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# A Proposal for an Accelerated Evaporation Treatment System for Alumina Industrial Soda Ash Wastes and Feasibility of the use Soda Ash and Sodium Percarbonate for $CO_2$ Capture and Phenol Oxidation in Water

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*Abstract-* Increasing interest in $CO_2$ sequestration is linked to the need for cost-effective alternatives of In Situ Combustion Pilot Projects (ISCPP) which is an ongoing field testing effort aimed at assessing its efficacy in increasing the recovery factor of heavy oil reservoirs from Hugo Chávez Orinoco Oil Belt (Venezuela). A proposal is presented for an accelerated evaporation system for volume decreasing of caustic liquor in alumina industrial artificial lagoons, obtaining sodium carbonate (soda ash) and its later utilization in the synthesis of sodium percarbonate ($Na_2CO_3 \bullet 1.5H_2O_2$) (SPC). The caustic liquor evaporation rate was of 0.146 mm/h using only radiation in the evaporation system. When only artificial wind was used the evaporation rate was 0.196 mm/h, which means an increase of 34% in the evaporation rate. Results indicate that simultaneous application of radiation and wind into the evaporation panels allowed to attain a maximum caustic liquor evaporation rate of 0.302 mm/h, which represents an increase of 107% in the evaporation rate. The obtained salt is rich in sodium carbonate, which was used as raw material to accomplish SPC synthesis. The reaction was possible, with a yield of 46.7% and SPC content of 68% for every 100 g of sample obtained. SPC synthesis seems to be an alternative to waste disposal by adding value to caustic liquor.

*Keywords: by-products (caustic liquor "soda ash" and red mud); bayer process; in situ combustion; mineral carbonation; sodium percarbonate (SPC).*

## I. INTRODUCTION

The Bayer process, developed by Karl Josef Bayer 130 years ago, is a widely applied commercial technology to obtain calcined alumina from bauxite ore (Habashi, 2004; Habashi, 2016). For alumina extraction, bauxite is crushed and subjected to alkaline digestion with a caustic soda (NaOH) solution at high temperature. Impurities generated from the extraction are separated in the form of a solid insoluble earthy brick red, called red mud; and a caustic liquor (soda ash about 13%) from spent alkaline solutions (Pérez et al., 2010). The process continues to obtain anhydrous alumina from the calcinations of aluminum trihydroxide as a final commercial product, leaving as environmental liabilities red mud and spent alkaline solutions (pH 10-13). Red mud is highly alkaline, corrosive and contains several oxides and salts, which are toxic to biota. Due to its particle-size distribution from fine powder to granular material sediment is highly mobile in aqueous environments and hazardous to human health, causing respiratory, cardiovascular and eczema-rosacea skin problems (Nagy, Szabó, and Vass 2013).

In Situ Combustion Pilot Project (ISCPP) is an enhanced oil recovery process which consists on burning a portion of the reservoir contained oil in order to generate thermal energy that allows an increased displacement and production of the rest oil.

The crude oil is mobilized by such thermal 'brooming' sweep/scavenge and pushed towards the drainage points located hundreds of meters from the air injection well (Figure 1) (Perozo et al., 2011). The ISCPP generates significant emissions of carbon dioxide ($CO_2$) contributing to global warming and harzarduos hydrogen sulfide gas ($H_2S$), during its operation (Perozo et al., 2011).

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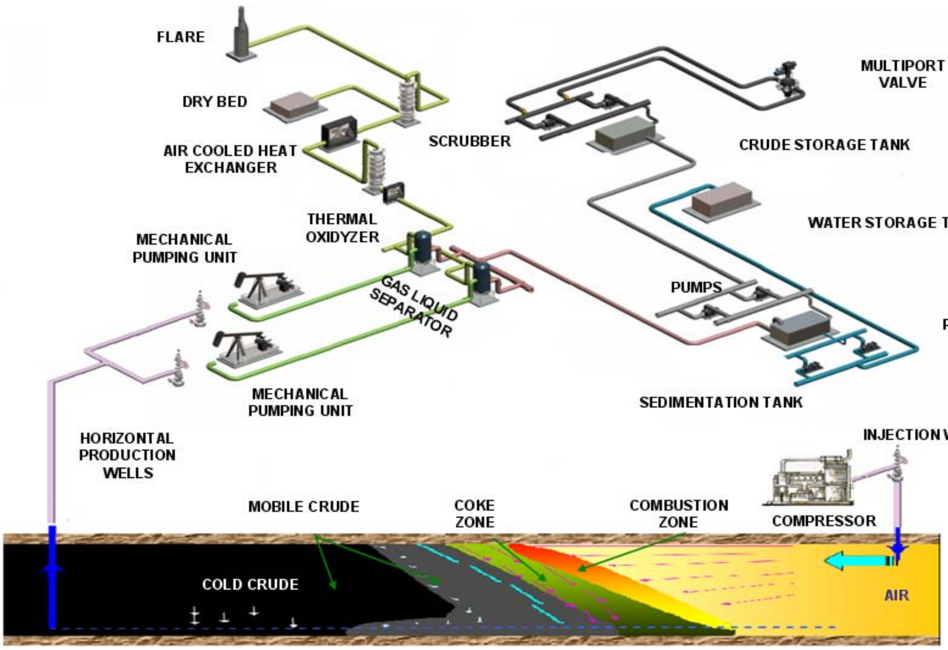

*Figure 1:* In situ combustion and surface facilities block diagram (Perozo et al., 2011)

Due to venezuelan environmental regulations, it is not allowed to release to the atmosphere this kind of gases. This causes the need to apply processes to treat the gas stream at the surface for the removal of these contaminants in order to satisfy environmental standards (Pérez et al., 2010; Perozo et al., 2011).

Present work shows results of laboratory scale studies using an accelerated evaporation system (intensive) of caustic liquor (waste) in alumina industrial artificial lagoons, to obtain sodium carbonate and its later utilization in the synthesis of sodium percarbonate (SPC); as an environmental cost-effective solution that could allow to minimize hazards and environmental liabilities by reducing alkalinity and volume of lagoons. Feasibility of the use sodium percarbonate from the reaction between $Na_2CO_3$ and hydrogen peroxide ($H_2O_2$), with a performance similar to advanced oxidation process for the treatment of waters contaminated with phenols (Bonenfant 2008, AOP's for Effluent Treatment 2009).

## II. Materials And Methods

### a) Caustic liquor samples used in the experiments

Two caustic liquor samples, CLL2 and CLL3 as well as a sample of red mud, were evaluated. Caustic liquor samples were analyzed for pH, NaOH concentration, $Na_2CO_3$ concentration and total alkalinity as $CaCO_3$ (Table 1). Caustic liquor was analyzed for determine pH, mineral phases via X-ray diffraction (X'Pert Pro equipment, mark PANAlytical) and thermogravimetric analysis (TG, mark SETARAM, KGP technologies) (Pérez et al., 2010). Evaporation rate was evaluated, starting from 4,500mL of caustic liquor samples in three experiments: one with CLL2, second with CLL3 and a third with both CLL2 and CLL3 caustic liquor samples.

*Table 1:* Caustic liquor average composition in samples

| Composition | unit | L2 | L3 |
|---|---|---|---|
| NaOH | mg/L | 6,157 | 3,870 |
| $Na_2CO_3$ | mg/L | 132,900 | 75,800 |
| NaCl | mg/L | 12,380 | 12,110 |

### b) Accelerated (intensive) evaporation system of caustic liquor to obtain sodium carbonate

*Components*

#### i. Tray and evaporation panel

A stainless steel tray (56.0 cm long by 46.0 cm wide and 4.0 cm deep) was used as caustic liquor reservoir. Some steel barriers were incorporated to increase the liquor retention time in order to help incidence of simulated meteorological variables (wind and radiation). Likewise, evaporation panel was made of stainless steel in a hexagonal grid form (honeycomb) to increase the surface area and therefore liquids retention in its weft. Dimensions of each individual panel were: 21.5 cm wide, 28.0 cm long and 1.3 cm deep, giving a total approximately 57.0 $m^2$ surface area.

#### ii. Sprinkler and peristaltic pump

The sprinkler used to recirculate caustic liquor from the reservoir tank and the tray to the evaporation pads was Monarch F-80 type. The sprinkler transforms a pressurized liquid fluid into spray, which is called a water curtain, whose shape will depend on the nozzle of the sprinkler and the pressure capacity of the outlet. A peristaltic pump with a regulated flow rate of 120 mL/min was used. Masterflex L/S. Coputerized Prive. Model 77250-62. Hoses: Masterflex 6409-24 TYGON®. Mfg by Norton.

iii. *Wind speed meter*

Therno - Anemomether: EXTECH Instruments.

iv. *Radiation meter*

Radiometer Light Meter. LI-COR (Quantum radiometer/photometer). Model LI-189.

v. *Relative humidity/temperature meter and devices to simulate meteorological factors*

Brand Hanna, model HT-9218. A 100W/120V incandescent lamp was used for the simulation of radiation, and a two-speed, 120V, 6 inches fan for wind.

vi. *Evaporation capacity*

Three experiments were carried out in which different meteorological factors were simulated. The Penman equation (Equations 1, 2 and 3) describes evaporation (E) from an open water surface. Penman's equation requires daily mean temperature, wind speed, air pressure and solar radiation to predict evaporation rate.

$$\text{Eto} = k.\ [R_n + (1\text{-}W)f(u).\ (e^0 - e)] \qquad \text{(Eq.1)}$$

$$\kappa = 10/\lambda_v \qquad \text{(Eq.2)}$$

$$\lambda_v = (595\text{-}t_m) * 0{,}51 \qquad \text{(Eq.3)}$$

$$(1\text{-}W) = \gamma/(\Delta+\gamma) \qquad \text{(Eq.4)}$$

$$\Delta = 33.8693\ [0.05904\ (0.0078t_m + 0.8072)^7\text{-}0.0000342] \qquad \text{(Eq.5)}$$

$$\gamma = (C_p.P)/(0.62198.\lambda_v) \qquad \text{(Eq.6)}$$

Where

$Et_o$ = reference evaporation rate (mm/day); k = adjustment factor; $R_n$ = net radiation (mm/day); (1-W) = radiation weighting factor; $f(u)$ = wind function; $e^o$ = saturated vapor pressure in air (mbar); e = vapor pressure of free flowing air (mbar), $\lambda_v$ = heat vaporization of water at mean temperature; $t_m$ = annual mean temperature; $\Delta$ = slope of the steam saturation curve; $\gamma$ = psychrometric constant; $C_p$ = specific heat capacity; P = atmospheric pressure.

*Experiment 1:* Control, no radiation, no winds. *Experiment 2:* Conditions of a cloudy day and nights. *Experiment 3:* Conditions of a clear sunny day. Images of experiments are shown in Figure 2.

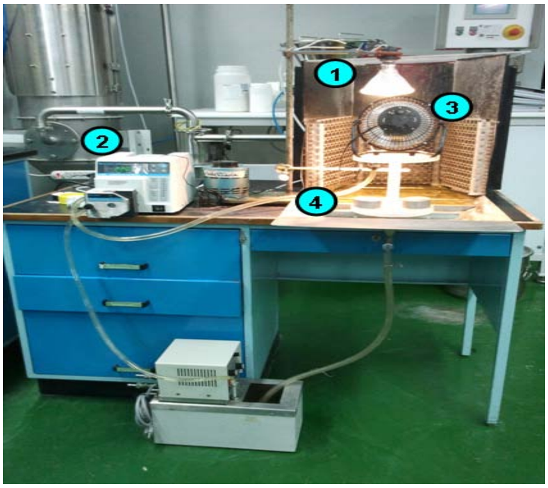

*Figure 2:* Experimental assembly: 1) lamp; 2) peristaltic pump; 3) fan; 4) tray and evaporation panel

Table 2 shows temperature and wind conditions applied in each experiment, positive (+) and negative (-) symbols respectively indicate whether or not the corresponding environmental condition were applied.

*Table 2:* Experimental design of wind and radiation application

| Experiment | Radiation | Wind |
|---|---|---|
| 1 | - | - |
| 2 | - | + |
| 3 | + | + |

Each experiment was monitored at two-hour intervals when it was determined:relative humidity, temperature and caustic liquor volume.

c) *Saturated caustic liquor sample with $CO_2$ (g)*

Impure salts of sodium carbonate were obtained after saturation of caustic liquor with $CO_2$ and water evaporation

d) *Feasibility of SPC synthesis as alternative for final disposition caustic liquor*

SPC is an additional compound produced in the reaction of sodium carbonate with hydrogen peroxide, which can be done via dry, spray granulation, crystallization and electrochemical processes (Gerd et al., 1975; Ruiz et al., 2009). During the crystallization process, sodium carbonate is formed by reacting solutions of sodium carbonate and hydrogen peroxide in a crystallizer, possibly in combination with salting out additives (HERA, 2008). SPC is a moderately strong oxidizer, widely used in detergent formulations and cleaning products. SPC was prepared by mixing impure sodium carbonate (25 g) and hydrogen peroxide at 30 wt/vol % (34 mL). After 20 min, 20 mL of denaturalized ethanol was added. The SPC adduct formed as white granule-shaped crystals and were dried for 4 hours at room temperature (Equation 7).

$$Na_2CO_3 + 1{,}5\ H_2O_2 = Na_2CO_3{\bullet}1.5H_2O_2 \qquad \text{(Eq. 7)}$$

Sodium percarbonate was evaluated in base to the availability of active oxygen and its oxidation capacity of phenol in aqueous solutions (Vesper et al., 1994; Kabalka et al., 1990).

$$Na_2CO_3{\bullet}1.5H_2O_2 = Na_2CO_3 + 1{,}5\ H_2O + 1{,}5/2\ O_2 + \text{heat} \qquad \text{(Eq. 8)}$$

## III. Results And Discussion

### a) *Experiment 1*

Results of evaporation rate in Experiment 1 (control experiment), in which no meteorological factors were simulated (Table 2), have been taken as reference for estimation of evaporation rate increase to compare with following experiments. Figure 3, shows average evaporation rate for experimental and theoretical results and accumulation of the caustic liquor evaporation as a function of time. Relative humidity is inversely related to temperature. With increase of temperature, saturation pressure (maximum pressure before condensation occurs) increases resulting in a decrease of relative humidity (Rolle 2006). Observed maximums in saturation pressure are caused by both properties and depends on the time of the day at which each mean is measured; in the morning lower temperatures and therefore higher relative humidity values were registered. This trend varied with the time of the day. The variation of the evaporation rates among different performed assays is caused by aspersion loses generated during the impact of the aspersed liquor and the evaporation panel, which in turn caused a significant difference between experimental and theoretical results. The latter do not cover the observed loses (Frost 1955). The gradual evaporation of the water contained in the caustic liquor caused an increase in the carbonate concentration. For that reason, the liquid turned denser and more difficult to asperse. With an increase in the aspersion there is also an increase in the surface area of the panel that is covered causing a thinner liquid film which favors the action of the meteorological factors. This phenomenon was also observed in Experiments 2 and 3.

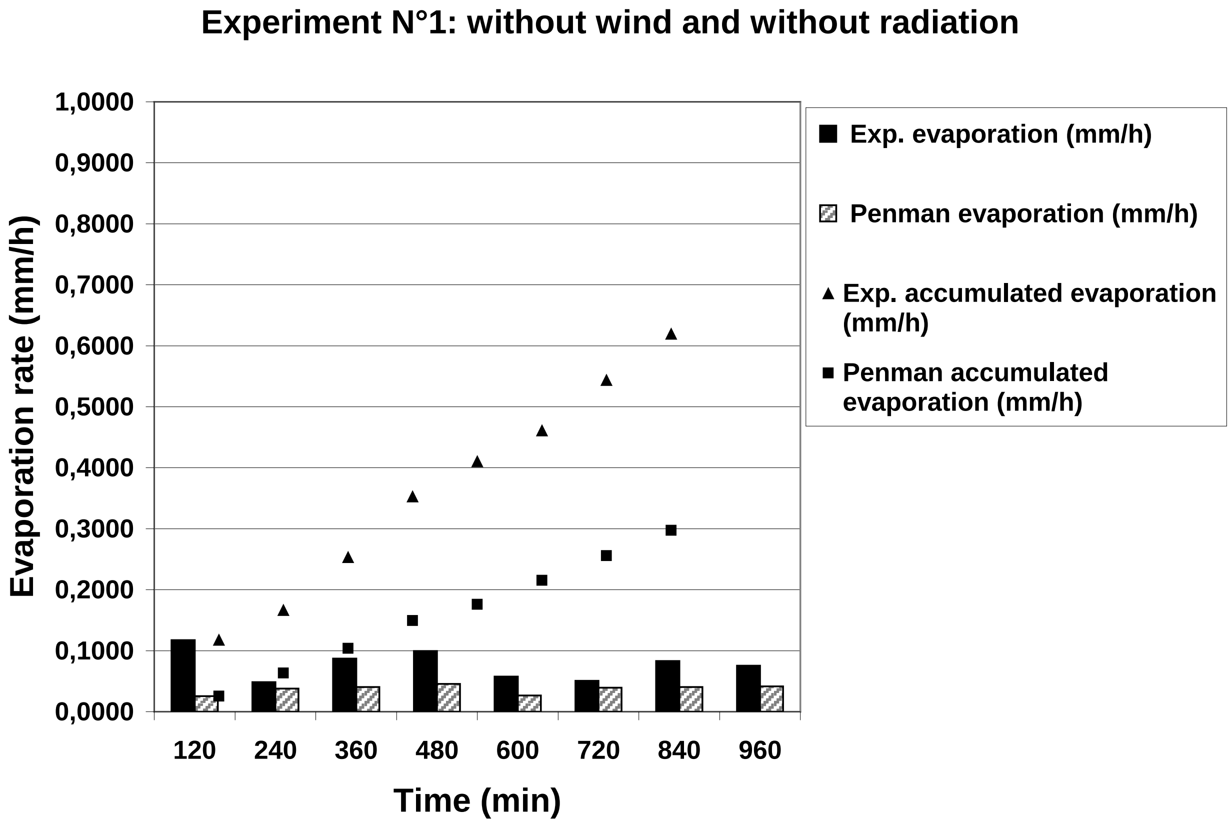

*Figure 3:* Evaporation rate: without wind and without radiation

*b) Experiment 2*

To simulate one-day weather conditions without solar radiation and only with wind (cloudy day: nights), a fan with regulated speed of 0.5 km/h was used. This is illustrated in Figure 4. An increase of 38% with respect to the control was obtained in the evaporation rate. The result was theoretically very approximate since wind spped is the variable that influences the evaporation rate in the Penman equation in tropical conditions. This variable was in a great extend below actual data for wind speed on tropical conditions. The decrease in the wind speed was so to avoid the caustic liquor loss that it generates and to obtain the evaporation yielded by the system. Drag-out losses are favored by decreases in the drop size; an increase in wind speed and temperature increases evaporation processes. Moreover, the provided wind speed kept a constant renewal of vapor saturated air layers. This trend was also observed in Experiment 3.

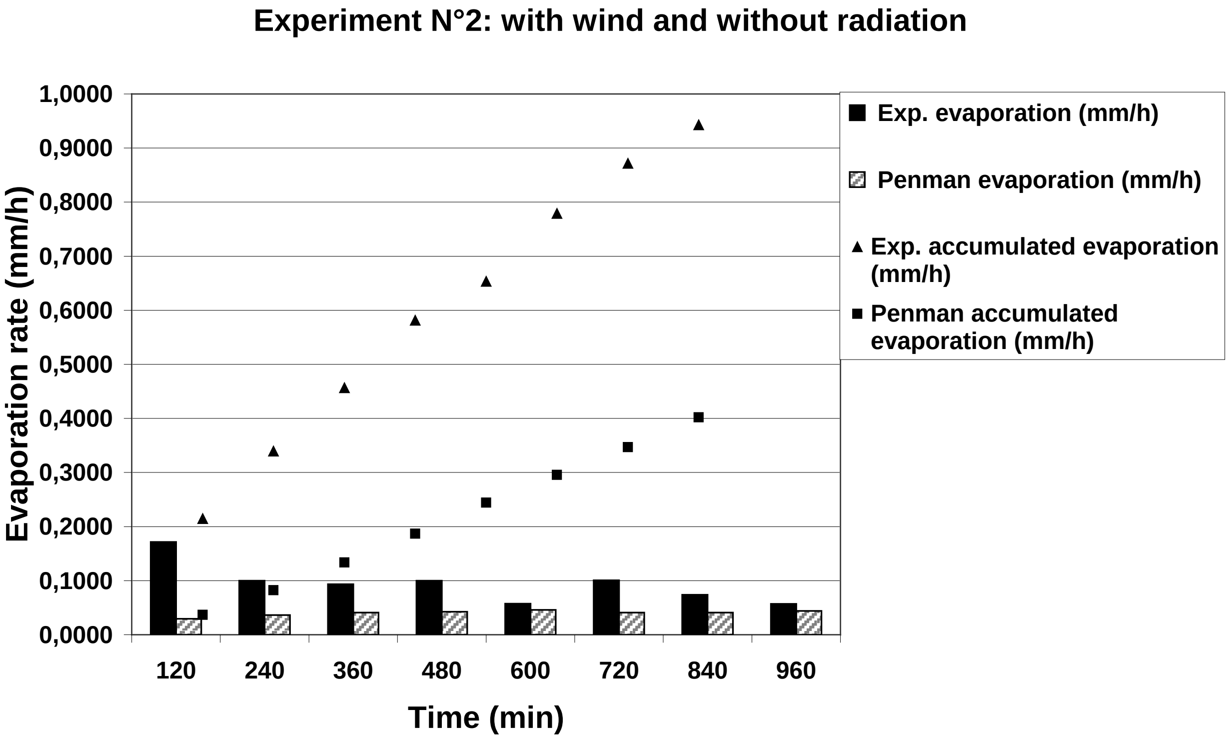

*Figure 4:* Evaporation rate: with wind and without radiation

*c) Experiment 3*

For the simulation of the meteorological conditions of a clear sunny day a speed-regulated fan (0.5 km/h) and a lamp 15.3 W/m$^2$ were employed. Figure 5 shows the rates of caustic liquor reduction through evaporation. An increase in evaporation rate of 107% with respect to Experiment 1 and of 34% respect to Experiment 2 was obtained. Aspersion on the panel favored evaporation since a thinner layer of liquid covered the surface area. This is explained by the fact that more thermal energy form radiation is adsorbed increasing temperature and the kinetic energy of the molecules. This acceleration in turn provides the energy required to overcome intermolecular energy of attraction. The evaporation process increase of the superficial area given by the panels was observed in Experiments 1 and 2. Similarly, wind accelerated evaporation by the renewal of water vapor saturated boundary layers as observed in the Experiment 2.

*Saturated caustic liquor sample with $CO_2$ (g) and feasibility of sodium percarbonate synthesis ($Na_2CO_3$•$1.5H_2O_2$) as alternative for final disposition of caustic liquor*

*Total alkalinity determination:* caustic liquor sample Caustic liquor samples have a low concentration of caustic soda (NaOH) compared to high content of soda ash ($Na_2CO_3$). As indicated in Table 3, on the initial composition of both samples (caustic soda concentration was between 3,870 and 6,157mg/L; and soda ash was between 75,800 mg/L to 132,900 mg/L). Table 3 shows alkalinity values obtained by acid-base titration of caustic liquor samples (Pérez et al., 2010).

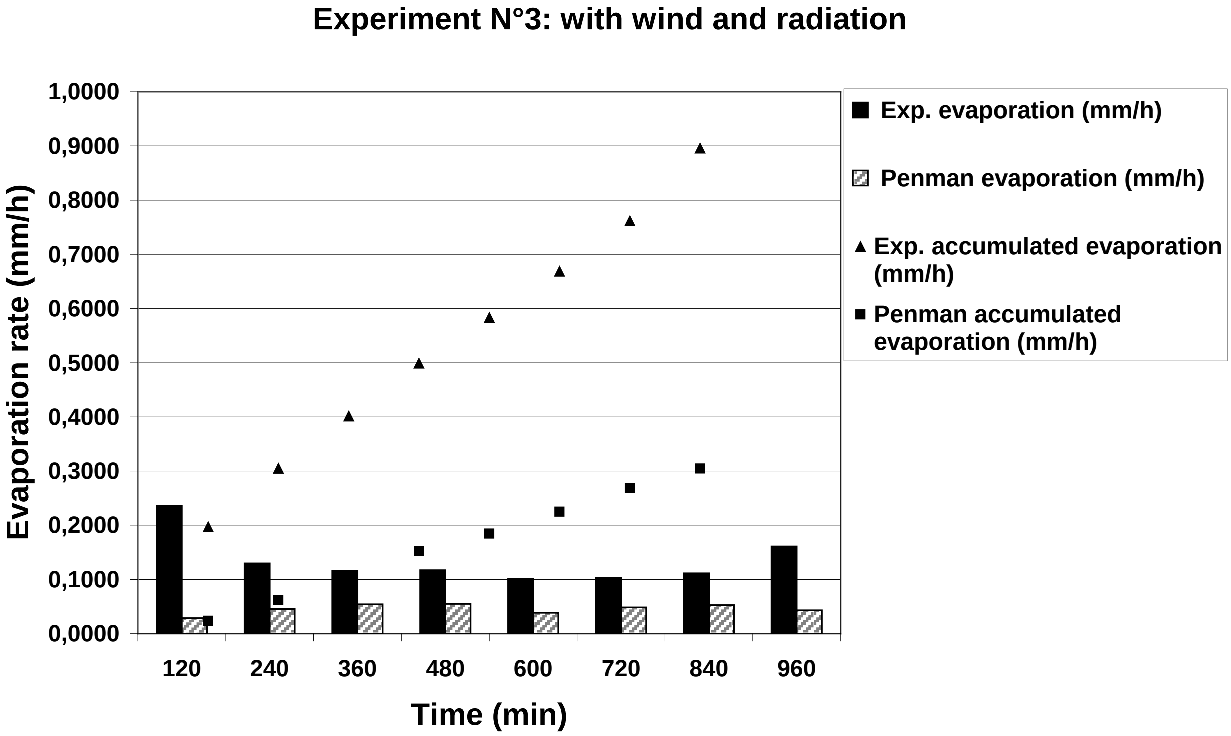

*Figure 5:* Evaporation rate: with wind and radiation

*Table 3:* Average initial values of alkalinity of the caustic liquor in samples

| Total alkalinity as | CLL2 (mg/L) | CLL3) (mg/L) |
|---|---|---|
| $Na_2CO_3$ (soda ash) | 150,343 | 93,538 |
| $CaCO_3$ | 139,516 | 87,737 |

*d) $CO_2$ (g) capture capacity*

Determinations of total alkalinity, pH, carbonate and hydroxide, showed that liquor sample CLL2 has an absorption capacity of 7.9 kg $CO_2$ (g) /$m^3$ and total alkalinity, as carbonate, of 1,359,865 ± 15,149 mg/L (Potenciometric method). Sodium carbonate precipitation in liquor sample CLL2 using pure $CO_2$ (g), for maximum precipitation of carbonate and hence the maximum capture of $CO_2$ (g) is presented in Table 4. Maximum neutralization values will reach a pH 8.3, which is the value of pH given atmospheric gas carbon dioxide when dissolved in water (carbonate equilibrium) (Kaneko and Okura 2002; Pérez et al., 2010).

*Table 4:* Amount of precipitated $Na_2CO_3$ caustic liquor samples

| Test | $Na_2CO_3$ (g) |
|---|---|
| Sample LL2-1 250 mL | 4.7639 |
| Sample LL2-2 250 mL | 4.7519 |
| Average | 4.7579 |

*e) Identification of precipitated soda ash*

Precipitated salt after saturation of caustic liquor samples with $CO_2$ (g) was identified by X-ray diffraction as sodium carbonate heptahydrate ($Na_2CO_3.7H_2O$).

*Feasibility of sodium percarbonate synthesis ($Na_2CO_3$•$1.5H_2O_2$) as alternative for final disposition of caustic liquor*

Precipitated $Na_2CO_3.7H_2O$ from the saturated caustic liquor samples was used to obtain SPC with a yield of 46.7% and a concentration of 68.0% SPC is produced from caustic liquor with impurities of ferric oxide and sodium salts from bauxite-goethite-hematite digestion process. Table 5 shows, the production of dissolved oxygen (DO) of reference bleacher with a content of SPC 33% and the SPC synthesized in distilled water.

*Table 5:* Dissolved oxygen (DO) from synthesized SPC and a reference bleacher

| SPC synthesized (*) (g ± 0.0001) | DO (mg/L) | Bleacher reference (*) (g ± 0.0001) | DO (mg/L) |
|---|---|---|---|
| 10.1752 | 33.44 | 10.0878 | 17.52 |
| 10.0549 | 36.77 | 10.3142 | 18.08 |
| 10.0822 | 35.64 | 10.2276 | 18.01 |
| 10.0480 | 34.81 | 10.0145 | 17.09 |

*(*)Distilled water: 2.84 mg/L DO*

The effect of SPC on the oxidation of standardized phenol aqueous solutions was investigated.Table 6 shows, the oxidation of phenol by synthesized SPC at ambient temperature. The high amount of phenol oxidized (82.47 %), indicates that synthesized SPC is an efficient oxygen source for phenol oxidation to hydroquinone (McKillop and Sanderson 1995).

*Table 6:* Capacity of SPC on the oxidation of standardized phenol aqueous solutions

| Test | Check | SPC synthesized (g ± 0.0001) | $[Phenol]_0$ (mg/L) | [Phenol] (mg/L) After the oxidation | Oxidized phenol (%) | Reaction Time (min) | Oxidized phenol average (%) |
|---|---|---|---|---|---|---|---|
| 1 | a | 0.9003 | 3.9740 | 1.2632 | 68.20 | 13 | 66.70 |
|  | b | 0.9009 |  | 1.3816 | 65.20 |  |  |
| 2 | a | 1.8002 | 4.1485 | 0.8819 | 78.70 | 28 | 79.06 |
|  | b | 1.8005 |  | 0.8554 | 79.40 |  |  |
| 3 | a | 2.7012 |  | 0.7194 | 82.60 | 39 | 82.47 |
|  | b | 2.7014 |  | 0.7345 | 82.30 |  |  |

## IV. Conclusion

1. The maximum rate of evaporation achieved in Experiment 3 corresponded to 2,646 mm/year, which is alike to the annualized average rate of an industrial lagoon system for alumina production of 2,182 mm/year. Results indicate that a combination of the natural evaporation and forced evaporation in a lagoon, is able to increase evaporation rate at 4,828 mm/year; minimizing the risk of overflow of the caustic liquor.
2. The feasibility of synthesis of SPC from impure sodium carbonate heptahydrate salt is possible, with a yield of 46.7 % and SPC with a purity of 68%. SPC synthesis looks as a treatment option that adds value to environmental liabilities of alumina industry.
3. The SPC purity obtained was sufficient to promote the oxidation of low concentration phenol aqueous solutions with a reaction time between 13 and 39 min.

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