

# ASSESSING POLYMER ELUTION AND HYDRAULIC CONDUCTIVITY OF BENTONITE-POLYMER COMPOSITE GEOSYNTHETIC CLAY LINERS PERMEATED WITH AGGRESSIVE SOLUTIONS

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**ABSTRACT:** Polymer elution from bentonite-polymer composite geosynthetic clay liners (BPC-GCLs) and the efficacy of using flow stress of polymer hydrogels as a screening tool were evaluated in this study using permeant solutions that alter hydraulic conductivity. For several of the BPC-GCLs that were tested, hydraulic conductivity increased during permeation, with polymer eluted in the effluent. BPC-GCLs with lower hydraulic conductivity ( $<10^{-11}$  m/s) produced smaller volumes of effluent with higher polymer content, whereas BPC-GCLs with higher hydraulic conductivity ( $>10^{-11}$  m/s) produced larger volumes of effluent with lower polymer content. Increases in intrinsic permeability were concomitant with increases in hydraulic conductivity, indicating that pore spaces opened as polymer was eluted. Opening of pore spaces was confirmed by macro- and microscopic imaging of flow paths and polymer arrangement. Flow stress of the polymers decreased with increasing moisture content, indicating that conditions that promote higher hydrogel moisture content are coincident with greater propensity for polymer elution. BPC-GCLs with polymers having higher flow stress had lower hydraulic conductivity, indicating that flow stress has potential as screening measurement for chemical compatibility of BPC-GCLs.

**Keywords:** Bentonite-polymer composite; geosynthetic clay liner; flow stress; polymer elution

## 1 INTRODUCTION

Geosynthetic clay liners (GCLs) are used for lining containment systems, such as solid waste landfills, waste impoundments, lagoons, and tailing impoundments. Conventional GCLs are traditionally composed of a layer of granular or powdered sodium bentonite (NaB) bound between two geotextiles. Montmorillonite, the primary mineral component of NaB, swells appreciably when hydrated, which reduces the size of pores through which flow is conveyed to create a low hydraulic conductivity barrier (Kolstad et al. 2004; Mitchell and Soga 2005). However, swelling of NaB can be suppressed by aggressive solutions that have high ionic strength or extreme pH ( $\text{pH}<2$  or  $\text{pH}>12$ ), leading to hydraulic conductivity that is 3-4 orders of magnitude higher than that of NaB permeated with more dilute solutions (Jo et al. 2001; Shackelford et al. 2010; Chen et al. 2019). Consequently, NaB-GCLs may not be appropriate for containment systems with more aggressive leachates.

Bentonite-polymer composite (BPC) GCLs can maintain low hydraulic conductivity in applications where NaB-GCLs are too permeable (Trauger and Darlington, 2000; Ashmawy et al. 2002; Benson et al. 2010; Scalia et al. 2011, 2014; Athanassopoulos et al. 2015; Scalia and Benson, 2016). Applications of BPC-GCLs include containment of mining and industrial process wastes (e.g., bauxite, also known as red mud) and coal combustion products (Athanassopoulos et al. 2015; Donovan et al. 2017; Tian and Benson, 2018; Chen et al. 2019). Unlike NaB-GCLs, which are sensitive to aggressive solutions, BPC-GCLs can maintain low hydraulic conductivity ( $10^{-13}$  -  $10^{-10}$  m/s) to solutions with high ionic strength and extreme pH (Athanassopoulos et al. 2015; Donovan et al. 2017; Scalia et al. 2018, 2014; Tian and Benson, 2018; Chen et al. 2019).

Different mechanisms control the hydraulic conductivity of NaB- and BPC-GCLs, as shown in Fig. 1. In NaB-GCLs, intergranular pores close when the bentonite granules swell, resulting in small and tortuous flow paths that correspond to low hydraulic conductivity. For this reason, swelling is a key indicator of hydraulic conductivity of traditional NaB-GCLs (Scalia et al. 2011; Tian et al. 2016a, 2017). For BPCs, the permeant solution hydrates the polymer to create a polymer hydrogel that fills pores between the bentonite granules, creating tortuous flow paths that are responsible for low hydraulic conductivity regardless of whether the NaB component swells (Scalia et al. 2014; Tian et al. 2016a, 2016b). Consequently, swell is not an effective indicator of hydraulic conductivity for BPC-GCLs (Chen et al. 2019).

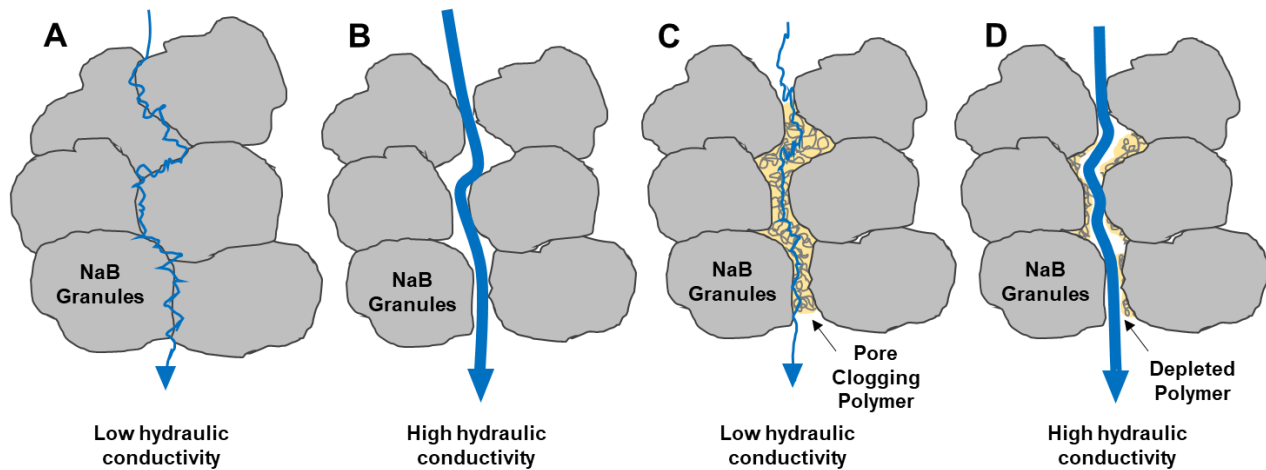


Fig. 1. (A) NaB granules swell in leachate with low to modest ionic strength, closing inter-granular pores and resulting in low hydraulic conductivity; (B) NaB granules permeated with aggressive leachate do not swell, resulting in larger inter-granular pores and higher hydraulic conductivity; (C) polymer component of BPC fills inter-granular pores, creating a low hydraulic conductivity barrier; (D) elution of polymer results in BPC with higher hydraulic conductivity.

BPCs maintain low hydraulic conductivity provided the polymer fills the pores in a BPC-GCL. However, the polymer may elute in a viscous effluent while a BPC-GCL is in service (Scalia et al. 2014; Tian et al. 2017). As elution continues, pores initially filled with polymer are opened and the hydraulic conductivity increases (Tian et al. 2017, 2019). The propensity for polymer elution and the corresponding increase in hydraulic conductivity are influenced by the chemistry of the permeant solution (Tian et al. 2016b, 2017). Commercially available BPC-GCLs contain numerous proprietary polymers, each of which interacts differently with leachates and therefore has different chemical compatibility.

Few indicator tests have been proffered for chemical compatibility screening between BPC-GCLs and leachates, and no test methods have been standardized. Water vapor sorption has been suggested as an indicator of interactions between BPC-GCLs and leachate; however, the relationship between water vapor sorption and hydraulic conductivity depends on the ionic strength of the solution, complicating interpretation of the test data (Akin et al. 2017). Viscosity of polymer-bentonite suspensions has also been suggested as a screening tool, but has yet to be related directly to hydraulic conductivity (Geng et al. 2016). Screening tests should represent the mechanisms controlling hydraulic conductivity of BPCs, such as polymer elution, which is controlled by the viscoelastic properties of the hydrated polymer. In this study, the relationship between the hydraulic conductivity of BPC-GCLs and polymer elution was investigated, along with the efficacy of using flow stress of the polymer hydrogel as a screening measurement for chemical compatibility between BPC-GCLs and aggressive leachates.

## 2 MATERIALS AND METHODS

Six commercially available BPC-GCLs were used in this study: BPC-GCL-N, BPC-GCL-S, BPC-GCL-P1, BPC-GCL-P2, BPC-GCL-P3L, and BPC-GCL-P3H. All of the GCLs contained proprietary polymers, NaB, and nonwoven upper and lower geotextiles bonded by needlepunching. Two of the BPC-GCLs contained a woven slit film geotextile on the interior to promote polymer retention (BPC-GCL-S and BPC-GCL-3H). BPC-GCL-N and BPC-GCL-S contained similar polymers. BPC-GCL-P1, BPC-GCL-P2, BPC-GCL-P3L, and BPC-GCL-P3H contained different polymers (P1, P2, and P3), and the bentonite and polymer components were obtained individually for each. BPC-GCL-3H contained a higher loading of polymer P3 than BPC-GCL-3L.

Hydraulic conductivity testing on each BPC-GCL was conducted in flexible-wall permeameters using the falling headwater, constant tailwater method in accordance with ASTM D6766. The average effective stress was 29 kPa and the average hydraulic gradient was 115. BPC-GCLs were hydrated with the permeant solution in the permeameter with the stress applied and the outflow line closed for 48 h prior to permeation. No backpressure was used to preclude geochemical changes that would not occur at natural porewater pressures. All tests were conducted until the termination criteria in D6766 were satisfied. Effluent from each hydraulic conductivity test was collected in 60 mL HDPE bottles. Polymer content of the effluent was determined based on total carbon (TC) analysis using the solid-state module of a TOC analyzer (Shimadzu TOC-L, Kyoto, Japan). Viscosity of effluent was measured using a rotational viscometer (Brookfield LVE, Middleboro, MA) paired with a low viscosity adapter.

Scanning electron microscopy (SEM) was used to assess structures of polymers in the pore space. Samples approximately 10 mm x 30 mm were cut from each BPC-GCL specimen after permeation and prepared following Tian et al. (2016b). The samples were flash frozen in liquid nitrogen and lyophilized (Labconco Model 7740020, Kansas City, MO). Smaller samples approximately 2 mm thick were removed from the freeze-dried material with a surgical scalpel and sputter-coated with gold. Images were obtained using a FEI Quanta 650 SEM (ThermoFisher Scientific, Hillsboro, Oregon).

Flow stress of hydrated polymers P1, P2 and P3 was determined by conducting shear-strain-amplitude sweeps with controlled-shear deformation using a parallel plate rheometer (Anton Paar MCR 302, Austria). Flow stress is the viscoelastic property of a hydrogel that describes the sol-gel transition, i.e., the shear stress at which the viscoelastic gel transitions from a relatively immobile solid to a mobile liquid, which is more likely to be eluted from the pore space. Lower flow stress means that less force is required to cause elution of the polymer gel. The flow stress depends on polymer conformation, solution chemistry, and moisture content, all of which can be altered by the leachate. Amplitude sweeps were completed over a range of 1-10,000% shear strain using a 25-mm-diameter sand-blasted parallel plate. Storage modulus and loss modulus were measured during the amplitude sweep, and flow stress was computed as the stress at which the storage modulus became less than the loss modulus.

### 3 RESULTS AND DISCUSSION

#### 3.1 Hydraulic conductivity and polymer elution

An initial set of duplicate hydraulic conductivity tests was conducted with BPC-GCL-N and BPC-GCL-S using 20 and 100 mM CaCl<sub>2</sub> solutions. The initial hydraulic conductivity of BPC-GCL-N was less than  $1 \times 10^{-11}$  m/s with 20 and 100 mM CaCl<sub>2</sub>, whereas the initial hydraulic conductivity of BPC-GCL-S was less than  $1 \times 10^{-10}$  m/s with 20 mM CaCl<sub>2</sub> and less than  $6 \times 10^{-10}$  m/s with 100 mM CaCl<sub>2</sub> (Fig. 2). With the exception of BPC-GCL-N with 20 mM CaCl<sub>2</sub>, which maintained hydraulic conductivity below  $10^{-11}$  m/s throughout the duration of testing, all specimens eventually reached a hydraulic conductivity greater than  $10^{-8}$  m/s.

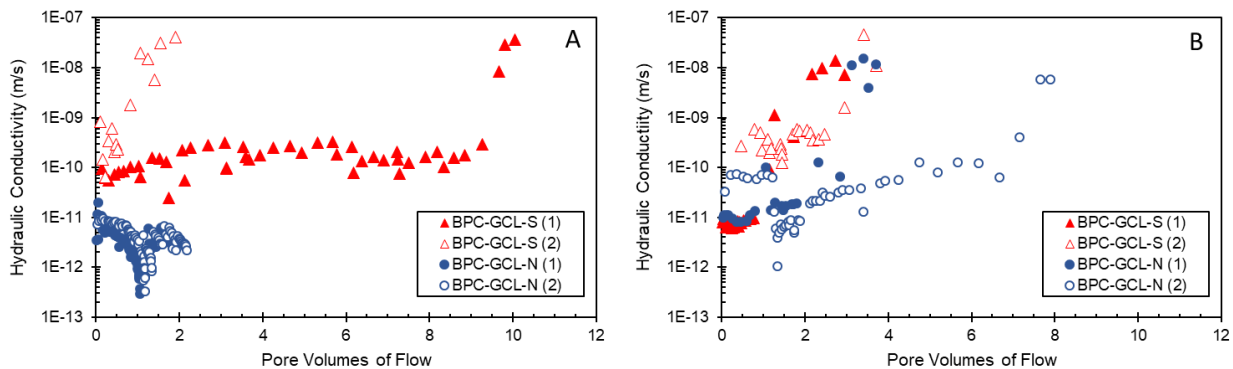


Fig. 2. Hydraulic conductivity of BPC-GCL-S and BPC-GCL-N permeated with (A) 20 mM CaCl<sub>2</sub> and (B) 100 mM CaCl<sub>2</sub>. Numbers in parentheses indicate replicates.

Throughout the duration of testing, polymer content of the effluent was determined by TC analysis. The relationship between hydraulic conductivity and polymer content of the effluent is shown in Fig. 3. Polymer content of the effluent decreases as the hydraulic conductivity increases, which can be explained conceptually by elution of polymer from the pore space, as shown in Fig. 4. Initially, when the hydraulic conductivity is low, the flow rate is low and small volumes of effluent with high concentrations of dissolved polymer are produced. The polymer concentration is high because there is a high ratio of polymer to solution in the pore space and the retention time is long, allowing polymer to dissolve into the flowing pore water (Fig. 4A). In contrast, when the hydraulic conductivity is high, larger volumes of effluent are conducted that have lower polymer concentration because the solution moves quickly through pore spaces depleted of polymer, reducing the amount of polymer contacted by solution and the contact time (Fig. 4B).

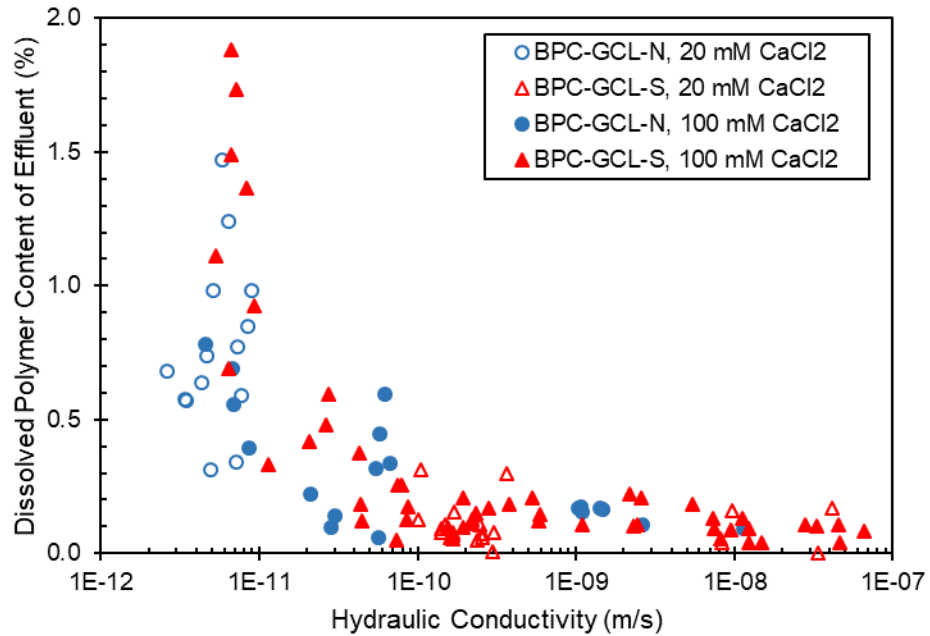


Fig. 3. Dissolved polymer content of effluent as a function of hydraulic conductivity when the effluent was collected.

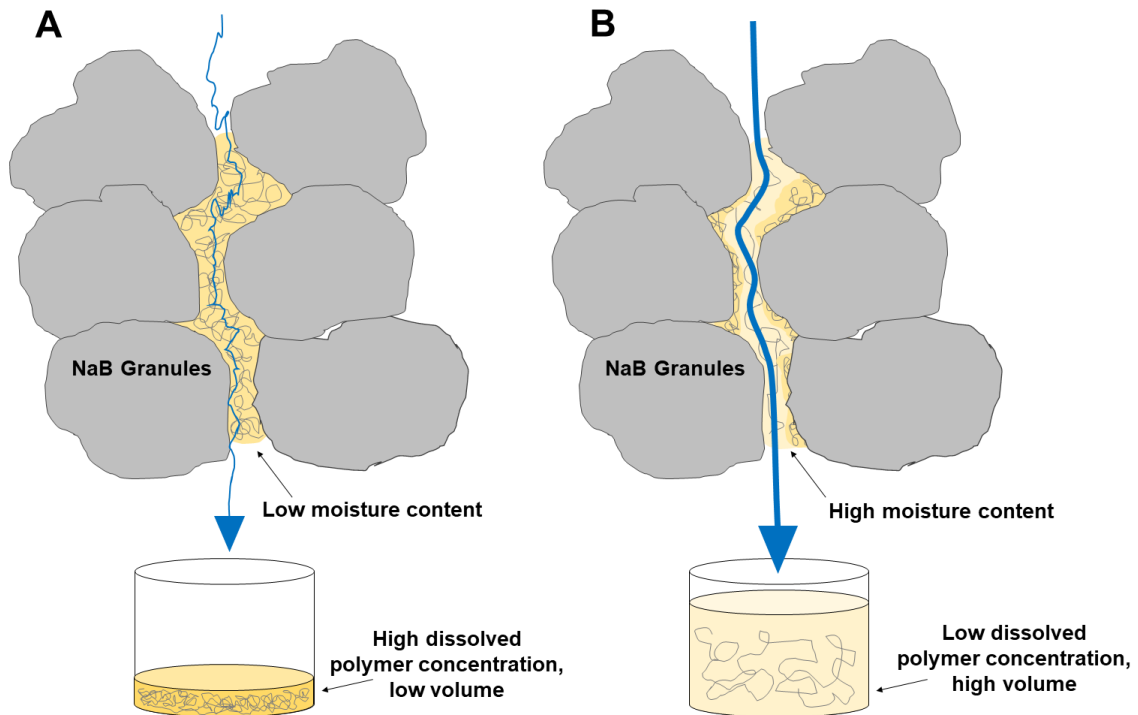


Fig. 4. Conceptual model showing how properties of the polymer hydrogel and effluent change during polymer elution from a BPC-GCL. (A) High polymer to solution ratio in the pore space at low hydraulic conductivity leads to increased contact between the pore water and the polymer gel, and higher polymer content of the effluent; (B) Lower polymer to solution ratio when the hydraulic conductivity is higher leads to less contact between the pore water and the polymer gel, and lower polymer content of the effluent.

Changes in hydraulic conductivity occur in response to changes in the porous medium and/or the fluid properties. To evaluate which mechanism is responsible for the alterations in hydraulic conductivity shown in Figs. 2 and 3, intrinsic permeability was calculated using the viscosity of the effluent:

$$k = \frac{k_i \rho g}{\mu} \quad [1]$$

where  $k$  is the hydraulic conductivity,  $k_i$  is the intrinsic permeability,  $\rho$  is the density of the eluent,  $g$  is the acceleration due to gravity, and  $\mu$  is the measured dynamic viscosity. Intrinsic permeability represents the geometry of the pore space (size, shape, and tortuosity) and is independent of the hydrodynamic properties of the permeant liquid.

An example of changes in intrinsic permeability as a function of pore volumes of flow (PVF) is shown in Fig. 5 for BPC-GCL-S permeated with 20 mM CaCl<sub>2</sub>. The intrinsic permeability increases concomitantly with hydraulic conductivity, indicating that the change in hydraulic conductivity is predominantly due to opening of the pore space due to polymer elution.

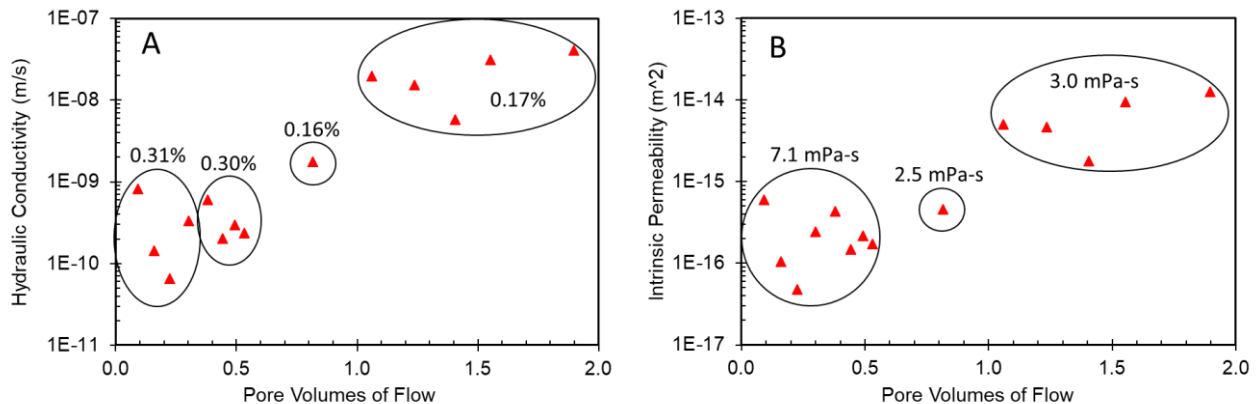


Fig. 5. (A) Hydraulic conductivity and (B) intrinsic permeability of BPC-GCL-S permeated with 20 mM CaCl<sub>2</sub>. Called out data represent (A) dissolved polymer content and (B) viscosity of effluent.

The opening of large pores results in preferential flow paths through which the majority of flow passes, yielding high hydraulic conductivity. Specimens that had hydraulic conductivity greater than  $5 \times 10^{-9}$  m/s were permeated with rhodamine dye, disassembled, and visually inspected for stained flow paths. Flow paths on the order of 1-10 mm wide were present in isolated locations in each specimen (Fig. 6). The number of flow paths per specimen ranged from 1-5, with several specimens having only 1-2 preferential flow paths. The low number of flow paths indicates that polymer is eluted from isolated locations rather than uniformly across the BPC-GCL.

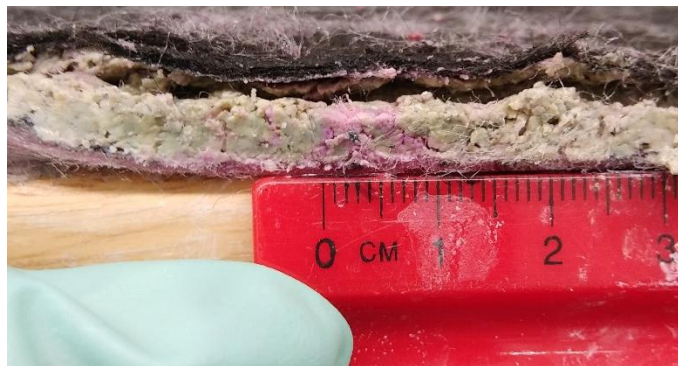


Fig. 6. Example of preferential flow path dyed pink in BPC-GCL-N.

To further investigate changes in the polymer arrangement, the BPC-GCLs were analyzed using SEM. BPC-GCL specimens that were hydrated, but not permeated, were used to examine initial conditions. Two polymer structures were observed in the initial conditions: sheets (Fig. 7A) and honeycombs (Fig. 7B). Stringy structures (Fig. 7C) were observed in addition to sheet and honeycomb structures in BPC-GCLs that had been permeated and underwent an increase in hydraulic conductivity. The stringy structures appear to be remnants of the sheet or honeycomb structures. The sheet and



honeycomb structures appear to completely fill the pore space, whereas the stringy structures do not. All three structures could be found in the same specimen after permeation, but stringy structures were never present prior to permeation. The presence of all three structures in the specimens with high hydraulic conductivity indicates that polymer loss does not occur uniformly across a BPC-GCL.

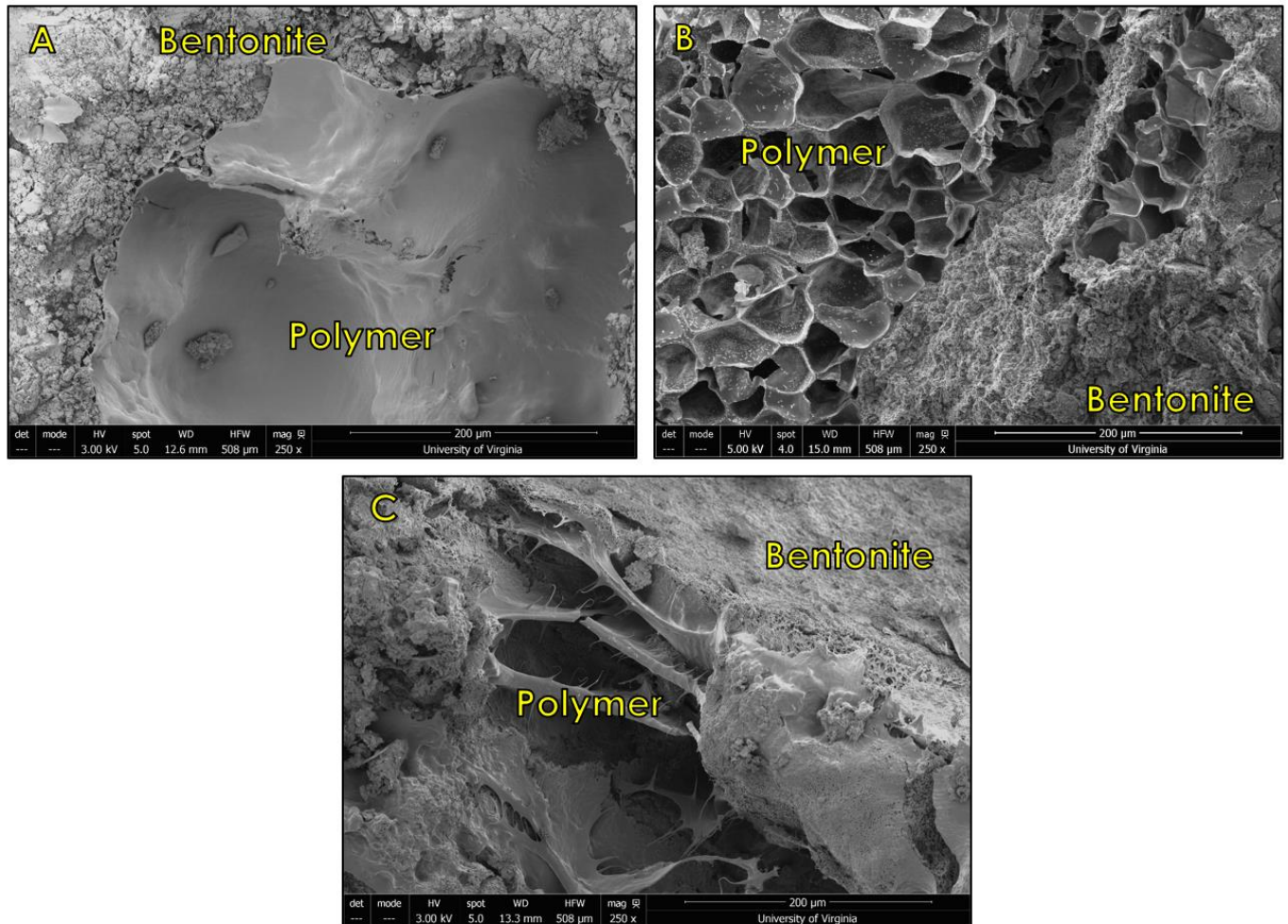


Fig. 7. (A) Sheet, (B) honeycomb, and (C) stringy polymer structures found in BPC-GCLs

### 3.2 Flow stress as a screening tool for polymer elution

No index tests are currently available to quickly screen BPC-GCLs for propensity for polymer elution. Gel flow stress was evaluated as an indicator of the propensity for elution and increases in hydraulic conductivity under the premise that the viscoelastic properties of gels affects mobility in the pore space.

A second set of hydraulic conductivity tests was conducted to evaluate the efficacy of flow stress as a screening test. These tests were conducted using BPC-GCL-P1, BPC-GCL-P2, BPC-GCL-P3L, and BPC-GCL-P3H, for which the polymer in each was available separately. Hydraulic conductivity and flow stress tests were conducted using 50 mM CaCl<sub>2</sub>. Hydrogels were created by hydrating polymers P1, P2, and P3 with the 50 mM CaCl<sub>2</sub> to moisture contents of 200, 500, 1000, and 2000%, representing a range of hydration states in the pore space of BPC-GCLs. Hydraulic conductivities of the BPC-GCLs are summarized in Table 1. The flow stresses of the polymer gels are shown as a function of moisture content in Fig. 8.

Table 1. Hydraulic conductivity of BPC-GCLs to 50 mM CaCl<sub>2</sub>

BPC-GCL	Current Hydraulic Conductivity (m/s)
BPC-GCL-P1	$5.4 \times 10^{-12}$
BPC-GCL-P2	$2.5 \times 10^{-8}$
BPC-GCL-P3L	$6.3 \times 10^{-9}$
BPC-GCL-P3H	$2.2 \times 10^{-11}$

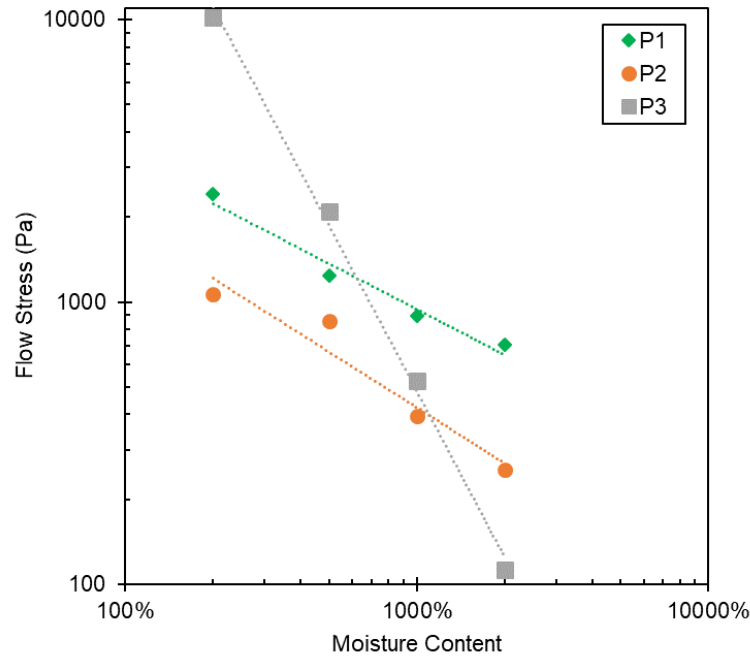


Fig. 8 Flow stress (Pa) as a function of moisture content (%) for three hydrogels.

The trends with flow stress in Fig. 8 are consistent with the hydraulic conductivities of the BPC-GCLs to 50 mM CaCl<sub>2</sub>. For example, the hydraulic conductivity of BPC-GCL-P2 ( $2.5 \times 10^{-8}$  m/s) was several orders of magnitude higher than that of BPC-GCL-P1 ( $5.4 \times 10^{-12}$  m/s). Polymer P2 has a consistently lower flow stress than polymer P1 (Fig. 8), indicating that P2 is more easily eluted than P1 when hydrated with 50 mM CaCl<sub>2</sub>. The hydraulic conductivity of BPC-GCL-P3L ( $6.3 \times 10^{-9}$  m/s) was much higher than that of BPC-GCL-P3H ( $2.2 \times 10^{-11}$  m/s), even though both contain polymer P3. The flow stress of polymer P3 is very sensitive to moisture content (Fig. 8), indicating that small changes in the polymer moisture content could have a significant effect on the mobility of the hydrogel in the pore space. BPC-GCL-P3L has lower polymer loading of polymer P3 than BPC-GCL-P3H, and therefore the moisture content of the P3 gel should be higher in BPC-GCL-P3L than BPC-GCL-P3H. Consequently, the polymer in BPC-GCL-P3H should be more difficult to elute.

The agreement between high flow stress and low hydraulic conductivity indicates that flow stress may be a viable indicator of chemical compatibility if the moisture content of the hydrogel in the BPC can be identified a priori. This topic is an area of active research. Hydraulic conductivity tests are continuing on the four BPC-GCLs to investigate whether flow stress is indicative of the longevity of low hydraulic conductivity.

#### 4 SUMMARY AND CONCLUSIONS

Tests were conducted to evaluate the relationship between hydraulic conductivity and polymer elution from BPC-GCLs, and whether the flow stress of polymer hydrogels can be used as a screening measurement to evaluate propensity for polymer elution and chemical incompatibility. The findings illustrate that the hydraulic conductivity of BPC-GCLs is controlled by polymer gels in the pore space when aggressive leachates suppress swelling of the bentonite fraction. When the polymer forms a gel that fills pores between bentonite granules, and that gel is retained in the pores in the presence of leachate, low hydraulic conductivity is maintained. If the polymer gel is eluted, the hydraulic conductivity increases as

larger pores open for flow. Elution of polymer generally occurs in localized regions, forming preferential flow paths that are responsible for high hydraulic conductivity. SEM images demonstrated that polymer that originally fills the pore space is altered by elution during permeation, and dye studies confirmed that polymer elution results in the preferential flow paths in BPC-GCLs with high hydraulic conductivity.

Flow stress of hydrogels in BPC-GCLs appears to be an effective indicator of chemical compatibility. Hydrogels with higher flow stress corresponded to BPC-GCLs with lower hydraulic conductivity. When comparing between two different polymers, the polymer with a consistently higher flow stress resulted in a BPC-GCL with lower hydraulic conductivity. For two BPC-GCLs containing different quantities of the same polymer, lower hydraulic conductivity was obtained in the BPC-GCL with the higher polymer loading, a condition that yields lower gel moisture content and higher flow stress.

A direct relationship between flow stress and hydraulic conductivity was not determined because a method has yet to be developed to determine the moisture content of the gel in the pore space of BPC-GCLs. Research is underway to develop methods to measure the in-situ moisture content of gels in BPC-GCLs that may lead to flow stress as an effective screening tool for chemical compatibility of BPC-GCLs.

## ACKNOWLEDGEMENT

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