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AN IMPROVED CONTAMINANT RESISTANT CLAY FOR ENVIRONMENTAL CLAY LINER APPLICATIONS

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Abstract—Testing shows that many of the present commercially available bentonite products used for clay liner/soil sealant applications may be susceptible to chemical degradation by certain contaminants. Testing also confirms that a recently developed contaminant resistant clay (CRC) is resistant to various contaminants that would otherwise attack and degrade the present commercially available products. The tests that were used to determine its effectiveness were American Petroleum Institute (API) fluid loss, rigid wall hydraulic conductivity, flexible wall hydraulic conductivity and a newly developed top loading filter press (TLFP) test (LSK method).

Key Words—API Fluid Loss, Bentonite, Chemical Degradation, Contaminant Resistance, Exchangeable Cations, Flexible Wall Permeability, Flocculation, Geosynthetic Clay Liner, Hydraulic Conductivity, Prehydration, Rigid Wall Permeability.

INTRODUCTION

Because of its high swelling and low permeability characteristics, sodium bentonite clay is used in many current clay liner and soil/bentonite sealant products. Certain contaminants (especially Ca, Mg, NH_4, K) may degrade bentonite by causing the clay to flocculate, resulting in higher permeability rates (Rogers 1963; Egloffstein 1994). Calcium is a ubiquitous contaminant and appears to be one of the more damaging contaminants to sodium bentonite. Testing has shown that seawater also has detrimental effects on the sealant characteristics of bentonite clay liners as well as high concentrations of various organic contaminants (Egloffstein 1994). To minimize the potential for degradation, some of the clay liner products contain chemically treated bentonites (Ruhl 1994). There has been at least 1 event where a commercial geosynthetic clay liner (GCL) in a secondary containment application was degraded by Ca contamination (Dobras and Elzea 1993).

Like some of the present commercially available clay liner bentonite products, the new CRC also contains chemical additives to increase contaminant resistance. However, what makes this new clay different is that the CRC has undergone proprietary processing that allows the chemical additives to be more effective at preventing contaminant degradation of the bentonite. The result is the new CRC that demonstrates improved resistance to various contaminants.

When determining the hydraulic conductivity of bentonite samples using contaminated permeants, it is common practice to hydrate the samples with either distilled or tap water prior to being exposed to the contaminated permeants. However, in field applications, GCLs and/or soil/bentonite liners (SBLs) may not be prehydrated with freshwater and must therefore hydrate with whatever fluid is available. In many cases, this fluid may have percolated through the salt-rich cover soil or cover stone and may have picked up substantial quantities of dissolved salts, lime or metals. The GCLs and/or SBLs may also be required to hydrate with a fluid containing high concentrations of seawater salt if the application is located near a salt deposit or body of seawater. The clay liner may also come in contact with other contaminants such as various acids and organics commonly found in landfill leachates. For this reason, the permeability testing was performed where the samples were prehydrated with a contaminated solution prior to being exposed to a more severely contaminated permeant. Three different types of permeability apparatus were used: the rigid wall permeameter, the flexible wall permeameter and the TLFP. The first phase of the permeability testing used tap water as both the prehydrant and the permeant to establish baseline performance data. To evaluate the resistance of samples to various contaminants, more severe conditions were employed.

MATERIALS

For all of the testing, current conventional or commercially available products were compared to the new CRC clay. The selection of commercial samples included bentonite products from various bentonite suppliers and clay liner and GCL manufacturers. The names and manufacturers of these commercial products were withheld and were issued new code names (listed in Table 1).

METHODS

API Fluid Loss Test

During the development of the new CRC, a quick indicator test was needed to test a clay's reaction to

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Table 1. Sample descriptions.

Sample name	Description
Commercial Sample 1	A commercially available bentonite product used in clay liner/soil sealant applications. Also the bentonite material used in Commercial GCL 2 (listed below).
Commercial Sample 2	A commercially available bentonite product used in clay liner/soil sealant applications.
Commercial Sample 3	A commercially available bentonite product used in clay liner/soil sealant applications. Also the bentonite material used in Commercial GCL 1 (listed below).
Commercial Sample 4	A commercially available bentonite product used in clay liner/soil sealant applications.
Commercial Sample 5	A commercially available bentonite product used in clay liner/soil sealant applications.
Commercial Sample 6	A commercially available bentonite product used in clay liner/soil sealant applications.
Commercial Sample 7	A commercially available bentonite product used in clay liner/soil sealant applications.
CRC	Plant production sample of the new CRC.
Commercial GCL 1	Commercially available GCL.
Commercial GCL 2	Commercially available GCL.
CRC GCL	GCL containing the new CRC.

various contaminants. API 13A (1993), section 4, Method for Measurement of Fluid Loss, was used as this quick indicator test. Initially (to establish baseline data), all sample slurries were prepared by dispersing 22.5 g of clay sample into 350 mL of deionized water. After the mixed slurry was aged for 16 h, the slurry was remixed and tested for fluid loss. Solutions containing various contaminants were then substituted for the deionized water in the slurry preparation and tested for fluid loss. An effective sample clay slurry would be able to form a good filter cake in the filter cell and minimize fluid passage. A poor clay or a clay that had been degraded by contamination would not be able to build up a sufficient filter cake and would therefore allow more fluid to pass through. Consequently, lower fluid loss values indicate superior performance. For the testing, samples that normally come in granular form were tested as received. However, the GCL samples were opened and the bentonite material within was removed and tested.

TLFP Test (LSK Method)

This new test was developed to measure the quantity of permeant that would pass through a clay sample layer on an accelerated basis. A predetermined weight of granular bentonite sample was placed into the cell over the main filter screen and filter paper (Figure 1). The sample was then covered with another filter paper and a sand layer. Prehydration fluid was poured over the sand layer: just enough to fully hydrate the underlying clay sample layer. After 16 h of hydration, a permeant solution containing dye was then poured on top of the hydrated sample and the cell was sealed. Pressure was then applied to the cell at 100 psi (approximately 690 kPa) and the subsequent filtrates were weighed at timed intervals. Due to the low flow volumes normally generated during this test, the amounts of filtrates are generally too small to be accurately measured volumetrically and are therefore weighted. Also, weighing the filtrates produces more accurate results. Because the TLFP tests measure the amount of fluid passing through a sample layer, lower filtrate values indicate more favorable sealant characteristics. If a sample's reaction to a permeant results in degradation, the filtrate values should increase and continue to increase (if the degradation continues). During the test runs, the pressure was never completely released from the cells to prevent possible disruption of the sample cake seal. Dye was added to the permeant to indicate when and where, within the sample cake, the permeant completely penetrated the sample layer. The dye was carefully selected so that it would not be absorbed by or react with the clay and/or any constituents of the permeant.

To evaluate the contaminant resistance of all clay samples, 3 cycles of TLFP tests were performed. The 1st cycle used tap water as both the prehydrant and the permeant. The 2nd cycle consisted of prehydrating the samples with 1000 ppm aqueous $CaCl_2$ solution followed by 1 wt% or 10,000 ppm aqueous $CaCl_2$ solution as the permeant. The total cumulative weight of filtrates was divided by the total running time in hours in order to acquire the flow rates of the samples.

To measure the resistance of the samples to seawater, a 3rd set of tests was performed using an actual ocean water sample as the permeant. The samples were prehydrated with tap water.

Rigid Wall Hydraulic Conductivity Test

The procedure for conducting the rigid wall hydraulic conductivity (permeability) testing was similar to ASTM D2434-68 (1974), Method B (falling head). Two cycles of rigid wall permeability testing were performed on several of the samples. The 1st cycle consisted of mixing the bentonite sample with a standard silica sand (7.5 wt%) and then adding tap water to raise the total moisture content up to 15 wt%. The mixture was then compacted in accordance with ASTM D698 (1990) into the rigid wall permeameter cell to a 2-in thickness and covered with sand. Tap water was used as the permeant to obtain baseline hydraulic conductivity values.

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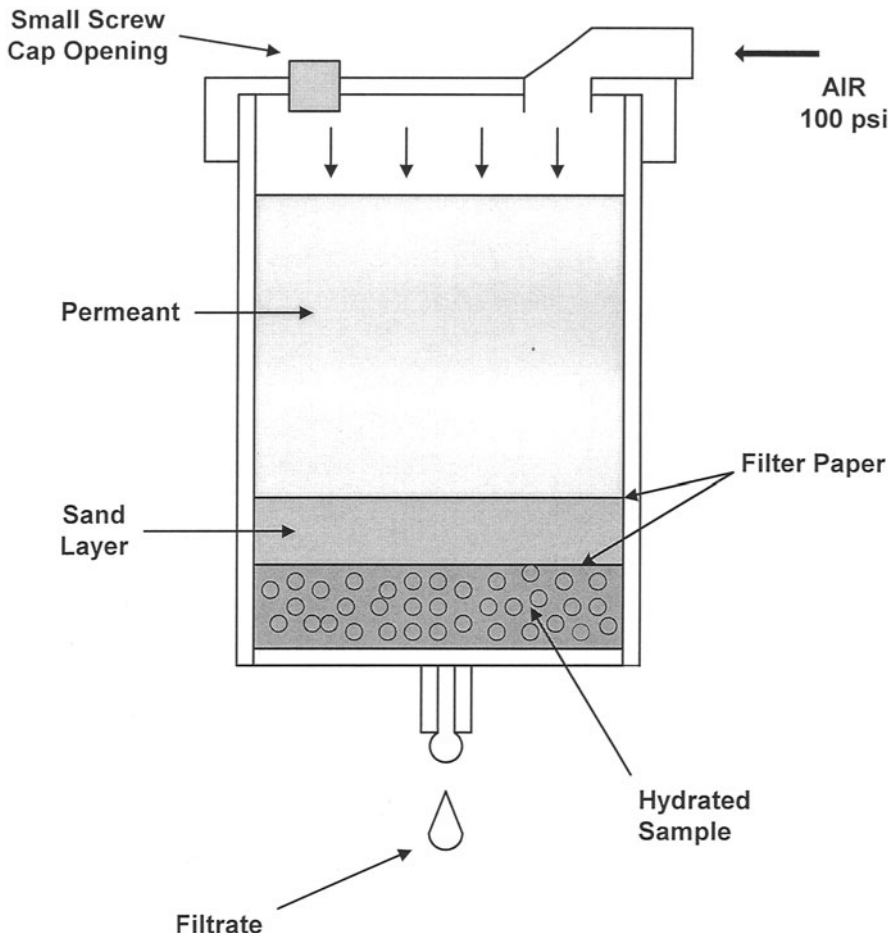


Figure 1. TLFP test (LSK method).

The 2nd cycle of rigid wall permeability testing involved mixing the bentonite sample with a standard silica sand (7.5 wt%) and then adding 1000 ppm aqueous $CaCl_2$ solution to bring total moisture content up to 15 wt%. As with the 1st cycle, the mixture was compacted into the rigid wall permeameter cell to a 2-in thickness and covered with sand. However, with this 2nd cycle, 1000 ppm $CaCl_2$ aqueous solution was poured into the permeameter, and the compacted sample was allowed to hydrate in the calcium-contaminated solution for 3 d. After the 3 d, the permeant was changed to a more concentrated 1 wt% or 10,000 ppm aqueous $CaCl_2$ solution. The sample test runs of this 2nd cycle were continued until a significant increase in permeability was observed.

Flexible Wall Hydraulic Conductivity Test

Flexible wall hydraulic conductivity testing was performed in accordance with ASTM D5084-90 (1990). Sample preparation was performed according to the Geosynthetic Research Institute (GRI) Test Method GCL-2 (1993). For the flexible wall permeability testing, all samples were in GCL form. The GCLs used for the testing were composed of bentonite clay product sandwiched between 2 geotextiles. The first 2 samples were commercially available GCLs. The 3rd sample was a GCL that contained the new CRC. The 1st cycle of testing used tap water only as the permeant to establish a baseline hydraulic conductivity for each sample. The test runs were terminated after each sample achieved steady rate flow (approximately 2 weeks). In the 2nd cycle, the GCL samples were prehydrated for 2 d with 1000 ppm aqueous $CaCl_2$ solution. The permeant was then changed to 1 wt% aqueous $CaCl_2$ solution. The duration of the 2nd cycle test runs depended on the samples' reaction to the contaminated solutions. The test runs were terminated when the permeability values significantly increased (indicating calcium degradation of the bentonite). A 3rd cycle of testing was performed using 1 wt%

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Table 2. API fluid loss test comparing samples in contaminated solutions.

Sample	Fluid loss API (mL) deionized water	Fluid loss API (mL) 1000 ppm $CaCl_2$	Fluid loss API (mL) 1000 ppm NH_4NO_3	Fluid loss API (mL) 1000 ppm KCl	Fluid loss API (mL) 1000 ppm NaCl
Commercial Sample 1	9.7	12.1	—	—	—
Commercial Sample 2	13.8	23.1	—	—	—
Commercial Sample 3	11.6	21.2	13.0	14.4	13.5
Commercial Sample 4	10.7	16.5	13.8	14.0	14.2
Commercial Sample 5	18.0	19.7	—	—	—
CRC	8.0	7.5	7.8	7.9	7.6

Table 3. API fluid loss test comparing samples in additional contaminated solutions.

Sample	Fluid loss API (mL) 1% seawater salt solution	Fluid loss API (mL) 1000 ppm phenol	Fluid loss API (mL) 1000 ppm acetic acid	Fluid loss API (mL) 1000 ppm acetone
Commercial Sample 1	25.2	—	17.0	—
Commercial Sample 2	58.0	—	24.7	—
Commercial Sample 3	40.1	11.8	18.8	11.7
Commercial Sample 4	19.7	12.8	17.8	12.3
CRC	8.8	8.3	7.5	8.2

seawater salt solution as the prehydration fluid. After 2 d of prehydration, the permeant was changed to 3.8 wt% seawater salt solution. The sample test runs of the 3rd cycle were terminated when the hydraulic conductivity values significantly increased (indicating seawater degradation). The samples that maintained a steady flow rate during the 2nd and 3rd cycle test runs were monitored continuously until equipment limitations necessitated their termination. The exception to this was GCL 2, which was prematurely terminated during its 3rd cycle (seawater permeant) test run due to mechanical problems with the permeameter cell.

Free Swell

An analysis was made of the GCL samples after their 2nd cycle test runs to assess the effects of the $CaCl_2$ contamination on the bentonite clay within. To measure the swelling capabilities of the clay in the GCL samples after exposure to the contaminants, free swell testing was performed in accordance with American Colloid Company (ACC) Procedure 1010 (1990). In this swell test, 2.0 g of dry sample bentonite was slowly and gradually added to 100 mL of deionized water in a graduated cylinder. After 24 h of hydration, the volume of swelled clay was visually measured in the cylinder at the clay/water interface. After their 2nd cycle flexible wall test runs, the GCLs were dried, and the bentonite within was extracted and reduced to granular particle size. For comparison, free swell tests were also conducted using the bentonite material from the GCLs prior to any exposure to contaminants.

Exchangeable Cations

Chemical analyses of the GCL samples after their 2nd cycle flexible wall test runs ($CaCl_2$-contaminated) were also performed to measure any changes in the chemical composition of the bentonite clays. Most GCLs and soil bentonite liner products use Na-based bentonites because of their high swelling, low permeability characteristics. The initial concentrations of Na in these bentonites are generally higher than those of Ca or Mg. The exchangeable cations were extracted from the clays by dispersing the samples into 3.6 wt% solution of benzyltrimethylammonium chloride (BTA) and then filtering. The amount of exchangeable Na^+, Ca^{2+} and Mg^{2+} in the filtrate was measured by the use of an atomic absorption spectrophotometer. The results are expressed in meq/100 g.

RESULTS AND DISCUSSION

API Fluid Loss Test

During the development of the new CRC, the API fluid loss test was used as a screening device to obtain quick results of the samples' reaction to various contaminated solutions. Contaminated solutions were substituted for the deionized water normally used in the sample slurry preparation. The fluid loss data in Tables 2 and 3 indicate that the new CRC produced much lower fluid loss values when dispersed in contaminated solutions than the commercially available products. With some contaminated solutions, the fluid loss values were almost equal to the deionized water values. This indicates that the CRC was not severely affected by the contaminants and was able to build up sufficient filter cakes to prevent more fluid from passing through. Testing also showed that the CRC was more resistant to organic contaminants as well as dissolved salts and metals.

TLFP Test (LSK Method)

The results in Table 4 and Figure 2 indicate that the CRC sample was able to resist Ca degradation and produce very low filtrate rates over a 55-h period. However, Commercial Sample 3 was degraded by Ca and was no longer able to function as a barrier to fluids. This was evident as it completely lost all of its

Table 4. Average flow rates from TLFP test.

Sample	Average flow rate (g/h) tap water baseline 1st cycle	Average flow rate (g/h) prehyd. 1000 ppm $CaCl_2$ 1% $CaCl_2$ as permeant 2nd cycle
Commercial Sample 3	4.3	34.3
CRC	0.4	0.3

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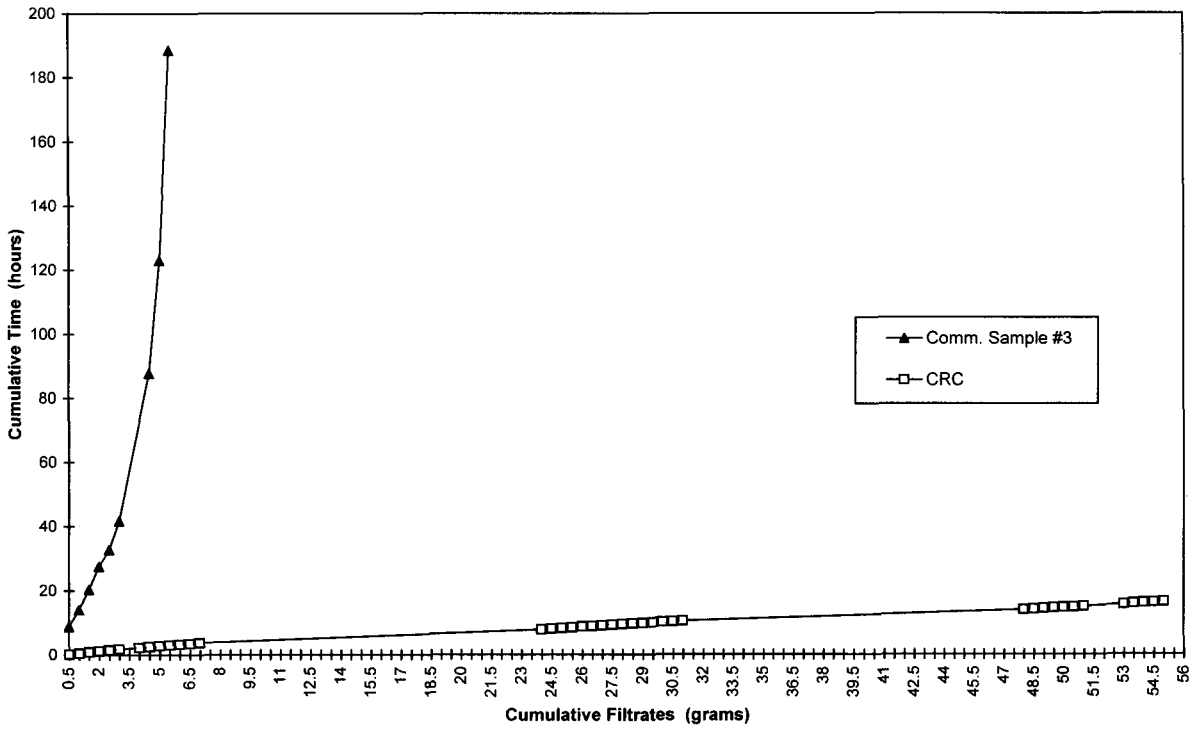


Figure 2. TLFP test (LSK method) prehydrated with 1000 ppm $CaCl_2$/1% as permeant.

permeant after only 5 h of running time. The results from these tests were later confirmed by similar results from both the rigid wall and flexible wall hydraulic conductivity testing. Consequently, it is apparent that the TLFP test (LSK method) can be used to quickly obtain preliminary sealant or permeability characteristics of a clay.

Table 5 and Figure 3 indicate that Commercial Sample 3 was degraded by the ocean water and lost its barrier qualities. This is evident by the higher flow rates and by its having passed all of its ocean water permeant after 24 h of exposure. It should also be noted that the commercial product had the benefit of being fully hydrated with tap water prior to its exposure to the ocean water. In comparison, the CRC sample was able to endure for over 4 d of running time with minimal filtrate loss. The testing shows that the CRC sample was much more resistant to ocean water as well as Ca. It should be noted that Figures 2 and 3 plot *cumulative* filtrates against *cumulative* time. Although the data from the CRC testing have a slope (low), their linear relationship indicates no increase in flow rate.

Table 5. Average flow rates from TLFP test using ocean water as permeant.

TLFP test (LSK method) Average flow rates (prehydrated with tap water/ocean water sample as permeant)	
Sample	Average flow rate (g/h)
Commercial Sample 3	8.0
CRC	0.2

An important distinction between the new TLFP test (LSK method) and the API fluid loss test is that the API method uses mixed slurries in its cells while the TLFP test (LSK method) uses a discrete layer of clay. By being mixed into a slurry, the clay and chemical additives have the advantage of dispersing and hydrating more efficiently. In actual field applications of GCLs and SBLs, the bentonite does not have this advantage. The TLFP test (LSK method) allows the granular bentonite product to hydrate on its own and in place as it would in an actual GCL or clay liner application. This test is similar to the rigid wall hydraulic conductivity test with the exception that the permeant is forced through the sample clay layer quickly by using 100 psi of head pressure. As a result, any changes in the sealant characteristics of the clay (due to degradation) can be observed much sooner than in other permeability tests. Even though clay/soil mixtures can be used in the filter press cells, pure bentonite layers were used to simulate GCL applications. While the TLFP test (LSK method) measures the flow of fluid through a sample, the filtrate values should not be utilized with Darcy's Law to calculate hydraulic conductivity values. Additional research is needed to determine if this test can be used to obtain hydraulic conductivity values.

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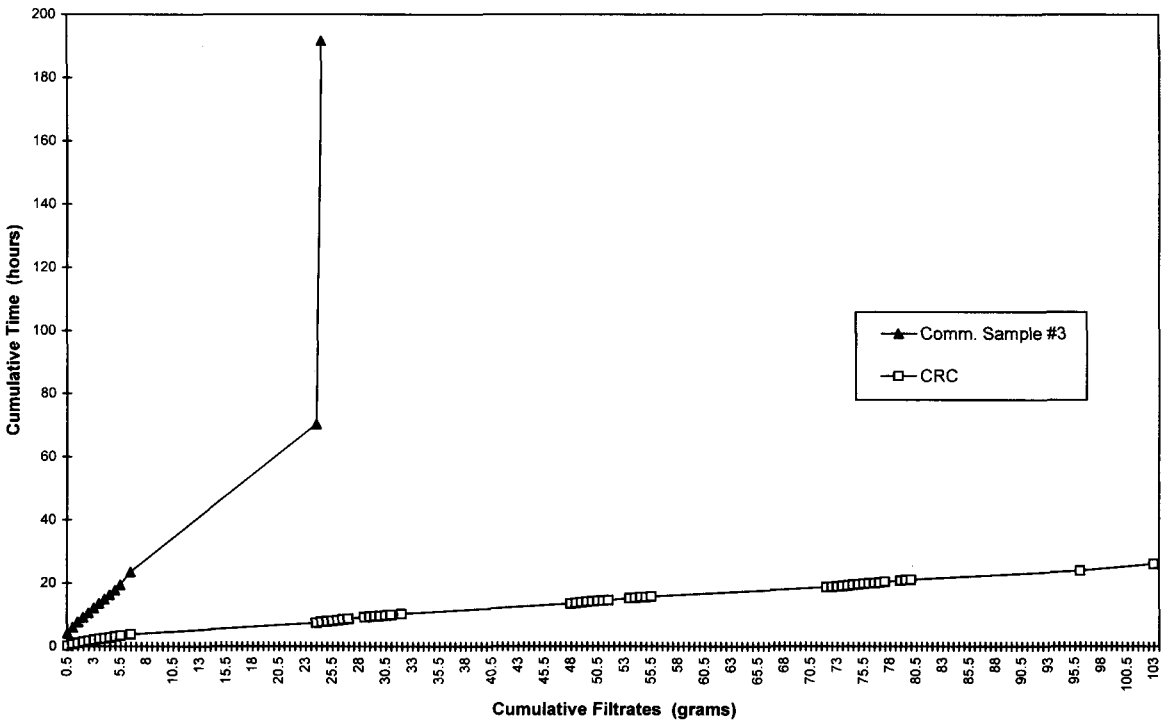


Figure 3. TLFP test (LSK method) prehydrated with tap water/ocean water as permeant.

Rigid Wall Hydraulic Conductivity Test

Table 6 indicates that the 1st cycle tap water hydraulic conductivity values are fairly low among all of the tested samples. However, there is a sharp increase in the hydraulic conductivities of the commercially available samples where $CaCl_2$-contaminated solutions were used as both the prehydrant and the permeant. After 1 to 3 weeks, the Ca degraded the commercial samples and caused the hydraulic conductivities to increase, while the CRC yielded a much lower permeability value during its 2nd cycle run, and maintained it for over 2 mo. Comparing the results from the 2 cycles, it is apparent that the commercial products suffered degradation from the Ca-contaminated solutions and were no longer able to perform as barriers to fluids. It was evident that the CRC sample was much more resistant to the contaminated solutions, and was able to maintain hydraulic conductivity values as low or lower than tap water rates.

Table 6. Rigid wall hydraulic conductivity tests using compacted clay/sand mixtures.

Hydraulic conductivity (cm/s) (compacted clay/sand mixtures in rigid wall permeameters)		
Sample	Tap water (cycle 1)	Prehydr. 1000 ppm $CaCl_2$ 1% $CaCl_2$ as permeant (cycle 2)
Commercial Sample 2	7.5×10^{-8}	1.3×10^{-6}
Commercial Sample 3	3.1×10^{-9}	1.3×10^{-7}
Commercial Sample 4	1.4×10^{-8}	1.5×10^{-7}
Commercial Sample 5	4.7×10^{-9}	1.5×10^{-6}
Commercial Sample 7	9.4×10^{-9}	5.4×10^{-7}
CRC	6.0×10^{-8}	9.4×10^{-9}

Flexible Wall Hydraulic Conductivity Test

The hydraulic conductivity results summarized in Table 7 indicate that GCL 1 and GCL 2 were degraded by the $CaCl_2$ solutions as can be seen by the higher 2nd cycle hydraulic conductivity values when compared to the lower tap water baseline values. This relationship was also seen with the 3rd cycle (seawater) test runs where GCL 1 suffered degradation from the seawater. GCL 2 had been in its 3rd cycle test run for only 4 weeks when the test had to be prematurely terminated due to equipment problems. However, when referring back to the API fluid loss test results (Table 3), Commercial Sample 1 (the bentonite material used in GCL 2) produced a relatively high fluid loss value when tested in 1 wt% seawater salt solution. This indicates that the sample experienced degradation by the seawater solution. When considering the effect of 1 wt% seawater solution upon Commercial Sample 1, it can be deduced that similar degradation would likely have occurred with GCL 2 if its 3rd cycle seawater test run had been longer. Also, during the 2nd cycle test run with $CaCl_2$, it was not until after 5 weeks that GCL 2 started to show significant increases in hydraulic conductivity. Perhaps GCL 2 would have shown

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Table 7. Flexible wall hydraulic conductivity test of GCL samples.

Sample	Hydraulic conductivity (cm/s) (GCL forms in flexible wall permeameters) Tap water (cycle 1)	Prehydrated 1000 ppm $CaCl_2$/1% $CaCl_2$ as permeant (cycle 2)	Prehydrated 1% seawater salt/ 3.8% seawater salt as permeant (cycle 3)
Commercial GCL 1	8.0×10^{-10}	8.0×10^{-8}	1.4×10^{-8}
Commercial GCL 2	6.0×10^{-10}	8.0×10^{-8}	1.5×10^{-9}
CRC GCL	3.6×10^{-10}	3.8×10^{-10}	3.4×10^{-10}

the same trend during the seawater test had it been run longer.

When comparing the hydraulic conductivity test runs of the new CRC GCL, it can be seen that there is almost no difference between the 1st cycle tap water test run and the 2nd cycle $CaCl_2$ test. This indicates that the new CRC within the GCL was not significantly affected by the severe $CaCl_2$ contaminants and was able to maintain hydraulic conductivity values nearly equal to tap water baseline values. The data also show that the CRC sample was able to hydrate in the contaminated solution without any significant degradation to its performance. Although the hydraulic conductivity of the CRC GCL (during its 2nd cycle) was low and appeared to be in a steady-state condition for 2 mo, the test was terminated to make the permeameter available for other sample runs. To verify the results of this test run, the 2nd cycle was repeated and resulted in a slightly lower hydraulic conductivity of 2.5×10^{-10} cm/s. In the 3rd cycle seawater test run, the new CRC GCL was able to tolerate the seawater conditions and produce low hydraulic conductivity results nearly equal to the tap water values. Figures 4 and 5 illustrate the increasing hydraulic conductivity values of the 2 Commercial GCL samples during the 2 test runs. It is also evident that the new CRC GCL produced lower hydraulic conductivities that remained stable over much longer test periods.

Free Swell

The results in Table 8 indicate that the swelling capabilities of the Ca-contaminated clays taken from GCL 1 and GCL 2 were very low when compared to the higher free swell values of the virgin material. This confirms that the $CaCl_2$ contamination deteriorated the swelling characteristics of those bentonite products. However, the exposed clay from the CRC GCL still maintained its swelling capabilities, indicating that the

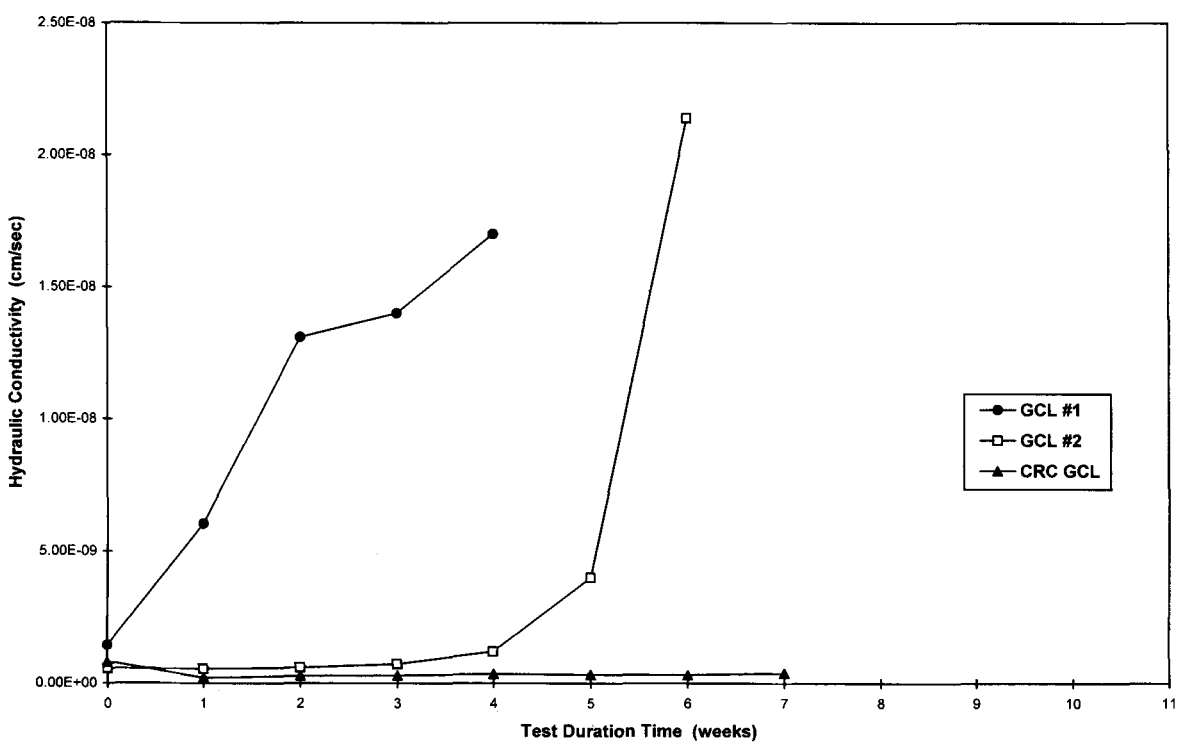


Figure 4. Flexible wall hydraulic conductivity test, prehydrated with 1000 ppm $CaCl_2$/1% $CaCl_2$ as permeant.

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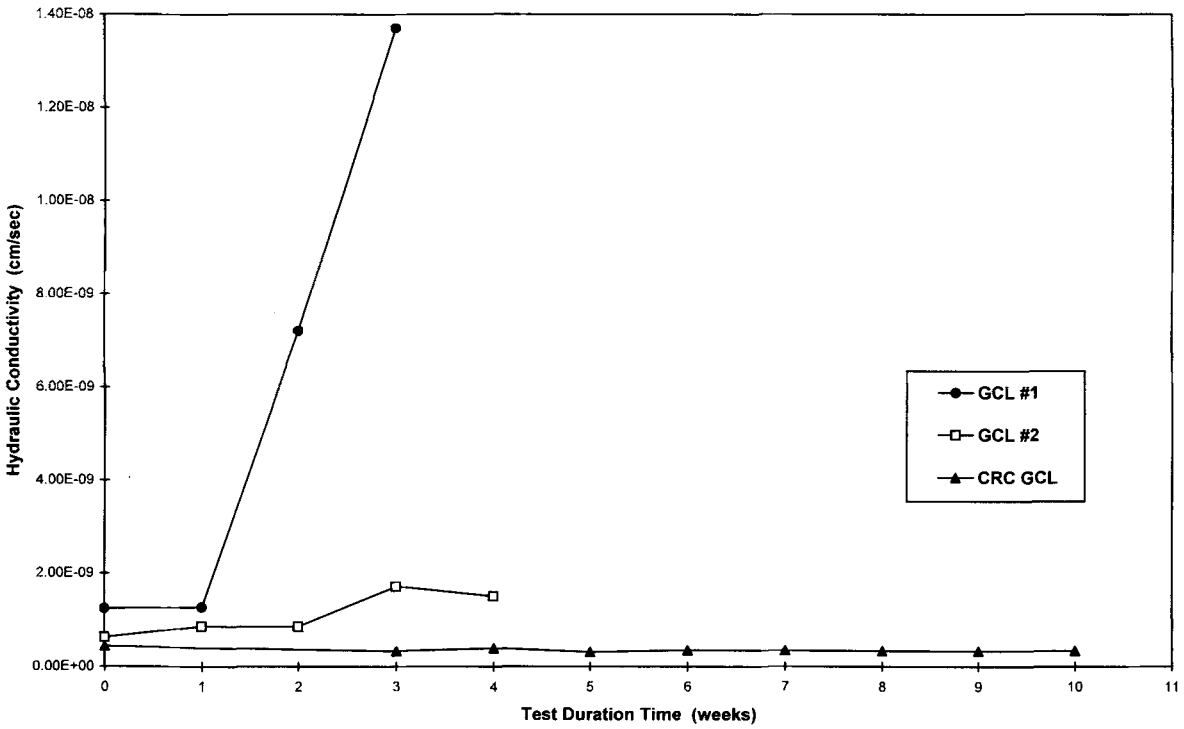


Figure 5. Flexible wall hydraulic conductivity test, prehydrated with 1% seawater salt solution as permeant.

CRC was not significantly affected by the Ca solutions.

Exchangeable Cations

The data from Table 9 show that the Ca-exposed clays from GCL 1 and GCL 2 contained lower Na concentrations while the Ca concentrations were comparatively high. This suggests that the bentonite clays in both GCLs were not able to prevent the exchange of Na ions for the Ca. The high amount of Ca correlates with the high permeability and the low swelling capacity of both GCLs. In contrast, the clay taken from the CRC GCL still showed a high Na concentration relative to the Ca. This confirms that the new CRC was able to resist the extensive Ca exchange (which the other 2 GCLs experienced), resulting in the continuing low permeabilities and high swelling characteristics.

Additional Discussion

Field applications of the new CRC have confirmed its performance in several containment applications where contaminated liquids were present. One case study involved the application of over 350,000 ft^2 of CRC GCL to contain alkaline wastewater generated from the treatment of acid mine drainage in northern California (Trauger 1996). In this system, acid drainage from an abandoned mine was collected and treated in large storage lagoons. The leachate from the sludge generated by the treatment process contained significant concentrations of salts and metals. Other applications of the CRC GCL include sites in New Jersey and in Nassau, US Virgin Islands. At both of these sites, the CRC GCL was used as a barrier to protect building structures from the influx of seawater.

Table 8. Free swell tests of clays taken from GCL samples before any exposure to contamination and after the flexible wall 2nd cycle test runs with $CaCl_2$.

	Free swell (mL/2 g)	
Sample	Before exposure to $CaCl_2$	After exposure to Ca or 2nd cycle run
Commercial GCL 1	30	7
Commercial GCL 2	33	8
CRC GCL	24	25

Table 9. Exchangeable cation analysis of the clays taken from the GCL samples tested in the 2nd cycle flexible wall test runs with $CaCl_2$.

	Exchangeable cations		
Sample	Na^+ (meq/100 g)	Ca^{2+} (meq/100 g)	Mg^{2+} (meq/100 g)
Commercial GCL 1	15	67	8
Commercial GCL 2	19	59	4
CRC GCL	47	14	6

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CONCLUSIONS

The comprehensive testing program described in this paper has demonstrated that the commercially available products that are used in GCLs and SBLs can be susceptible to certain contaminants. The testing also showed that the new CRC was resistant to the contaminants that would ordinarily attack and degrade these present commercial bentonite products. Field testing has also confirmed that CRC can be used in applications where the usage of the present commercial products may not be suitable. It has also been discovered that the new TLFP test (LSK method) can be used to obtain quick preliminary results pertaining to the sealant characteristics of bentonite clay samples.

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