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Recovery of sodium from bauxite residue by pressure filtration and cake washing

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ABSTRACT

Bauxite residue, the main waste fraction of alumina production, is extremely hazardous to the environment, as well as to human health. After the recovery of the pregnant liquor, the washed and thickened bauxite residue is typically pumped as thick slurry to a residue disposal area. The use of filter presses for the final washing, in order to further enhance the recovery of alkali and aluminum, together with effective deliquoring, may be beneficial in many cases. In this study, a vertical filter press ($A = 0.1 \text{ m}^2$) was used for the filtration and washing of industrial bauxite residue. The effectiveness of various washing sequences, as well as variation in the local washing results in the filter cakes, were compared. The results show that Na recovery of 98 % can be obtained at a wash ratio as low as 1.4. It was also observed that not only the wash ratio is important: temperature and pressure have a significant effect on the dissolution of Na.

Keywords

Bauxite residue, red mud, filtration, washing, dissolution, sodium

1. Introduction

The production of alumina from bauxite ores by the Bayer process generates bauxite residue as the main waste fraction. The amount of bauxite residue (red mud) generated while producing one ton of alumina is typically about 1-2 tons, and the residue inventory is, according to numbers from the year 2007, increasing by approximately 120 Mt/a (Power et al., 2011). The increase of the residue production rate has been rapid, because only 70 Mt/a was produced in the early 1990s (Green et al., 1994). Among the most significant producers of alumina are currently Australia, China and Brazil, which together are responsible for nearly 60 % of the annual alumina production globally (Sparks, 2010; Samal et al., 2013). Toxicity to the environment (Milacic et al., 2012) and risks to animals and humans restrict the use and disposal of this strongly alkaline residue ($\text{pH} > 13$ in many cases). Concerning waste management, dry stacking (solid content = 48-55 %) and dry cake disposal (solid content > 70 %) are currently regarded as the preferred disposal methods, with a share of approximately 65 %. Wet disposal in large ponds (lagoons)

accounts for most of the rest, while the possibilities for direct marine disposal are inevitably vanishing. Both lagooning and direct marine disposal were the primary disposal methods until the 1970s, after which dry disposal methods became more popular (Power et al., 2011; Rai et al., 2012). In the case of lagooning, the major risks are associated with the mechanical instability of old dam constructions and the hazardous properties of the bauxite residue (Lorber and Antrekowitsch, 2011).

The composition of bauxite residues varies depending on the bauxite ore and the Bayer process. Typically present are hematite, alumina, titania, silica, CaO, Na₂O, K₂O, as well as several other components (Atasoy, 2007; Liu and Wu, 2012). Rare earth minerals are also typically present in bauxite residues. The major caustic-insoluble product formed in the Bayer process is the so called Bayer-sodalite, a sodium aluminum silicate, the exact composition of which depends on the purity of the digestion liquor (Chvedov et al., 2001). Bauxite residues also have a high salt content, and therefore high electrical conductivity. Sodium (Na⁺) is the dominating metal ion in the liquid phase (Gräfe et al., 2009). The solid particles in bauxite residues are fine: particles larger than 10 µm represent about 50 % or less of the particles (Lorber and Antrekowitsch, 2011; Liu and Wu, 2012; Johnston et al., 2010). The bulk density and the specific surface area of bauxite residues are high (Liu and Wu, 2012; Gräfe et al., 2009; Gräfe et al., 2011).

A large number of potential applications for the residues have been recognized and demonstrated. Some studies have focused on the most apparent value of the residues: the metals that are present as valuable oxides and alumina (Atasoy, 2007; Zhong et al., 2009). Bauxite residue is sometimes neutralized in order to 1) reduce its environmental impact on the surroundings of the disposal area and 2) sequester CO₂ (Rai et al., 2012; Kirwan et al., 2013; Si et al., 2013; Yadav et al., 2010). Neutralization with seawater or CO₂ are probably the most promising neutralization methods to be applied in alumina refineries (Gräfe et al., 2009; Brunori et al., 2005). The utilization of bauxite residue, even after a long period of time, is enabled by proper dry disposal. In order to minimize unnecessarily waste disposal, several applications for solid residue have been suggested, including material technology (Mymrin and Vazquez-Vaamonde, 2001), chemical industry (Alvarez et al., 1999; Schwarz and Lalik, 2012), water and soil treatment (Liu et al., 2011; Sahu et al., 2010), and recovery of valuable compounds (Agatzini-Leonardou et al., 2008; Li et al., 2009; Zhu et al., 2012; Lee and Pandey, 2012; Liu et al., 2009). However, there is hardly any significant use for the residue currently (Lee and Pandey, 2012).

Separation of bauxite residue from the sodium aluminate-rich liquid phase is typically conducted in a multi-stage countercurrent washing/thickening process, using polymeric flocculants (Gräfe et al., 2011). In order to reduce the moisture content after the final washing/thickening stage, vacuum drum filters have been typically used (Sparks et al., 2010; Borges et al., 2011). Filtration is performed, because dry cake disposal of bauxite residue requires filtration of the thickened residue, to obtain a cake with significantly reduced moisture content: approximately 30-35 % (most vacuum filters) and 25-28 % (typical pressure filters). The recovery of alkali and aluminum can be further improved by cake washing and minimizing the residual moisture left in the cake. Hyperbaric filters operating at pressure differences of 2-6 bar have been used for this purpose for

approximately two decades (Paramguru et al., 1994). The use of filter presses, demonstrated earlier by Rousseaux et al. (2008) and Kinnarinen et al. (2015), is most likely to become more popular in the future, because filter presses can operate at even higher pressure differences, which is important when the amount of cake and the loss of process chemicals are minimized.

In this study, deliquoring and washing of industrial bauxite residue is investigated by using a vertical filter press. The alkali recovery, filtration capacity and the local differences in the washing results are evaluated through three different washing sequences. Experiments without cake washing are also performed for comparison. Additionally, dissolution of sodium from the solids at various conditions is studied.

2. Material and methods

2.1. Preparation and characterization of the slurry

A slurry sample ($V = 120$ L), received from an industrial alumina production facility, was mixed with three pitched-blade turbines in a well-sealed, temperature-controlled tank at 65 °C. During the tests, four slurry samples were taken to ensure that the slurry composition did not change significantly. The Na concentrations in the liquid phase of the slurry, measured before and after the experiments, were 15.2 and 15.9 g/kg. The pH of the slurry, measured four times during the tests, was 13.05 ± 0.01 . The total solids (TS) content of the slurry was 406 g_{solids}/kg_{slurry}. The particle size distribution of the solids suspended in the slurry is presented in Fig. 1, which shows that the solids particles were relatively fine: the median particle size was 2.32 μm , and 10% of the particles were smaller than 0.87 μm . The mineralogical composition of the slurry was determined with X-ray diffraction (XRD). The most abundant mineral phases identified in the slurry were hematite, anatase, boehmite, gibbsite, silicon oxide, sodium aluminum titanium silicate, sodium magnesium titanium oxide, sodium iron titanium oxide, goethite, and faujasite.

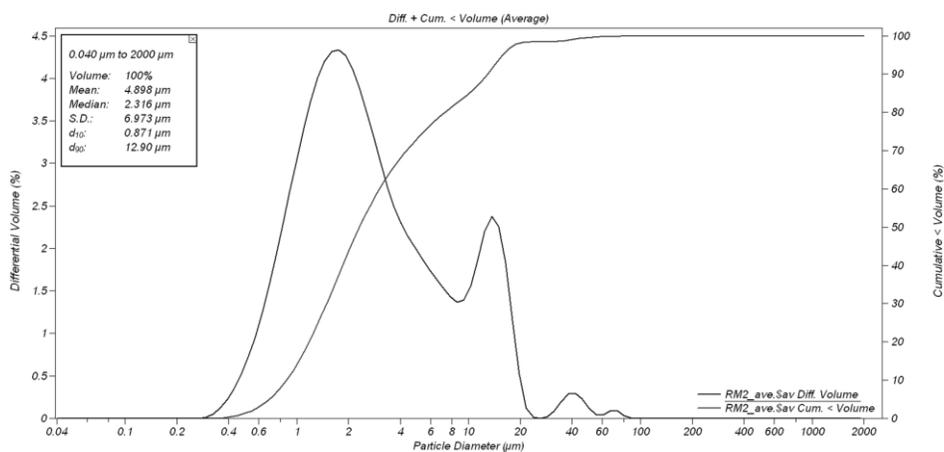


Fig. 1 Particle size distribution of slurry solids, measured with a Beckman Coulter laser diffraction analyzer.

A scanning electron microscope equipped with an energy dispersive spectrometer (SEM-EDS) was used for slurry characterization. Several SEM pictures were taken from a completely dried slurry sample (Fig. 2), in order to investigate the suspended solids and the dissolved solids, which were crystallized when the sample was dried. The elemental composition of the slurry was evaluated based on the SEM-EDS spectrum. The following elements were detected at significant amounts: O, Fe, Na, Al, Ti, and Si.

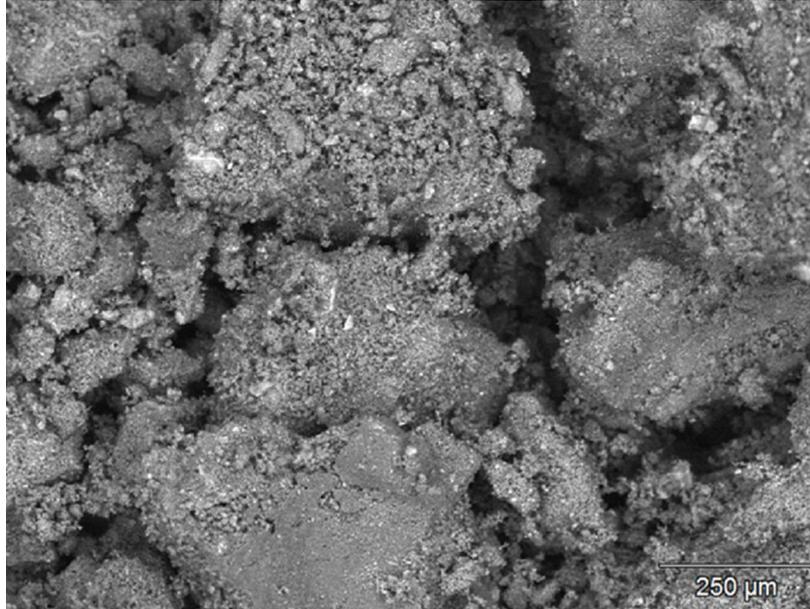


Fig. 2 SEM picture of a dried slurry sample.

2.2. Operation of the test unit

A Larox® PF 0.1 vertical filter press (Fig. 3), was used to carry out the solid-liquid separations and to compare the performance of cake washing. The test unit contained only one filter chamber ($A = 0.1 \text{ m}^2$) and there was an elastomer pressing diaphragm in the top of the chamber. The test unit was operated at a constant pumping pressure ($\Delta p \approx 6 \text{ bar}$), and the final cake squeezing was performed at 12 bar.

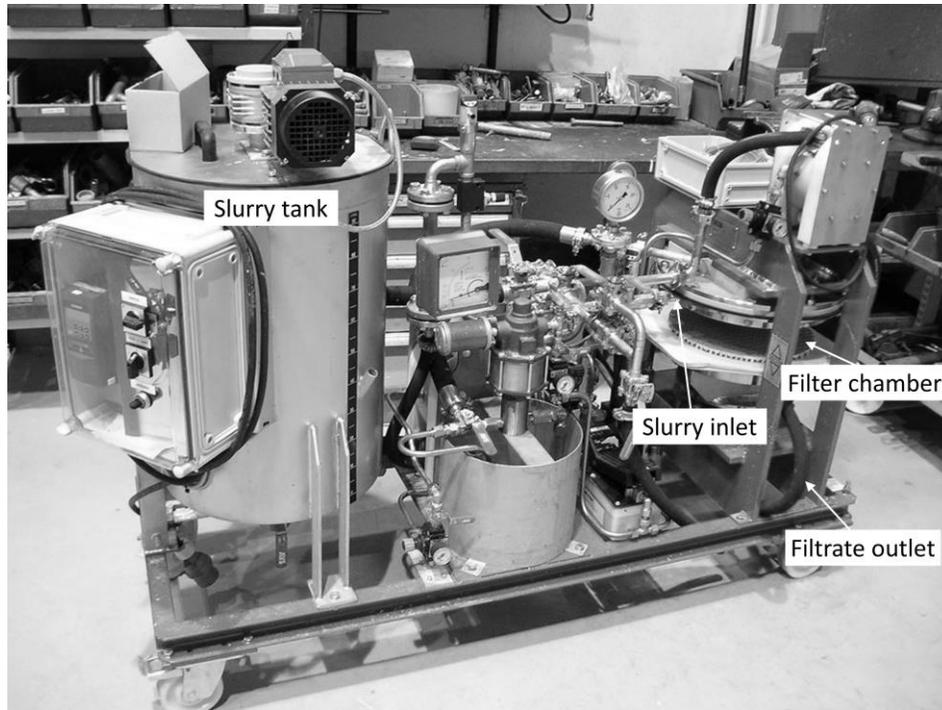


Fig. 3 Larox PF 0.1 filter press ($A = 0.1 \text{ m}^2$).

In addition to cake squeezing with the pressing diaphragm pressurized with water, air (5.1 bar on average) was available for the cake deliquoring in the final stage of operation. The average air flow rates in the beginning and end of air deliquoring were 250 and 270 L/min, respectively. The number of experiments performed with the vertical filter press was 14, out of which 11 included cake washing.

2.3. Washing sequences

Three filtration tests without cake washing were performed. The cake washing tests were divided into three categories, based on the washing sequence applied:

- I) Hose washing: Pumping → Hose washing → Pressing → Air drying (5 tests)
- II) Hose washing followed by classic cake washing: Pumping → Hose washing → Pressing I → Cake washing → Pressing II → Air drying (4 tests)
- III) Classic cake washing: Pumping → Pressing I → Cake washing → Pressing II → Air drying (2 tests)

In the PF tests, the target pumping time was 2.5 min. In tests where classic cake washing was included in the washing sequence, the first pressing was performed at only 10 bar, in order to avoid significant consolidation of the cake prior to the cake washing stage.

2.4. Sampling from filter cakes and filtrates

Three samples were taken from each filter cake, from certain locations, as shown in Fig. 4. The samples were extracted using a specific sampling tool, and homogenized and analyzed for solid content and Na concentration.

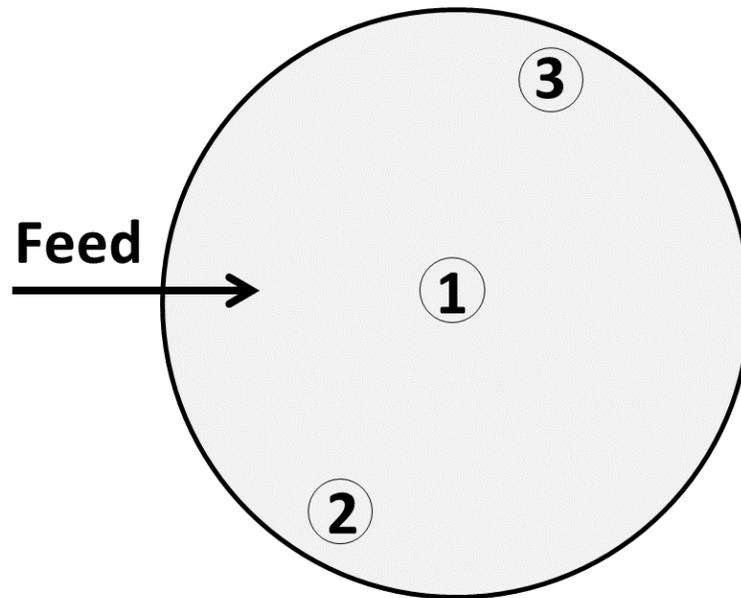


Fig. 4 Sampling from filter cakes.

Filtrate samples were taken from all stages of each filtration experiment. In the case of hose washing, for instance, the number of filtrate samples taken from each cycle was four. All filtrate samples were then analyzed for sodium content as described in detail in Section 2.6.

2.5. Leaching tests

The objective of the leaching tests carried out at laboratory scale was to investigate the extent of sodium dissolution from the bauxite residue, using large wash ratios at 20 and 65 °C, as shown in Table 1. Each batch for the leaching tests was prepared by adding bauxite residue slurry in water to obtain diluted slurries containing approximately 5-40 % of the original slurry and 2.0-16.2 % of total solids (TS). The total weight of each batch was 40.0 g, and the exact mass of bauxite residue slurry was recorded and the exact wash ratios for the leaching experiments were calculated.

Table 1. Levels of variables in the leaching tests.

Test	Slurry content, target (wt.%)	Solids content TS, target (wt.%)	T (°C)	Wash ratio, real (kg _{water} /kg _{solids})
1	40	16.2	20	3.8
2	20	8.1	20	10.2
3	10	4.1	20	23.1
4	5	2.0	20	47.5
5	40	16.2	65	3.8
6	20	8.1	65	10.1
7	10	4.1	65	22.6
8	5	2.0	65	48.2

2.6. Tests with a Nutsche pressure filter

A small scale pressure filter (Nutsche), with a filtration area of 19 cm², was used in order to evaluate the mass balance of the washing process at a moderate pressure difference ($\Delta p = 6$ bar). Only two tests were carried out with this filter. The wash ratios were 2.2 and 4.4 kg_{water}/kg_{solids}. Nitrogen was let into the filter to set the filtration pressure. The slurry temperature was 65 °C, and the jacketed filter was kept at the same temperature during the experiments.

2.7. Analytical methods

The particle size distribution of the solid material was measured with a Beckman Coulter 13 320 analyzer. The concentrations of suspended (TSS) dissolved (TDS) and total (TS) solids in the slurry were measured by drying the slurry samples in an oven at 105 °C until a constant weight was obtained. This drying method was also used for determining the total solid contents of the cakes.

The sodium concentration in the slurry, cake samples and filtrates was measured with atomic absorption spectroscopy (AAS) using a Thermo Scientific iCE 3000 AA spectrometer. The slurry and filtrate samples were mixed and settled, and the clear supernatant was filtered through a syringe filter of 0.2 µm pore size. To analyze a cake sample, 10 g of the original cake was reslurried with roughly 90 g of water. After this, the samples were shaken to ensure homogeneous quality of the slurry, and allowed to stabilize and settle without agitation for at least 24 hours prior to filtration and analysis.

As stated above, the elemental composition of the slurry was measured with a Jeol JSM-5800 SEM-EDS. The mineralogical composition of the slurry was determined using X-ray diffraction (XRD). A Bruker D8 Focus diffractometer with the following specifications was applied: CuK α radiation, 40 kV voltage, 40 mA current, step size 0.05°, and one step per second. The range of measurement for the spectrum was 2 - 70°.

3. Theory

Two types of cake washing sequences were applied, either separately or successively in the same sequence. The hose washing process represented a combination of reslurry washing and displacement washing, depending on the local state of cake formation and compression in the filter. The classic cake washing sequence aimed at an ideal displacement washing.

An illustrative example of displacement and reslurry washing, according to Svarovsky (1981), showing the solute concentration relative to the original solute concentration (c/c_0) in the filtrate, is presented in Fig. 5. Curve A1 shows an example of a typical washing curve obtained by displacement washing, whereas dotted line A2 represents an ideal displacement. A dilutive type of washing, reslurry washing, performed in one stage, is illustrated by curve B. In the case of displacement washing, the washing result depends largely on the properties (particle size, liquid content, and homogeneity) of the filter cake, which in turn are influenced by upstream processing and the previous operational stages in the filter unit. Regarding the reslurry washing, the liquid content of the cake determines the wash water consumption. During the pumping stage, when the cake starts to form in the filter chamber, a relatively large amount of filtrate (mother liquor) is removed from the slurry. The wash water requirement for the hose washing is therefore significantly smaller than in the direct dilution of the slurry prior to filtration.

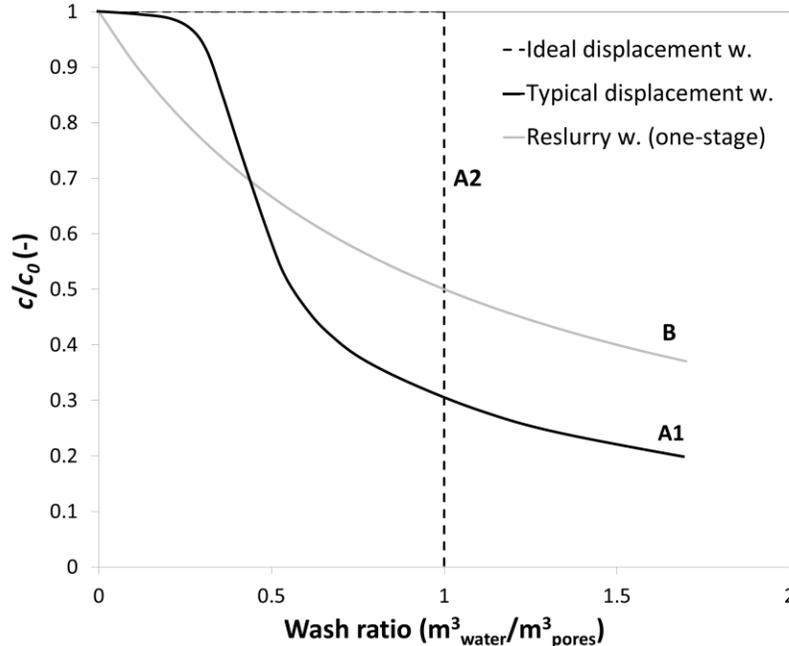


Fig. 5 Comparison of cake washing methods using volumetric wash ratios. Displacement washing: a typical (A1) and an ideal (A2) washing curve. Reslurry washing when equilibrium is reached: one-stage washing of a cake similar to that in displacement washing (B).

On the basis of Fig. 5 it may look that hose washing is not a serious alternative to conventional displacement washing. The hose washing sequence was, however, investigated in this study because of its benefits: 1) the hoses are washed immediately after the pumping stage, which reduces the long-term risk of hose blocking, 2) in the case of compressible cakes, the cake resistance is kept at a minimum, 3) the capacity can be increased, as one pressing stage can be skipped.

In this study, the mass-based wash ratio WR_M was defined as the ratio of the mass of wash liquid $m_{wash\ liquid}$ to the mass of dry solids in the cake (Eq.1).

$$WR_M = \frac{m_{wash\ liquid}}{m_{cake}(1 - X_{cake})} \quad (1)$$

where X_{cake} is the residual moisture content of the filter cake and m_{cake} is the mass of the filter cake.

The alkali recovery R_{Na} was calculated from:

$$R_{Na} = \frac{m_{Na,filtrates}}{m_{Na,slurry}} \quad (2)$$

where R_{Na} is the alkali recovery, $m_{Na,filtrates}$ is the total mass of soluble Na in the filtrates and $m_{Na,slurry}$ is the mass of Na in the feed slurry. The filtrates include the primary filtrate, i.e. mother liquor, and the wash filtrates collected during and after the cake washing stage.

The conventional method for calculating the recovery of Na from bauxite residue slurry is to compare the amount of soluble Na in the washed filter cake with the amount of soluble Na in the feed slurry. The large amount of analyses performed during this study, however, showed clearly that this method is highly unreliable due to the uncertainties related to 1) representative sampling from the filter cakes, 2) inaccuracy of the analysis of the cake samples, and 3) changes in the solubility of the ‘insoluble’ components during the filtration cycle. For the above-mentioned reasons, the alkali recovery values were calculated based on the quantities and Na concentrations of slurries and filtrates.

As was mentioned above, the results of this study revealed that the amount of soluble Na increased during the deliquoring process. This observation was confirmed by the mass balance calculations. To demonstrate the dissolution phenomenon, a new parameter; ‘Extra Na in filtrates’ (g_{Na}/kg_{slurry}), was introduced, and it was defined by equation:

$$c_{Na,extra} \left(\frac{g_{Na}}{kg_{slurry}} \right) = c_{Na,filtrates} \left(\frac{g_{Na}}{kg_{slurry}} \right) - c_{Na,slurry} \left(\frac{g_{Na}}{kg_{slurry}} \right) \quad (3)$$

The gross capacity of the filter unit, C_G , with respect to the rate the solids are deliquored, excluding the technical time required for filter opening and closing, cake discharge, cloth washing, etc., is given as:

$$C_G = \frac{m_{cake}(1 - X_{cake})}{A_{filter}t_{operation}} \quad (4)$$

where A_{filter} is the effective filtration area and $t_{operation}$ is the total time of the operational stages (filtration, 1st pressing, washing, 2nd pressing, air drying).

4. Results and discussion

4.1. Alkali recovery

The recovery of Na could be significantly enhanced by cake washing (Fig. 6). Irrespective of the washing sequence applied, the improvement in the Na recovery was very good. When it comes to the wash water consumption, classic cake washing can be recommended. The maximum wash ratio used in this study was only 1.4, which is quite a typical ratio for the countercurrent decantation systems at current alumina refineries. It is probable that the wash ratios that can be readily and economically used in filter presses at alumina refineries are smaller than 1, approximately 0.8, which equals about 2 times the void volume of the cake.

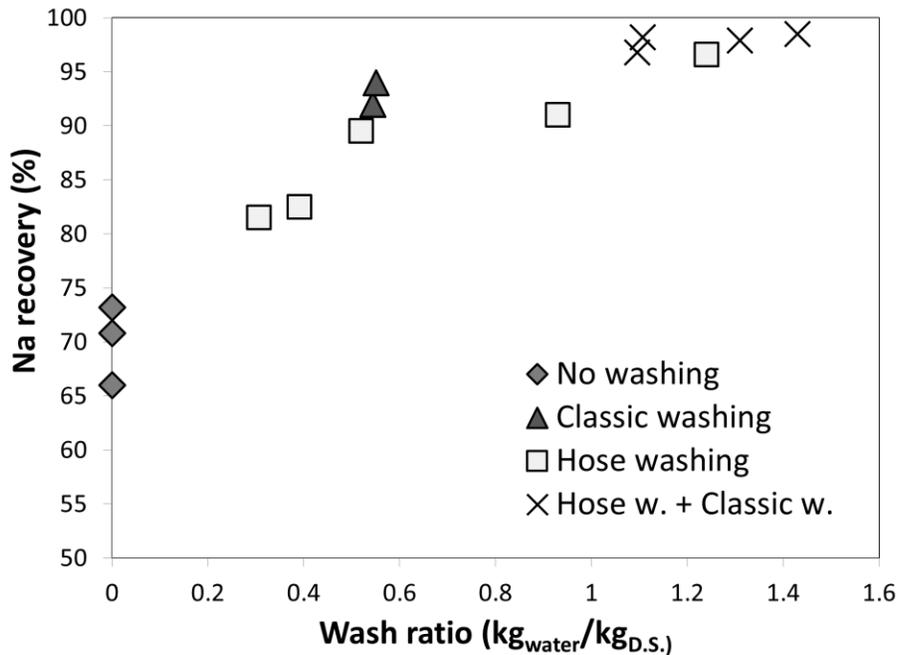


Fig. 6 All tests: Alkali recovery values obtained at various wash ratios (kg of water / kg of dry solids in the cake) using different cake washing sequences.

The values of sodium recovery obtained with the use of the vertical filter press were excellent. In an earlier study of the authors (Kinnarinen et al., 2012), a horizontal membrane filter press (MFP) with vertical plate assembly was used for the filtration and washing of a similar slurry. The washing results obtained with the horizontal MFP were

good, and the Na recovery was almost 90 % at a wash ratio lower than 2 $\text{kg}_{\text{water}}/\text{kg}_{\text{D.S.}}$. However, it is easy to conclude that the use of the vertical filter press with a horizontal cake position helps to improve the efficiency of the cake washing process significantly. As can be approximated on the basis of Fig. 7, Na recovery of 95 % can be obtained by the hose washing method, using a wash ratio of 1. This wash ratio can be regarded as relatively realistic for most refineries, although even lower wash ratios (about 0.7) are often aimed at.

Regarding the mass balance of the process, some interesting observations about the dissolution of Na were made: the results showed that different relative amounts of Na can be recovered at 65 °C during 1) leaching at atmospheric pressure, 2) filtration with a Nutsche-type pressure filter, and 3) filtration with a PF 0.1 pressure filter (Fig. 7). The results indicated clearly and consistently that different proportions of Na became soluble during the filtration cycle. This means that deliquoring and washing of bauxite residue with a filter press actually improves the recovery of sodium that can be recycled back into the process, which also helps to reduce the leaching of sodium from the cake after the final disposal. It was shown that process temperature and pressure have an important role in the dissolution of Na from the solid phase (Fig. 7). The Na recovery also increased with the wash ratio, probably as a result of short-term reduction in the local pH. In the case of the slurry used in the experiments, the phenomena illustrated in Fig. 7 together increased the alkali recovery values by approximately 10-20 %. It must be emphasized at this point that the leaching results presented in Fig. 7 are interesting only from the phenomenological point of view, because the use of such high wash ratios is not possible in the refinery scale. Additionally, liberation of Na as a result of mechanical shear caused by cake pressing may play an important role, and should be studied in closer detail in the future.

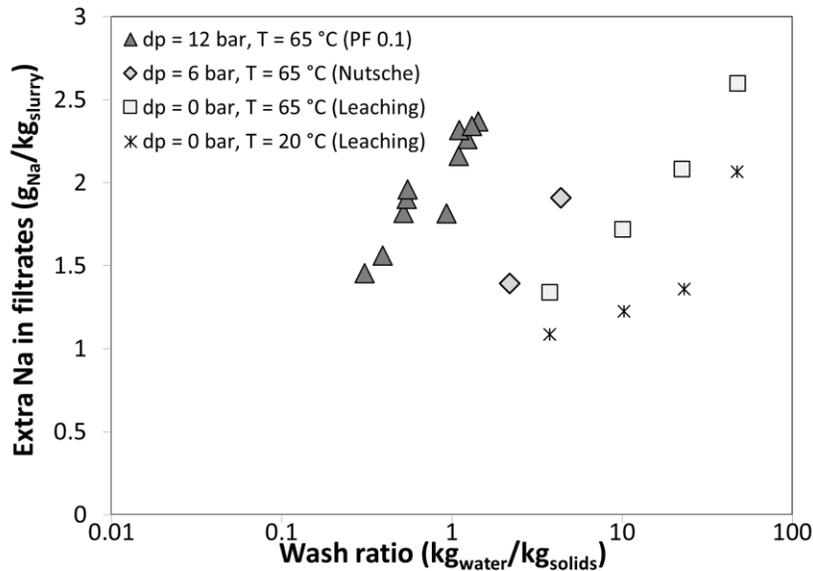


Fig. 7 Comparison of Na dissolution at different conditions.

The presence of certain mineral phases in the bauxite residue may explain the above-described observations related to the dissolution of sodium. For instance, some of the desilication products formed during bauxite processing have been reported to be soluble in

water. The theory about Na^+ exchange, proposed by Thornber and Binet (1999), is likely to apply in this case as well. They observed that during sequential washing of bauxite residue with water, the release of Na^+ was related to the total alkalinity of the obtained wash solution. Thornber and Binet (1999) also state that particulate desilication products represented the most important source of Na^+ , which was apparently exchanged from the cages of these particles. The pH where these desilication products start to dissolve, has been reported to be as high as near 9 (Gräfe et al., 2009, 2011).

Snars and Gilkes (2009) observed that the concentration of dissolved Na^+ in bauxite residue slurry was strongly dependent on the pH. They found that the concentration of dissolved sodium was 6-7 times higher at pH 3, compared with that at pH 11. Snars and Gilkes (2009) also present composite buffering curves for bauxite residue. The buffering curves indicate that the high initial pH was caused by NaOH, while the buffering effect at pH 6-8 resulted from dissolution of calcite and sodalite. According to Gräfe et al. (2011), typical Na containing buffering solids present in bauxite residues include natron-decahydrate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$), hydroxysodalite ($\text{Na}_6[\text{Al}_6\text{Si}_6\text{O}_{24}] \cdot 2\text{NaOH}$) and cancrinite ($\text{Na}_6[\text{Al}_6\text{Si}_6\text{O}_{24}] \cdot 2\text{CaCO}_3$). Another potential source of Na^+ could be NaOH entrapped in the solid particles.

4.2. Gross capacity

The obtained alkali recoveries together with the corresponding gross filtration capacities are presented in Fig. 8. The gross capacity in this case was defined according to Eq. (4), i.e. the technical time and the dead time of the filter unit were omitted. It is clearly shown that the gross capacity is notably reduced when the washing result is improved. In industrial cake washing, the applied process is always a trade-off between capacity and washing efficiency. From an economical point of view, a cake washing process is optimally designed to fulfill the requirements of national laws, while obtaining an acceptable alkali recovery, taking into consideration the cost of wash water and taking care of environmental aspects.

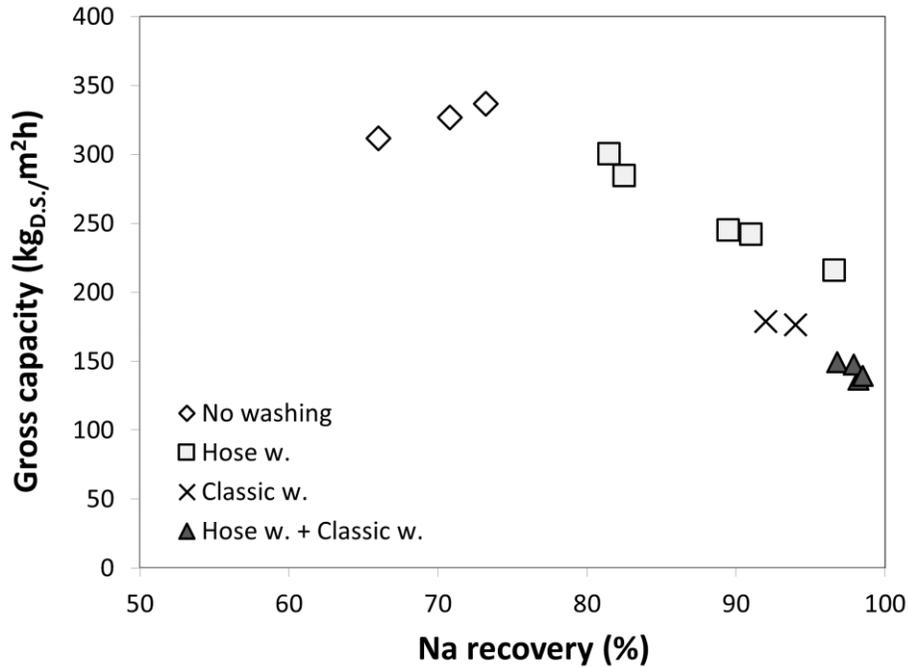


Fig. 8 Comparison of cake washing sequences: Alkali recovery against gross filtration capacity.

The obtained gross capacities for the cake washing experiments can be regarded as very good, taking into account the fact that also the cake washing results were excellent. In an earlier study performed by Rousseaux et al. (2008), focusing on filtration of a different bauxite residue slurry with comparable solid concentrations and conditions, except a lower temperature (30 °C in most tests), the capacities were significantly lower, approximately 50-90 kg m⁻² h⁻¹. Rousseaux et al. (2008) used a horizontal press-filter with vertical plates, but they did not perform cake washing.

4.3. Evenness of cake washing

A summary of local washing results for the filtration tests is presented in Table 2. As shown in Table 2, the relative standard deviations of the residual Na concentrations in the three selected measurement points, calculated for wet cakes, ranged from 3 to 21 %. The residual moisture contents in the cake samples varied between 25.3 and 27.7 %. The average moisture contents in points 1, 2 and 3 were 26.5, 26.7, and 26.6 %, respectively. There was no significant correlation between the moisture and the Na contents of the cake samples. The average Na concentrations in sampling points 2 and 3, which were located on the edge of the cake, were 6 and 14 % lower, respectively, than in point 1 that was located in the center of the cake. Comparison of the washing sequences regarding the evenness of washing reveals that the classic washing sequences may result in the lowest deviation, probably because of reduced channeling of the wash water in pre-squeezed filter cakes during displacement washing.

Table 2. Local differences in washing results with respect to sodium concentrations and their standard deviations (Points 1-3 in Fig. 4).

Test	Washing sequence	Min. (g/L)	Max. (g/L)	Mean (g/L)	Stdev. (g/L)	Relative stdev. (%)
4	hose w.	5.24	6.97	6.39	1.00	15.61
5	hose w.	5.73	6.63	6.28	0.48	7.62
6	hose w.	5.10	5.56	5.33	0.23	4.33
7	hose w.	5.02	6.02	5.63	0.53	9.42
8	hose w.	3.56	5.52	4.59	0.98	21.46
9	hose w. + classic w.	3.02	4.63	3.77	0.81	21.43
10	hose w. + classic w.	3.21	3.92	3.57	0.36	10.02
11	hose w. + classic w.	3.06	4.53	3.88	0.74	19.28
12	hose w. + classic w.	3.55	3.77	3.65	0.11	2.94
13	classic w.	3.37	4.15	3.76	0.39	10.33
14	classic w.	3.92	4.14	4.05	0.11	2.82

5. Conclusions

This study aimed at effective use of a vertical filter press for the filtration and washing of bauxite residue. The performance of three different washing sequences was evaluated. Additionally, variation in local washing results was investigated. The highest alkali recovery, 98 %, was obtained at the wash ratio of 1.4, by using a combination of hose washing and classic cake washing methods. However, the results with classic cake washing were almost as good (up to 94 %), while the quantity of wash water consumed was reduced to about 0.55. The dissolution of sodium from the filter cake was shown to depend on a few process variables. A more detailed study should be performed to fully understand the dissolution phenomena taking place in the filter. In the filter unit investigated in this study, the distribution of wash water on the cake in the filter unit was quite uniform. The average Na concentrations in the sampling points located on the cake edge were lower than those in the center of the cakes, respectively. This implies that the cake solidosity and the local flow resistance were higher in the cake center.

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