

FINAL TECHNICAL REPORT
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Project Title: **ELECTROCHEMICAL METHOD FOR CHLORIDE
REMOVAL FROM WET FGD LIQUIDS**

ICCI Project Number: 07-1/ER9
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ABSTRACT

The objectives of this project were to explore the operating characteristics of an electrochemical method to remove chlorides from wet flue gas desulfurization (FGD) slurries and to estimate the implications of those characteristics for chloride removal at a full-scale power plant. Experiments were conducted with a laboratory-scale system; the energy efficiency of chlorine separation was measured as a function of several parameters relevant to the operation of wet FGD systems. The key conclusion from this project is that the chloride removal system would provide the greatest recycled water benefit for FGD systems that must maintain low chloride levels (approximately 5000 ppm or less) to meet wastewater disposal requirements. In this situation, the chloride removal system could maintain low chloride levels, and the purge stream (or blowdown) flow could be reduced to the amount needed to regulate suspended solids and metals. A preliminary estimate of the capital costs for a 530-MW plant with a FGD slurry chloride concentration of 3500 ppm was \$21,000 per gpm of avoided wastewater treatment capacity. The concept appears feasible and would be a candidate for further development; however, given the high cost estimates of recycling and/or treating water with this approach, the market needs to be thoroughly evaluated before more development work is justified.

EXECUTIVE SUMMARY

Wet flue gas desulfurization (FGD) systems have become standard equipment for utilities consuming Illinois basin coal. While primarily intended to remove SO_2 , these scrubbers also capture HCl which is generated in significant quantities during the combustion of Illinois coal. The SO_2 absorbed in the scrubber is removed from the system as a precipitated solid, but in the recycle loop of a wet FGD system, there is no inherent mechanism to remove the accumulated chlorides, except for a periodic or continuous purge stream of high-chloride-content solution and replenishment with low chloride water. This project evaluated an exploratory concept for the direct removal of FGD slurry chlorides using an electrochemical method. The proposed concept is similar to other industrial electrochemical processes; however, there are differences specific to the characteristics of FGD slurries that have not been evaluated before.

Laboratory-scale experiments of direct chloride removal from simulated FGD slurry samples were conducted to gather data regarding the operating characteristics of the proposed concept. The process's energy efficiency as a function of several FGD slurry parameters including chloride concentration and inhibited or forced oxidation operation was determined. The results suggest that the scrubber operating mode, i.e., forced, inhibited, or natural oxidation, and the resulting neutralization chemistry, i.e., precipitated CaSO_3 or CaSO_4 , did not have a significant impact on electrolyzer operation, but an increased dissolved oxygen level did have a negative impact on process efficiency.

An engineering analysis was also performed to estimate the size and power consumption of a system designed to remove chlorides at a 530-MW power plant. The analysis indicated that the proposed concept would be most competitive for situations where the purge flow (or blowdown) is high in order to maintain a low slurry chloride content (approximately 5000 ppm or less). For these cases, investment in the cell would have the greatest return in terms of recycling FGD water. The purge flow could then be reduced to the amount needed to regulate suspended solids or metals. Depending on the actual process efficiency and operating costs for the plant, it may be possible for the savings in water consumption to equal the lost revenue for the electricity consumed by the process. However, for most cases, it is expected that the water savings will be less than the lost electricity revenue. For the assumptions used in this analysis, the break-even point could be achieved for a slurry chloride content of 3500 ppm. Since the potential for water recycling will not result in significant savings, capital investment in the proposed concept must be justified as being either a less expensive or more practical alternative to conventional water treatment technology. For example, investment in this process could reduce the purge flow and also the needed capacity of the downstream wastewater treatment plant. The estimated capital cost corresponding to a chloride concentration of 3500 ppm was \$21,000 per gpm of avoided wastewater treatment capacity.

In summary, the concept is feasible and would be a candidate for further development. However, given the high cost estimates of recycling and/or treating water with this approach, the market needs to be thoroughly evaluated before more development work is

justified. It is possible that further research could improve the process performance and reduce the estimated costs.

OBJECTIVES

The objectives of this project were to explore the operating characteristics of an electrochemical method to remove chlorides from wet flue gas desulfurization (FGD) slurries and to estimate the implications of those characteristics when applying the concept at full-scale. In order to meet the stated objectives, a scope of work was outlined consisting of the following tasks:

- Task 1 – Establish a research contract with ICCI
- Task 2 – Assemble experimental system
- Task 3 – Conduct experiments
- Task 4 – Perform an engineering analysis of the proposed concept
- Task 5 – Prepare final report

INTRODUCTION AND BACKGROUND

Illinois Basin coal has a high sulfur content and a moderate-to-high chlorine content. Most of the Illinois coal mined is used for utility electricity production and is typically used in the immediate vicinity of the Illinois Basin or at points east of Illinois. Implementation of the Clean Air Interstate Rule in 2005 has mandated that many eastern U.S. plants that fire high-sulfur coal be retrofitted with FGD equipment; therefore, any plant that consumes Illinois Basin coal probably already has or will have an SO₂ scrubber. While primarily intended to remove SO₂, these scrubbers also capture HCl which is generated in significant quantities during the combustion of Illinois Basin coal. The SO₂ absorbed in the scrubber is removed from the system as a precipitated solid, typically calcium sulfite or sulfate, but in the recycle loop of a wet FGD system, there is no inherent mechanism to remove the accumulated chlorides, except for a periodic or continuous purge stream of high-chloride-content solution and replenishment with low-chloride water. Previous studies have examined chloride removal from FGD systems [1] prior to the construction of second- and third-generation FGD systems, in the mid 1990s, but an effective solution was not identified. Instead, during the evolution of utility FGD systems, chlorides have been dealt with by increasing the corrosion resistance of the construction materials and using purge flow to limit slurry chloride concentrations.

This project evaluated an exploratory concept for the direct removal of FGD slurry chlorides using an electrochemical method. The method is based on chlor-alkali processing where a concentrated solution of sodium chloride is electrolyzed to evolve chlorine gas at the anode and create sodium hydroxide in solution. The chemistry of the concept under evaluation is analogous to chlor-alkali processing; for instance, the initial form of the dissolved chloride is calcium chloride, and when electrolyzed, chlorine gas is evolved, and calcium hydroxide is left in solution. However, there are significant differences specific to the characteristics of FGD slurry that have not been evaluated before.

The proposed integration of the electrolyzer within the recycle loop of a wet FGD system is shown in Figure 1. As shown in the figure, the liquid fraction exiting the slurry

thickener, which could be a hydroclone or clarifier, is routed through an electrochemical cell where a portion of the chlorides is oxidized and evolved as chlorine gas. Some amount of purge flow and conventional wastewater treatment and disposal will still be needed to control suspended solids and dissolved metals, but the amount could be significantly reduced since the electrolyzer could regulate slurry chloride levels. Initial estimates based on typical chlor-alkali cell efficiencies suggested that the electricity per unit mass of chlorine removed with electrolysis was of the same order of magnitude as the electricity needed to operate a modern wet FGD system per unit mass of removed sulfur.

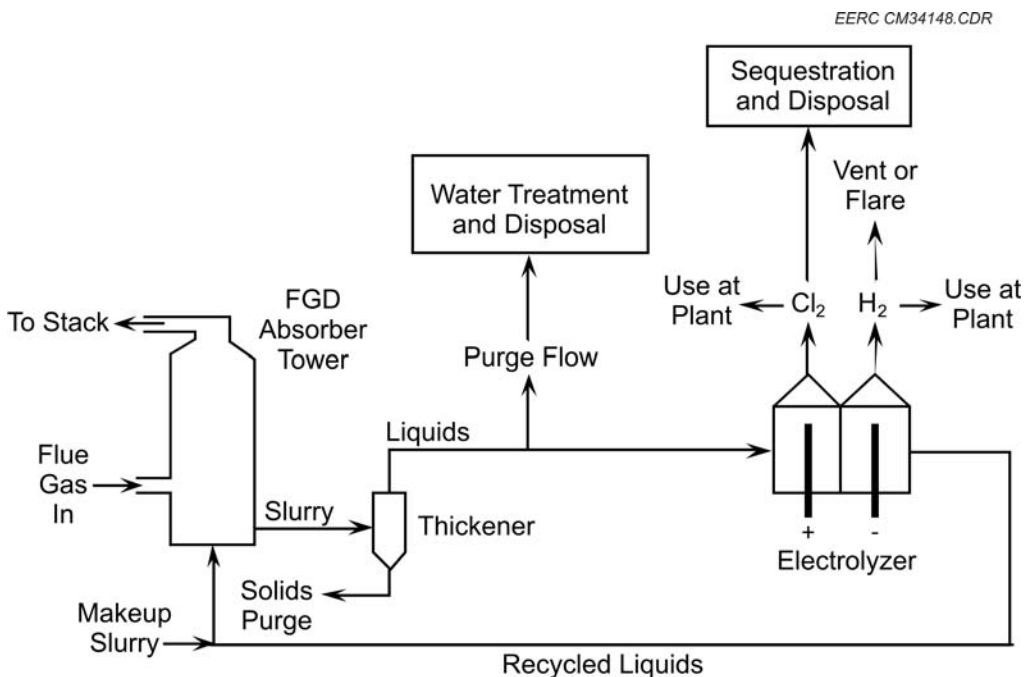


Figure 1. Integration of electrochemical chloride removal.

EXPERIMENTAL PROCEDURES

Laboratory-scale experiments of electrolyzing simulated FGD slurry samples were used to gather data regarding the operating characteristics of the proposed concept. The experimental setup is shown in Figure 2 and consisted of a custom electrolysis cell, circulation pump, and impinger for offgas capture. The cell's anode was ruthenium oxide-coated titanium (similar to the composition used in chlor-alkali processing) and had an approximate surface area of 14.3 cm². The cathode material was a stainless steel mesh and had a surface area greater than that of the anode. The electrodes were separated by a Nafion[®] N117 ion exchange membrane.

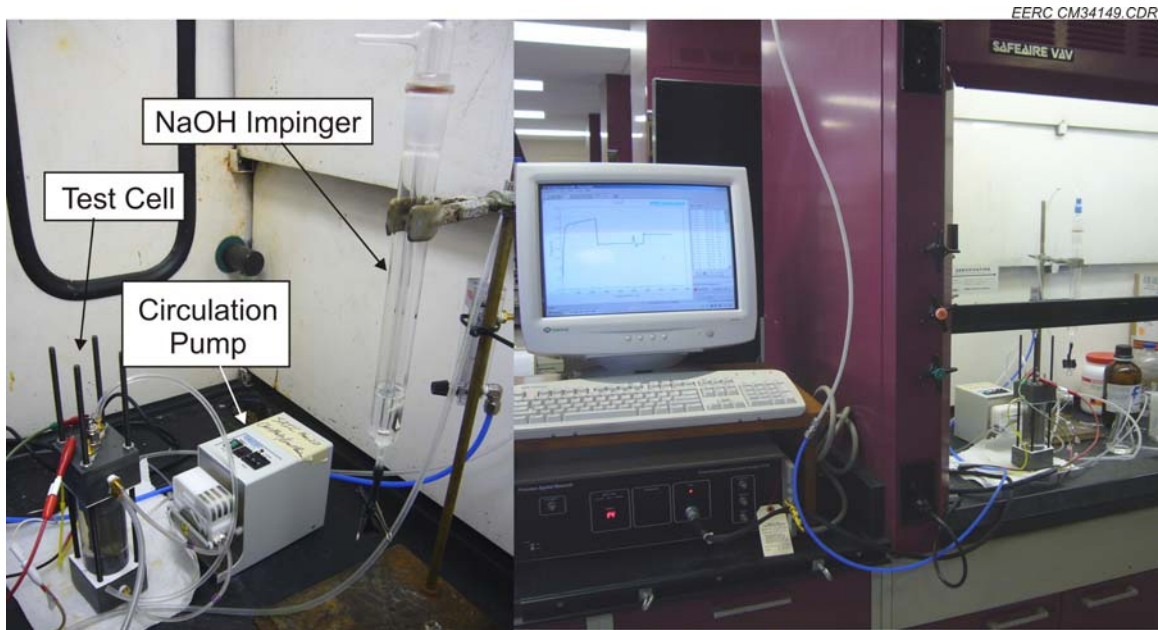


Figure 2. Experimental setup, left; galvanostat and control computer, right.

Power was supplied to the cell using a Princeton Applied Research galvanostat (model 263A); the power supply's control software recorded the total cell potential with time. Chlorine gas evolved at the anode was desorbed by sparging the electrolyte with nitrogen or air, depending on the test being conducted. The effluent containing the chlorine gas was then bubbled through an impinger filled with a sodium hydroxide solution to capture the chlorine. Postrun measurements of the chloride concentration in the impinger solution were used to determine the quantity of chlorine removed from the slurry. A mass balance check was also performed for each run by measuring the chloride reduction in the anode solution. Given the small uncertainty regarding the cathode products, the evolved hydrogen gas was not collected.

The experiments used a simulated FGD slurry containing the primary neutralization products of a limestone or lime wet FGD system, specifically CaCl_2 , H_2SO_3 , H_2SO_4 , CaSO_3 , and CaSO_4 . For the simulated slurry, the desired chloride concentration was obtained by the addition of CaCl_2 to a saturated solution of calcium hydroxide. The mixture was then neutralized with either H_2SO_4 or H_2SO_3 (depending on whether forced or inhibited oxidation was being evaluated) until all of the base was neutralized and the desired pH was obtained. The result was a saturated solution of calcium sulfate (or sulfite) with the required chloride concentration at the desired pH. The starting simulated FGD slurry had the specifications outlined in Table 1.

Table 1. Starting Specifications for the Simulated FGD Slurry

Chloride Concentration	55,000 ppm
Solids	Saturated CaSO ₄ or CaSO ₃
pH	5.7 (adjusted with H ₂ SO ₄ or H ₂ SO ₃)

A summary of the testing is provided in Table 2. Each test started with a consistent chloride concentration and solution pH. The tests were operated in batch mode so the chloride concentration declined throughout each test as chlorine was removed from the solution. Within reasonable time limits, most tests were continued until the ending chloride concentration was 5000 ppm or less. Intermediate measurements for the ending chloride concentration were obtained by performing identical runs of shorter duration.

Table 2. Summary of Independent Test Conditions

Starting Chloride Concentration, ppm	Starting pH	Current Density, mA/cm ²	Precipitate	Sparging Gas
55,000	5.7	7	CaSO ₄	N ₂
55,000	5.7	30	CaSO ₄	N ₂
55,000	5.7	80	CaSO ₄	N ₂
55,000	5.7	30	CaSO ₄	Air
55,000	5.7	30	CaSO ₃	N ₂

Testing began with optimization of the current density; three current levels were tested over a consistent range of chloride concentrations, and the overall energy efficiency for each was determined. The baseline slurry conditions were representative of a forced oxidation system; i.e., they contained a fully oxidized CaSO₄ precipitate. The impact of an inhibited oxidation system was tested by using CaSO₃ precipitate, and the dissolved oxygen content was varied by sparging with air instead of N₂ for one test.

RESULTS AND DISCUSSION

Task 3

The experimental test results are summarized in Figure 3, which is a plot of the cell's measured energy efficiency as a function of chloride concentration for the test conditions of Table 2. The current efficiency, i.e., chlorine produced versus the quantity of electricity consumed, was determined to be high for all runs (greater than 95%), so the recorded time scale for the data in Figure 3 has been replaced with the approximate solution concentration based on the amount of electricity consumed.

As shown in Figure 3, the 7 mA/cm² condition and several of the 30 mA/cm² conditions provide a repeatable efficiency versus concentration curve. The highest current density tested, 80 mA/cm², had a significantly lower cell efficiency, beginning at approximately 50%. This suggests an optimum somewhere between 30 and 80 mA/cm² for this particular cell. The cell efficiency was strongly related to the chloride concentration of the solution since the dissolved CaCl₂ was the primary source of conductive ions in the

simulated slurry. The slurry is only mildly acidic, and the solubility of CaSO_4 (or CaSO_3) is low, so the only significant number of ions in solution are from the CaCl_2 .

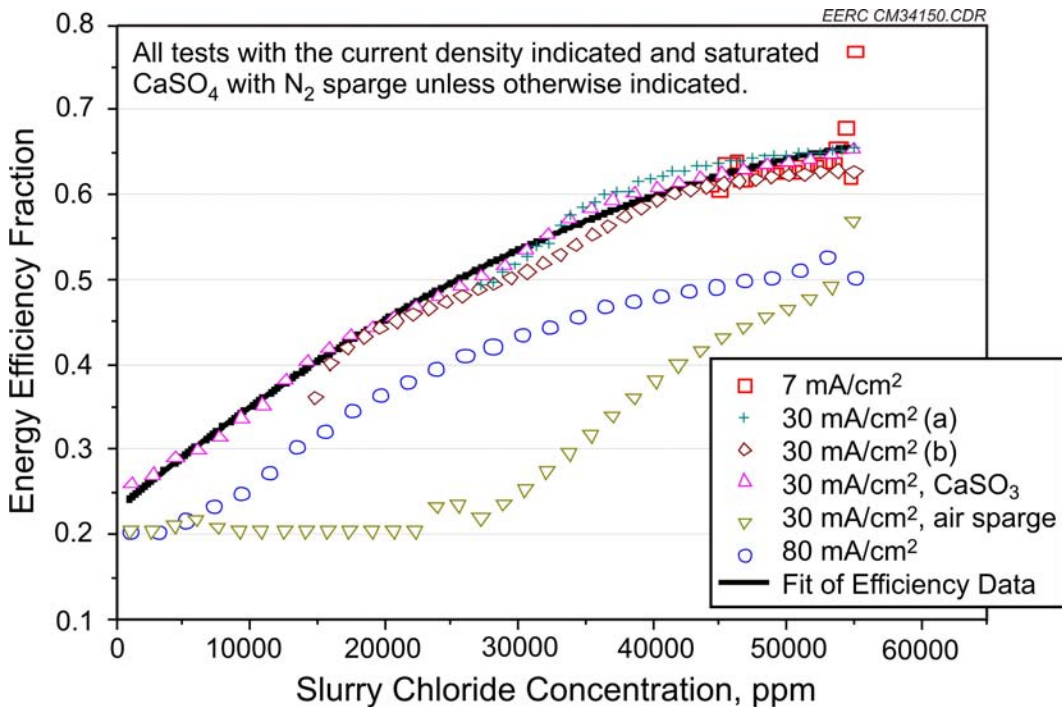


Figure 3. Experimental cell energy efficiency results.

The high dissolved oxygen condition in Figure 3 (denoted as the air sparge test) also had a much lower cell efficiency trend compared to the other results at an equal current density of 30 mA/cm^2 . For this run, the calculated current efficiency for chlorine evolution was still high, 96%, but the cell voltage was significantly higher because of the dissolved oxygen. Conversely, the condition with saturated calcium sulfite did not result in any significant difference compared to the corresponding calcium sulfate data. These results suggest that the scrubber operating mode, i.e., forced, inhibited, or natural oxidation, and the resulting neutralization chemistry, i.e., precipitated CaSO_3 or CaSO_4 , do not have a significant impact on electrolyzer operation. However, in forced oxidation scrubbers where the dissolved oxygen content may still be high, cell efficiency may benefit from a degassing of the solution. The efficiency data of Figure 3 appear to flatten at a minimum efficiency of approximately 20% for the air-sparged data; however, this is actually due to the maximum recordable cell potential being encountered during the test.

In the cathode compartment, a fine white precipitate settled out of solution after testing; thermogravimetric analysis of the precipitate confirmed that it was calcium hydroxide. Some of this precipitate appeared to form a surface layer on the cathode itself, but during the relatively short duration of the experiments, this calcium hydroxide coating did not hinder the cathode operation. However, for full-scale, long-term applications, this issue

will have to be resolved, possibly with a cathode designed to resist surface precipitation or seeding of the cathode solution with previously precipitated crystals. Formation of suspended $\text{Ca}(\text{OH})_2$ crystals is preferred since in the full-scale application the precipitate would be recycled to the FGD absorber and used again to neutralize HCl or SO_2 .

Another observation during the tests was that due to the relatively low current densities, the evolution of chlorine gas did not have rapid bubble formation, which may hinder chlorine separation in the full-scale application. This was overcome in the laboratory setup by desorbing the chlorine gas with the N_2 (or air) sparge of the anode compartment. At the cathode, visible hydrogen bubbles formed and disengaged from the electrode.

As mentioned in the previous section, the experimental cell used an ion exchange membrane to separate the anode and cathode compartments. Ion exchange membranes have generally failed in chlor-alkali applications that contain calcium and magnesium contaminant cations [2]. In these instances, the transfer of calcium or magnesium through the membrane results in sudden precipitation of solid hydroxide as these cations encounter the basic cathode solution. During these experiments, this did not appear to be a problem since there was always an excess of calcium in the cathode solution and precipitation of calcium hydroxide occurred at the cathode where the hydroxide was generated rather than in the membrane.

Task 4

The experimentally measured cell performance from Task 3 was used to estimate the size and power consumption of a chloride removal cell sized to handle a full-scale power plant firing a representative Illinois basin coal. The assumed integration of the cell into the plant was according to the diagram of Figure 1, in which the FGD recycle liquids from either a hydroclone or clarifier are routed through the cell before being returned to the FGD absorber. The FGD purge stream is split from the overflow liquids prior to entering the electrochemical cell. In this simplified arrangement, the mass flow rate of chlorine entering the slurry from the flue gas has two possible exits from the system, either as an evolved gas from the electrochemical cell or as a dissolved ion in the purge stream. Key parameters for the hypothetical plant are provided in Table 3.

Table 3. Assumed Plant and Economic Details Used for the Engineering Analysis

Plant Setup	
Plant Size	530 MW
Coal	Bituminous (3.1% S, 1000 ppm Cl)
Chlorine Feed into the Scrubber	333 lb/hr
FGD Overflow Liquid Recycle Flow	561,000 lb/hr
Economic Assumptions	
Plant Water Acquisition and Disposal Costs	\$4 per 1000 gal
Plant Electricity Consumption Cost	\$0.05 per kWh
Cell Capital Costs	~\$10,000 per m^2 of electrode area

Table 3 also contains economic data used to calculate costs for the various scenarios. Plant water acquisition and disposal cost was based on the upper end of current rates [3], and the electricity consumption cost represents the average value of lost revenue by consuming the power on-site. The cell capital cost is an estimate based on current prices for similar electrochemical equipment and a conservative “ground-up” estimate based on the calculated amount of electrode and membrane surface area needed. Cell efficiency was calculated during the analysis using the fit of experimental data presented in Figure 3.

The fundamental relationships between cell size and power consumption as functions of the slurry chloride concentration and the avoided purge flow are shown in Figures 4 and 5. The avoided purge flow is the water that is saved by chloride removal with the electrolyzer rather than using the purge stream exclusively. Cell size (represented by the required electrode surface area) is shown in Figure 4 and is the leading indicator of electrolyzer capital costs. The power consumption values of Figure 5 provide an indication of the ongoing operational expenses. In each figure, the water flow saved by the electrolyzer (avoided purge flow) ranges from zero, corresponding to the case of all chlorine entering with the coal being removed by the purge stream, to a maximum value where all of the chlorine is removed by the electrolyzer and no purge flow is required (in reality some purge flow would always be required so this is a theoretical end point).

For the cell size results of Figure 4, the lines of constant chloride removal (50% and 100%) are horizontal, indicating identical cell sizes regardless of chloride concentration; however, different amounts of water are saved because of the variable chloride content (note that any electrolyzer would need to be sized to handle the full FGD liquid recycle flow regardless of the amount of chlorine removed). Because of the differences in the amount of water saved, the most favorable ratio of cell size to the quantity of water saved occurs for the lowest chloride concentration, which is 5000 ppm in Figure 4. Using the estimate of cell capital costs presented in Table 3, the normalized capital expense for the 5000 ppm case in Figure 4 was \$30,000 per gpm of avoided purge flow.

In the cell power results of Figure 5, the benefit of improved cell efficiency is highlighted by the lines of constant chlorine removal. At higher chloride concentrations, more chlorine can be removed with less power input because of the higher cell efficiency. However, the most favorable balance between power (electricity costs) and water consumption (avoided water costs) is again with the lowest chloride concentration. Using the costs for electricity and water presented in Table 3, the ratio of avoided water costs to electricity costs for the 5000 ppm case is approximately 0.75, meaning that even under the most favorable circumstances in Figure 5, the savings in water will not recover all of the lost revenue for the consumed electricity. The break-even point between electricity costs and water savings for this analysis is with a chloride concentration of 3500 ppm. This implies that the economic justification for applying the proposed concept must come from avoided incremental investment in the primary wastewater treatment process.

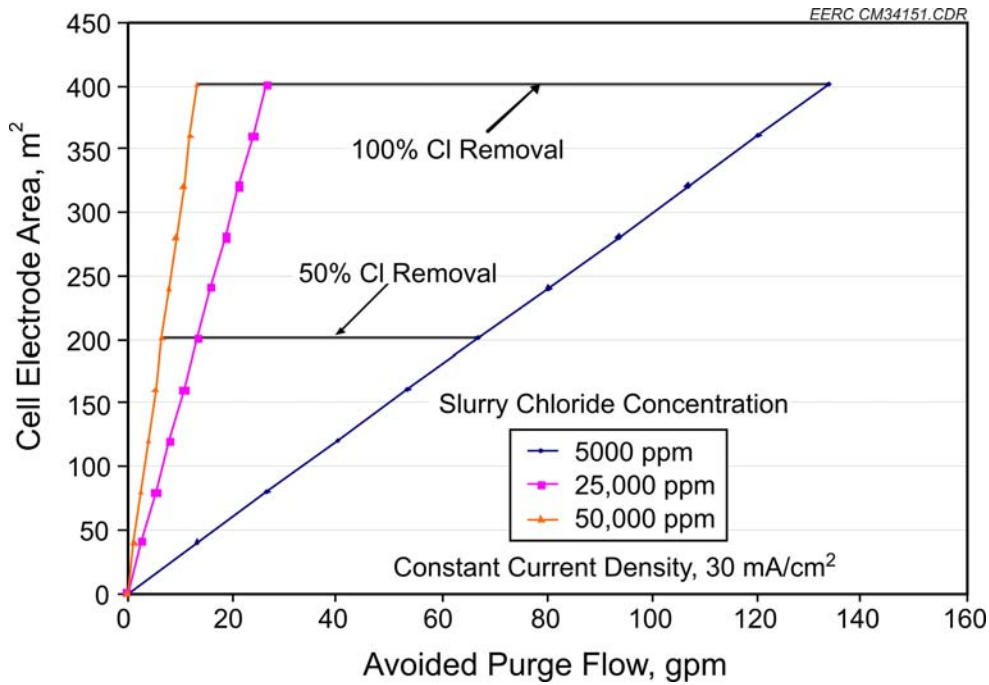


Figure 4. Estimated cell size versus saved water flow and chloride concentration.

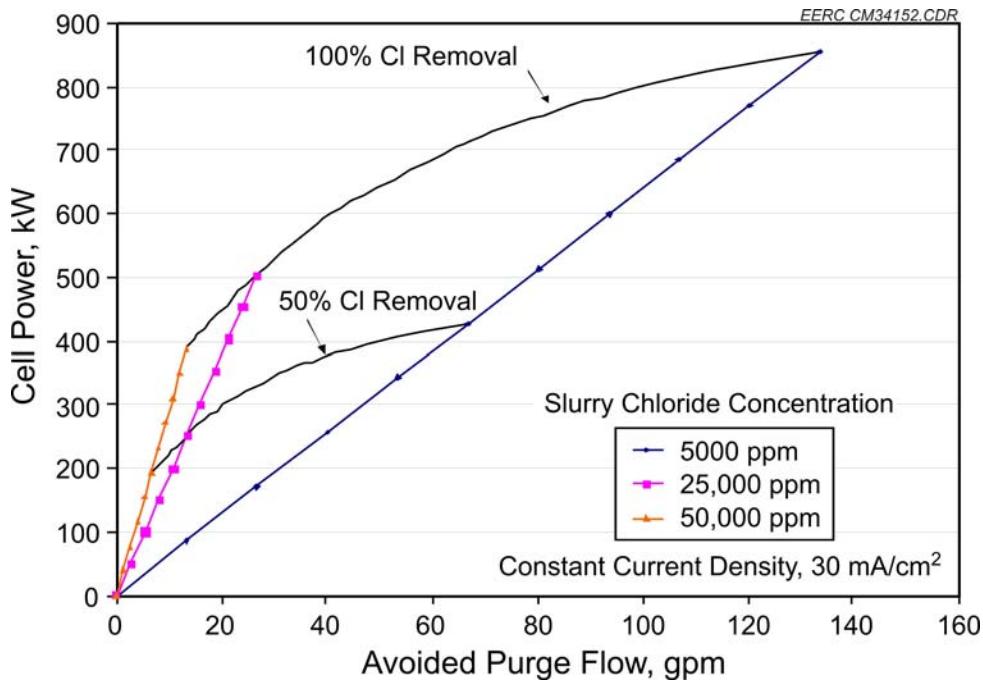


Figure 5. Estimated cell power requirement versus saved water flow and chloride concentration.

Given these characteristics, it appears that integrating a FGD slurry electrolyzer would have a potential advantage for systems that must treat and dispose of a low-chloride-content wastewater, i.e., where purge flow is high just to maintain acceptable chloride levels. In this configuration, the electrolyzer could maintain low chloride levels and produce a concentrated chloride-only waste stream. The reduced purge stream flow would then regulate suspended solids and dissolved metals that would be treated with the plant's wastewater treatment system. In this case, the added investment in a slurry electrolyzer would avoid costs associated with constructing a larger capacity wastewater treatment system. For cases where higher chloride content can be tolerated in the wastewater system and where the purge flow is not excessively high, then the slurry electrolyzer option would be less competitive and other means of concentrating all wastewater impurities, e.g., reverse osmosis or vapor compression evaporation, would become more competitive.

Plant Integration

The electrolysis process releases the chlorides in solution as chlorine gas which must be disposed of or, possibly, put to alternate uses at the plant, as indicated in Figure 1. As for disposal, absorption and neutralization of the chlorine with a low-cost basic solution will be the most feasible option. It may be possible to use a small portion of the fresh limestone slurry prepared for the FGD system or a portion of the calcium hydroxide slurry that is typically used as the first stage of on-site water treatment [4]. A final possibility may be neutralization with a fly ash slurry, which for Illinois Basin coal could result in appreciable quantities of iron chloride. However, all of the chloride salts that will form are highly soluble, so the plant must have disposal facilities for concentrated brines, not just dry ash or dewatered sludge.

Some plants with once-through cooling with river, lake, or sea water use significant quantities of chlorine gas as a biocide to keep condenser surfaces free from fouling organisms. Depending on the site-specific details, it may be possible to use a portion of the chlorine gas generated at the anode to supply the chlorination needs of the plant. Using a range of chlorine consumption data reported for once-through utility condensers [5], the consumption could range as high as 10% of the chlorine fed with a high-chlorine coal for locations where extreme fouling control is needed but would typically be in the range of 1%–2% for less severe duty.

The hydrogen product produced at the cathode is not a regulated emission so it could be vented or flared depending on the ignition risk. A possible integration and use of the hydrogen at the plant is to supply makeup hydrogen for the generator cooling system. Many utility generators use hydrogen as the heat-transfer fluid of choice because of its high thermal conductivity and acceptable dynamic fluid properties. Hydrogen leaks from these cooling systems and makeup gas must be constantly supplied. Makeup hydrogen can be supplied by compressed gas cylinders or generated on-site with a water electrolysis system. Based on the sizing estimates provided by one manufacturer of hydrogen electrolyzers [6], the makeup hydrogen needed for a 500-MW plant is approximately $1 \text{ Nm}^3/\text{hr}$, which is less than 2% of the expected output for an electrolyzer

removing all of the chlorine entering with the coal in Table 3. The hydrogen produced through electrolysis of the FGD slurry should be very pure; however, it will need to be thoroughly dried and perhaps filtered for fine particulates before it could be used for generator cooling.

If integration of the chlorine electrolyzer allows operation at a lower chloride concentration than would otherwise be feasible, it will result in a less corrosive scrubber slurry. In this environment, scrubber components would last longer or could be constructed from less expensive materials. While this is a distinct benefit, at this point in the deployment of wet FGD technology at power plants, construction decisions have already been made about materials of construction, and new technology currently in development will probably not alter those decisions.

CONCLUSIONS AND RECOMMENDATIONS

Overall the concept is feasible and would be a candidate for further development. However, given the high cost estimates of recycling and/or treating water with this approach, the market needs to be thoroughly evaluated before more development work is justified. Specific conclusions regarding the concept are provided below:

- The proposed concept would be most competitive for situations where the purge flow is high to maintain a low slurry chloride content. For these cases, investment in the cell would have the greatest return in terms of recycled water. In this situation, the cell would function as an extraction device forming a concentrated stream of chloride-only waste for disposal, and it should be compared to technological alternatives such as reverse osmosis or vapor compression evaporation that concentrates all wastewater impurities.
- Depending on the actual operating costs and performance of the electrochemical cell, it may be possible for the savings in water consumption to equal the lost revenue for the electricity consumed by the cell. However, for most cases, the water savings will be less than the lost electricity revenue. For the assumptions used in this analysis, the break-even point could be achieved for a slurry chloride content of 3500 ppm.
- Capital investment in the proposed concept must be justified as being either a less expensive or more practical alternative to conventional water treatment technology. For example, investment in the electrochemical cell could reduce the purge flow and also the needed size of the downstream wastewater treatment plant. The estimated cell capital cost corresponding to a chloride concentration of 3500 ppm was \$21,000 per gpm of avoided wastewater treatment capacity.
- There are possible synergistic uses for the produced chlorine and hydrogen at a power plant; specifically, the chlorine can be used as a biocide to prevent condenser fouling, and the hydrogen could provide the makeup gas for generator cooling. However, the demand for either of these purposes is only expected to be

1%–2% of the totals that would be produced, so a method for gas disposal will be needed. The hydrogen can be vented or flared fairly easily, but the chlorine will require neutralization and disposal. Plants with a wet FGD system and on-site water treatment should have access to a suitable calcium-based neutralization agent.

If there appears to be a justifiable market for the technology, then there are several areas of research that make the concept more competitive. Specific recommendations for future development work are provided below:

- Investigate increases to the operating current density. Even small gains to current density could result in significant reductions in electrode surface area needed that would translate into lower capital costs. Gains could come from improvements to mass transfer between the electrolyte and electrodes.
- Increase cell efficiency through optimized cell design. By optimizing the cell geometry and reducing the electrode to membrane distance, it should be possible to gain an incremental improvement to efficiency which will reduce the power required to operate the cell and, possibly, lower capital costs.
- Evaluate alternative anode coatings that would improve performance and lower overall cost.
- Perform an extended test with FGD slurry obtained from a power plant to provide some indication of life cycle costs and to determine the secondary impacts to slurry chemistry.
- Identify a cost-effective solid sorbent for the chlorine gas that could be landfilled. This would simplify the disposal issues since most plants already have means for fly ash disposal.

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