



Fluid Indicator Test (FIT) for Screening the Hydraulic Conductivity of Enhanced Bentonites to Inorganic Aqueous Solutions

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Abstract. Over the past 30 years, geosynthetic clay liners (GCLs) have been demonstrated to be a valuable barrier technology in numerous environmental containment applications. However, past research has also shown that the hydraulic conductivity (k) of GCLs is sensitive to the electrolyte concentration of the permeant liquid. At low effective stresses, a sufficiently high electrolyte concentration has the potential to result in a multiple order of magnitude higher k than the same GCL exhibits to water. To expand the boundaries of GCL functionality, recent research has focused on the use of enhanced (e.g., polymer modified) bentonites (EBs) for improving the resilience of bentonites, and GCLs, subjected to high concentration electrolyte solutions. These EBs have frequently incorporated anionic polymers (e.g. sodium polyacrylate) into the bentonite using a plethora of methods, (e.g. dry mixing, wet mixing, in-situ polymerization.) However, existing standard indicator tests (swell index, fluid loss, modified Atterberg limits) for GCL k may not always be effective for EB-GCLs. A rapid fluid indicator test (FIT) for screening high k versus low k of EB-GCLs and unmodified sodium bentonites, is proposed. Preliminary tests were conducted on granular sodium bentonite and an EB (bentonite polymer composite). Results of the FIT were compared with the swell index and k for these materials over a range of CaCl_2 solutions. The results support that a passing FIT generally correlates to low k for the same fluid under low stress conditions.

Keywords: Geosynthetic clay liners · Enhanced bentonite · Fluid indicator test

1 Introduction

Geosynthetic clay liners (GCLs) are used in environmental containment systems due to the low hydraulic conductivity (k) of sodium bentonite (NaB) to water [1, 2]. Unfortunately, NaB may not maintain low k when exposed to inorganic waste leachates with high concentrations of dissolved ions, particularly at low effective stresses [1, 2]. Enhanced bentonites (EBs), a composite material composed of NaB blended with polymers or high molecular weight organic molecules, are a potential solution to the instability of natural bentonite in many containment applications [3–5]. Enhanced bentonites have shown improved performance (lower k , lower rates of diffusion, greater membrane efficiency) relative to unamended NaB when exposed to high concentration

inorganic aqueous solutions, but the mechanisms underlying these improvements are largely unknown [5].

The k of GCLs containing NaB is controlled primarily by the swelling of NaB component [1, 2, 5]. The test used to measure the swelling capability of a NaB in a specific solution is the swell index (SI) defined by ASTM 5890 [6]. The SI is used to screen the k of GCLs when both tests are conducted with the same solution [2, 7]. However, swelling (and Atterberg limits or fluid loss) do not universally correlate to the k of EB-GCLs [5]. For example, the relationship between k and SI is shown for a NaB and an EB-GCL (bentonite polymer composite, BPC) in Fig. 1. For NaB, a low SI correlates with increased k ($>1 \times 10^{-10}$ m/s). However, the opposite trend was observed for BPC, where the lowest SI correlates to lower k ($\ll 1 \times 10^{-11}$ m/s). These data provide an illustration of how the SI test may not be an effective indicator test for EB-GCLs.

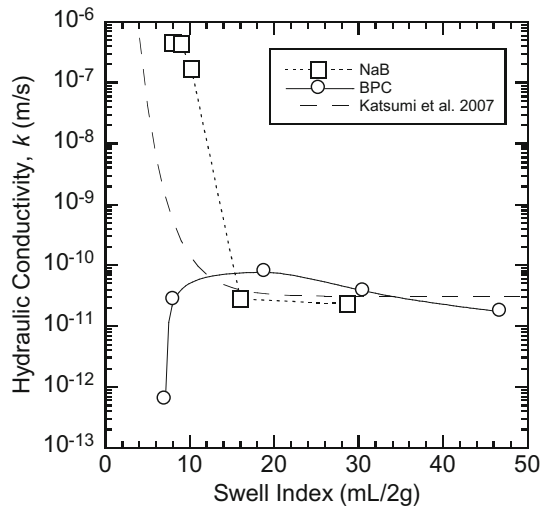


Fig. 1. Swell index as a function of CaCl_2 concentration for NaB and BPC, reproduced from [5], and the logarithmic trend fitted by [7].

The purpose of this study was to develop a rapid and simple indicator test for GCL k in various aqueous inorganic solutions that is effective for both GCLs containing unmodified bentonites and EBs. A new method is described and compared to k and SI in 5, 20, 50, 200, and 500 mM calcium chloride (CaCl_2) solutions.

2 Materials

The NaB used in this study, Volclay CG-50, was obtained from the Colloid Environmental Technologies Company (CETCO) of Hoffman Estates, IL, USA, and is the same NaB used in Bentomat GCLs as well as other studies [5]. Bentonite-polymer

composite is prepared by free radical initiated additional polymerization acrylic acid within a bentonite slurry to create a covalent crosslinked polymer network. Properties of NaB and BPC (cation exchange capacity, mineralogical composition, polymer content) are reported in [5].

Solutions of 5, 20, 50, 200, and 500 mM CaCl_2 were used in this study. The electrical conductivities of these solutions were 1.22 mS/m (5 mM CaCl_2), 4.50 mS/m (20 mM CaCl_2), 10.4 mS/m (50 mM CaCl_2), 38.5 mS/m (200 mM CaCl_2), and 86.5 mS/m (500 mM CaCl_2). The pH of these solutions were near neutral (5.7–6.9). Solutions were prepared by dissolving calcium chloride di-hydrate ($\text{CaCl}_2 + 2\text{H}_2\text{O}$) in deionized water, and were stored in collapsible carboys with no headspace.

3 Methods

The fluid indicator test (FIT) apparatus is a pseudo-rigid wall permeameter as shown in Fig. 2. A hollow acrylic cylinder (outside diameter, $D_o = 63.5$ mm) contains sandwiched layers of mesh aluminum screens, filter paper, and NaB or EB forming a pseudo-GCL (with filter paper in lieu of geotextiles; specimen diameter, $D = 38.1$ mm). The cylinder base is fitted with 6.35-mm outside diameter tubing that is used to supply or receive water for hydration and flow. A second hollow acrylic cylinder (outside diameter, $D = 38.1$ mm; inside diameter, $D_i = 25.4$) sits above the pseudo GCL and within the outer acrylic cylinder. The 38.1-mm diameter cylinder is filled with the selected liquid in both the hydration and testing phases. Weights are added to the top of the inner acrylic cylinder to apply a total normal stress of 6 kPa to the pseudo GCL. A flexible membrane and O-rings surround the connection between the two cylinders. A flexible membrane and O-rings surround the connection between the two cylinders.

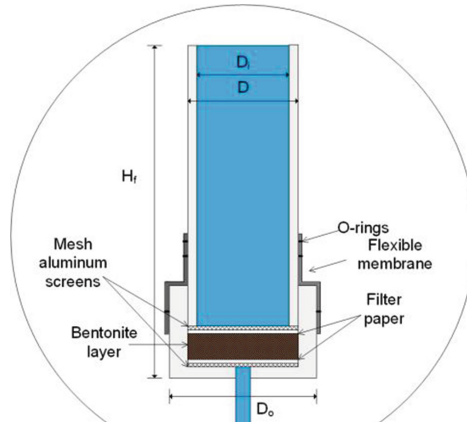


Fig. 2. Fluid indicator test (FIT) apparatus set up.

The FIT is run in two stages, a hydration stage (Fig. 3a), and a flow stage (Fig. 3b). The FIT apparatus (Fig. 2) is placed on a stand with height, H_s , of 225 mm, fitted with a hole for the 6.35-mm outside diameter tubing. The tubing is connected to a vertical

burette at a height, H_B of 400 mm above the lab floor. To control the flow of solution, a valve is included near the base of the burette. After the pseudo-GCL is assembled within the FIT apparatus, and weight(s) are placed atop the apparatus yielding an applied normal stress of 6 kPa, a small amount (<10 mL) of solution is added to burette to removal of air bubbles from the tubing. During this step, the bentonite should not be allowed to contact the solution (i.e., the bentonite should not be allowed to hydrate). Once all air is removed from the tubing, solution is added to the burette, hydrating the bentonite from the bottom up until the system reaches equilibrium at a solution height, H_w of 1.0 m above the lab floor. The system is then left to hydrate for 24 h while the bentonite hydrates under the 6 kPa total normal stress.

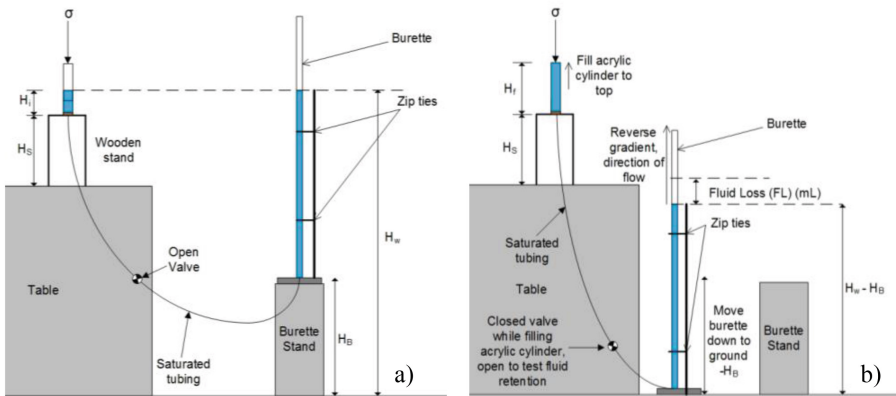


Fig. 3. Fluid indicator test (FIT) (a) setup (left) and (b) measurement (right).

After 24 h hydration period, the valve is closed, and the burette is moved to the lab floor ($-H_B$). The acrylic cylinder above the pseudo-GCL is filled with solution through a hole in the weights (~ 100 mL total volume, $H_f = 18$ cm). To initiate flow, the valve is opened and the volumetric flow through the pseudo-GCL is measured for a period of 5 min. The fluid retention (FR) is calculated by subtracting the flow through the pseudo-GCL from 100 mL. If $FR = 100$ mL, the specimen passes the FIT and is anticipated to potentially have a low k to the testing solution under low effective stress conditions. If the $FR < 100$ mL the specimen fails the FIT, and is anticipated to potentially have a high k to the testing solution under low effective stress conditions.

4 Results

The results of the FIT test for the NaB and BPC are shown in Fig. 4 and summarized in Table 1. These results are compared with the k and SI for the same material in the same solution [5]. The FIT test generally captures hydraulic compatibility (low or high k to a given solution) of both NaB and EB. Two outliers are evident in Fig. 4. The NaB tested with 50 mM CaCl_2 solution produced a passing result for the FIT but when hydrated

and permeated with the same solution produced a high k . The reason for this outlier is unknown and is under investigation. The BPC with 500 mM CaCl_2 solution also produced an anomaly with a failing FIT but a low k . This outlier is explained by the polymer (sodium polyacrylic acid) behavior in the 500 mM CaCl_2 solution. Polymer in BPC acts to clog the pores of the GCL [5] via a tacky polymer hydrogel within the bentonite matrix. When hydrated with 500 mM CaCl_2 , the network of polymer within BPC interconnects and tightens resulting in shrinkage of the specimen away from the sidewalls, producing sidewall leakage upon execution of the FIT test. Sidewall leakage was proven for this experimental combination by dyeing the specimen during the FIT as shown in Fig. 5. Preliminary tests of BPC with 500 mM CaCl_2 under a higher total stress (20 kPa) produced a passing FIT, correlating to the low measured k of this material-permeant solution combination. The higher load enabled maintenance BPC edge seal preventing sidewall leakage.

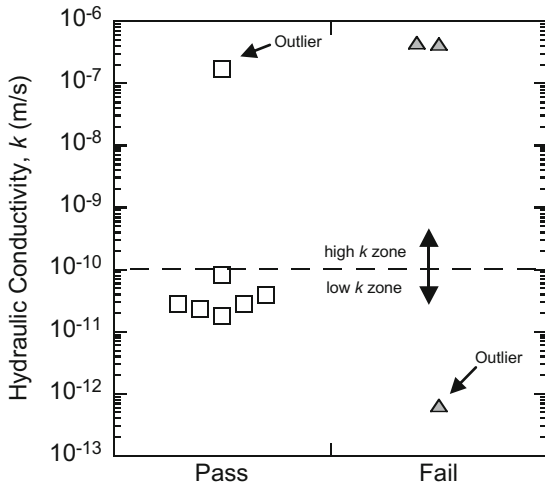


Fig. 4. Hydraulic conductivity (k) as a function of fluid indicator test (FIT) pass or fail for NaB and BPC. FIT passes when fluid retention = 100 mL, FIT fails when fluid retention <100 mL).

5 Discussion

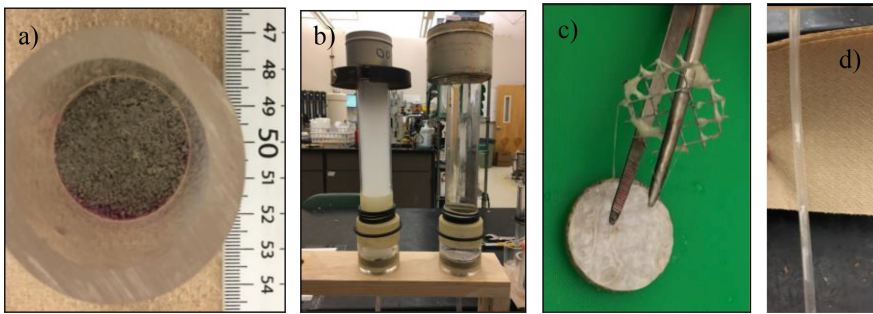
The FIT results shown in Fig. 4 illustrate the potential use of this method as an indicator for high or low k of NaBs and EBs with potential permeant solutions. For comparison SI as an indicator test, an empirical equation for k based on SI from the literature [7] was used calculate the expected a predicted $k(k_c)$ from SI

$$\log(k_c/c) = \exp(a(\text{SI} - b)) \tag{1}$$

where k_c is in cm/s, and a, b, and c are fitting parameters equal to -0.31 , $8.69 \text{ mL}/2 \text{ g}$ solid, and $3.09 \times 10^{-9} \text{ cm/s}$, respectively, and SI is the measured swell index in mL/2 g.

Table 1. Swell index (SI), hydraulic conductivity (k), and fluid retention (FR) of Na-bentonite (NaB) and bentonite-polymer composite (BPC) in CaCl_2 solution. SI and k data taken from [5].

Conc. CaCl_2 (mM)	NaB			BPC		
	SI (mL/2 g)	k (m/s)	FR (mL)	SI (mL/2 g)	k (m/s)	FR (mL)
5	28.7	2.3×10^{-11}	100	46.7	1.8×10^{-11}	100
20	16.0	2.8×10^{-11}	100	30.5	3.9×10^{-11}	100
50	10.2	1.7×10^{-7}	100	18.8	8.1×10^{-11}	100
200	9.0	4.3×10^{-7}	97	8.0	2.8×10^{-11}	100
500	8.0	4.5×10^{-7}	94	7.0	6.5×10^{-11}	0

**Fig. 5.** Photographs of polymer effects on FIT: (a) preferential flow of BPC with 500 mM CaCl_2 ; (b) visible dispersion of polymer in inflow column of BPC tested with 50 mM CaCl_2 (left) versus no visible dispersion of BPC tested in 200 mM CaCl_2 (right); (c) crosslinked polymer on inflow side of BPC tested with 200 mM CaCl_2 ; (d) polymer eluted into outflow line of BPC tested in 200 mM CaCl_2 .

Measured k , k_m , are compared to those calculated using Eq. 1, k_c , in Fig. 6. The data plotted shows agreement between k_c and k_m when SI is above 14 mL/2 g. This swell index is close to the lower limit (15 mL/2 g) that [7] noted was related to low k . A SI of 14 mL/2 g is also noted by [8] as a minimum SI associated with low k at low effective stress. The poor correlation zone shown in Fig. 6 illustrates that Eq. 1 predicts a k that is > two orders of magnitude lower than k_m for NaB specimens with a SI < 14 mL/2 g. Equation 1 also predicts a k_c > one order of magnitude higher for BPC specimens with a SI < 14 mL/2 g.

Unlike the SI test, the FIT generally matched low and high k of the materials tested. The FIT test was also qualitatively useful in enabling observations of BPC polymer behavior. As shown in Fig. 5b–d, polymer elution was visible in the testing and breakdown of the FIT with CaCl_2 solutions. In relatively dilute solutions (≤ 50 mM CaCl_2), the polymer was dispersed into solution upon filling the acrylic inflow cylinder (Fig. 5b). In 200 mM CaCl_2 , the creation of solid polymer precipitates was visible (Fig. 5c). As shown previously in Fig. 5, the BPC FIT tested in 500 mM CaCl_2 caused catastrophically cross-linked polymer chains within the pore matrix such that no

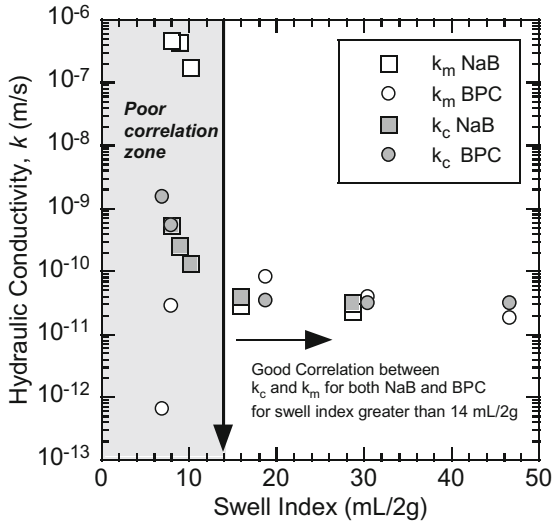


Fig. 6. Hydraulic conductivity (k) as a function of swell index (SI) for sodium bentonite (NaB) and bentonite polymer composite (BPC). Measured k , (k_m) from [5]; calculated k , (k_c) calculated using Equation A reported in [7] with SI from [5] used to calculate k_c .

dispersion or elution occurred. The photographs shown in Fig. 5 illustrate the potential utility of visual observations of EB hydration in different aqueous solutions for an indicator of polymer migration, and behavior. Efforts to standardize quantification of mobilized polymer are ongoing.

6 Conclusions and Recommendations

A fluid indicator test (FIT) was developed as a rapid indicator test to predict the hydraulic conductivity (k) of geosynthetic clay liners (GCLs), and GCLs containing enhanced bentonites (EBs). Results of the FIT illustrate the pass/fail criterion developed provided a better indication of high and low k for bentonite-polymer composite (BPC) than the swell index (SI) test. The FIT also allowed for qualitative observations of polymer behavior (e.g. elution and crosslinking) that were not observed upon inspection of the SI test. The FIT requires further testing to prove if the pass/fail criterion can accurately predict the high or low measured k of other EBs produced with different polymers and with other material preparation techniques. Additional study is also needed to with more complex aqueous inorganic solutions that may be more representative of field solutions.

References

1. Ruhl JL, Daniel DE (1997) Geosynthetic clay liners permeated with chemical solutions and leachates. *J Geotech Geoenviron Eng ASCE* 123(4):369–381
2. Shackelford C, Benson C, Katsumi T, Edil T, Lin L (1000) Evaluating the hydraulic conductivity of GCLs permeated with non-standard liquids. *Geotext Geomembr* 18(2–3):133–161
3. Onikata M, Kondo M, Kamon M (1996) Development and characterization of a multiswellable bentonite. In: Kamon M (ed) *Environmental geotechnics*. A A Balkema, Rotterdam, pp 587–590
4. Di Emidio G (2010) Hydraulic and chemico-osmotic performance of polymer treated clays. Ghent University, Faculty of Engineering, Ghent
5. Scalia J, Benson C, Bohnhoff G, Edil T, Shackelford C (2014) Long-term hydraulic conductivity of a bentonite-polymer composite permeated with aggressive inorganic solutions. *J Geotech Geoenviron Eng ASCE* 140(3):04013025-1–04013025-13
6. ASTM D5890 (2018) Standard test method for swell index of clay mineral component of geosynthetic clay liner. ASTM International, West Conshohocken
7. Katsumi T, Ishimori H, Ogawa A, Yoshikawa K, Hanamoto K, Fukagawa R (2007) Hydraulic conductivity of nonprehydrated geosynthetic clay liners permeated with inorganic solutions and waste leachates. *Soils Found* 47(1):79–96
8. Scalia J, Benson C, Finnegan M (2018) Alternate procedures for swell index testing of granular bentonite from GCLs. *Geotech Test J Rev* (2018)