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Assessment of the influence of aqueous solutions chemistry on the water absorbency of recycled superabsorbent polymers

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ABSTRACT

This study aims at understanding the effect of water chemistry on the absorbency of superabsorbent polymers (SAP) that can be used for densification of tailings slurries. For this purpose, the absorbency of recycled SAP from unused hygienic diapers was determined for different aqueous solutions by varying their sulphates and salts concentrations and their pH. A SAP dosage of 4 g/L was mainly used. Experimental results obtained 72 hours after the SAP addition into the solutions showed that the absorption rate is at its maximum in deionized water. However, the addition of cations from salts in the solution has shown a drastic reduction in the SAP's absorption rate. The ensuing pH of the solution showed that the acidity had an inhibitory effect on the SAP's absorption rate.

RÉSUMÉ

Cette étude vise à comprendre l'effet de la chimie de l'eau sur le pouvoir absorbant de polymères super absorbants (SAP) pouvant être utilisés pour la densification des pulpes de résidus miniers. À cette fin, le pouvoir absorbant des SAP recyclés à partir de couches hygiéniques non utilisées a été déterminé pour différentes solutions aqueuses en faisant varier leurs concentrations en sulfates ainsi qu'en sels et leur pH. Une dose de SAP de 4 g/L a été principalement utilisée. Les résultats obtenus après 72 heures de résidence du SAP dans les solutions ont montré que la capacité d'absorption est maximale dans l'eau dé-ionisée. Cependant, l'addition de cations provenant de sels à la solution a montré une réduction drastique de la capacité d'absorption du SAP. Le pH des solutions a montré que l'acidité avait un effet inhibiteur sur la capacité d'absorption du SAP.

1 INTRODUCTION

Mineral processing in the concentrator generates tailings that are usually stored in ponds at a pulp density or solids content ranging between 30 - 45% by weight (Aubertin et al. 2002; Aubertin and Chapuis 1991; Vick 1990).These ponds are built to serve as retention and containment of these tailings in impoundments. The tailings deposition is usually made from one or more discharge points at the crest of the dikes ((Vick 1990); (Blight 2009). These points of discharge are moved periodically to fill the parks evenly.

The deposition of tailings in the form of a pulp with a solids content varying between 30–45% is often accompanied by a problem of water management which can be at the source of tailings dam failure. In fact, the main causes of tailings are dam failure, slope instability (static failure), seepage and internal erosion, foundation conditions, foundations with insufficient investigations, overtopping, structural inadequacies, inadequate or failed decants, liquefaction (earthquake - seismic instability), mine subsidence, external erosion (Aubertin et al. 2002; Poncelet 2012). In addition, the conventional deposition of tailings in the form of pulps is accompanied by the phenomenon of hydraulic segregation of particles. This is because coarse particles tend to settle closer to the containment dams, while the finer ones settle further away from the discharge point.

To overcome the physical instabilities and hydraulic segregation mentioned above, researchers have looked at alternative methods of tailings deposition and the most common are those focusing on the tailings densification. Robinsky (1975) was the first to suggest an increase of the density of tailings before their deposition in order, among other things, to reduce their susceptibility to instabilities. Today, it is possible to densify tailings up to a pulp density of 85%. The maximum density of the pulp varies according to the rheological and hydrogeotechnical properties of the tailings, as well as according to the method used to densify them (Benzaazoua et al. 2004; Paterson 2004; Boger et al. 2006; Meggyes and Debreczeni 2006; Ouattara 2011;). One can distinguish thickened, paste and filtered tailings that can be obtained using high density thickeners, deep cone and conventional thickeners and disc filters, respectively.

A new method that has not been sufficiently explored in the mining industry is the use of superabsorbent polymers (SAP) for the densifying of mine tailings. Indeed, SAPs are hydrophilic materials capable of storing large quantities of water between their macromolecular chains (Bahaj et al., 2009). Superabsorbents are substances usually consisting of a white powder similar to table sugar (Zohuriaan-Mehr and Kabiri 2008). The high absorption rate of SAP allows them to store huge quantities of water. Because of this characteristic, SAPs were particularly used in the 1960s in the field of agriculture in the United States to improve the water retention property of cultivated soil (Zohuriaan-Mehr and Kabiri 2008). For several years now, the PSAs have found application in various fields other than agriculture in particular, for the manufacture of disposable diapers, in medicine, in the field of horticulture and in the protection of the environment through their power. sequestering heavy metals (Gao 2002).

The use of SAP in densifying tailings is worthy of more considerations. Some preliminary work on the densifying efficiency of SAP for mining tailings was carried out at the Research Institute of Mine and Environment. The absorption rate of a given SAP for tailings water may however depend on many factors, including the chemical properties of the water. Various authors have studied the evolution of the absorption rate in different pH buffer solutions (Bao et al. 2011; Lee and Wu 1997; Li et al. 2009; Li et al. 2004; Pourjavadi et al. 2004; Wu et al. 2012a; Wu et al. 2012b). Regarding the influence of the ionic charge of the solution, Ismi et al. (2015); Li et al. (2004); Zheng et al. (2007) have worked on the behavior of sodium polyacrylate in contact with aqueous solutions in the absence and in the presence of metal ions.

The purpose of this article is to study the absorption rate and kinetics of SAP in different controlled solutions, i.e., deionized water, and acid and basic salt solutions containing different cations.

2 CHARACTERISTICS OF SUPERABSORBENT POLYMERS

The type of polymer used in these tests is sodium Polyacrylate recovered by Recyc PHP Inc. (Drummondville, Québec, Canada) from unused defective hygienic diapers. Sodium polyacrylate in solid form (in powder form) contains sodium atoms attached to oxygen atoms. The bond can be broken by dissolving the polymer in water, as seen in Figure 1.

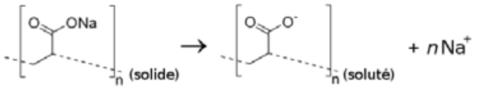

Figure 1. Chemical illustration of the dissolution of sodium polyacrylate in water

In the solid state, the polymer chains are folded back on themselves. As each oxygen atom is connected to a sodium atom, both of them have zero charge, and there are no attractive or repulsive forces between the different pieces of a chain. In water, the chain is stripped of the sodium atoms that have become free Na^+ ions. The chain then becomes negatively charged (at the level of the oxygen atoms) and the different pieces of chain repel, the chain unfolds, thus allowing the water molecules to be inserted. In this article the polymers used are granular particles. When placed in aqueous solutions, the polymers absorb very large quantities of water in a short time. The wet density of the gel formed is high due to the large amount of water absorbed. This amount of absorbed water can be quantified in terms of the absorption rate of the polymers that can be defined as follows:

$$K_{SAP}(g/g) = \frac{M_{SAP-w} - M_{SAP-d}}{M_{SAP-d}} \qquad [1]$$

where K_{SAP} is the absorption rate (g water / g SAP), M_{SAP-d} is the dry polymer mass (g) and M_{SAP-w} is the mass (g) of the wet polymer (gel formed after adsorption of water).

3 EXPERIMENTAL METHOD

3.1 Materials

Two types of SAP were used to carry out this study, namely the SAGM85F SAP type which diameter is between 100 µm and 400 µm and the SAGMD06K SAP type which diameter varies between 300 µm and 1100 µm. Hereafter, these SAPs are called fine-grained and coarse-grained SAP, respectively. The objective was to first compare their absorption rate and then decide which type of SAP to use in further work.

The absorption rate of SAP in deionized water was compared with SAP absorption rates in other chemically controlled solutions. They first included solutions of potassium sulphate (K_2SO_4) and aluminum ($Al_2(SO_4)_3$) with sulphate contents ranging from 2.5 g/l to 10 g/l; then salt solutions of potassium chloride (KCl) and magnesium ($MgCl_2$) with concentrations between 0.5 g/l and 5 g/l; and finally acidic and basic solutions with pH ranging from 1.4 to 11.7.

3.2 Method of determining the absorption rate

SAPs were added directly to a given volume of solutions. To determine the absorption rate using Eq. [1], the final weight of the swollen polymers after absorption (M_{sap}-w) was measured after removing any interstitial water that may remain in the mixture, leaving only the mass of swollen polymers containing the absorbed water. This was done using a pressure filter (Figure 2). For this purpose, the swollen SAP was deposited on filter paper and placed at the bottom of the metal cylinder of the filter. Air pressure of about 200 kPa was applied for 30 minutes to allow drainage of interstitial water. The wet sample obtained was weighed. The mass of the filter paper in the wet and filtered state was previously weighed and deduced from that of the final sample obtained (see Figure 3). Other authors have used the vacuum filtration method (Ismi et al. 2015) and some others the method of gravity dewatering (Bahaj et al. 2009).

The applied filtration method was previously verified to ensure that there was no loss of absorbed water caused by the applied filtration pressure by comparing the results of the latter with those obtained using a vacuum filter, which uses suction to drain interstitial water in SAP. Both methods gave the same results, except that vacuum filtration yielded results in a longer time (about 25 times the filtration time with the pressure filter).

The reliability of the filtration process using the filter press was performed by measuring the final mass of the swollen polymer samples (prepared under the same conditions; i.e. the same amount of SAP in the same amount of deionized water) when the pressure in the pressure filter varied between 50, 100, 150 and 200 kPa. The results showed that pressures ≤ 200 kPa had no influence on the absorption rate of SAP.

Figure 2. Pressure filter

Figure 3. Initial filter paper and hydrogel SAP after filtration

3.3 Adsorption test of SAP in deionized water

The influence of the mass of deionized water used in the mixture was first evaluated by determining the variation of the absorption rate according to the variation in quantities of water for the same conditions (same amount of SAP, same residence or contact time). For this, several mixtures were prepared by adding 0.8 g of fine and coarse SAP to amounts of deionized water varying from 100 ml to 600 ml. The absorption rate was measured after a residence time of 30 minutes for all mixtures.

3.4 Adsorption test of SAP in metal salt solutions

These tests aimed to verify the behavior of SAP in solutions containing metal chlorides and sulphates. Three types of solutions containing metal chlorides were prepared: solutions containing potassium chloride (KCl), sodium chloride (NaCl), and magnesium chloride ($MgCl_2$). The solutions containing metal sulphates include potassium sulphate (K_2SO_4), sodium sulphate (Na_2SO_4) and aluminium sulphate ($Al_2(SO_4)_3$).

For potassium chloride and potassium sulphate solutions, several samples at different concentrations were prepared to assess the effect of varying concentrations: 0.5 g/l, 1 g/l. 2.5 g/l and 5 g/l for KCl and 2.5, 5, 10 g/l for K_2SO_4. For the remaining solutions, a single concentration was used, namely 5 g/l, for comparison purposes and for studying the effect of the presence of monovalent, divalent or trivalent cations on the absorption rate. The solutions were prepared in 1 l bottles of deionized water by gradually adding the salt powders. The amount of sulfates (SO_4^{2-}) to be added was determined using stoichiometric ratios with the typical following equation (valid for K_2SO_4):

$$C_{K_2SO_4}(g/l) = \left(\frac{C_{SO_4}}{M_{SO_4}}\right) \times M_{K_2SO_4} \qquad [2]$$

where $C_{K2SO4:}$ is the mass concentration of potassium sulphate (g/l), C_{SO4} the mass concentration of sulphate (g/l), M_{SO4} the molar mass of sulphate (mol/l) and M_{K2SO4} the molar mass of potassium sulphate (mol/l).

A quantity of 0.8 g of coarse SAP was then added to the different sulphate solutions. The SAP absorption rates in each mixture was then measured for different residence periods.

4 RESULTS AND DISCUSSIONS

4.1 Influence of the quantity of deionized water on the adsorption rate K_{SAP}

For these tests, samples were prepared with the same amount of fine and coarse SAP (0.8 g) but added to 100, 200, 300, 400, 500 and 600 ml of deionized water. Figure 4 shows the variation of the absorption rate with increasing amount of water. Adsorption rates K_{SAP} were determined after a contact period of 30 minutes. It can be observed for both coarse and fine SAP, that the absorption rate increases with the volume of water and reaches a maximal value around 300 ml of water. Afterwards, the rate of absorption seems to remain almost constant. In other words, for quantities of water greater than 300 ml, we observe water coming out of the filter press. Given these results, the amount of solution was set at 500 ml for deionized water and at 200 ml for salt solutions. The latter is fixed at 200 ml because previous tests have shown that the absorption rates K_{SAP} remain very low in these solutions.

We can also note in Figure 4 the absorption rate in coarse grains is higher compared to that obtained in fine grains. Around 210 g/g for coarse grains and 136 g/g for fine grains in 400 ml of deionized water and 0.8 g of SAP. These results made it possible to focus on the use of coarse SAP for the rest of the tests.

The results in Figure 4 also show that grain size of the SAP has a significant influence on K_{SAP}: the coarser the SAP, the higher the absorption rate. For this reason, some tests were performed using only the coarse SAP.

4.2 Influence of the amount of SAP on the absorption rate K_{SAP} in deionized water

These tests were carried out by adding different amounts of SAP of 0.4; 0.6; 0.8 and 1.2 g of coarse SAP in 500 ml of deionized water. The absorption rates K_{SAP} were determined at residence times of 0.5 h, 1.5 h and 24 h to evaluate the absorption kinetics with increasing SAP dosage. The results are presented in Figure 5. One can notice that the absorption rate decreases with increasing SAP dosage for a given residence time. To confirm this, deionized water and K_{SAP} were determined at residence times of 1.5 h. A value K_{SAP} of about 175 g/g was obtained. This led us to the conclusion that the absorption rate decreases slightly when the amount of SAP increase. Ismi et al. (2015) reached to the same conclusion in their work.

Figure 5 shows that the absorption rate remains almost constant, with a very slight decrease, over the residence time for a given SAP dosage.

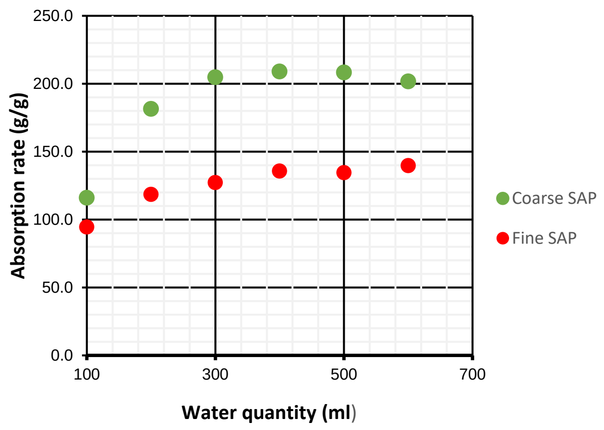

Figure 4. Variation of the absorption rate of 0.8 g of SAP (fine and coarse) with respect to the amount of water (≤ 600 ml) after a contact time of 30 minutes

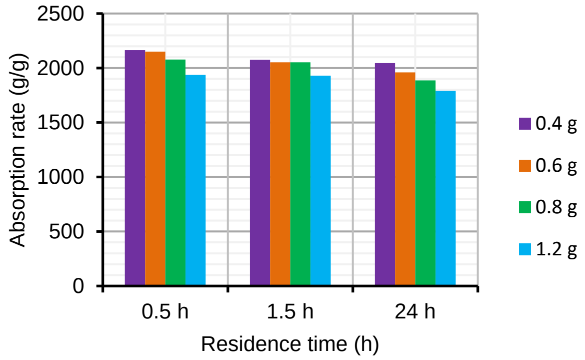

Figure 5. Evolution of the absorption rate as a function of the residence time (0.5 h, 1.5 h and 24 h) for different quantities of polymers added in 500 ml of water

4.3 Absorption kinetics of SAP in aqueous solutions

4.3.1 Deionized water

In a first step, tests were carried out by adding 0.4 g and 0.8 g of coarse SAP in 500 ml of deionized water in order to have control samples. The results obtained (figure 6) show that the absorption rate at residence times of 90 min (1.5h), 1440 min (24 h) and 4320 min (72h), are respectively 208, 205, 200 g/g for a dosage of 0.4g SAP and 205, 189, 200 g/g for a dosage of 0.8g SAP. In this figure, one can notice that the PSA absorb large quantities of solutions in the first 20 minutes and then releases the water to stabilize at a constant absorption rate. This observation was also made by Bahaj et al. (2009) in their study.

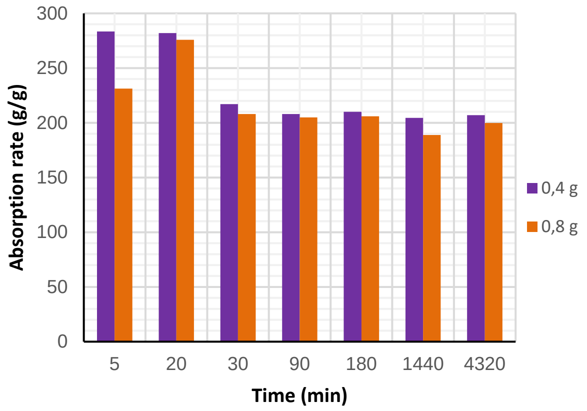

Figure 6. Evolution of the absorption rate of coarse SAP in 500 ml of deionized water for 0.4 and 0.8 g of SAP

4.3.2 Effect of the concentration in KCl and K_2SO_4

Solutions containing potassium chloride (KCl) and potassium sulphate (K_2SO_4) were used in order to evaluate the influence of salts on the absorption rate. These amounts of SAP were added in 200 ml of solutions with KCl concentrations of 0.5; 1; 2.5 and 5 g/l and K_2SO_4 concentrations of 2.5; 5; 10g/l. Figures 7 and 8 show the evolution of the absorption rate of coarse SAP KCl and K_2SO_4 , respectively, as a function of residence time (from 10 min to 72h). It can be observed that the absorption rate decreases when the concentration of salt increases regardless of the residence time considered. The absorption rates obtained in the potassium chloride solutions are lower than those of the deionized water but higher than those of the potassium sulphate solutions. This decrease can be explained by the insertion of the K^+ ions between the polymer chains, thereby causing a screen effect on the carboxylate groups, which reduces the intensity of the electrostatic repulsions between the negative charges (-COO) (Ismi et al. 2015). Indeed, chloride solutions absorb better than sulphate solutions. This may be due to the low (acidic) pH in potassium sulphate solutions (3.67 for 2.5 g/l, 3.46 for 5 g/l and 3.18 for 10 g/l) compared to the high pH (close to neutral) in the solutions of salt (7.2 for 0.5 g/l, 7.62 for 1 g/l, 7.43 for 2.5 g/l and 7.8 for 5 g/l). This conclusion will be confirmed or invalidated below by studies carried out on acidic and basic solutions. Referring to the work of (Li et al. 2009) who concluded, in their study of the influence of pH on a superabsorbent composite obtained by grafting laponite on a nonionic polyacrylamide, that the absorption rate is low in acidic pH, one could a priori confirm this statement. Other authors such as ((Wu et al. 2012a; Zhao and

Schiraldi 2005) have reached the same conclusion in their studies.

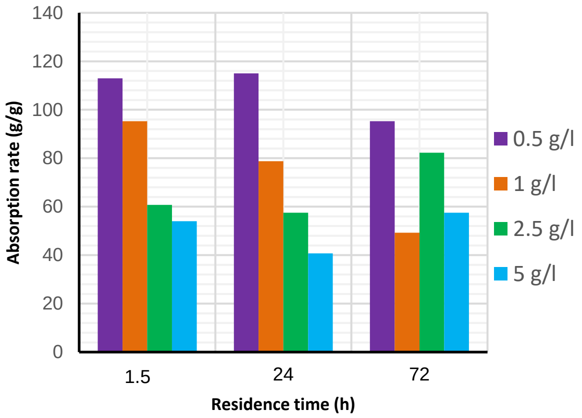

Figure 7. Evolution of the absorption rate of 0.8 g of coarse SAP added in 200 ml of potassium chloride solution at concentrations of 0.5; 1; 2.5 and 5 g/l

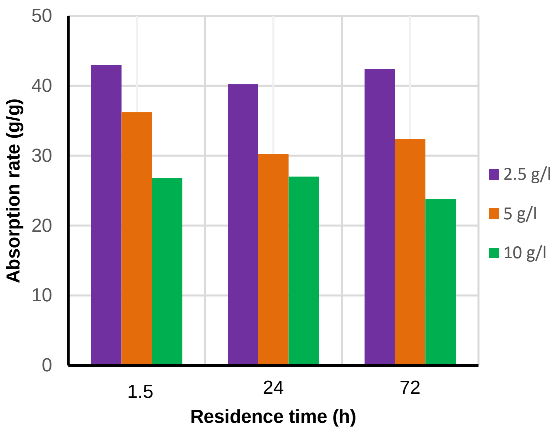

Figure 8. Evolution of the absorption rate of 0.8 g of coarse SAP added in 200 ml of potassium sulphate solution at sulphate concentrations of 2.5; 5 and 10 g/l

4.3.3 Effect of the type of cation in the solution

The effect of the type of cations present in the absorption solution has been studied using solutions of chloride such as sodium chloride, potassium chloride and magnesium chloride; then sulphate solutions such as sodium sulphate, potassium sulphate and aluminum sulphate. The cations released in solution are sodium (Na^+), potassium (K^+) both monovalent, magnesium (Mg^{2+}) and aluminum (Al^{3+}). These tests were carried out in order to investigate the influence of the latter on the absorption rate of SAP. Indeed the SAP of our study which chemical formula is COONa (Sodium Polyacrylate), once in solution, releases Na^+ to attract water molecules. Therefore, it would be interesting to focus on the influence of an excess of cations in solution.

These tests were carried out by adding 0.8 g of SAP in 200 ml of solutions for a residence time of 24 h and a concentration of 5 g/l. The results of Figure 9 show us that the absorption rates obtained in the solutions of sodium and potassium chlorides are high compared to those obtained with the solution of magnesium chloride.

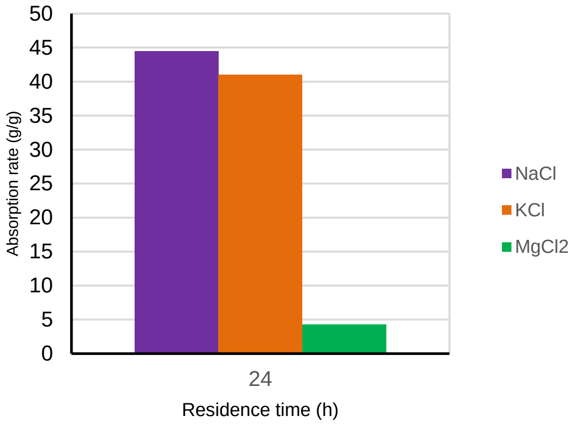

Figure 9. Absorption rate of 0.8 g of coarse SAP added in 200 ml of potassium, sodium and magnesium chlorides solutions at a concentration of 5 g/l for a residence time of 24 h

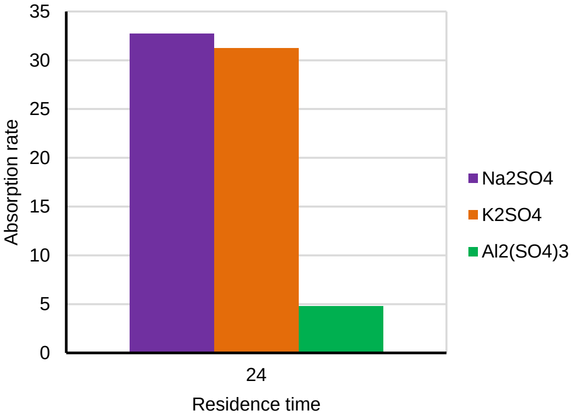

Figure 10. Absorption rate of 0.8 g of coarse SAP added in 200 ml of potassium, sodium and aluminium sulphates solutions at a concentration of 5 g/l for a residence time of 24 h

Similarly, the absorption rates obtained in the solutions of sodium and potassium sulfates are high compared to those obtained with the solution of aluminum sulfate as shown in Figure 10. The recorded absorption rate in the presence of divalent and trivalent cations is almost 8 to 10 times less important compared to that measured in the presence of monovalent cations. This is explained by the fact that the complexation of the carboxylate groups is possibly added to the screen effect observed in previous section. The formation of intermolecular complexes leads to additional crosslinking of the polymeric network and prevents the expansion of the polymer network (Ismi et al. 2015).

4.3.4 Influence of the pH of the solutions

These tests involved solutions with different pH values to evaluate their influence on the absorption rate of SAP. For the preparation of acidic and basic solutions, solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used, respectively. A pH meter was previously calibrated whit acidic, basic and neutral solutions and then set in continuous mode to be able to display the pH of the solution while gradually adding HCl or NaOH. The results are illustrated in Figures 11 and 12. These results show that the absorption rate is lower in the acidic solutions than it is in basic solutions and that it is low for both pH conditions compared to that obtained for deionized water (pH: 6.3).

For pH values ranging from 1.39 to 6.3, a rapid and abrupt increase in the maximum absorption rate from 3.5 to 207 g/g was observed after 72 hours of residency. This strong increase reflects the hypersensitivity of the phenomenon of SAP swelling with pH. For pH values ranging from 6.3 to 10.6, the absorption rate decreased from 207 to 153 g/g was observed but increase to 185 g/g for pH 11.71.

The work of Bahaj et al. (2009) reached the same assessment. It can be concluded that pH has a significant effect on the absorption rate of superabsorbent polymers. Wu et al. (2012b) also observed this effect in their study on the sensitivity of cellulose-based superabsorbent composites [poly (acrylic acid - co-acrylamide)] to changes in the pH of the medium.

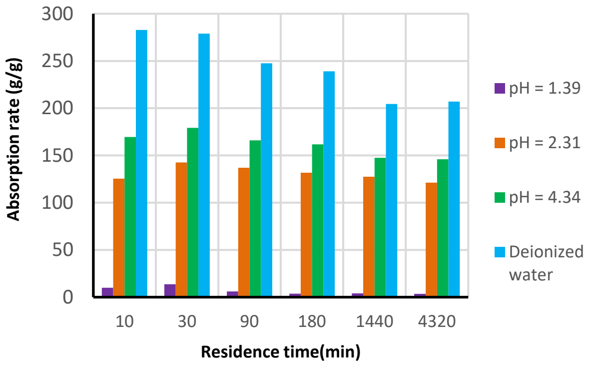

Figure 11. Absorption rate of 0.4 g of coarse SAP added in 100 ml of acidic solutions (HCl) and comparison with deionized water

5 LATEST REMARKS AND CONCLUSION

The objective of this analysis was to study the behavior of SAP in different controlled solutions in order to be able to determine their possible use for densification of tailings ponds. The experimental results have shown that indeed the aqueous solutions containing chloride and sulphate metal salts have a negative influence on the absorption rate of the recycled SAP used. The influence of the metal valence was also assessed: SAP better absorbed less aqueous solution in the presence of divalent and trivalent cations than monovalent cations.

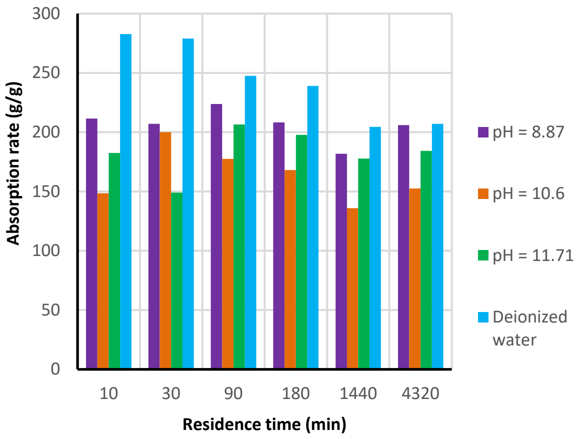

Figure 12. Evolution of the absorption rate of 0.4 g of coarse SAP added in 100 ml of basic solutions (NaOH) and comparison with deionized water.

Acidity leads to a large decrease in the absorption rate of SAP. The more acidic the solution, the lower the absorption rate which, however, gradually increases as the pH approaches neutrality. The basic solutions have a less pronounced effect compared to acidic solutions.

Results presented in this study are still preliminary, but provide an overview of the influence that mining water chemistry could have on the absorption rate of SAP. Moreover, it should be noted that several parameters have not been taken into account, namely the influence of the temperature of the absorption medium and the presence of solid grains. In order to comply with the reality of the mining industry, studies on actual tailings are underway at the UQAT Research Institute for Mines and Environment.

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