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Enhancement of the Exchangeable Sodium Percentage of Bentonites through Dialysis

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TECHNICAL NOTE

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Enhancement of the Exchangeable Sodium Percentage of Bentonites through Dialysis

Reference

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ABSTRACT

Dialysis has been used over the past decades as a laboratory procedure to homoionize the exchange complex of bentonites and remove the excess soluble salts naturally contained in the bentonite pores. However, if the bentonite fabric is in an aggregated state, reduced pore accessibility limits the replacement of interlayer exchangeable cations by those in the dialyzing solution, thus undermining the efficiency of the homoionization process. To overcome these limitations, this study introduces a novel dialysis procedure that incorporates a preliminary treatment phase prior to homoionization. The additional phase consists of dialysis cycles with deionized water, aimed at promoting tactoid dispersion and improving accessibility of interlayer cations. A powdered bentonite was used to assess the improvement in sodium homoionization achieved with the new method compared with the conventional procedure. The modified dialysis procedure increased the exchangeable sodium percentage from 35 % to 78 %, accompanied by a corresponding decrease in the exchangeable calcium percentage.

Keywords

bentonite fabric, clay purification, dialysis, exchangeable cations, geo-environmental barriers, laboratory testing, remediation, waste disposal

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Introduction

Modifying the composition of the exchange complex in bentonites, widely employed as part of engineered barrier systems for the control of contaminants in the subsoil, is crucial

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for laboratory investigations aimed at understanding how the type and abundance of exchangeable cations affect water and solute transport through such barriers and their mechanical behavior. In particular, when monovalent cations (typically sodium, Na^+, and potassium, K^+) dominate over divalent cations (typically calcium, Ca^{2+}, and magnesium, Mg^{2+}), bentonites tend to exhibit a dispersed fabric, thus reducing the hydraulic conductivity (Katsumi et al. 2007; Shackelford, Sevick, and Eykholt 2010; Manassero 2020; Guarena, Dominijanni, and Manassero 2024) and increasing the water uptake and retention ability (Seiphoori, Ferrari, and Laloui 2014; Della Vecchia, Scelsi, and Musso 2019). Moreover, for the same salt concentration of the permeant solution, replacing divalent with monovalent cations on the exchange sites increases the thickness of the diffuse double layers. This promotes osmotic swelling (Mesri and Olson 1971; Di Maio 1996; Musso et al. 2017) and enhances the semipermeable membrane behavior of bentonites (Kemper and Rollins 1966; Shackelford and Lee 2003; Mazzieri, Di Emidio, and Van Impe 2010; Tong, Sample-Lord, and Bohnhoff 2021). Alongside modifying the exchange complex composition, removing excess soluble salts, which are naturally contained in pore spaces and are not electrostatically bound to mineral surfaces, also facilitates laboratory investigations on the fundamental mechanisms governing bentonite behavior.

Several laboratory procedures have been established in the literature to saturate the bentonite exchange sites with a single cation, commonly referred to as homoionization or clay purification, and to reduce the concentration of soluble salts in the pore solution. These procedures include washing and centrifuging (e.g., Mesri and Olson 1971; Di Maio 1996) as well as dialysis-based techniques (e.g., Kemper and Rollins 1966; Elrick et al. 1976; Armando et al. 2004; Segad et al. 2012). Using dialysis, the bentonite is placed inside a tube made of a selectively permeable membrane, sealed at both ends, and immersed in a bath solution (dialysate). The membrane, usually comprising regenerated cellulose or a comparable material with a molecular weight cut-off (MWCO) between 1 and 10 kDa, allows passage of water and dissolved solutes and retains the clay particles. Two treatment phases can be distinguished based on the composition of the dialysate: (1) dialysis using a concentrated salt solution, and (2) reverse dialysis using deionized water (DW). Dialysis may consist only of the first phase when modification of the exchangeable cations is required, of the second phase when removal of excess soluble salts is the objective, or of both phases in sequence when homoionization and desalination of the pore solution are necessary. Throughout such treatment phases, the dialysate is continuously stirred and periodically refreshed to sustain the concentration gradient that drives solute diffusion across the membrane.

Notwithstanding the importance of evaluating the extent of homoionization attainable through dialysis, this aspect has received limited attention so far. Sample-Lord and Shackelford (2016) were the first to examine this issue for a GCL-grade granular bentonite by investigating the relation between the concentration of the dialysate, i.e., aqueous sodium chloride (NaCl) solutions ranging from 0.1 to 1.0 M, and the resulting extent of Na^+ homoionization. The degree of homoionization was quantified by the exchangeable sodium percentage (ESP), representing the proportion of exchange sites occupied by Na^+. Their results showed an increase in ESP from 47 % before dialysis to 89 % after treatment with the highest-strength (1.0 M NaCl) dialysate, thereby demonstrating that dialysis is an effective laboratory method for altering the composition of the exchange complex of bentonites.

This paper seeks to further evaluate the effectiveness of dialysis, employing the same treatment phases used by Sample-Lord and Shackelford (2016), and to propose an alternative dialysis-based purification procedure designed to enhance its performance in cases where the bentonite exhibits an aggregated fabric with Ca^{2+} as the dominant interlayer cation. Bentonite was selected as the focus of this study because it is the reference active clay used in geoenvironmental barrier systems, including geosynthetic clay liners and slurry trench cutoff walls. Its high smectite content results in a large cation exchange capacity (CEC) and in a strong dependence of volumetric swelling, hydraulic conductivity, membrane behavior, and water retention on the composition of the exchange complex. Consequently, laboratory procedures capable of producing purified (homoionized) bentonites are of particular interest for establishing reproducible relationships between their physicochemical, fabric, and engineering properties. Although the same methodology could potentially be extended to other clay-rich materials, evaluating its applicability to different mineralogical systems is beyond the scope of the present study.

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Materials and Methods

BENTONITE AND DIALYSATES

The powdered bentonite tested in this study, supplied by Laviosa Chimica Mineraria (Livorno, Italy), is used in manufacturing a commercial needle-punched geosynthetic clay liner. The mineralogical composition of the bentonite was investigated by powder X-ray diffraction (XRD), performed by means of a Philips diffractometer (PW1730 X-ray generator, PW 1050/70 goniometer, and CuKα radiation). Semi-quantitative analysis of the XRD results, which are shown in **figure 1**, indicated the dominance of smectite (≈82 % by weight), and the presence of feldspars (≈7 %), mica/illite (≈6 %), Fe-oxides (≈3 %), calcite (≈2 %), and quartz (<1 %) as accessory minerals.

The water content in the air-dry state (ASTM D2216-19, *Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*), the liquid limit (ASTM D4318-17e1, *Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils*), and the swell index (ASTM D5890-25, *Standard Test Method for Swell Index of Clay Mineral Component of Geosynthetic Clay Liners*) were found to be equal to 11 %, 540 %, and 29 mL/2 g, respectively. The CEC, determined by the methylene blue titration method (EUBA 2002, *Methodology for the Determination of the Methylene Blue Value of Bentonite*) to enhance the accessibility of the bentonite exchange sites (Dominijanni et al. 2019), was equal to 98.9 meq/100 g. The concentrations of the exchangeable cations, determined by displacement with ammonium acetate (ASTM D7503-18, *Standard Test Method for Measuring the Exchange Complex and Cation Exchange Capacity of Inorganic Fine-Grained Soils*), were equal to 34.9 meq/100 g for Na^+, 7.0 meq/100 g for Mg^{2+}, and 13.5 meq/100 g for Ca^{2+}. The relative abundances of the bound Na^+, Mg^{2+}, and Ca^{2+} on the exchange complex of the bentonite, calculated as the ratio of the measured concentrations of the bound cations to the CEC, were found to be equal to 35.3 %, 7.1 %, and 13.6 %, respectively.

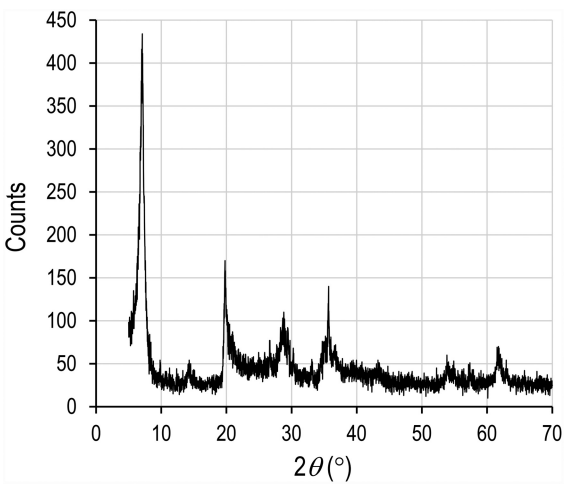
DW and an aqueous solution of 1.0 M sodium nitrate ($NaNO_3$) (ACS reagent, purity ≥99 %, Merck KGaA, Darmstadt, Germany) were used as the dialysates. The ionic concentrations in the liquid samples collected during the experimental activity were measured by means of a Dionex ICS 1000 Ionic Chromatograph.

DIALYSIS PROCEDURES

The powdered bentonite was subjected to two purification procedures, i.e., dialysis A and dialysis B. Dialysis A was similar to that considered by Sample-Lord and Shackelford (2016), hereafter referred to as dialysis SLS. A 1.0 M $NaNO_3$ solution was selected as the dialysate to purify the bentonite with respect to sodium, with the

FIG. 1

X-ray diffraction pattern of the powdered bentonite.



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concentration chosen to match the upper limit of the range of salt concentrations examined by Sample-Lord and Shackelford (2016). Dialysis A consisted of a phase of Na^+ homoionization with a frequency of dialysate replacement of 10 d and a total duration of 70 d, followed by removal of soluble salts by DW, daily replaced for a total duration of 33 d. Dialysis B was identical but preceded by a reverse dialysis phase with DW for the purpose of breaking up the bentonite tactoids. In fact, the bentonite fabric is known to be affected by the chemical composition of the dialysate (Armando et al. 2004), such that delamination of the tactoids into their individual unit layers is expected to occur when the bentonite is exposed to DW. This delamination favors the substitution of the bound cations, initially isolated in the interlayer voids of the tactoids, with Na^+ contained in the dialysate during the subsequent Na^+ homoionization phase.

The dialysate strength, the duration of the homoionization phase, which comprised seven dialysis cycles of 10 d each, and the longer reverse dialysis phase were expected to provide equal or greater effectiveness than the original procedure (dialysis SLS). Details of the procedures used in the present study and the one implemented by Sample-Lord and Shackelford (2016) are summarized in **Table 1**.

The bentonite specimen was prepared, for each of the two purification procedures, by evenly distributing 60 g (oven-dry mass) of powdered bentonite into four dialysis bags (i.e., 15 g of bentonite per dialysis bag). The bags were made of regenerated cellulose membrane tubing (flat width = 76 mm and MWCO = 14 kDa, Merck KGaA, Darmstadt, Germany). To prepare the bags, four 300 mm-long sections of the tubing were cut and soaked in DW for 24 h to remove impurities. After soaking, one end of each bag was sealed with a plastic clamp, and the powdered bentonite and dialysate were introduced sequentially through the open end until the filled length reached approximately 150 mm. The open end was then sealed with a second plastic clamp. The four filled dialysis bags were placed in a glass jar containing 7 L of dialysate, which was continuously agitated using a magnetic stirrer (2 mag AG, München, Germany) at a stirring speed of 500 rpm and at a controlled room temperature of approximately 22°C. The resulting ratio of dry bentonite mass to dialysate mass was approximately 1:117, comparable to the ratio of 1:280 reported by Sample-Lord and Shackelford (2016). Upon completion of the purification process, the bentonite paste was removed from the bags, and its water content was determined by oven drying. The dialyzed bentonite was then characterized using the ammonium acetate displacement method (ASTM D7503-18) to quantify the concentrations of bound Na^+, Mg^{2+}, and Ca^{2+}. The determination of the concentrations of the bound cations for the dialyzed bentonite was performed in two replicates.

Results and Discussion

Figure 2 shows the occupation of the bentonite exchange sites, expressed as the percentages of exchangeable Na^+, Mg^{2+}, and Ca^{2+}, before and after purification by the two dialysis procedures. For dialysis A, the percentages of exchangeable Na^+, 33.5 % (±4.9 %), and Mg^{2+}, 11.9 % (±1.6 %), which were averaged over the two replicates,

TABLE 1

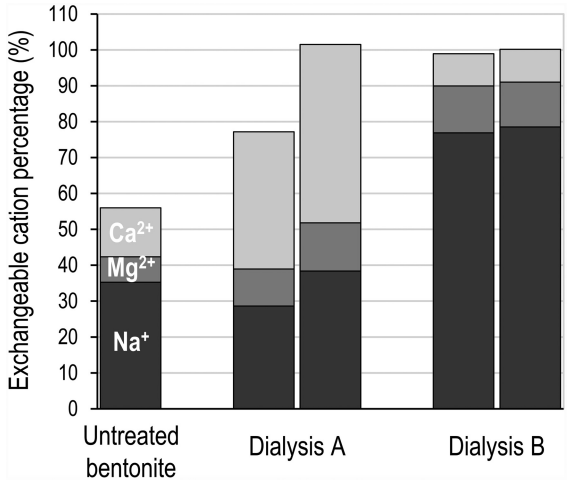
Comparison of the treatment phases included in the Sample-Lord and Shackelford (2016) dialysis protocol (dialysis SLS) with those incorporated into the dialysis A and dialysis B protocols used in this investigation

			Dialysis SLS	Dialysis A	Dialysis B
Phase (0) breakup of tactoids	Dialysate				Deionized water
	Total duration	(d)			33
	Frequency of dialysate replacement	(d/cycle)			1
Phase (1) Na^+ homoionization	Dialysate		NaCl (0.1–1 M)	$NaNO_3$ (1 M)	$NaNO_3$ (1 M)
	Total duration	(d)	7	70	70
	Frequency of dialysate replacement	(d/cycle)	1	10	10
Phase (2) removal of soluble Na^+	Dialysate		Deionized water	Deionized water	Deionized water
	Total duration	(d)	13–14	33	33
	Frequency of dialysate replacement	(d/cycle)	1	1	1

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FIG. 2

Relative proportions of Na^+, Mg^{2+}, and Ca^{2+} retained on the bentonite exchange complex in the as-received material and following purification using the dialysis A and dialysis B procedures.



remained close to their initial values of 35.3 % and 7.1 %, respectively, whereas the percentage of exchangeable Ca^{2+} increased markedly from 13.6 % to 43.9 % (±5.7 %). Although this outcome appears to contrast with the findings of Sample-Lord and Shackelford (2016), it can be interpreted by considering the change in the bentonite fabric during purification and the consequent variation in the accessibility of the bentonite pores.

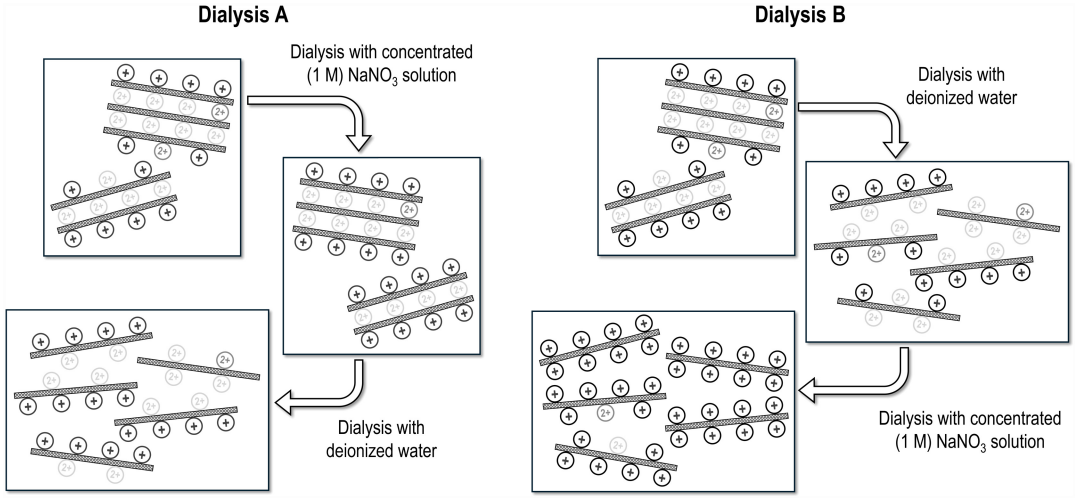
When the bentonite exhibits an aggregated microstructure, divalent cations tend to be concentrated within the interlayer voids (i.e., the voids between adjacent montmorillonite unit layers that make up the tactoids), whereas monovalent cations are primarily adsorbed on the external tactoid surfaces (Laird 2006). Under these conditions, the cations that are readily displaced by ammonium ions (NH_4^+) during the ASTM D7503-18 procedure are those located on the external surfaces, and only these cations contribute to the measured exchangeable cation percentages. Based on this interpretation, the uncharacterized portion of the exchange complex of the untreated bentonite (100 %−35.3 %−7.1 %−13.6 % = 44.0 %) can be attributed to the interlayer voids that were inaccessible to NH^{4+} displacement.

As pictorially shown in **figure 3** for dialysis A, treatment of the bentonite specimen with the concentrated $NaNO_3$ solution resulted primarily in the replacement of divalent cations located on the external tactoid surfaces by Na^+ from the dialysate, whereas the divalent cations residing on the internal tactoid surfaces remained largely unaffected. During the reverse dialysis with DW, the excess soluble salts were removed and the bentonite fabric underwent a structural change, namely the breakup of the tactoids into their constituent unit layers. This fabric evolution did not further alter the composition of the exchange complex. Under these new conditions, however, the divalent cations originally confined within interlayer voids became accessible to NH_4^+ displacement, leading to (1) an almost complete characterization of the exchange complex (89.3 % on average) in the dialyzed bentonite, and (2) the observed increase in the measured percentage of exchangeable Ca^{2+}.

Based on the bar chart shown in **figure 2**, the differing effectiveness of the treatment phase aimed at purifying the bentonite with respect to Na^+, with and without the preliminary DW treatment (dialysis B and dialysis A, respectively), becomes apparent. Whereas the original dialysis procedure produced no meaningful change in the occupation of the bentonite exchange sites, the modified procedure increased the percentage of exchangeable Na^+ from 33.5 % to 77.7 % (±0.8 %), accompanied by a corresponding decrease in the exchangeable Ca^{2+} percentage from 43.9 % to 9.1 % (±0.1 %). As shown in **figure 3**, the same conceptual framework used to interpret the behavior observed in dialysis A can be extended to explain the results obtained for dialysis B. Preliminary exposure to DW caused the bentonite fabric to become dispersed without altering the exchange complex composition relative to the untreated material. This dispersion exposed both the external and internal tactoid surfaces,

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FIG. 3 Evolution of the bentonite fabric and exchange complex composition throughout the dialysis A and dialysis B treatment phases (Na^+ shown in dark gray, Mg^{2+} in medium gray, and Ca^{2+} in light gray).



making the divalent cations originally bound to the latter sites readily replaceable by Na^+ when the bentonite was subsequently dialyzed with the concentrated $NaNO_3$ solution. Consequently, by the end of all treatment phases comprising dialysis B, the exchange complex of the bentonite was predominantly saturated with Na^+.

Conclusion

The use of purification procedures is a fundamental prerequisite when testing active clays, such as bentonites, for the characterization of their physicochemical, transport, and mechanical properties. These procedures aim to both occupy the majority of the exchange sites with a single cation and effectively rinse the specimen of any excess soluble salts. In this context, the present study evaluated the effectiveness of two dialysis procedures (dialysis A and dialysis B) for the purification of a GCL-grade powdered bentonite. These methods closely follow the approach examined by Sample-Lord and Shackelford (2016) with some modifications aimed at enhancing the purification efficiency.

Because of the aggregated fabric of the tested bentonite specimen and the preferential segregation of divalent cations in the interlayer voids, the Na^+ homoionization treatment phase of dialysis A, which lasted ten times longer than the procedure evaluated by Sample-Lord and Shackelford (2016), did not significantly modify the ESP compared to the untreated bentonite. Dialysis B, comprising a preliminary treatment phase aimed at delaminating the bentonite tactoids through dialysis with DW, resulted in a notable increase (from 35 % to 78 %) in the ESP.

Besides the importance of greater control over the testing conditions in the laboratory, the development of purification procedures for the saturation of the bentonite exchange complex with monovalent ions can be regarded as an opportunity for the mining industry to move toward more sustainable practices. As a result of the growing demand for bentonite with a high swelling potential in oil and gas exploration, construction, and environmental remediation activities, the exploitation of high-purity sodium bentonite deposits has been steadily increasing over the past five decades (Black et al. 1981; Lofty et al. 1986, 1991; Taylor et al. 1995; Taylor, Hillier, and Benham 2005; Stockwell et al. 2000; Brown et al. 2010, 2015, 2019; Idoine et al. 2025). In contrast, calcium bentonite deposits have not experienced a similar increase in their exploitation, because of the inability of the currently used activation methods (e.g., mixing with sodium carbonate) to achieve an equivalent performance

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to that of natural sodium bentonites. Therefore, the possibility of homoionizing calcium bentonites with respect to sodium ions through innovative treatment methods, comprising purification via dialysis, may allow for a more rational and sustainable exploitation of natural bentonite deposits.

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DISCLOSURE OF POTENTIAL CONFLICTS OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

DATA AVAILABILITY STATEMENT

The data, models, and/or code used for this study are available from the corresponding author upon request.

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