

PREVENTION OF ACID MINE DRAINAGE BY AVOIDING ACID FORMATION THROUGH PRODUCT RECOVERY WITH F-LLX™

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ABSTRACT

Acid mine drainage (AMD) water from abandoned flooded mines becomes acidic on air contact and precipitates unsightly orange solids in streams, harms wildlife, and resists purification. Current AMD water treatment processes generate large volumes of solid hazardous wastes and leave the water with toxic sulfate contamination. However, these components are useful commodities of increasing demand (Fe, Al, Zn, Ni, Co, Mg). Quality water and fertilizer demands are also rising worldwide. Barriers to AMD treatment are huge: medium TDS (thousands of mg/L), large flows (500-50,000 L/min), and high $\text{SO}_4^{=}$ concentrations. Slow reacting metals and low value per liter raise equipment size and costs due to long processing times. Thousands of kilometers of rivers are continuously contaminated worldwide with projections to thousands of years. We have found that Flotation Liquid Liquid Extraction (F-LLX) technology can cost effectively purify most coal mine pool water by purifying in ~100% yield, with minimal waste generation, while recovering metals as processable gypsum-free concentrate, and produces sulfate co-product values useful to fertilizers. This paper describes the F-LLX process and the identification/validation/demonstration program, including continuous bench scale piloting, a 40-120 L/min field demonstration, including process chemistry and flow diagram, products, engineering assessment, scalability, and water product.

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INTRODUCTION

Acid Mine and Acid Rock Drainage (AMD/ARD) seriously degrade natural streams in the coal mining states of Pennsylvania (PA) and Ohio (OH) in the USA (>10,000 km). Similar situations exist in the Western USA and many areas of the world. In PA, 144 major (up to 38,000 L/min) AMD sites exist with more projected as abandoned mines continue to fill and overflow from ground water intrusions. Active mines avoid AMD by treating such seepages in conventional processing by pH adjustment and flocculation of metal hydroxide/gypsum sludge for landfill disposal resulting in creation of toxic waste tailings “ponds” and landfills. Unfortunately, the current liming process leaves the water clear and colorless but still unusable due to its very high $\text{SO}_4^{=}$ content. It is desirable to avoid producing these wastes, wasting the useful metals in the wastes, losing the land fill and pond real estate for their storage, and wasting scarce state funds used to create them. These annual costs will be required for hundreds of years to come. On the other hand over 99% of the AMD TDS content consists of useful commodities in growing demand worldwide: land, water, metals (Fe, Al, Mg, Mn), and S (as $\text{SO}_4^{=}$), with lower levels of higher values (e.g., Ni, Co, Zn and Cu). AMD/ARD represent mineral sources that have already been mined, milled, leached to yield dissolved values ready for rapid automated processing into concentrates using small foot print operations paid for by water, land and sulfate products. Critically, $\text{SO}_4^{=}$ must be removed from the water to < 600 mg/L for discharge, but preferably to < 250 mg/L (potable water) and still lower for industrial operations. To date economical and effective $\text{SO}_4^{=}$ removal from water at the large scales needed has not been available. This paper presents a field demonstrated technology that appears to meet this need.

The Pennsylvania Department of Environmental Protection (PA-DEP), looking for an economical alternative treatment technology for the AMD problem that produced clean water and metal products instead of wastes for disposal, contacted Winner Global, LLC. Winner Global then approached Battelle in their search for an alternative technology. PA-DEP provided the following requirements for the desired process: The technology must: 1) purify the water to US EPA surface water dischargeable requirements with respect to a broad spectrum of toxic metal ions, and remove the very high concentrations of $\text{SO}_4^{=}$ down to US EPA surface water dischargeable requirements (primary and secondary standards), 2) convert these commodities into product concentrates that could readily be used, preferably sold, to industry and agriculture, 3) eliminate waste water production by providing a very high (e.g. 95-100%) water recovery yield as useful and dischargeable water, 4) eliminate the current massive and continuous solid waste sludge formation and disposal characteristic of conventional “Treatment” systems, 5) be scalable to large flows (500-50,000 L/min) with a relatively small physical footprint, 6) operate the process at cold (ground water) temperatures, with variable feed water composition, handle trace organics expected from coal mine pool waters, and yield product water of dischargeable pH (5-9). In prior work Battelle confirmed that more economical sulfate removal technology was needed worldwide for AMD/ARD water which presents the following challenges: 1000-5000 mg/L TDS, high Fe (hundreds of mg/L), and > 2000 mg/L $\text{SO}_4^{=}$ making conventional treatments ineffective, expensive, and/or impractical.

Technology need

Sulfate ion has proven very difficult to remove practically and economically from AMD [1]. This technical difficulty arises due to its high flow rates of medium to high concentrations of $\text{SO}_4^{=}$, ($[\text{SO}_4^{=}]$ >600 to several thousands of mg/L). The technology need is to reduce the $[\text{SO}_4^{=}]$ low enough to enable surface discharge of the water, < 600 mg/L, but preferably lower still to enable its

use for potable water (< 250 mg/L), and most desirably to < 100 mg/L for regions of poor ground and surface water quality where purer water is of value to prevent “sulfur” odors, mineral scaling/staining, and sulfide corrosion of hardware. The difficulty in removing $\text{SO}_4^{=}$ from water is due to the high charge density of $\text{SO}_4^{=}$ ion that provides favorable hydration energy thermodynamics. Only the minerals barite, jarosite, gypsum and ettringite offer medium to low sulfate solid solubilities for consideration as precipitation agents for sulfate ion but all have serious limits in the case of AMD purification [1].

Water recovery yield is also a serious problem since large water flows are involved and will flow unabated for hundreds of years so any small but continuous waste stream flowing from a treatment operation will and has caused significant environmental problems. Reverse osmosis, RO, is hampered by cost, membrane fouling, and low purified water yield [1]. Water purification rate is another major consideration due to the large mine pool flow rates involved. Bioprocess swale areas and granular limestone filled trenches require weeks of water retention time that result in very large process foot prints with expensive real estate requirements and maintenance [1].

AMD value extraction process (VEP) using F-LLX: program overview

Winner Global contracted Battelle to identify technology to meet the above AMD VEP need. Battelle performed an 18-month phased program that started with lab scale concept generation and ended in a successful 40-115 L/min Field Demonstration. Phase I (Concept Evaluation) used a hydrometallurgical process chemistry science approach to identify an efficacious concept for AMD water prevention with VEP features that met the downselection criteria with a single unit operations. Phase II evaluated and selected a range of key design parameters including extractant formulation, flow configurations (residence time and phase ratios), water balance control, pH control and other process control set points, identification of VEP product candidates, materials of construction confirmation, and upset causes and recovery means. The simplest field operable process configuration [Flotation-Liquid Liquid Extraction (F-LLX)] (Figure 1) was taken forward for technology demonstration purposes at a closed mine site. This configuration was first operated at extended steady-state bench top scale pilot trials (Phase III) to refine the mass balance and VEP product composition data set to use for Preliminary Engineering Assessment and needed to modify conventional hardware that was available for a field demonstration. Phase III demonstrated the F-LLX process at continuous bench pilot scale at steady-state and 200 mL/min mine pool feed water flow (Table 1). Phase IV was the Field Demonstration. An inactive coal mine pool water at St. Michael, PA provided the mine pool water for this Field Demonstration. The three organizations then collaborated to demonstrate the process capability at field conditions at 40-115 L/min. This demonstration showed that detailed equipment design work for a commercial plant (at least 400-2000 L/min) is justified and of minimal technical risk. This scale up design is underway by Winner Global, LLC. (Phase V).

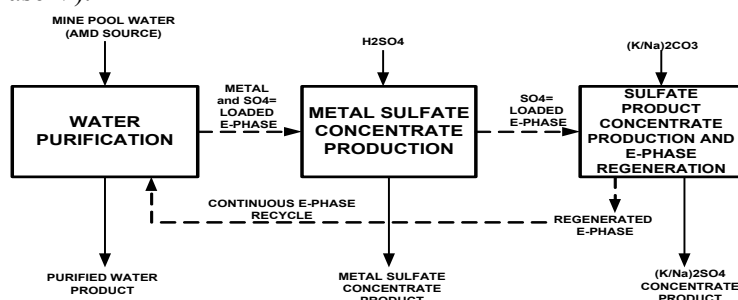


Figure 1: AMD VEP™ F-LLX™ process block flow diagram

METHODOLOGY

AMD VEP™ F-LLX™ process chemistry identification: general description

The AMD VEP™ F-LLX™ process [3] uses “forced ion pairing” (FIP) using integrated thermodynamics chemistry approach to quickly and simultaneously remove cations and anions from mine pool waters. (The term “integrated thermodynamics” chemistry was selected to indicate process conditions that involve three chemical reactions coupled energetically so the favorable free energy changes of at one reaction drives other reactions with small or unfavorable free energy changes). Ferrous sulfate is the bulk of the mass to be removed [2] (Table 1). With FIP sulfate ion is forced to partition into a non aqueous phase of a liquid-liquid extraction (LLX) system to the extent that the sulfate ion concentration, $[\text{SO}_4^-]$, in the extracted feed water (raffinate) is reduced to 20-30 mg/L and the Fe and other toxic cations are reduced to <0.25 mg Fe/L as required for drinking water purposes. F-LLX process hardware maximizes commercial-off-the-shelf (COTS) hardware to enable rapid and low cost deployment of the technology.

Table 1: Typical Mine Pool Performance Targets and Results from Phase III Bench Scale Pilot Testing

AMD Feed	Coal Mine Pool Water Feed (across four sites)	Target	Results
SO_4^-	2000 mg/L	250-500 mg/L	<30 mg/L
Fe	200 mg/L	<3 mg/L	<0.25 mg/L
Al	1-200mg/L	<1 mg/L	<0.2 mg/L
pH	2-5	6.5 to 8.5	7.5 to 7.8
O&G	0	<100 mg/L	<10 mg/L

Physically, the key process step of F-LLX involves mixing the mine pool aqueous solution with a specially formulated, nonflammable, water immiscible, extractant phase (“E-phase”) in mixer-settler hardware modified to handle the unique conditions of F-LLX.

Three thermodynamically integrated sets of chemical reactions occur simultaneously during this short (45-90 sec) mixing time to cause sulfate ion (SO_4^-) and toxic metal ions, using anionic colloid/floc process chemistry, to become associated with the neutral E-phase (Figure 1) within a few minutes and thereby purifying the water with respect to these and similar anions and cations, and thereby significantly lowering TDS. During this step, flow rate ratios are used to concentrate these values by 400-600%. Even higher concentration factors are reached during the concentrate isolation step by internal recycle. In this manner the water impurities are not only removed from the water to the extent that the water is reusable or dischargeable, but are simultaneously concentrated by large factors so that values are concentrated and separate sulfate and metal sulfate products of value are co-produced with lowered TDS water. Significant solid or liquid wastes do not form. Critically, these concentration factors are achieved in minutes, which has the impact of providing a small site footprint. To maximize process efficiency most working capital (E-phase) is kept continuously flowing either purifying water or generating concentrate products.

The E-phase formulation is manipulated by the fluid handling hardware to provide neutral oxide/hydroxide metal ion colloid formation with simultaneous $\text{SO}_4^{=}$ extraction required for charge balance. This synchronization of chemical reactions is robust and solids tolerant and the key to the low cost of the AMD VEP process. The E-phase consists of a homogenous formulation of extractant, modifier, carbonate ion, and hydrocarbon diluent. These hydrometallurgically proven compounds are water immiscible, nonflammable, low cost, and not discharged to the environment since they are retained within process hardware. Low polarity and viscosity of the E-phase formulation drives rapid phase separation when mixed with mine pool water. The extraction chemistry is very fast ($\sim 45\text{-}90$ sec) and removes $\text{SO}_4^{=}$ using a liquid quaternary ammonium ion, R_4N^+ (Aliquat® 336, Cognis, Inc.). Although water immiscible liquid R_4N^+ extractants are useful for extracting a number of anions, $\text{SO}_4^{=}$ is only poorly extracted. Also, for mine pool water purification the TDS needs to be lowered markedly to achieve the goal of producing a useful water product. Hence liquid ion exchange (LIX) is not useful. Therefore, for the AMD VEP process it was necessary to modify the process chemistry such that *three* very fast primary chemical reactions occur simultaneously in the same liquid-liquid contactor reactor integrated thermodynamically: carbonate raises pH and converts to CO_2 gas, anionic oxo/hydroxide metal ion colloids form rapidly at the higher pH that associate with the cation present, R_4N^+ , leaving ion pairing between $\text{SO}_4^{=}$ and the R_4N^+ to occur. This concerted reaction is illustrated in Figure 2.

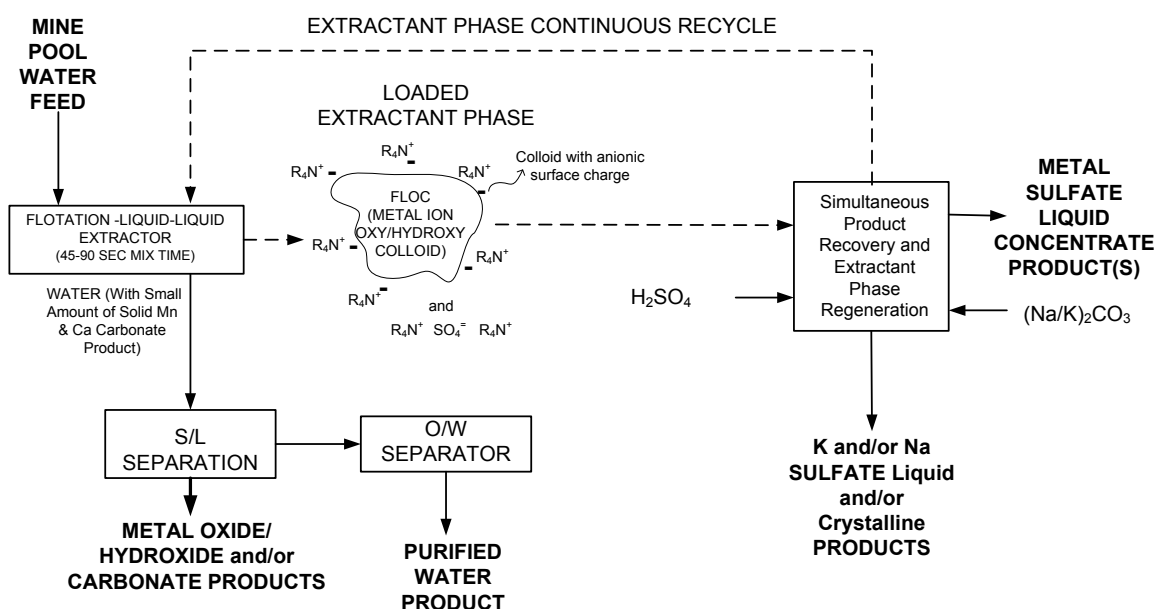
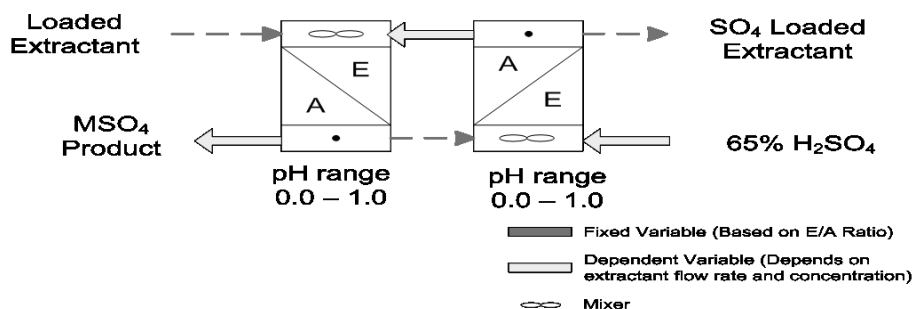


Figure 2: F-LLX VEP Process chemistry schematic illustrating “forced” $\text{SO}_4^{=}$ extraction by integration of thermodynamic driving forces of precipitation, flocculation, CO_2 gas evolution, and charge neutralization

TDS reduction in mine pool water (prevention of AMD/ARD water)

F-LLX is able to reduce the TDS levels of waters for essentially 100% of the mine pool water (no waste water is produced) by simultaneously co-extracting the anions and cations present in the water. In this manner, F-LLX achieves low cost deionization at very high flow rates. These ions are captured from the aqueous solution in a water immiscible phase, the “extract” (E-phase). For coal mine pool water, the cations removed by F-LLX are normally ions of useful metals consisting of a group from the following list (highest quantity in bold): **Fe**, **Al**, Cu, Ni, Ag, Zn, Co, Ca, **Mg**, Cd, **Mn**, Pb, Hg, and Cr, where Fe, Mn, and Al represent metal ions with the needed colloid forming

capability and present in high enough concentration to co-aggregate other trace metal ions to form negatively charged colloids. These are used to produce the colloidal floc of the extract. The primary anion as previously mentioned is $\text{SO}_4^{=}$. The F-LLX process also adjusts the pH of the water into dischargeable range as these ions are extracted. If present, Ca is removed as calcite particulate. When this pH change occurs, the transition metal cations listed above hydrolyze into colloidal oxo/hydroxo compounds to coagulate with those of Fe, Mn, and Al. These ions form amorphous or highly open metal hydroxide gel absorbents, which in turn are excellent scavengers of trace metal ions. Such metal hydroxide coagulants are widely used in the industrial water purification industry for this purpose. It is important that the colloid be removed before the gel-like structure dehydrates



(collapses) too far and thereby loses some of the absorbed ions held within. With AMD VEP this colloid is produced with a small negative charge so it partitions into the E-phase and can be quickly separated from the water phase, which in turn is purified. This colloid layer is not allowed to proceed to conventional very slow coagulation and flocculation. Instead the E-phase with colloidal component is fed immediately to the metal ion (Figure 3) and sulfate (Figure 4) strippers that use sulfuric acid to reverse the colloidal chemistry and aqueous carbonate to regenerate the extractant. By internally recirculating this acid and carbonate the metal ions and sulfate concentrations build to many times their original concentrations forming separate products.

Figure 3: Metals stripping process flow schematic; phase III data at steady-state

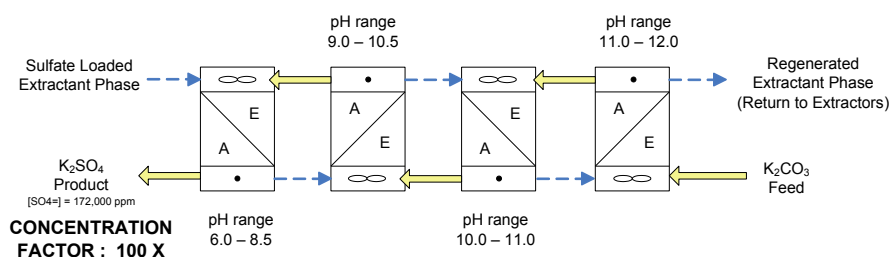


Figure 4: Sulfate product recovery with extractant phase regeneration process schematic

Like any LLX operation, it is important to maintain the E-phase within the process equipment for F-LLX, as well as using conventional means such as water/oil separators, traps, and phase splitters, and avoiding the use of high shear mixing. The most important control of E-phase retention within the system is to pass the purified water through an effective oil/water separator and recycling the collected E-phase. Depending on the fate of the purified water, odor sorbent treatment may also be desirable. TOC levels in process raffinate should always run $< 10 \text{ mg/L}$ (as oil and grease).

Bench scale pilot plant: refinement and design data collection for field demonstration

The objectives of Phase III testing included the following: 1) Refine the AMD F-LLX purification system process configuration and control, 2) Operate the system continuously for three weeks while meeting stated performance objectives, 3) Refine the use of process chemicals, 4) Maximize the system production rate, 5) Characterize the system performance with respect to process controls, 6) Develop preliminary operating procedures for start up, shut down, and troubleshooting for the 200 mL/min pilot-scale unit. The scope of Phase III also included collecting critical process operational information relevant to the design of the Phase IV field-scale Demonstration plant, including: 1) The required number of extraction, decanter, and recovery/E-phase regeneration stages, including the justification for them. 2) The needed extractant/aqueous (E/A) flow rate ratio for the extraction stages, with estimates for the decanter and product recovery units. For the recycle units, an internal recycle flow rate range is also needed. 3) Extractor mixing and settling times, and a range of acceptable mixer tip speeds. 4) The rate of process chemical usage (sulfuric acid and Na/K carbonate soda ash). 5) Product characterization, especially purified water, metal sulfate concentrate, and sodium sulfate concentrate. Characterization of any solid product(s) produced. 6) Identification of any waste streams generated and, if so, their composition, fate, and flow rate.

RESULTS AND DISCUSSION

Determination of design parameters

Sulfate Ion Extraction Range Determination

To evaluate $\text{SO}_4^{=}$ removal in $\sim 98\%$ yield or to < 250 mg/L from mine pool water it was useful to first screen a synthetic surrogate AMD feed water based on Na_2SO_4 . The sulfate loading efficiency, achievable raffinate residual $[\text{SO}_4^{=}]$, and achievable product $[\text{SO}_4^{=}]$ were determined using McCabe-Thiele diagrams for extraction and stripping for the $[\text{SO}_4^{=}]$ expected for mine pool feed waters. Therefore it was determined that four extraction stages could effectively take the $[\text{SO}_4^{=}]$ down to 50 mg/L in the Extraction operation and also be fully regenerated in 4 strip stages. It was necessary to establish conditions such that sulfate concentrate product solubility limits are not surpassed.

In Phase III, continuous operation of the bench pilot-scale F-LLX process at steady-state conditions for three weeks was performed to demonstrate robustness, evaluate the performance in detail, and refine process configuration, controls, and operational procedures for startup, operation, shutdown, and upset recovery. The process was operated through daily start-up and shutdown cycles and was found to exhibit rapid on/off capability.

Therefore, these specific tests, targeting dischargeable water as the product, indicated that the composition of the solution met the required discharge standards with respect to residual $\text{SO}_4^{=}$ and toxic metals, and probably could be treated using an extended F-LLX process, to achieve drinking water standards.

The test results were used to generate a set of recommended design and operational parameters for field testing including the projected consumption of process chemicals and the production rate of major products. These products were characterized and met the expected process performance criteria. Operating procedures for start-up and shutdown of the 200 mL/min pilot-scale unit also were developed.

Field Demonstration

With the pilot success, the work proceeded to on site Field Demonstration. Demonstration is required to justify the much greater cost of process optimization needed for the engineering design of commercial plants, expected to range from 400 to 40,000 L/min mine pool water feed. Battelle already possessed substantial LLX contactor equipment, tanks, and pumps which were assembled into a functional F-LLX configuration at Battelle's West Jefferson, OH facility (Figure 5). This equipment was not ideal for the process but it represented a low-cost means to quickly exhibit and reduce the risk of the technology (Figure 6). The flow rate objective was about 40-120 L/min of mine pool water with an optimal steady-state operation at about 60-80 L/min and where the main throughput bottleneck were an extractor aqueous overflow stage transfer line. Air introduction wasn't controlled in the extractors as it was in the pilot tests allowing more ferric iron formation than necessary resulting in an undersized mixer-settler for the first metal sulfate production unit. The extractors were oversized relative to the rest of the hardware (capable of 180 L/min hydraulic flow). The mine pool water to feed the trailer mounted facility was drawn from a shallow well drilled into the pool specifically for that purpose at the inactive coal mine. The operation consisted of green field site preparation, construction and delivery of a trailer mounted facility, start-up, and then operation as three 10-day field trials or "campaings."



Figure 5: Portable 40-117 L/min AMD VEP™ F-LLX™ field demonstration unit

Figure 6: Field demonstration plant in operation

Demonstrated Results

The Field Test was conducted in October through December 2007. Total operational time was 800 test hours and total volume of mine pool water processed was 1.1M gallons at mine pool feed water at flow rates ranging from 40-120 L/min. This Field Test demonstrated that raffinate purity produced from the AMD VEP Demonstration Plant was suitable for potable water production plant feed. Specifically, we achieved oil and grease (O&G) target of ≤ 10 mg/L with negligible extractant amounts lost with purified water (easily removed with granulated activated carbon) in conventional surface water fed municipal/ industrial potable water production plants; maintained produced water product of pH 7.4 – 7.8; demonstrated effective contaminant removal in less than optimally designed contactors; residual Sulfate = 120 to 580 mg/L (50 – 89% removal and all dischargeable to surface stream); readily achieved <250 mg/L steadily when system was running as designed (Note: Bench-scale unit demonstrated sulfate residuals of 20-30 mg/L); residual iron was 5 to 15 mg/L for the Field unit (89 – 96% removal). This residual was significantly higher than the pilot testing and was traced to too short a residence time in the metal sulfate strippers caused by higher than ferric ion content, which caused by excessive introduction of air into the extractor contactors (Figure 7). This was remedied in follow-on operations by introduction of a larger metal ion stripper to enable full recovery of the ferric and ferrous iron in the extract. Properly sized strippers reduce the iron level to <0.3 mg/L. (Note: Lab- and bench-scale units demonstrated routine iron residuals of <0.25 mg/L). This adjustment also removed the iron residual from the plant's process water (taken from the raffinate) and therefore the iron rust coloration from the potassium sulfate product.

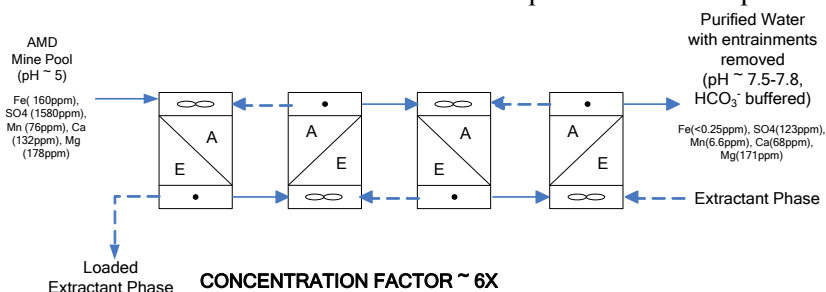


Figure 7: Extraction process flow schematic with mass balance; phase III data at steady-state

The production of potassium sulfate was very robust. The Liquid Concentrate assayed 10% K_2SO_4 , the solid Filter Cake: >93% solids with the crystals (Figure 8) being >98% pure K_2SO_4 (by XRD). Iron Sulfate, with other metals, was produced as a liquid concentrate of up to 6.7% as $FeSO_4$ (2.5% total Fe). This solution is an item of commerce if further purified. Process operating conditions yield maximum $FeSO_4$ concentration by maximizing iron as ferrous vs ferric form.

Figure 8: K_2SO_4 Salable Sulfate Product.

A comparison for F-LLX vs COTS Technologies for sulfate ion removal was made (Figure 9). Total Life Cycle Cost Estimates were then determined and these showed the substantial economic advantage nature of the F-LLX technology relative to alternatives (\$/1000 m³ AMD water treated): F-LLX (1600), Lime/Dewatered (4800), RO Membrane (13,000) and Bio Treatment (4200). Costs Include Capital. Limestone treatment alone is not included in this comparison since it does not remove $SO_4^{=}$, a critical requirement. Recently Winner Global has reduced the F-LLX capital costs much further.

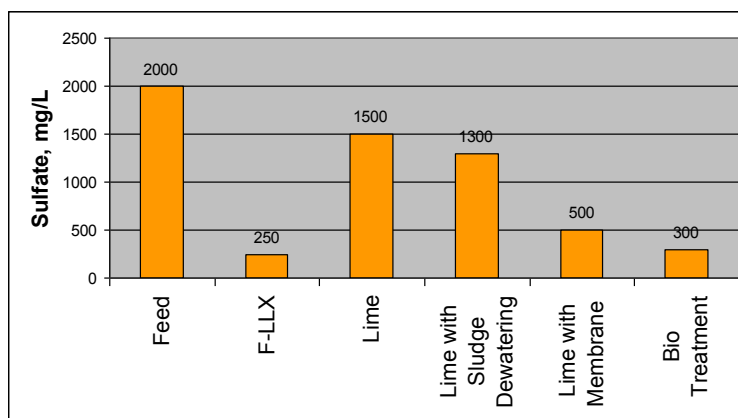


Figure 9: Sulfate Ion Concentration Reduction Demonstrated in Phase IV Field Testing Results and Engineering Assessment Comparison of F-LLX with current alternative technologies. Drinking water secondary standard is < 250 mg/L [$SO_4^{=}$] and discharge standard is < 600 mg/L [$SO_4^{=}$].

CONCLUSIONS

The transformation from AMD VEP™ F-LLX™ concept identification, through bench scale pilot testing, and then to field demonstration in 18 months was challenging but successful. Most process parameter set points were determined at the bench-scale pilot plant which produced purified water far surpassing Fe and $SO_4^{=}$ residuals targets of 3 and 500 mg/L, respectively, needed for discharge to surface waters. In fact potable level purities of these contaminants were met routinely at the bench scale (< 0.25 and < 30 mg/L, respectively). The Field Demonstration verified F-LLX

scalability to the 40 to 120 L/min range while producing dischargeable water purity and high yield water recovery effectiveness. AMD VEP F-LLX technology achieved simultaneous and fast water purification and extraction of metals and sulfate that are key to low cost and small footprint characteristics by using process chemistry that integrated LLX with flotation and precipitation chemistries that enable a small plant footprint through rapid process extraction and strip kinetics. In addition, it was found that AMD VEP based on F-LLX has an attractive life-cycle potential, it reduces the cost for $\text{SO}_4^{=}$ removal from AMD well below membrane alternatives, is cost competitive with lime treatment that needs land disposal of waste sludge, directs AMD would-be pollution into usable products, and provides high yield of purified water essentially without any waste production.

Features and benefits of F-LLX

F-LLX technology appears to have the potential for reduced plant size because of vastly improved kinetics and avoidance of a need for flocculation and clarification, multiple treatments of the feed water, or internally recycling the feed water. Processing times (from many hours in ponds/clarifiers to 45-90 sec contact time in mixers and uses weir overflow liquid transfers, giving a few hours maximum of water residence in plant) provide low power and emissions footprints. The fluid flow feature is expected to provide smooth scalability from < 200 L/min to 20,000 L/min (then replicate as needed). Critically, the high purity of the product water enables its use for many applications (industrial, potable plant feed, watershed/tourism restoration, and agriculture), with production of useful chemical co products without waste sludge.

Next steps

With the successful demonstration of the F-LLX technology at the bench pilot (0.2 L/min) and demonstration (40 to 120 L/min) continuous operation, the continued development, scale up and commercialization investments are justified. Refinement of plant design at larger scales at still lower capital costs has been accomplished. The immediate goal is to construct and operate a “first of its kind” AMD VEP commercial plant based on F-LLX technology. Commercial scale design of about (2,000 L/min) mine pool water flow rate is seen as a reasonable commercial scale and on the path to even larger needs of 20,000-40,000 L/min plants.

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