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MULTIPLE CATION-EXCHANGE CAPACITY MEASUREMENTS ON STANDARD CLAYS USING A COMMERCIAL MECHANICAL EXTRACTOR[1]

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Abstract—Sequential cation-exchange capacity (CEC) measurements were obtained from standard clays using a mechanized, variable-rate leaching device. The device consists of a motorized screwjack and as many as 24 leaching tubes coupled to 60-ml plastic syringes. Controlled withdrawal of the syringe plungers produces a vacuum that permits samples in the leaching tubes to be extracted at a uniform rate. A single, 8-hr leaching of clays with 35 ml of salt solution was found to be comparable to multiple saturations or displacements using a centrifuge. CECs consistent with published values were obtained for reference 2:1 clay minerals using both acetate and chloride salts of Na, Ca, and Mg. Potassium-exchange capacities were also successfully measured following *in situ* thermal treatment of samples in the leaching tubes. Variations in measured CECs for kaolin-group minerals due to salt intercalation were minimized by using chloride rather than acetate salts and by washing with a dilute aqueous solution of the saturating cation following initial saturation. The mechanical extractor significantly reduced the effort required to perform conventional CEC determinations without sacrificing analytical precision.

Key Words—Apparatus, Cation-exchange capacity, Ion-exchange method, K-fixation, Leaching tube, 2:1 clay minerals.

INTRODUCTION

Most methods of determining the cation-exchange capacity (CEC) of soils and clays require multiple centrifugations of the sample to achieve cation saturation, removal of excess salt, and displacement of the index cation. As a result, these methods are usually time consuming, labor intensive, and subject to errors associated with salt removal and/or sample loss during the many decantation steps.

Recently, a mechanical extractor was developed to obtain controlled leaching of soil samples for various chemical analyses including CEC determinations (Holmgren *et al.,* 1977). The commercially available units (Concept Engineering Co., Box 2555, Lincoln, Nebraska 68502) consist of 12 or 24 extraction assemblies and a motorized mechanical holder. The device can be set to operate overnight with variable extraction rate and automatic shutoff. The design of the extraction assembly allows uniform sample-solution contact time regardless of texture and makes each saturation, wash, or displacement step more effective by placing the sample in continual contact with fresh solution. In addition, problems of sample loss are minimized, and a single sample can be subjected to multiple analyses without physical disturbance. In this paper, multiple CEC measurements obtained using this instrument are presented for a variety of reference clays and are compared to available published data.

MATERIALS AND METHODS

Samples

Reference CMS and API clays were obtained from the Source Clay Repository of The Clay Minerals Society and Ward's Natural Science Establishment, Rochester, New York, respectively (Table 1). Samples of the CMS source clays were generally used without pretreatment; however, $CaCO_3$ was removed from samples SWy-1, STx-1, and SHCa-1 by leaching with pH 5 NaOAc-HOAc buffer prior to CEC measurement. Selected samples of the CMS source clays were also wet sedimented to separate the <2-μm fraction using an automatic fractionator (Rutledge *et al.,* 1967). The clay separates were subsequently Mg-saturated, frozen, and freeze-dried.

Additional pretreatments were employed for the CMS special clays and the API clays. Particles of the Llano, Texas, vermiculite (VTx-1) were hand-picked from the sample, treated with pH 5 buffer to remove residual magnesite, and subsequently ground in ethanol to pass a 60-mesh sieve. Special clay samples CCa-1, SCa-2, SWa-1, IMt-1, ISMt-1, SapCa-1, SepNev-1, and API samples No. 12 and No. 35 were also ground in ethanol and passed through a 60-mesh sieve. The <2-μm fractions of specimens SWa-1, SapCa-1, SepNev-1, and NG-1 were separated using wet sedimentation in an automatic fractionator. The clay separates were then Mg-saturated, frozen, and freeze-dried.

General procedure

(1) Consistent with recommendations of the manufacturer, 0.5–2 g of clay were accurately weighed, mixed

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Table 1. Mineralogy and source of samples.

Sample	Mineralogy[1]	Source
	CMS source clays	
KGa-1	Well-crystallized kaolinite	Washington County, Georgia
KGa-1, <2 μm	Well-crystallized kaolinite	
KGa-2	Poorly crystallized kaolinite	Warren County, Georgia
KGa-2, <2 μm	Poorly crystallized kaolinite	
SWy-1	Na-montmorillonite, minor quartz and calcite	Crook County, Wyoming
SWy-1, <2 μm	Montmorillonite	
STx-1	Ca-montmorillonite, minor quartz and calcite	Gonzales County, Texas
STx-1, <2 μm	Montmorillonite	
SAz-1	Ca-montmorillonite, minor silica phase	Apache County, Arizona
SAz-1, <2 μm	Montmorillonite	
SHCa-1	Hectorite, calcite (27%), minor quartz	San Bernardino County, California
SHCa-1, <2 μm	Hectorite	
Syn-1	Synthetic mica/smectite	NL Industries
PFl-1	Palygorskite, minor quartz	Gadsden County, Florida
	CMS special clays	
CCa-1	Chlorite	Flagstaff Hill, El Dorado County, California
SCa-2	Montmorillonite	Otay, San Diego County, California
SWa-1	Ferruginous smectite, minor quartz	Grant County, Washington
SWa-1, <2 μm	Ferruginous smectite	
VTx-1	Vermiculite (hand-picked flakes)	Llano County, Texas
IMt-1	Illite, quartz, minor feldspar	Silver Hill, Montana (shale, Cambrian)
ISMt-1	Illite/smectite, quartz, minor feldspar	Montana (Mancos Shale)
NG-1	Quartz (80–85%), nontronite (15–20%), minor feldspar and goethite	Hohen Hagen, Germany
NG-1, <2 μm	Nontronite, minor goethite and quartz	
SapCa-1	Saponite, ≤3% diopside	Ballarat, California
SapCa-1, <2 μm	Saponite	
SepNev-1	Sepiolite, minor calcite	Two Crows, Nevada
SepNev-1, <2 μm	Sepiolite	
	API Clays	
API-12	Halloysite, gibbsite	North Gardiner mine, Bedford (Huron), Indiana
API-35	Illite, illite-smectite, quartz, minor calcite, feldspar, and smectite	Fithian, Illinois

[1] Mineralogy was determined or obtained from published data as follows: CMS source clay data from van Olphen and Fripiat (1979); CMS special clay data from information supplied by Source Clays Repository of The Clay Minerals Society and by X-ray powder diffraction; API data by X-ray powder diffraction.

with 0.50 g of Celite powdered paper pulp to maintain permeability, and quantitatively transferred to a 60-ml sample tube which contained 1.00 g of compressed paper pulp as a filter (Figure 1). Sample transfer was achieved using 5–10 ml of ethanol from a wash bottle. Distilled water was avoided to minimize sample dispersion and loss of permeability. Each sample tube was then attached to the mechanical extractor and coupled to a standard extraction syringe.

(2) Using a wash bottle, about 20 ml of solution containing the index cation was added to the sample tubes. The extractor was then activated and adjusted so that saturating solution was drawn through the samples at a rate of about 2 ml/min (suction was applied to the sample tube as the syringe plunger was withdrawn by a mechanized screw jack during system operation). When about 5 ml of solution remained above each sample, the extractor was turned off and adjusted for a slow extraction rate (≃0.1 ml/min). A reservoir tube was then attached to the top of each sample tube and filled to the 35-ml mark with saturating solution. The extractor was reactivated and allowed to run unattended (usually overnight) until an automatic shutoff switch was engaged (after about 8 hr). The saturating solution collected in the extraction syringes was discarded, and the syringe and reservoir tubes were rinsed with distilled water. The extractor was returned to its initial position and the syringes reattached.

(3) Excess salt was removed by washing as in step (2) except that a fast extraction rate (0.5–1.0 ml/min) was used throughout.

(4) The index cation was displaced using a slow extraction rate as in step (2) except that the displaced solution was retained, quantitatively transferred to a volumetric flask, and diluted to mark with displacing solution. The CEC was then determined by analyzing

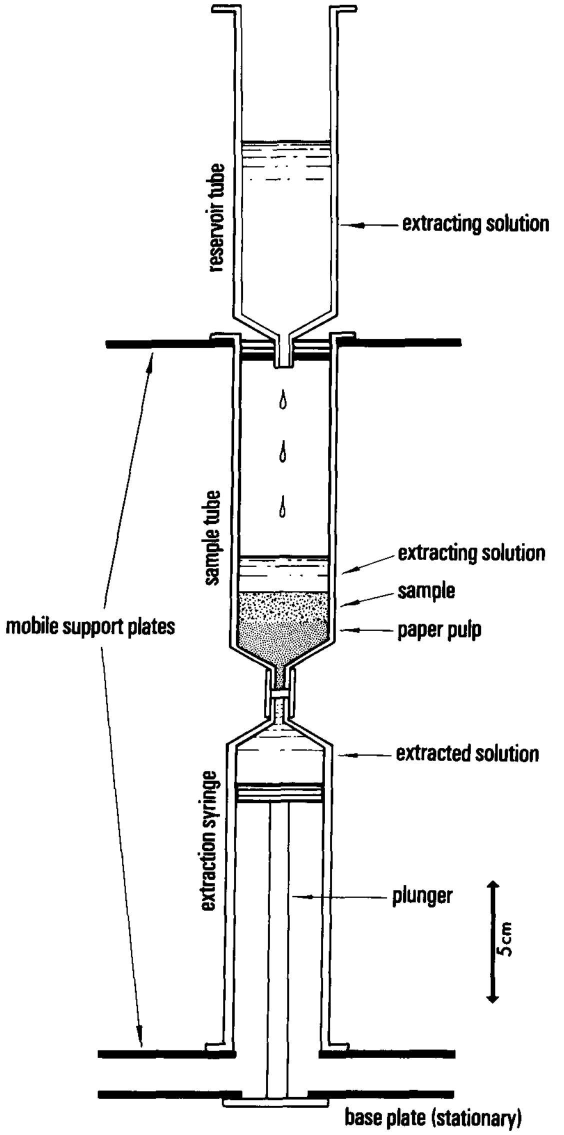

Figure 1. Cross section of extraction assembly, mechanical extractor.

the displaced solution for the quantity of saturating cation with a correction for any cation retention by a blank containing only Celite and paper pulp.

Specific procedures

CECs were measured using 1 N solutions of Na, Mg, Ca, and K. Buffered (pH 7) solutions of all cations were made by preparing solutions which were 0.8 N with respect to chloride (Cl) and 0.2 N with respect to acetate (OAc). Unbuffered solutions prepared from the pure Cl salts (adjusted to pH 7) were also used for Na, Ca, and K determinations. In general, multiple CECs were determined sequentially using the same sample in the order Na, Ca, Mg, K. Measurements were replicated as many as 3 times using separate samples. Displaced Ca and Mg were determined using atomic absorption spectrophotometry; Na and K were analyzed by flame emission spectroscopy. All CECs were expressed on an oven-dry (110°C) basis.

The specific experimental procedure for each CEC determination is outlined in Table 2. For example, a CaEC-1 determination was made by first saturating a sample with Ca using a solution that was 1 N Ca, 0.8 N Cl, and 0.2 N OAc. It was then washed once with a solution made by diluting the saturating solution 1:100 with distilled water. Next, the sample was washed once with 95% ethanol. Finally, Ca was displaced with a solution that was 1 N Mg, 0.8 N Cl, and 0.2 N OAc.

The procedures listed in Table 2 are modifications of centrifuge-based techniques. For example, the NaEC procedures are similar to that recommended by the U.S. Salinity Laboratory Staff (1954) except that Ca was used instead of NH_4 as the displacing cation to avoid possible NH_4 entrapment in vermiculite or the smectites prior to KEC determinations. The MgEC, CaEC, and KEC procedures are modifications of those proposed by Alexiades and Jackson (1965), Jackson (1975), Coffman and Fanning (1974), and Wada and Harada (1969). Alexiades and Jackson (1965) and

Table 2. Solution and thermal parameters for cation-exchange capacity (CEC) measurements.

CEC designation	Saturating solution	Washings			Thermal treatment	Displacing solution
		1st	2nd	3rd		
NaEC-1	1 N Na, 0.8 N Cl, 0.2 N OAc	EtOH	EtOH	–	–	1 N Ca, 0.8 N Cl, 0.2 N OAc
NaEC-2	1 N Na, 0.8 N Cl, 0.2 N OAc	EtOH	EtOH	EtOH	–	1 N Ca, 0.8 N Cl, 0.2 N OAc
NaEC-3	1 N Na, 1 N Cl	EtOH	EtOH	EtOH	–	1 N Ca, 1 N Cl
MgEC	1 N Mg, 0.8 N Cl, 0.2 N OAc	0.01 N Mg, Cl + OAc	EtOH	–	–	1 N Ca, 0.8 N Cl, 0.2 N OAc
CaEC-1	1 N Ca, 0.8 N Cl, 0.2 N OAc	0.01 N Ca, Cl + OAc	EtOH	–	–	1 N Mg, 0.8 N Cl, 0.2 N OAc
CaEC-2	1 N Ca, 1 N Cl	0.01 N Ca, Cl	EtOH	–	–	1 N Mg, 1 N Cl
KEC-1	1 N K, 0.8 N Cl, 0.2 N OAc	EtOH	EtOH	EtOH	110°C	1 N NH_4, 0.8 N Cl, 0.2 N OAc
KEC-2	1 N K, 1 N Cl	EtOH	EtOH	EtOH	110°C	1 N NH_4, 1 N Cl
KEC-3	1 N K, 1 N Cl	0.04 N K, Cl	EtOH	EtOH	110°C	1 N NH_4, 1 N Cl

Table 3. Experimental (Na, Mg, Ca) and reported cation-exchange capacities for standard clays.

Sample	Cation-exchange capacities (meq/100 g)							Reported CEC (meq/100 g)
	NaEC-1	NaEC-2	NaEC-3	MgEC	CaEC-1	CaEC-2	CEC[1]	
IMt-1	14.3	13.6	14.2	14.7	15.2	15.4	14.7 ± 0.8	15[2]
API-35	18.1	17.1	17.5	18.5	18.2	—	18.0 ± 0.8	25[3], 20[4]
ISMt-1	35.4	38.4	35.1	37.1	37.0	37.3	36.7 ± 1.6	
Syn-1	—	—	61.5	—	—	62.9	61.9 ± 2.2	67–161[5]
NG-1, <2 μm	97.8	97.8	94.0	96.9	98.2	95.9	97.0 ± 2.2	
SWa-1	98.6	103	101	99.8	101	104	101 ± 2	103[6]
SWa-1, <2 μm	—	—	107	—	—	108	107 ± 1	
SWy-1	80.1	79.2	81.3	80.0	79.0	—	79.8 ± 1.2	74–79[5]
SWy-1, <2 μm	—	88.7	88.5	85.8	87.1	86.0	87.0 ± 1.6	89[7], 91[8]
STx-1	86.3	86.4	81.9	84.4	85.0	81.0	84.2 ± 2.8	80–88[5], 85[9]
STx-1, <2 μm	—	89.8	86.7	89.8	90.6	86.4	88.3 ± 2.5	
SHCa-1	44.7	48.2	43.6	48.0	47.5	—	46.5 ± 2.3	40–46[5]
SHCa-1, <2 μm	—	87.2	87.5	87.2	93.1	90.1	89.2 ± 2.5	82[7], 86.5[10]
SapCa-1	—	—	73.6	—	—	73.9	73.6 ± 0.2	73.8[11]
SapCa-1, <2 μm	—	—	80.1	—	—	81.3	80.4 ± 0.9	
SAz-1	124	126	126	121	126	127	125 ± 3	112–129[5], 120[12]
SAz-1, <2 μm	—	128	131	125	131	131	130 ± 2	133[7], 120[13]
SCa-2	121	124	127	119	124	123	123 ± 3	114[7], 121[14], 122[15]
VTx-1	220	231	219	212	209	197	212 ± 10	210[9]
PFl-1	20.4	19.9	19.2	19.3	20.0	20.6	20.0 ± 1.1	16–25.4[5], 19.7[16]
SepNev-1	—	—	17.4	—	—	—	17.4 ± 0.9	
SepNev-1, <2 μm	—	—	19.8	—	—	—	19.8 ± 1.5	
CCa-1	1.0	0.5	0.5	1.5	1.3	0.5	1.0 ± 0.5	
KGa-1	10.7	1.4	0.7	2.7	3.1	1.6	—	1.7–2.4[5]
KGa-1, <2 μm	—	1.7	1.3	2.5	3.5	1.8	—	
KGa-2	11.4	2.4	0.7	3.7	3.7	2.8	—	2.8–3.8[5]
KGa-2, <2 μm	—	2.3	3.6	4.0	4.2	3.5	—	
API-12	52.7	—	17.6	50.3	43.2	18.7	—	10.6[3]

[1] CEC = average across all determinations with standard deviation.

[2] Hower and Mowatt (1966).

[3] Kerr *et al.* (1950).

[4] Koppelman and Dillard (1977), <10 μm.

[5] van Olphen and Fripiat (1979), p. 199.

[6] Goodman *et al.* (1976).

[7] Grim and Kulbicki (1961), <2 μm.

[8] Sposito *et al.* (1983), SWy-1, <0.5 μm.

[9] Egashira *et al.* (1982) "Llano vermiculite," <2 μm.

[10] Schoonheydt (1984), SHCa-1, <2 μm, average sum of cations, Co-hectorite and Ni-hectorite.

[11] Post (1984).

[12] Komarneni and White (1983).

[13] Ames *et al.* (1983).

[14] Maes *et al.* (1979).

[15] Weaver and Pollard (1973).

[16] Golden *et al.* (1985).

Jackson (1975) used Cl solutions in their determinations, whereas Coffman and Fanning (1974) employed OAc solutions buffered at pH 7. Wada and Harada (1969) used both Cl and OAc systems for selected samples.

In general, modifications to the referenced centrifuge methods related primarily to the number of saturation, wash, or displacement steps employed. A single, overnight saturation or displacement on the extractor was found to be equivalent to multiple saturations or displacements using a centrifuge. Because of problems commonly associated with removal of excess salt, including the formation of mineral-salt complexes, hydrolysis of the saturating cation, and sample dispersion, a variety of wash procedures were evaluated (Table 2). In all tests, a final wash with ethanol was employed rather than a measurement of the entrained salt solution as preferred by some investigators. Entrained-solution determinations are especially useful for samples containing variable charge clays, and it should be noted that this approach is totally compatible with the syringe system employed with the mechanical extractor.

Table 4. Potassium-exchange capacity measurements for standard clays.

Sample	Potassium-exchange capacities (meq/100 g)				Reduction[2] %
	KEC-1	KEC-2	KEC-3	KEC[1]	
IMt-1	13.5	13.2	13.7	13.5 ± 0.5	8
API-35	16.7	17.9	—	17.1 ± 1.2	5
ISMt-1	30.6	30.8	30.7	30.7 ± 0.5	16
Syn-1	—	—	46.2	46.2 ± 0.5	25
NG-1, <2 μm	71.9	70.5	70.1	70.9 ± 2.1	27
SWa-1	83.6	86.9	82.9	83.9 ± 1.9	17
SWa-1, <2 μm	—	—	86.8	—	19
SWy-1	81.2	81.9	73.3	77.4 ± 7.9	3
SWy-1, <2 μm	87.2	76.6	78.4	79.8 ± 6.1	8
STx-1	78.3	78.0	73.7	76.4 ± 2.6	9
STx-1, <2 μm	78.7	78.2	74.8	76.6 ± 2.2	13
SHCa-1	40.4	40.7	38.3	39.6 ± 1.2	15
SHCa-1, <2 μm	64.4	77.0	76.1	73.4 ± 6.1	18
SAz-1	98.6	102	102	100 ± 2	20
SAz-1, <2 μm	98.7	102	103	102 ± 2	22
SCa-2	88.7	89.3	88.6	88.8 ± 1.9	28
VTx-1	10.7	2.6	2.1	5.6 ± 4.7	97
PFl-1	36.4	29.0	18.1	—	—
CCa-1	1.1	—	—	1.1 ± 0.4	—
KGa-1	3.8	3.4	2.0	—	—
KGa-1, <2 μm	6.4	8.7	2.4	—	—
KGa-2	7.4	7.9	3.2	—	—
KGa-2, <2 μm	12.8	11.2	4.1	—	—
API-12	73.6	35.4	20.3	—	—

[1] KEC = average across all determinations with standard deviation.

[2] [(CEC − KEC)/CEC] × 100; CEC from Table 3.

Thermal treatment

KEC determinations were made following measurement of Na, Ca, and Mg CECs. After K saturation and removal of excess salt, the sample tubes were detached from the extractor and heated to 110°C overnight (the sample tubes are made of polypropylene and can be safely heated to 135°C). Following heat treatment, the sample tubes were reattached to the extractor, and the exchangeable K was displaced with NH_4.

RESULTS AND DISCUSSION

The measured Na, Mg, and Ca CECs are presented in Table 3. The CEC values for the 2:1 minerals are in good agreement with available published values. For a given 2:1 mineral, there is also good agreement between CEC values irrespective of the saturating cation (Na, Mg, Ca) or wash procedure. Hence, results can be averaged across all treatments to obtain meaningful error terms.

Similarly, the KEC values for the 2:1 clays (Table 4) show good agreement irrespective of saturating solution (i.e., presence or absence of acetate) and wash procedure. The values, however, are lower (by as much as 97%) than the corresponding average CECs obtained using other saturating cations. Because of the great affinity of the vermiculite interlayer for soluble K, the difference between the CaEC and KEC [(CaEC − KEC/154) × 100] has commonly been used as a measure of vermiculite content in mixed clay systems (Alexiades and Jackson, 1965; Coffman and Fanning, 1974). Data obtained in the present study indicate that the smectites may also entrap variable quantities of K depending upon the source and magnitude of their layer charge. Horváth and Novák (1975) have likewise concluded that the ability of smectites to "fix" K is directly related to their total charge.

For the kaolin samples, substantial differences exist between the measured CEC values (Tables 3 and 4). In most tests, acetate salts yielded higher CECs than did the corresponding chlorides. Previous studies (e.g., Garrett and Walker, 1959; Wada and Harada, 1969) have demonstrated that the differential intercalation of salts can cause serious variations in measured CECs for the kaolin-group minerals. Although kaolinite does not form complexes as readily as halloysite, both minerals are clearly more susceptible to intercalation by acetates than by chlorides (Wada, 1959, 1961; MacEwan and Wilson, 1980). Previous research has also shown that the kaolins form complexes more readily with K or NH_4 salts than with salts of Li, Na, or the common divalent cations (Garrett and Walker, 1959; MacEwan and Wilson, 1980). Measured CECs for the reference kaolins examined in this study follow the general order K > Ca ≃ Mg > Na. This trend is more pronounced for treatments involving acetate salts than for chlorides.

Differences in the kaolin CECs may also be determined in part by the relative effectiveness of the wash procedure in removing intercalated salts. Garrett and Walker (1959) observed that washing with a liquid in which the saturating salt was sparingly soluble (such as ethanol) failed to achieve adequate removal. In Table 3, this effect is demonstrated by the fact that comparable NaECs were obtained for most specimens using either two (NaEC-1) or three (NaEC-2) ethanol washes; however, three washes yielded a five- to six-fold reduction in the values for samples KGa-1 and KGa-2. Wada and Harada (1969) also found that CEC measurement errors can be reduced for kaolin-group minerals if dilute (<0.2 N) saturating solutions are used or if samples are washed with a dilute aqueous solution of the saturating cation following initial saturation. The value of the latter approach is reflected in the KEC data collected in the present study. For the kaolinite samples, similar results were obtained using the acetate and chloride salts of K when ethanol was employed as the sole wash solution (KEC-1 vs. KEC-2 in Table 4). In contrast, the measured KECs were reduced by more than 50% when samples were equilibrated with dilute KCl prior to final washing with ethanol (KEC-3). This result suggests that the wash procedure may be more critical than the particular salt employed when evaluating the KEC of kaolinites.

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