction of approximately 11 kPa is frequently required to provide adequate solids removal. A cyclonic separator body induces final separation of droplets from the vent gas. If required, a mist eliminator can be installed prior to gas discharge to recover residual condensed droplets; however, fouling of the mist eliminator and the necessity for cleaning should be considered.

Ideally, scrubber water should be relatively free of particulate matter and should be used in a single-pass system for best performance (i.e., not recycled). Recirculating the scrubber solution can be problematic with respect to fouling and scaling, similar to the condenser system. Any chemical interaction between the water and gas must be considered, as well as the potential for any component of the scrubber water to be stripped by the gas and emitted from the process.

Special systems. In some cases, process off-gas may contain contaminants that require additional treatment, as they are not effectively removed by conventional process equipment. Examples of such contaminants are elemental sulfur and mercury, which can be volatilized under high temperatures and oxidative conditions (Krumins et al. 2013). Elemental sulfur can be removed using condenser systems with slurry to promote agglomeration of sulfur particles (Fraser and McCombe 2011). Pressure oxidation processes have used carbon-bed technology for removal of mercury to meet regulatory emissions targets.

The necessity of any special treatment requirements should be assessed on a case-by-case basis from an analysis of the process feed material, pilot test off-gas analysis, or stack tests during operation.

Oxygen safety. Pressure oxidation and pressure oxidative leach process vents typically contain unused oxygen. Residual oxygen can concentrate as a result of condensing steam, increasing the risk of oxygen-enriched metal ignition (e.g., titanium). Therefore, where applicable, the process vent and downstream equipment design needs to consider selection of ignition-resistant materials of construction, appropriate instrumentation, and process controls such that the risk of oxygen-induced ignition is mitigated. An example of ignition risk mitigation is controlling the autoclave overpressure to an oxygen concentration of less than 35% using the measured autoclave vapor temperature and pressure (Frischmuth et al. 2014). Readers should refer to the best practices for oxygen safety and design principles from organizations such as the Compressed Gas Association and the European Industrial Gases Association.

Ancillary Systems and Services

Oxygen. Pressure oxidation and the pressure oxidative leach process typically require a supply of high-purity (>90%) oxygen. Depending on the quantity required, oxygen is either supplied as delivered liquid oxygen (for small plants and pilot work) or produced on-site from an energy-intensive (450–550 kW·h/t O_2) air separation plant. On-site production is most commonly from vacuum pressure swing adsorption (VPSA) or a cryogenic separation process, where the former is typically used for capacities less than 500 t/d. The VPSA process uses molecular sieves to filter out nitrogen, water (humidity), and hydrocarbons and typically produces a product at approximately 93 vol % O_2. The cryogenic process distills oxygen and nitrogen from air based on differences in boiling points and can produce oxygen at greater than 99% purity (McMullen et al. 2014).

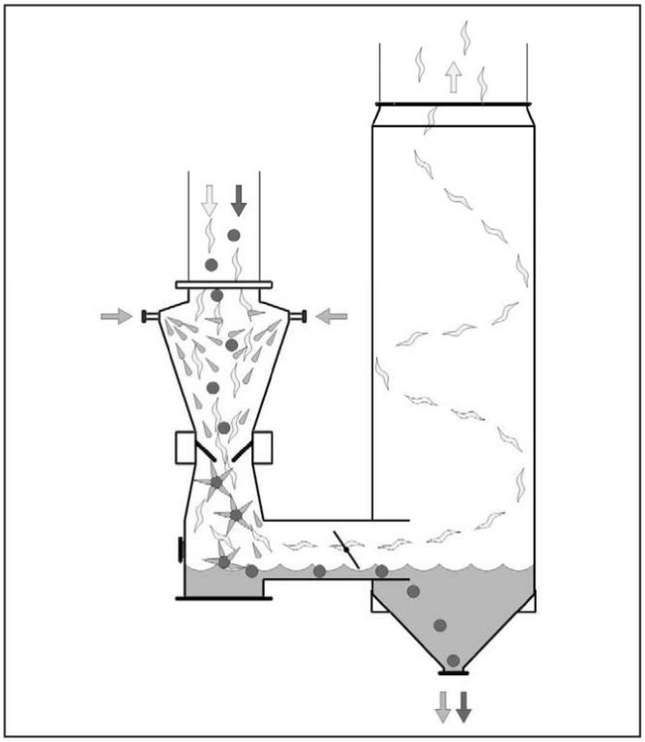


Figure 26 High-energy wet scrubber

Although the oxygen purities of both processes are relatively high, they are an important consideration for processes with sensitivity to oxygen partial pressure. The lower the oxygen purity, the higher the system operating pressure (and cost) must be to achieve the same oxygen partial pressure in the autoclave. Furthermore, additional impurities result in greater venting requirements to maintain oxygen partial pressure and increase the size of gas handling equipment.

Acid. Sulfuric acid is a principal reagent for acidic pressure leaching of laterites and can either be imported or produced by burning elemental sulfur in an on-site acid plant. On-site production has the added advantage of producing steam and power for the process. Acid injection to the reactor vessel is accomplished using tantalum-lined titanium dip pipes (lances) located to introduce the acid into well-mixed zones. The exothermic heat of dilution results in a temperature increase that affects material corrosion resistance; therefore, the lances are typically water cooled.

For pressure oxidation, most processes are self-sufficient in generating acid through the oxidation of sulfide minerals. However, in the presence of carbonates, pre-acidification of the feed may be necessary. This can be achieved through direct acid contact prior to autoclave feed, the recovery of acid from the autoclave discharge in a countercurrent wash circuit, or a combination thereof, depending on the sulfur-to-carbonate ratio of the feed material.

Services and utilities. High-pressure systems typically require a supply of water, air, and steam (continuous or intermittent) at a pressure exceeding that of the process. It is imperative that these systems be designed to avoid backflow of corrosive material from the high-pressure process.

Seal systems (e.g., for agitation) must be carefully designed to contain the process pressure and operated such that pressure cycling, wear, and corrosion of the seal components are minimized. Barrier fluids often use viscosity modifiers such as diethylene glycol or glycerin to maintain adequate lubrication at the seal mating faces.

Safety instrumented system. The ideal approach to reduce risk in a metallurgical process plant is to design safe and stable processes. However, because of the large quantity of potential energy inherent to pressurized processes, such as pressure leaching or pressure oxidation, reducing risk through elimination or substitution is limited, and engineering or administrative controls are often required. The identification and control of hazards is typically undertaken during the early stages of project execution using a hazard and operability study and layer-of-protection analysis based on the equipment design and process control developed during prior studies. If the residual risk from either activity exceeds acceptable levels, the additional protection of a safety instrumented system (SIS) may be required.

The SIS is a highly reliable instrumentation and control system with the sole purpose of monitoring critical process variables and initiating a controlled shutdown in the event of unsafe process deviations. An SIS, separate and redundant to the process control system, contains one or more safety instrumented functions, typically consisting of a field device (sensor) to detect an unsafe condition, a logical operation (logic solver) to initiate one or more specified actions, and a final control device (final element) to execute the specified actions (Pearson et al. 2014). The design, implementation, operation, and maintenance of the SIS follow internationally recognized standards such as IEC 61508 and IEC 61511/ANSI/ISA-84.00.

Materials of Construction

Table 8 highlights the typical differences in materials use for constructing the different types of processes. Pressure acid leaching has the highest relative cost per ton of material feed to the circuit due to the quantity of exotic (expensive) materials of construction needed for corrosion resistance at elevated temperatures and free acid concentrations of 25–50 g/L. This is followed by chloride-assisted oxidative leaching, which requires exotic materials that are resistant to chloride-induced stress corrosion cracking and pitting, although at a lower temperature and pressure. Acidic pressure oxidation (without chlorides) is the second-lowest relative cost, followed by alkaline pressure oxidation or caustic digestion, which use conventional carbon steels and 316 stainless steel.

General Cost Considerations

Capital costs. Capital expenditure costs for pressure hydrometallurgical facilities vary greatly with process conditions (pressure and temperature), materials of construction (equipment, piping, valves), and site location (site access, availability of skilled trades, supporting infrastructure, and site conditions). Much of the process equipment is designed for the specified plant throughput, with materials of construction that are specifically selected to be cost-effective yet suitable for unique process conditions. Such considerations include temperature, acidity/alkalinity, oxidation–reduction potential, solids abrasion, settling velocity, presence of multiphase flow, presence of flashing flow, presence of supersonic flow (such as vent gas depressurization), and mass transfer requirements.

Table 9 compares the direct capital costs of four pressure oxidation projects/studies completed by the authors between

Table 8 Typical materials of construction of pressure hydrometallurgical process facilities

Process	Pressure Acid Leach	Cl^--Assisted Oxidative Leach	Acidic Oxidative Leach	Alkaline/Caustic Leach
Relative Cost	**Highest** →			**Lowest**
Slurry Preheaters				
High-pressure vessel shell/head	C-Mn-Si steel	NA	C-Mn-Si steel	C-Mn-Si steel
Low-pressure vessel shell/head	C-Mn-Si steel	NA	Alloy 2507	C-Mn-Si steel
Membrane	Halogenated butyl rubber	NA	Vinyl ester resin	None
Refractory brick	Acid-proof fire clay	NA	Acid-proof fire clay	Acid-proof fire clay
High-pressure steam duct	Ti Grade 12	NA	Ti Grade 12	316 stainless steel
Low-pressure steam duct	Alloy 2507	Ti Grade 16	Alloy 2507	316 stainless steel
Interstage heater feed pumps	High-Cr iron	NA	CD4MCu	High-Cr iron
Autoclave Feed Pumps				
Diaphragm housing	Cast Ti Grade 3/5	CD4MCu	CD4MCu	Nodular cast iron
Check valves	Cast Ti Grade 3/5	CD4MCu	CD4MCu	Stellite-coated steel
Diaphragm	EPDM	Butyl rubber	EPDM	EPDM
Drop legs	Ti Grade 12	NA	Alloy 255	C-Mn-Si steel
Suction accumulator	Ti Grade 12	Alloy 2507	Alloy 2205	C-Mn-Si steel
Discharge dampener	Ti Grade 12	Alloy 2507	Alloy 2507	C-Mn-Si steel
Slurry strainers	Ti Grade 2/12	Alloy 2507	Alloy 2205/2507	C-Mn-Si steel
Reactor Vessels				
Vessel shell/heads	C-Mn-Si steel	C-Mn-Si steel	C-Mn-Si steel	C-Mn-Si steel
Membrane	Ti Grade 17—explosion welding cladding	Bituminous mastic	Homogeneously bonded lead or bituminous mastic	—
Refractory brick	NA	Silica/carbon/fire clay	Acid-proof fire clay	Acid-proof fire clay
Mortar	NA	Silicate/litharge-glycerol	Silicate/litharge-glycerol	Calcium salt
Oxygen/acid lances	Ta-clad Ti Grade 12	Alloy 255 or Alloy 2507	Ta-2.5W or Ta-clad Cu alloy	Inconel 625
Dip pipes/internals	Ti Grade 12	Ti Grade 16	Ti Grade 12	316 stainless steel
Vent piping/valves	Ti Grade 12	Alloy 59	Ti Grade 12/Ti-45Nb	316 stainless steel
Agitators	Ti Grade 5/12	Alloy 59/Ti Grade 12	Alloy 625/Ti Grade 12	316 stainless steel
Pressure Letdown				
Vessel shell/heads	C-Mn-Si steel	C-Mn-Si steel	C-Mn-Si steel	C-Mn-Si steel
Membrane	Halogenated butyl rubber	Bituminous mastic	Vinyl ester resin	—
Refractory brick	Acid-proof fire clay	Acid-proof fire clay	Acid-proof fire clay	Acid-proof fire clay
Mortar	AR-20-C vinyl ester	—	VET94 vinyl ester	—
Letdown valve body	Ti Grade 12	Ti Grade 12	Ti Grade 12	Hastelloy C
Letdown valve trim	Ceramic SiC	Ceramic SiC	Ceramic SiC	Ceramic SiC

NA = not applicable

2012 and 2016 that are representative of various types of processes, plant sizes, and site locations. All costs exclude indirect project costs such as engineering, procurement, and construction management services; construction equipment and services; camp costs; freight, transportation; and owner's costs and exclude any normalization for escalation, currency exchange, bulk material unit rates, labor rates, or labor productivity. As shown in the table, unit capital costs per metric ton of feed vary widely from project to project. An alternative metric is the unit capital cost per ton per day of sulfide sulfur oxidized by the facility (US\$ per t/d S^{-2}). However, these unit costs also vary considerably depending on the infrastructure, buildings, utilities, and electrical services needed to support the process facility, and Table 9 illustrates this variability.

Building costs (local excavation, fill, concrete, architectural, structural steel) for pressure hydrometallurgical facilities vary according to the area and volume of process buildings. For prefeasibility level studies, these can be estimated parametrically using a standard unit cost per building area (US\$/m^2) for building foundations and containment areas and a standard unit cost per building volume (US\$/m^3) for architectural and structural steel in specific locales, based on wind and snow loads, seismic zone, and ambient temperature range.

The direct cost of piping and valves for pressure acid leach and pressure oxidation facilities is generally 45%–55% of the installed mechanical equipment cost. A significant proportion of this cost is for severe service ball valves used for autoclave vessel and service isolation. The cost ratio is more valid for a range of design pressures from ASME Class 300 to 900, as the cost of piping, valves, and pressure equipment is equally affected by design pressure.

The cost of low-voltage (<600 V) and medium-voltage (4.16–13.8 kV) three-phase electrical systems varies as a function of connected load from US\$800–US\$1,000 per kilowatt. High-voltage systems (>38 kV) with redundant power supplies (substations, transformers, and switchgear) vary from US\$1,500–US\$2,000 per kilovolt-ampere of connected load; therefore, it is important to define the available supply voltage

Table 9 Comparison of direct capital costs of pressure oxidation facilities

Design Consideration	Project 1	Project 2	Project 3	Project 4
Environment	Tropical, highlands	Temperate, mountains	Arctic Circle	Arid, high elevation
Design oxidation capacity	High	Medium	Medium	Low
Feed to pressure oxidation plant	Whole ore	Concentrate	Concentrate	Concentrate
Unit capital expenditure, US$ per t/d run-of-mine	13,000	6,000	6,000	11,000
Unit capital expenditure, US$ per t/d S^{-2}	190,000	130,000	580,000	330,000
Distribution of Costs by Category, % of Total Direct Cost of Pressure Oxidation Facilities Only				
Earthworks, mass excavation, %	Excluded	0.2	0.6	0.1
Local excavation, fill, concrete, %	1.7	2.9	9.5	7.1
Architectural, %	3.5	2.5	0.7	0.5
Structural steel, %	5.7	7.9	13.4	5.9
Mechanical equipment, %	51.6	55.1	42.2	36.9
Piping and valves, %	24.4	28.6	17.3	28.5
Electrical systems, %	8.6	2.5	12.1	18.4
Instrumentation and control, %	4.5	0.4	4.0	2.6

and capacity when preparing the scope of work for an electrical estimate.

Instrumentation and control system costs vary with the type of control system (distributed control system or programmable logic controller), field communications network (analog, digital, HART, Fieldbus Foundation), and input/output (I/O) count. A typical pressure hydrometallurgy circuit has 100 control loops and 1,500 discrete I/O points per train. The average installed unit costs in the second quarter of 2016 were $1,500 per control loop and $1,000 per discrete I/O point, including control hardware, cable and tray, termination, continuity check, calibration, and software programming.

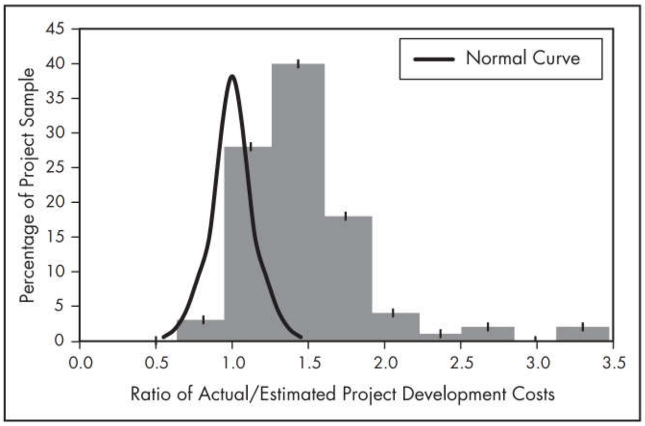
A 2016 study of construction cost overruns on mining and metallurgical projects identified a strong direct correlation between cost overruns and project duration and weaker indirect relationships between cost overruns and project size (plant capacity), the presence of local skilled trades, and existing infrastructure (Pearson et al. 2016). The study examined the ratio of actual project completion costs to estimated project development costs for a sample of 98 mining and metallurgical projects completed between 1997 and 2015. The frequency distribution of project overruns generated from this study is shown in Figure 27. The outcome of this study reaffirmed the historical norm, showing a shift in the mean of the sample distribution (1.35) from that of a normally distributed population (1.0), implying a systematic bias toward underestimating project development costs. One possible explanation for this is that although the number of unit operations in these metallurgical facilities is comparable to that of the chemical process industry, they can be more complex because of the natural variation in the plant feed. These facilities require a great deal more custom engineering and more detailed execution planning due to the remoteness of the host mine sites. Hence, they are more susceptible to cost overruns associated with procurement and construction.

Oftentimes, the cost overrun can be attributed to the underestimation of bulk quantities such as excavation, engineered fill, concrete, steel and cable, and the labor hours associated with their movement and installation. This growth in bulk quantities can occur at several stages of a project: between basic engineering and detailed engineering due to better definition of vendor-supplied equipment; between completion of the project estimate and completion of purchasing activities due to revised take-offs from detailed engineering drawings; and between completion of bulk material purchasing and construction completion due to material loss, overpour, and wastage. Estimators typically include a growth allowance of 5%–20% for bulk materials in their project cost estimates, depending on the level of engineering and procurement definition at the time the estimate is prepared to account for growth in the bulk quantities.

Operating costs. Pressure leach and oxidation circuits are complex, and the operating costs are often notably higher than those of other mineral processing circuits. The comparatively higher cost is primarily associated with labor, maintenance, and reagents. Nonsulfide pressure leach circuits also require significant energy input to sustain the operating temperature, which is why large facilities are often integrated with a power plant or an acid plant to generate steam at a substantially lower cost than using fuel sources (e.g., diesel). The energy costs for a Bayer process typically represent approximately 35% of overall operating costs (Gorst et al. 2013).

Operations and maintenance labor costs often account for a significant proportion (>20%) of the operating costs for a pressure leach or oxidation facility. The costs are mostly due to the additional labor count required to manage, operate, and maintain the circuit compared to other mineral processing or hydrometallurgy facilities (e.g., a flotation concentrator or free-milling gold leach plant). Specifically, dedicated personnel include technical staff (e.g., metallurgists), area operations supervisors, control room operators, and a mechanical maintenance team. Electrical, instrumentation, process control, and reliability maintenance support are usually spread throughout multiple areas of the facility; however, additional personnel may be required to adequately support the pressure leach or oxidation circuit. Labor costs for a Bayer process are typically a lower proportion of overall costs—between 5% and 10% (Gorst et al. 2013). A project-specific labor plan and estimate of the labor rates are required to establish requirements and develop a cost estimate.

Pressure oxidation and pressure leach circuits typically include specialized process valves with exotic construction materials that increase regular maintenance consumable costs. A factor of 3.5%–4% of the total direct cost for the circuit is



Source: Pearson et al. 2016

Figure 27 Distribution of capital cost overruns for 98 mining and metallurgical projects (1997–2015)

often applied when estimating the maintenance consumable costs. The material cost is independent of contractor costs that are mostly associated with the annual or biannual shutdowns and an influx of personnel to complete the necessary vessel cleanout, refractory repairs, inspections, rebuilds, and other routine maintenance activities.

The reagent consumption and costs for pressure leaching and pressure oxidation circuits are mostly associated with the use of sulfuric acid, sodium hydroxide, or oxygen. Minor reagent consumption is associated with mechanical seal lubricant and demineralized water. Higher water treatment costs will be incurred if a boiler is required for continuous operation.

Sulfuric acid is consumed in acidic pressure leaching of nickel laterites, slurry conditioning for pressure oxidation, and pressure leaching of select base metals. An acid unit consumption of 200–500 kg/t of dry ore is a typical range for nickel laterite leaching (Kyle 1996), 25 t/t of nickel product (Crundwell et al. 2011), and up to approximately 75 kg/t of sulfuric acid (100%) for acidification ahead of pressure oxidation (Thomas 1994). The unit price of sulfuric acid is linked to global sulfur and fertilizer demand and can be quite volatile, ranging from US$50/t to more than US$150/t (excluding shipping). Operations typically arrange long-term supply contracts from sulfur and sulfuric acid producers such as copper smelters and oil refineries to ensure supply and reduce supply cost volatility.

Sodium hydroxide is predominantly used in the digestion of bauxite ores via the Bayer process and represents approximately 15% of operating costs (Gorst et al. 2013). The sodium hydroxide unit consumption is highly dependent on the reactive silica content of the ore but is ~100 kg/t of ore based on current minimization and recovery practices (Smith 2009).

High-purity oxygen (>90 vol %) is predominantly used for pressure oxidation and pressure oxidative leaching. The oxygen unit consumption is based on the oxidation reaction stoichiometry and efficiency. For a pyrite (FeS_2) feed material, the stoichiometric addition rate is ~1.9 kg O_2/kg S. The stoichiometry changes with the presence of minerals such as arsenopyrite (FeAsS) and stibnite (Sb_2S_3) or other sulfides. The oxygen usage efficiency (utilization) is the approach to the stoichiometric requirement and is dependent on oxygen mass transfer, sulfide reactivity, and the presence of noncondensable gases that increase the reactor vent rate. The use of high-purity oxygen reduces the noncondensable nitrogen content and improves oxygen utilization, although less significantly than reducing the carbonate content in the reactor feed. Typical oxygen utilization ranges from 85% to 95%; however, it can be less than 80% in the presence of carbonates.

The production of high-purity oxygen for use in oxidation processes typically requires 450–550 kW·h/t of oxygen, which corresponds to a unit cost of US$45–US$50 per metric ton at a power price of US$0.10/kW·h. This equates to US$94–US$104 per metric ton of sulfide sulfur in pyrite, assuming 90% oxygen utilization. In a sale-of-gas arrangement (i.e., the oxygen plant is owned and operated by others—sometimes referred to as "over the fence"), the unit cost of oxygen is often higher due to a monthly facilities charge, which is often a primary means for the vendor to obtain a return on capital and limit financial risk. The ability of a vendor to recover and sell nitrogen and argon may reduce the monthly operating fee; however, most mineral deposits are not located near other industries, so opportunities are limited, and supply costs are often elevated for this reason.

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