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Swelling ability, volume of cracks and hydraulic conductivity of a polymer modified bentonite subjected to wet and dry cycles

Michela DE CAMILLIS[1]

[1]*Ghent University, Department of Geotechnics, Ghent, BELGIUM*

ABSTRACT

Geosynthetic Clay Liners are widely used to isolate waste disposal facilities. However, long exposure to electrolyte solutions combined with temperature changes may impair their performance as barrier liners. Wet and dry cycles lead to desiccation and cracking of the bentonite. This study investigates the influence of wet and dry cycles with seawater on swelling ability, crack formation and permeability of a polymer modified clay, HYPER clay, and untreated bentonite. Untreated bentonite, bentonite treated with 2% and 8% polymer were evaluated through swelling tests, µCT scanning and hydraulic conductivity tests.

HYPER clay 8% presented the best performance. It swelled the most and its thickness was considerably larger compared to untreated clay. µCT analysis demonstrated the smaller volume of cracks of HYPER clays compared to untreated bentonite. In addition, the hydraulic conductivity of untreated bentonite increased within three cycles with seawater, while HYPER clay preserved low permeability.

Keywords: bentonite, polymer, wet/dry cycles, crack formation

1. INTRODUCTION

Geosynthetic clay liners (GCLs) are bentonite-based liners that are gaining acceptance as hydraulic barriers in containment and sealing applications (Petrov & Rowe, 1997). One important field of application is landfill capping systems. The aim of clay liners is to limit the infiltration of moisture, due to rainfall or water migration, through the barrier into the waste and to limit the release of leachate and gasses from the waste. GCLs are factory-manufactured clay liners containing a thin uniform layer of sodium or calcium bentonite sandwiched between two geotextiles or glued to a geomembrane.

The major significant component of the bentonite in GCLs is sodium montmorillonite. Sodium cations are able to bond with water molecules, increasing the interlayer space and forming tortuous flow paths. However, valence, concentration and dielectric constant of the hydrating solution influence the expansion of the diffuse double layer (DDL) of negatively charged clays (McBride, 1994). Accordingly, hydraulic conductivity and swelling of bentonite are related to the thickness of the DDL. A decrease of the thickness leads to an

* presenting author

increase of hydraulic conductivity resulting in particle attraction, shrinkage and cracking of clay (Shackelford et al., 2000). Therefore, bentonite is sensitive to chemical interactions with the hydrating liquid, and ion exchange can alter its physical properties (Meer & Benson, 2007). Several laboratory studies have investigated barrier performance deterioration of GCLs in contact with electrolyte solutions (Petrov & Rowe, 1997; Bouazza, 2002; Kolstad et al., 2004; Jo et al. 2005).

Nevertheless, the service life of a GCL cover can also be damaged by degradation due to seasonal temperature changes. Moreover, temperature as high as 70°C may occur due to daily thermal cycles when a geomembrane overlies a GCLs (Take et al., 2014). As a result, the hydraulic conductivity increases and loss of self-healing capacity occurs due to the combination of ion exchange and desiccation (Egloffstein, 2001). In particular, desiccation and contact with high electrolyte solutions are responsible for the collapse of the diffuse double layer and crack formation. Therefore, crack formation might not heal during rewetting due to the low swelling ability of the bentonite caused by the compression of the DDL. Several studies have been conducted to assess the effect of wet and dry cycles (Lin and Benson, 2000; Bouazza et al., 2006; Thiel et al., 2006; Bouazza et al., 2007; Rowe et al., 2011; Tang et al., 2011; Take et al., 2012; Hoor and Rowe, 2013; Mukunoki et al., 2014). Lin and Benson (2000) investigated the impact of wet and dry cycles on the swelling ability and hydraulic conductivity. The initial exposure to deionized water or tap water temporally delayed the reduction in swelling. On the contrary, the swelling capacity was reduced when the bentonite was wetted directly with 0.0125 M $CaCl_2$. The GCLs permeability increased of around two orders of magnitude after 4-6 wet and dry cycles due to crack formation and loss of self-healing capacity of the bentonite.

The GCL shrinkage caused by wet-dry cycles was investigated by Rowe et al. (2011). They found that the magnitude of shrinkage in the field is correlated to the water-retention of the GCLs, subsoil, number and duration of cycles.

Modified bentonites have been developed to improve bentonite performance in aggressive environments (Katsumi et al., 2008; Di Emidio, 2010; Malusis & McKeehan, 2013; Scalia et al., 2013). In this research, the HYPER clay technology has been investigated. HYPER clay is a polymer-treated bentonite created by combining natural bentonite with carboxymethyl cellulose (CMC). Once the CMC intercalates the clay platelets, the diffuse double layer is maintained open even in presence of factors that generally produce the collapse of the interlayer (Di Emidio, 2010).

The purpose of this investigation is to examine the impact of wet and dry cycles of HYPER clay and untreated bentonite on swelling ability, self-healing capacity, crack formation and hydraulic conductivity. The bentonites are hydrated with highly concentrated electrolyte solution such as seawater.

2. MATERIALS

Sodium bentonite (NaB) was used in this study and treated according to the HYPER clay procedure. Various physical and chemical properties of the bentonite are listed in Table 1.

Table 1. Material characterization

Property	NaB
Specific Gravity [-]	2.66
Liquid Limit [%]	660.55
Plastic Limit [%]	48.92
Plasticity Index [-]	611.62
CEC [meq/100g]	73
Smectites [%]	91
Quartz [%]	4
Feldspars [%]	2

The HYPER clay was prepared according to the procedure proposed by Di Emidio (2010). The principle of HYPER clay is to combine powdered Na-bentonite (NaB), referred to as base bentonite, with an anionic polymer (sodium Carboxymethyl Cellulose, Na-CMC). The treatment consists of dissolving the polymer in water and then adding the base clay. The material is mixed with a mechanical stirrer for 30 minutes to increase the specific surface area available for polymer adsorption. This slurry is then oven dried at 105°C for 16 hours to adsorb irreversibly the polymer. The HYPER clay is then ground first manually, using a mortar and pestle, and then mechanically. In this study, the NaB was combined with 2% and 8% of CMC, by dry mass of clay (HYPER clay 2% and HYPER clay 8% respectively). The dry mass per unit area of the bentonite was 7.5 kg/m^2 and the initial porosity was 0.718.

Deionized water was used as reference solution during the first wet cycle, for oedometer and hydraulic conductivity tests. Natural seawater, collected in the North Sea (Ostend, Belgium), was filtered through Grade 4 Whatman filter paper to remove coarse particles and stored in a tank. Table 2 shows the chemical characteristics of the solutions used in this study.

Table 2. Chemical properties of electrolyte solutions

Parameter	Deionized water	Seawater
EC (mS/cm)	0.002	44.8
Salinity (-)	0.0	28.6
pH	7.57	7.42
Na^+ (mg/L)	-	11517.9
K^+ (mg/L)	-	469.2
Mg^{2+} (mg/L)	-	1281
Ca^{2+} (mg/L)	-	478.5
Cl^- (mg/L)	-	19897
SO_4^{2-} (mg/L)	-	2352
HCO_3^- (mg/L)	-	183.1
CO_3^{2-} (mg/L)	-	18.0
NO_3^{2-} (mg/L)	-	43.4

3. METHODS

3.1. One-dimensional swell tests

One-dimensional swell tests were performed according to the procedure used by De Camillis et al. (2016). Samples of dry bentonite of untreated clay, HYPER clay 2% and HYPER clay 8% were poured in 70 mm diameter cells with initial porosity of 0.718 and 7.5 kg/m^2 dry mass. A sitting pressure of 1 kPa was used and the vertical swells were continuously recorded during hydration. The samples were subjected to six wet and dry cycles. Deionized water was the hydrating solution during the first cycle and seawater in the consecutive cycles. The specimens were allowed to swell for about 400 hours (16 days). After wetting, the samples were oven dried at 40°C until constant weight or the water content was between 10%-15%.

3.2. μCT scanning

μCT scanning is a non-destructive three dimensional (3D) imaging and analysis technique (Cnudde h Boone, 2013). With this method, the samples are fully reconstructed in 3D, based on a set of two dimensional (2D) projections or radiographs. μCT scans are carried out on the HECTOR system (High Energy μCT scanner, Optimized for Research), at the Centre for X-ray Tomography at Ghent University (UGCT), Belgium (Masschaele et al., 2013). Samples are placed on a rotating stage between the X-ray source and detector. After acquisition of the radiographs, the 2D projections are reconstructed into a stack of 2D slices through the object, building up the 3D image. The reconstruction step, as well as the subsequent image analysis on the images, are carried out using the in-house developed software tools Octopus Reconstruction (Inside Matters, Vlassenbroeck et al., 2007) and Octopus Analysis, formerly Morpho+ (Brabant et al., 2011), respectively. With VG Studio MAX®, a software tool of Volume Graphics, the analysed data could then be visualized in 3D.

In this research, μCT scans were performed on untreated sodium bentonite, HYPER clay 2% and HYPER clay 8%, after the third dry cycle and during the fourth wetting stage in order to assess the self-healing capacity and volume of cracks. In order to exclude side effects at the borders of the samples, the focus of this comparison lies within the central parts of the samples.

3.3. Hydraulic conductivity tests

Hydraulic conductivity tests were performed on untreated sodium bentonite, HYPER clay 2% and HYPER clay 8% according to the procedure of De Camillis et al. (2016). Tests were carried out in rigid wall permeameters with 71 mm diameter for NaB and HYPER clay 2% and 70 mm for HYPER clay 8%. The initial porosity of the samples was 0.718 and 7.5 kg/m^2 dry density. HYPER clay 8%, due to later shrinkage, was moved to a flexible wall 70 mm diameter permeameter during the second wet cycle to be able to permeate the sample. The specimen of HYPER clay 8% was confined with an effective stress of 15 kPa, in order to simulate in situ condition of a cover (Bouazza, 2002; Mazzieri and Pasqualini, 2006; Scalia and Benson, 2011). NaB and HYPER clay 2% recovered the initial diameter once rewetted, for this reason sidewall leakage did not occur.

The permeant liquids used were deionized water, during the first cycle, and seawater in the next cycles (as for swell tests). Termination criteria from ASTM D6766 were followed. The dry cycles were performed in a 40°C oven until constant mass was reached, or the water content was between 10%-15%.

4. RESULTS

4.1. One-dimensional swell tests

The first set of analysis investigated the impact of wet and dry cycles on swelling ability and self-healing capacity on untreated sodium bentonite, HYPER clay 2% and 8% through free one-dimensional swell tests in oedometer cells. Figure 1 outlines the swell related to the temporal behaviour of the tested samples. The first cycle in deionized water last 20 days. NaB and HYPER clay 2% were able to reach equilibrium while HYPER clay 8% did not yet achieve its maximum swelling. On average, the swelling capacity decreased with increasing the number of wet-dry cycles.

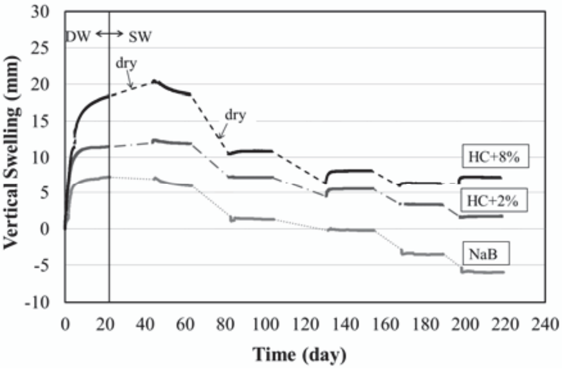

Figure 1. Temporal behavior of 1-D free swell tests at various wet-dry cycles of untreated sodium bentonite (NaB), HYPER clay 2% (HC+2%) and HYPER clay 8% (HC+8%)

Untreated sodium bentonite swelled the least compared to HYPER clays, reaching its maximum swelling of 7.3 mm in deionized water. However, the swell of NaB sharply decreased in contact with seawater. Therefore, these results are likely to be associated to the compression of the DDL which causes loss of self-healing and swelling capacities. As a consequence, the cracks formed during dry cycles might not be healed after rewetting.

HYPER clay 2% showed a clear trend of decreasing swelling ability with cycles. It swelled around 11.45 mm during the first cycle and it decreased up to 1.63 mm at the end of the sixth cycle.

As shown in Figure 1, HYPER clay 8% reported the best performance among the analysed samples. It swelled the most in deionized water (18.3 mm) and then the thickness decreased with the consecutive cycles. The most striking result to emerge from Figure 1 is that HYPER clay 8% thickness remained higher compared to untreated bentonite and HYPER clay 2% during further cycles with seawater. This behaviour may be linked to the presence of the polymer, which helps to keep the interlayer open (Di Emidio et al., 2015) and to enhanced water adsorption (Qui and Yu, 2007). The more surprising correlation is with sodium bentonite, the final swelling of HYPER clay 8% (7.22 mm) after six cycles is comparable to the maximum swelling in deionized water of NaB.

Swelling and adsorption capacity are strongly dependent on the electrolyte concentration of the hydrating liquid, such as seawater. Moreover, the combination with wet and dry cycles can further deteriorate the barrier performance of the bentonite. The diffuse double layer thickness is so compressed when it comes in contact with multi-valence and concentrated solutions. For these reasons, collapse of swelling ability was detected for NaB and the cracks, formed during desiccation, were not healed. On the contrary, the polymer treatment improves the swelling and healing capacity likely maintaining the DDL open in the long-term. The sample of HYPER clay 8% formed a rigid disk after the first dry cycle with seawater, which was maintained during the next cycles. Crack formation was detected on the edge where the sample was more in contact with the heat. However, HYPER clay 8% presented the first cracks during the fourth cycle.

4.2. µCT scanning

In order to quantitatively compare the amount of cracks and check for self-healing capacity of NaB, HYPER clay 2% and HYPER clay 8%, µCT scanning were performed in cooperation with Van Stappen J. and Cnudde V. at PProGress/UGCT - Department of Geology – SHE, Faculty of Sciences, Ghent University.

Figure 2. Specimens at the end of the third dry cycle and at the end of the fourth wet cycle

The analysis of the images was focused on the internal part of the samples to avoid edge effects. A cylindrical volume with a diameter of 35 mm and a height of 6.6 mm was chosen as a subsection to perform image analysis on. The samples were scanned at the end of the third cycle and during the fourth wet cycle (Figure 2).

Figure 3 presents the amount of cracks detected at the dry (end third dry cycle) and wet (fourth wet cycle) conditions. By keeping the dimensions of the cylindrical subsection identical, and by ensuring it is positioned at similar locations in each of the samples, the analysis of the crack opening can be used as a measure for the swelling of the clays.

It can be seen from figure 2 that NaB shows wider cracks throughout the full volume after the third dry stage. On the contrary, the cracks in the HYPER clays are mainly due to edge effects.

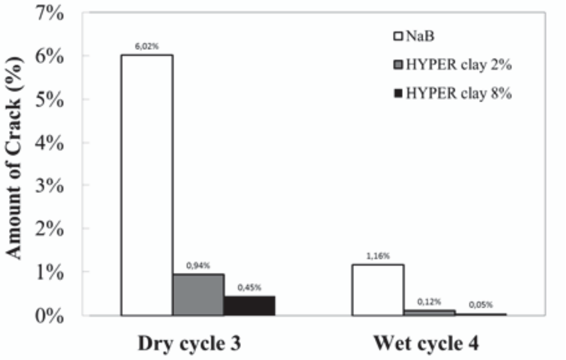

Figure 3. Amount of cracks versus dry and wet condition of untreated bentonite (NaB), HYPER clay 2% (HC+2%) and HYPER clay 8% (HC+8%)

The amount of cracks developed by NaB after the third dry was about 6% of total initial volume. Whereas the amount of cracks of HYPER clays were 0.94% and 0.45% for HYPER clay 2% and HYPER clay 8% respectively. The amount of cracks decreased for all the samples on wet conditions due to the swelling and healing of the bentonites. In particular, untreated bentonite presented an amount of cracks around 1.2% of the analysed subsection, comparable to the amount of cracks of HYPER clay in dry condition.

4.3. Hydraulic conductivity tests

Hydraulic conductivity tests were performed in order to assess the influence of wet and dry cycles on the self-healing capacity and permeability of samples of untreated sodium bentonite, HYPER clay 2% and HYPER clay 8%. Measured hydraulic conductivity (k) values are plotted as a function of wet-dry cycles in Figure 4.

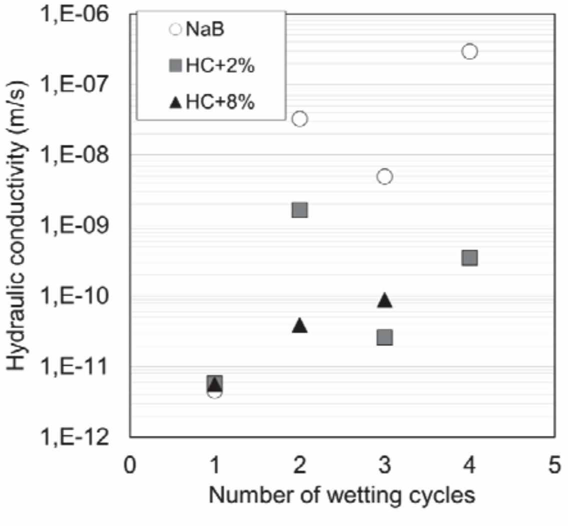

Figure 4. Hydraulic conductivities (*k*) of sodium untreated bentonite (NaB), HYPER clay 2% (HC+2%) and HYPER clay 8% (HC+8%) at each wetting cycles

The samples presented similar and low permeability in deionized water, which increased in the next cycles in seawater.

The greatest increase was observed for untreated sodium bentonite. NaB significantly increased its permeability up to 2.93 × 10^{-7} m/s during the fourth cycle. These results are in agreement with the DDL theory. Untreated bentonite forms aggregate structure once in contact with strong electrolyte solution due to the contraction of the DDL. As a consequence, self-healing and swelling capacity are weakened and the barrier performance of the bentonite is impaired.

On the other hand, HYPER clays showed lower permeability compared to untreated clay. The *k* of HYPER clay 2% was lower than the limit value of 10^{-9} m/s until the fourth cycle. The sudden increase in *k* during the second cycle for NaB and HYPER clay 2% might be related to the strong exposure of the samples to the heat in the oven during the first dry cycle. For this reason, cracks may not be completely healed. The exposure was then confined to better represent the gradual dehydration expected in the field.

HYPER clay 8% presented a gradual and constant increase of permeability. At the end of the third cycle *k* was 9.11×10^{-11} m/s. The difference between untreated clay and HYPER clays might be explained by the presence of the polymer intercalated in the clay particles, which remains over wet and dry cycles. Moreover, it is likely that the presence of the polymer helped to keep the diffuse double layer open during hydration.

However, wet-dry cycles likely showed lower impact on the polymer treated clays. The samples of HYPER clay presented few cracks during desiccation which were healed after rehydration, as can be noted from the low permeability.

5. CONCLUSIONS

The potential influence of wet and dry cycles in combination with cation exchange was investigated by means of swell test, µCT scanning and hydraulic conductivity tests. Samples of untreated sodium bentonite were compared to samples of HYPER clay 2% and 8%. The performance of these bentonites subjected to wet and dry cycles in contact with seawater was studied. Seawater is a highly concentrated electrolyte solution and represents an aggressive environment in the field.

The swelling ability increased with increasing polymer dosage in deionized water. Untreated sodium bentonite has swollen the least and its swelling ability was strongly affected from the consecutive wet and dry cycles. µCT analysis of untreated bentonite, HYPER clays 2% and 8% have shown the effect of the polymers on the healing capacity. HYPER clays presented lower amount of cracks both on dry and wet conditions and better healing capacity upon rewetting.

The effect of wet and dry cycles in seawater had a negative impact also on the hydraulic conductivity of the untreated bentonite. On the other hand, the hydraulic conductivity of HYPER clay 2% was 3.5×10^{-10} m/s, during the fourth cycle, 9.11×10^{-11} m/s for HYPER clay 8%, during the third cycle.

HYPER clay presented enhanced performance after being subjected to wet and dry cycles in seawater. The presence of the anionic polymer likely helps to keep the interlayer open, allowing greater water adsorption and swelling capacity. For this reason might be that the cracks were healed after rewetting and the permeability was maintained low.

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