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Research paper

## Cyclic organic carbonate modification of sodium bentonite for enhanced containment of hyper saline leachates

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## ABSTRACT

Two cyclic organic carbonates (COC), propylene carbonate (PC) and glycerol carbonate (GC), were investigated as saline-resistant modifying agents of Na<sup>+</sup>-montmorillonite using X-ray diffraction (XRD) and Fourier transform infrared (FTIR). PC has been studied previously and has been used as an effective amendment material of Na<sup>+</sup>-bentonite for saline applications. In this research GC is proposed as a more effective modifying agent for containing hyper saline leachates. Na<sup>+</sup>-montmorillonite was reacted with up to 1 N sodium chloride (NaCl) and calcium chloride (CaCl<sub>2</sub>) salt solutions to assess changes in the interlayer spacing (i.e., d-value of the 001 reflection in XRD traces) due to osmotic desiccation, as well as to investigate the mechanism and strength of bonding between GC/PC and Na<sup>+</sup>-montmorillonite by FTIR. GC/Na<sup>+</sup>-montmorillonite was strongly resistant against strongly saline sodic salt solution compared to PC/Na<sup>+</sup>-montmorillonite. CaCl<sub>2</sub> solution had a more detrimental effect on COC modified Na<sup>+</sup>-montmorillonite, however, GC/Na<sup>+</sup>-montmorillonite appeared to retain more intercalated COC than PC/Na<sup>+</sup>-montmorillonite when leached by strong calcic salt solutions.

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### 1. Introduction

Various mining and mineral processing industries produce large quantities of hypersaline liquid waste (Thiel and Smith, 2004; Smith, 2008). More recently, the advent of reverse osmosis plants in remote locations (e.g., from coal seam gas industry) results in large amounts of highly saline liquid waste stored in lined evaporation ponds (Fan et al., 2009; Pérez-González et al., 2016). Often, processing liquids from solid ore wastes, waste rock dumps or heap leach pads or brine ponds are stored in disposal impoundments lined with geotextiles, including geosynthetic clay liners (Breitenbach and Smith, 2006; Hornsey et al., 2010). Geosynthetic clay liners (GCL), have been widely used as components of hydraulic barriers in many waste containment applications, usually in combination with a polymeric liner referred to as geomembrane (Bouazza, 2002; Hornsey et al., 2010; Rowe, 2014) and within the past decade, have become an important component in engineered barriers in mining applications. A GCL product contains a thin layer of sodium (Na<sup>+</sup>)-bentonite contained within two layers of geotextile. The favourable geotechnical characteristics (e.g. high swelling and low hydraulic conductivity) of these liner materials originate

from the properties of their bentonite component (Egloffstein, 2001; Gates et al., 2009). As such, GCL-based materials have become one of the leading lining technologies in waste management and disposal facilities (Rowe, 2014).

Environmental or hydraulic barrier lining systems that incorporate GCL are not generally limited to cases where they are expected to be in contact only with clean water during their service lives. While a given GCL may have a very low hydraulic conductivity to water and low salinity waters (ionic strength:  $I < 0.1$  M), due to resulting chemical incompatibility, they often inadequately attenuate the transport of leachates having extremes of pH or ionic strength (Petrov et al., 1997; Petrov and Rowe, 1997; Shackelford et al., 2000; Jo et al., 2001; Kashir and Yanful, 2001; Kolstad et al., 2004; Jo et al., 2005; Benson et al., 2010; Gates and Bouazza, 2010; Bouazza and Gates, 2014; Liu et al., 2013, 2015). The chemical incompatibility associated with the GCL products, in part, stems from reactions that take place with Na<sup>+</sup>-montmorillonite (Mt), which is the active mineral component of Na<sup>+</sup>-bentonite. These reactions include a loss of crystalline swelling of Mt which results from the high osmotic strengths of hypersaline leachates (Norrish, 1954; Slade et al., 1991; Slade et al., 1991) leading to degradation in GCL hydraulic performance (Quirk and Schofield, 1955; Guyonnet et al., 2005). GCL performance is thus often limited by the ability of the Na<sup>+</sup>-bentonite component to swell and form and maintain strong gels (Bouazza and Gates, 2014).

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Osmotically induced desiccation and cation exchange reactions, which may occur in bentonites when exposed to saline and hypersaline leachates, can change the microstructure of the bentonite by removing the hydration water from the smectite interlayer space (Aylmore and Quirk, 1960; Suquet et al., 1975; Tessier, 1990; Guyonnet et al., 2005; Laird, 2006) and ultimately may result in increased porosity because of the collapse of the Mt gel structure (Norrish and Quirk, 1954; Quirk and Schofield, 1955; Slade and Quick, 1991; Slade et al., 1991). Furthermore, cations with higher charge and/or smaller ionic radii may cause ion exchange within Mt interlayers due to their greater affinity to the exchange sites (Guyonnet et al., 2005; Meer and Benson, 2007). Such effects have been identified as important in diminishing the tight seal that swollen bentonite normally forms when wetted by aqueous liquids (Shackelford et al., 2000; Chertkov and Ravina, 2001; Rowe, 2005; Gates and Bouazza, 2010; Bouazza and Gates, 2014). In consequence, questions on the long term compatibility of GCL due to exposure of saline and hypersaline leachates remain.

There therefore exists a need to (i) optimise GCL hydraulic behaviour when in contact with nonstandard liquids (Malusis and Shackelford, 2002; Shackelford and Lee, 2003), and (ii) develop possible solutions for applications where calcium-enriched leachates may have significant detrimental effects on the hydraulic performance of GCL (Guyonnet et al., 2005; Shackelford and Lee, 2003; Benson et al., 2010).

The objective of this study was to examine whether glycerol carbonate (GC) modified Na<sup>+</sup>-bentonite can overcome or minimise the adverse effects associated with osmotic desiccation as well as Ca<sup>2+</sup> for Na<sup>+</sup> exchange in hypersaline leachates. Previous studies have shown that PC-modified Na<sup>+</sup>-bentonite retains good swelling in up to 0.3 M CaCl<sub>2</sub> (Onikata et al., 1999) and has been proposed as an alternative material for liner systems (Katsumi et al., 2008). GC is a non-toxic cyclic organic carbonate solvent having good solubility in water and high dielectric permittivity (Chernyak, 2006). Moreover, GC can be relatively inexpensively synthesized by green chemical methods (Turney et al., 2013), and GC and its derivatives form stable intercalates with Na<sup>+</sup>-Mt (Gates et al., 2016).

The hypothesis tested here is that GC offers a stronger interaction with interlayer Na<sup>+</sup> than does PC, partly due to its higher permittivity (Chernyak, 2006), but also because it has an additional pendant OH functional group, which allows it to H-bond with both interlayer water and with the interlayer surfaces of Na<sup>+</sup>-Mt (Gates et al., 2016), thereby promoting resistance to the osmotically induced desiccation of saline leachates (Fig. 1). Incorporation of GC modified Na<sup>+</sup>-bentonite would thus be expected to improve the hydraulic performance of GCL materials to saline leachates.

Stability of glycerol carbonate is considered to be crucial when used as a modifying agent in an environmental liner application, especially where the liner may be exposed to variable conditions for considerable lengths of time. Any degradation due to heat, sunlight, oxygen or microbiological activity can reduce its efficiency in improving barrier performance of Na<sup>+</sup>-bentonites against saline leachates. While investigation of long-term chemical stability of the cyclic organic carbonates was out of the scope of this project, some preliminary research has been carried out in order to obtain basic information on the stability and possible degradation of glycerol carbonate and propylene carbonate.

**Table 1**  
Mineralogical properties of the natural sodium bentonite.

Property	Value
Bulk mineralogy (% of total) <sup>a</sup>	
Montmorillonite (Mt)	73 ± 1
Quartz	18 ± 1
Opal/cristobalite-tridymite	6 ± 1
Feldspar	3 ± 1
<0.2 μm <sup>b</sup> (% of fraction)	
Montmorillonite	98 ± 1
Opal/cristobalite-tridymite	2 ± 1
Particle size (% of bulk)	
<0.2 μm	53 ± 3
>0.2 μm	44 ± 3
CEC (meq/100 g)	
Bulk <sup>c</sup>	85
<0.2 μm <sup>d</sup>	104

<sup>a</sup> X-ray diffraction and Reitfeld analysis (performed at CSIRO Land and Water, Adelaide, Australia).

<sup>b</sup> The <0.2 μm fraction was separated by centrifugation (e.g., Gates et al., 2002).

<sup>c</sup> The methylene blue CEC tests were on bulk materials by CSIRO Land and Water (Adelaide, Australia) following the tetra-sodium pyrophosphate (TSP) pre-treatment method of Wang et al. (1996).

<sup>d</sup> The Ba CEC tests were conducted using X-ray fluorescence on oriented films of the <0.2 μm fractions by CSIRO Land and Water following in-house methods.

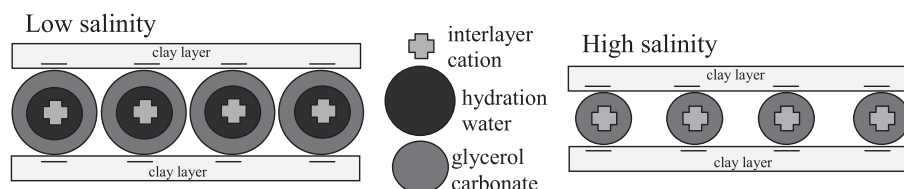
## 2. Materials and methods

A natural Na<sup>+</sup>-bentonite (Miles, Queensland, Australia) marketed by Sibelco (Melbourne, Australia), commonly used in GCL applications in Australia, was selected in this study. Mineralogical and chemical properties of the bentonite are summarized in Table 1 (see also Gates et al. (2016)).

Propylene carbonate (PC) (anhydrous, 99.7%) was purchased from Sigma-Aldrich and glycerol carbonate (GC) was synthesized according to the method developed by Turney et al. (2013). Basic chemical properties of GC and PC are summarized in Table 2.

To determine the specific mechanism of interaction between the cyclic organic carbonates (COC) and the bentonites, the effects of COC addition and exposure to salt solutions on COC-bentonite complexes were investigated by Fourier transform infrared (FTIR) and X-ray powder diffraction (XRD) techniques.

To reduce the amount of non-swelling components of the samples the bentonite was purified by sedimentation: 5 g bentonite was dispersed in 1 L distilled water by using an ultrasonic bath and the suspension was left undisturbed for two days to allow sedimentation of the non-swelling components. The upper half of the suspension was then decanted into a ceramic vessel and placed in an oven at 105 °C until an oven-dried material was obtained. The dry materials were hand ground with an agate mortar and pestle. This procedure effectively removed quartz and feldspar components, and significantly reduced the fraction of opal CT (Table 1), and was intended to improve investigations of the reactions of COC with the Na<sup>+</sup>-Mt component of Na<sup>+</sup>-bentonite.



**Fig. 1.** Proposed interactions of GC/Na<sup>+</sup>-montmorillonite with liquids having low and high salinity.

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