



Factor effect of operating conditions on the leaching of fly ashes from a pulp and paper mill



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ABSTRACT

The non-process-elements (NPE's) in the pulp and paper production, in particular chloride (Cl^-) and potassium (K^+), eventually accumulate in a sodium sulfate recovery cycle of a paper plant, especially on boiler fly ashes (flue gas cleaning). This accumulation often leads to fouling and corrosion. Thus, in order to reduce such problems, a leaching unit is typically integrated aiming to remove Cl^- and K^+ and recover sodium sulfate (Na_2SO_4) from boiler fly ashes. In this context, this research is focused on investigating the effects of temperature, ash concentration and pH on the separation efficiency (Cl^- and K^+ removal and Na_2SO_4 recovery) of recovery boiler fly ashes using design of experiments in a devised laboratory setup. As expected from the literature, the results obtained reveals that concentration has the most significant effect on the response variables followed by temperature; whereas pH, not mentioned in this application so far, also has a significant effect. The optimum operating condition, which yields 84 wt% of Na_2SO_4 recovery and 97 wt% of Cl^- and K^+ removal, is achieved at 60 °C, 50 wt% of ash and pH 9.0. For practical applications, this optimal condition reduces the accumulation of NPE's and mitigates the risks of fouling and corrosion.

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1. Introduction

Leaching is a unit operation typically employed aiming to separate inorganic compounds and heavy metals from municipal and industrial wastes. Such treatment allows to achieve technical requirements for either reuse or recycling purpose which may contribute to reduction of environmental impacts (Alam et al., 2017; Likon et al., 2011; Quina et al., 2009; Sun and Barlaz, 2015; Weibel et al., 2018)

The pulp and paper production are mostly delivered by the Kraft pulping process. In this process lignin is removed by sodium sulfide and sodium hydroxide up to 170 °C (Hu et al., 2018). The application of leaching plays an important role in the Kraft pulping process to recover valuable compounds and remove undesirable ones, especially from boiler fly ashes.

Residual fly ashes produced by this process have been highlighted due to their large amount generated and the several possibilities of their compounds to be treated and recycled. Kinnarinen and co-workers (Kinnarinen et al., 2016) have revised the main process treatment for fly ashes. According to the authors the fly ashes can be treated using ion change, leaching, crystallization

and electrodialysis. Comparing the treatment options and limitations, it was stated by (Kinnarinen et al., 2016) that leaching is simpler and less expensive for commercial usage.

The boiler fly ashes comprise mainly sodium sulfate (Na_2SO_4) and sodium carbonate (Na_2CO_3). Non-process-elements (NPE's), including chloride (Cl^-) and potassium (K^+), usually accumulate in the chemical recovering cycle, commonly in the aforementioned fly ashes - the elements Cl and K are bound in fly ashes as solid salts (NaCl and KCl) (Del'Antonio et al., 2011; Moreira et al., 2008). At recovery boilers, this NPE's accumulation often leads to fouling and corrosion (Ferreira et al., 2004; Jordan and Bryant, 1996). Consequently, it may compromise its steady state operation, thus, requiring more frequently cleaning cycles (Gonçalves et al., 2008; Jaretun and Aly, 2000). In order to reduce such problems, an ash leaching unit is usually integrated aiming to remove Cl^- and K^+ and recover Na_2SO_4 . Ferreira and co-workers (Ferreira et al., 2003) have tested three different methods to minimize NPE's accumulation such as: direct purge, leaching, evaporation/crystallization and ion exchange. Based on economic analysis, the authors have pointed out that leaching and ion-exchange techniques were the most feasible operations for treating fly ashes from the electrostatic precipitator.

Considering the solubility of inorganic compounds in water and the operational conditions for leaching, parameters such as ash

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Nomenclature

C	slurry concentration [%]	REC	recovery efficiency [%]
T	temperature [°C]	REM	removal efficiency [%]
RSD	rejected separation degree [%]		
PRD	product recovery degree [%]		

concentration and temperature are pointed out as determining factors to be evaluated. They can influence directly the degrees of reject separation (i.e., liquid supernatant rich in Cl^- and K^+ dissolved) and product recovery (i.e., remaining solid rich in Na_2SO_4 not dissolved).

Kinnarinen and co-workers (Kinnarinen et al., 2018) proposed a novel dissolution method in order to remove hazardous trace metals from industrial fly ashes. The authors have obtained removal efficiencies of Cl^- and K^+ ranging from 80 to 90 wt%, with increasing pH in a range of 9 to 12. Despite a different composition, a pH variation may also favor the removal of Cl^- and K^+ in the investigated system, being likely benefited by high values as well.

Another interesting work is reported by (Shi and Rousseau, 2001). The authors have investigated the salt crystallization from a solution rich in Na^+ , CO_3^{2-} and SO_4^{2-} ions. As a result, they found that a double salt Burkeite ($\text{Na}_2\text{CO}_3 \cdot 2\text{Na}_2\text{SO}_4$) can be formed. Therefore, this salt can also crystallize in the evaluated system, considering that it has the same species in solution. However, it is worth mentioning that this system has additional species (NPE's) that can also affect directly the thermodynamic equilibrium, which may influence the salt crystallization.

Given the lack of studies, it is proposed here to investigate the effects of temperature, ash concentration and, particularly, pH on the separation efficiency (Cl^- and K^+ removal and Na_2SO_4 recovery) in an experimental apparatus properly devised. Initially, the effect of ash concentration, pH and temperature on Cl^- and K^+ removal and on Na_2SO_4 recovery efficiencies was examined by means of a design of experiments. In sequence, the optimal conditions and the relation between the response variables and the significative process parameters were determined through statistical models. The implications of these findings on the operating conditions of a recovery boiler are also discussed.

2. Material and methods

2.1. Sampling and ash characterization

An amount of 4000.0 g of fly ashes with 0.18% moisture content (w.b) were collected at the outlet of an electrostatic precipitator (ESP) installed in an industrial recovery boiler from a pulp and paper mill (Kraft Pulping). By sample quartering, only 4.5 g and 40.0 g of ashes were subjected to characterization and leaching, respectively. For chemical ash characterization, the S8 TIGER wavelength dispersive X-ray fluorescence (WDXRF) spectrometer was employed in order to quantify its inorganic compounds. The

analyses were performed in triplicate. Table 1 shows the ash composition.

2.2. Ash leaching

According to the design of experiments presented in the Section 2.5, leaching experiments was performed in the following steps: slurry mixing (ash in water) with temperature and pH adjustment succeed by sedimentation and filtration (Fig. 1). The initial pH of solutions of boiler fly ashes were approximately 11.0. The slurry concentration of 30, 40 and 50 wt% of ash was provided with distilled water, heated to 30, 60 and 90 °C (heating plate) of suspension bulk temperature and mixed under 500 RPM during 5 min (magnetic stirrer). To reproduce similar experimental conditions stated in the Industrial process, the pH of 9.0, 10.0 and 11.0 was adjusted by sulfuric acid 98 wt% addition and measured using a pH meter with 0.01 of accuracy. For the subsequent solid-liquid separation, sedimentation was carried out in a graduated test tube with temperature control, having 0.1 °C of accuracy (thermostatic bath). Preliminary tests showed that 20 min were sufficient for phase separation. The rejected separation degree (RSD) was calculated as:

$$\text{RSD} = \frac{h_L}{h_T} \times 100\% \quad (1)$$

in which, h_T is the total volume and h_L is the liquid phase volume (i.e. the total volume less the solid phase volume).

The solid phase (i.e., concentrated slurry) was then filtered under vacuum. To obtain the dry mass of solid, the filtered solids were dried at 105 ± 3 °C during 24 h. Their dry mass was then measured in an analytical scale and the product recovery degree (PRD) was calculated as:

$$\text{PRD} = \frac{m_{DS}}{m_T} \times 100\% \quad (2)$$

in which, m_{DS} is the dried remained solid mass and m_T is the initial total mass.

In sequence, the supernatant liquid and the dried solids were subjected to chemical analysis.

2.3. Chemical analysis

To obtain the concentration of the following elements and species K^+ , Na^+ , Cl^- , CO_3^{2-} and SO_4^{2-} in the product and rejected, each sample was pretreated. Then, each sample of product and rejected

Table 1
Ash composition of three different ESP sampling points.

Species (wt%)	Sample point 1	Sample point 2	Sample point 3	Arithmetic mean	Standard deviation	Relative Standard deviation
K^+	1.64	1.85	1.80	1.76	0.11	6.3
Cl^-	2.14	2.14	2.14	2.14	0.00	0.0
$(\text{CO}_3)^{-2}$	15.35	14.98	15.19	15.17	0.19	1.3
Na^+	34.87	33.95	34.55	34.46	0.47	1.4
$(\text{SO}_4)^{-2}$	45.66	46.46	46.01	46.04	0.40	0.9
Total	99.70	99.40	99.70	99.60	-	-

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