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A COMPARISON OF METHODS FOR THE DETERMINATION OF CATION EXCHANGE CAPACITY OF SOILS

PORÓWNANIE METOD OZNACZANIA POJEMNOŚCI WYMIANY KATIONÓW I SUMY KATIONÓW WYMIENNYCH W GLEBACH

Abstract: The object of this study was to compare the results obtained with four methods of determination of cation exchange capacity (CEC) and sum of exchangeable cations (Ca, Mg, K) in soils. One of these methods is Kappen's method and the others methods are based on different extracting reagents: sodium acetate (pH = 8.2), barium chloride and hexaamminecobalt(III) chloride. Values measured with barium ions and hexaamminecobalt(III) ions as index cations are very comparable and these two methods can be considered as equivalent. Kappen's method gives overestimated results, especially for acid soils reach in organic matter and very calcareous soils. Sodium acetate, buffering the pH of the extracting solution, causes increase of numbers of negatively charged sites and particularly those bonded to organic matter and for this reason values obtained with this method are overestimated. Nevertheless, it is possible to correct this error for a given soil sample by regression equation considering pH of soil, clay and organic carbon content.

Keywords: cation exchange capacity (CEC), exchangeable cations, barium ions, hexaamminecobalt(III) ions

Introduction

The cation exchange capacity (CEC) and sum of exchangeable cations are an essential measurements in agronomy and soil science to estimate the physicochemical state of a soil, which may be a good indicator of soil quality and productivity. The cation exchange capacity (CEC) of a soil is a measure of the quantity of negatively charged sites on soil surfaces that can retain positively charged ions (cations) by electrostatic forces. Soil CEC is expressed in units of charge per weight of soil ($cmol(+) kg^{-1}$ or $mmol(+) kg^{-1}$ - centimoles or milimoles of charge per kilogram of dry soil). Soil negative charges are the result of isomorphic substitutions in phyllosilicate structures, non-compensated bonds at the edges of reticular plans or dissociation of functional groups (-OH, -COOH). These charges can be divided into permanent or fixed charges, which are independent of the pH and variable charges, which depend closely on the pH. The cations retained electrostatically are easily exchangeable with other cations in the soil solution and are readily available for plant

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uptake. Thus, CEC is important for maintaining adequate quantities of plant available calcium (Ca²⁺), magnesium (Mg²⁺) and potassium (K⁺) in soils. Under acid conditions (pH < 5.5), aluminum (Al³⁺) may also be present as an exchangeable cation.

Numerous methods are proposed to measure the cation exchange capacity (CEC) and sum of exchangeable cations in soils and many will give quite different results [1-5]. For all methods the first stage involved the saturation of the soil sample with an index cation. The results mainly depend upon the nature of this cation, the pH of extraction solution and the initial soil pH. The cation exchange capacity can vary greatly in soils rich in organic matter [6, 7], iron and aluminum hydrous oxides and kaolinitic clays [8] with predominantly variable charges. These charges depend upon the pH and ionic strength of the reagent used. Most of methods for determination cation exchange capacity imply that the cations extracted were exchangeable. In fact, some elements can be released from nonexchangeable sources, for instance calcium from dissolution of calcium carbonate. For this reason, the results of cation exchange capacity determination in calcareous soils may be significantly overestimated [4, 9-12]. The methods used can be grouped in four main categories: determination of CEC by summation of exchangeable cations; measurement of CEC at the soil pH (effective CEC_e); measurement of CEC at a given buffered pH; measurement of CEC at the pH for which the charge is zero (zero point charge (ZPC) or pH_0).

The determination by summation CEC_e is an estimate based on content of exchangeable Ca²⁺, Mg²⁺ and K⁺, which may be evaluation of the proportion and nature of the available elements in soil. However, if soil pH is < 5.5, significant quantities of exchangeable A^{3+} may be present but not able to be accurately measured with used extractants, causing an underestimate of the CEC. To overcome this, exchangeable acidity must be included in CEC. If soil pH is > 7.5 or if the soil has been recently limed this method gives overestimated results. Either the method using ammonium acetate at pH 7 or a method using an unbuffered solution of ammonium chloride. In the methods of measurement of CEC at the soil pH, the soil is saturated with an index cation. The exchange is carried out at a pH near the soil pH. After elimination of excess index cation with a diluted solution of the same ion, this cation is moved from soil by another cation. The determination of the moved cation concentration in extract enables determination of the CEC. In one of these methods [13, 14] the exchangeable cations are extracted with a not-buffered barium chloride solution. The exchangeable cations (Al³⁺, Fe²⁺, Ca²⁺, Mg²⁺, Mn²⁺, K⁺ and Na⁺) can be determined in the extract. The soil residue is extracted with a magnesium sulfate solution. The Mg^{2+} ions move the exchanged Ba^{2+} ions and $BaSO_4$ precipitates in the medium maintained at an ionic strength near that of the soil solution. The difference between the added Mg^{2+} and the Mg^{2+} which remains in solution gives the CEC. Magnesium concentration is determined by atomic absorption spectrometry (AAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES). Alternative procedure uses of hexaamminecobalt(III) (Cohex) cation as saturation cation [1, 15, 16]. In this procedure the soil is saturated by the hexaamminecobalt(III) cation $Co(NH_3)_6^{3+}$. Exchangeable Al³⁺, Fe²⁺, Ca²⁺, Mg²⁺, Mn²⁺, K⁺ and Na⁺ cations are determined directly in extract. The CEC is calculated by the difference between the initial quantity and the quantity remaining in solution. The calculated adsorbed Cohex ions correspond to the soil CEC in not-buffered medium at soil pH. Cobalt concentration is determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). The determination of CEC of the

soil at constant pH is used with mono- or divalent cations at different pH. In buffered medium, variations due to the soil pH are eliminated, but if the buffered pH is higher than the soil pH, they are likely to create negative charges on clay minerals and organic matter by dissociation of weak acid groups. The results are then overestimated, particularly in acid soils with variable charge. The most commonly used methods are: the ammonium acetate method at pH 7.0 [9, 17, 18] and sodium acetate method at pH 8.2 [4, 19]. In ammonium acetate method, the soil is saturated by the ammonium ion in a buffered medium at pH 7.0. The content of exchangeable cations is determined in extract by flame photometry, atomic absorption or ICP spectrometry. The excess of ammonium ion is eliminated by a solvent (ethanol) and subsequently the ammonium ion is moved by the potassium or sodium ion. To measure the CEC ammonium can be titrated by distillation and volumetric analysis or automated spectrocolorimetry. In sodium acetate method, sodium is saturated cation and is

moved by ammonium cation. The CEC is calculated on the basis of concentration of sodium ion in extract. In order to estimate the CEC is also proposed the use of instrumental methods [20], artificial neural network [21] and geostatistical methods [22].

The object of this study was to compare the results obtained with four methods of determination of cation exchange capacity (CEC) and sum of exchangeable cations (Ca, Mg, K) in soils. Two of these methods are analytical procedures published by the International Organization for Standardization: ISO 23470:2007(E) [15] and ISO 11260:1994(E) [14] methods. The one method is proposed by US Environmental Protection Agency: US EPA 9081 Method [19] and last is traditional Kappen's method [23].

Materials and methods

Thirty samples from different genetic horizons (ochric, luvic, argillic, cambic and horizons of parent rock) of the five soil profiles (four Stagnic Luvisols, one Eutric Cambisol), located on Siedlce Upland, were studied. Soil samples were air-dried and passed through a 2 mm sieve before being analyzed. Basic physicochemical properties of the soils were determined: granulometric composition by aerometric method; pH by potentiometric method; total carbon content on the CHNS/O 2400 autoanalyzer (Perkin Elmer, Waltham, USA); organic carbon content by oxidation-titration method [8]. Inorganic carbon content was calculated on the basis of the difference between the total carbon and organic carbon content. These properties are listed in Table 1.

Table	1
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Property	Clay	рН _{КС1}	pH _{H2O}	Ctotal	Corg	Cinorg
Unit	[%]	-	-	$[g kg^{-1}]$	$[g kg^{-1}]$	$[g kg^{-1}]$
Mean	20.3	-	-	24.3	2.45	21.8
Range	2-40	4.11-7.75	5.21	0.86-101.3	0.58-9.28	0-100
Std. Dev.	9.97	-	-	37.3	2.39	37.9

Basic physicochemical properties of the soils used in the studies

The hexaamminecobalt(III) chloride method

The sample of air-dried soil (2.5, 5 or 10 g; depending on the expected CEC) was weighted and transferred to a tightly stoppered polyethylene centrifuge tube of about 50 cm³ capacity. Then 50 cm³ of 0.0166 mol dm⁻³ hexaamminecobalt(III) chloride solution was added to the soil and was shaken on the rotary shaker for 1 h. After this time the tubes were balanced and centrifuged at 3000 g for 10 min. The cobalt concentration and exchangeable

cation contents (Al³⁺, Fe²⁺, Ca²⁺, Mg²⁺, Mn²⁺, K⁺ and Na⁺) in extract were determined by ICP-AES on Optima 3200RL (Perkin Elmer, Waltham, USA) spectrometer. The working parameters of the apparatus were: RF power - 1300 W, plasma argon flow rate - 15 dm³ min⁻¹, auxiliary argon flow rate - 0.5 dm³ min⁻¹, nebuliser argon flow rate - 0.8 dm³ min⁻¹, sample flow rate - 1.5 cm³ min⁻¹, integration time - 10 s. The analytical wavelengths were: K - 766.490 nm; Ca - 317.933 nm; Mg - 285.213 nm; Na - 330.237 nm; Fe - 238.204 nm; Al - 394.401 nm; Mn - 257.610 nm; Co - 228.616 nm. The CEC was calculated on the basis of the difference between initial concentration of cobalt in extraction solution and the quantity remaining in extract.

The barium chloride method

The 2.5 g of sample of air-dried soil was weighted and transferred to a tightly stoppered polyethylene centrifuge tube of about 50 cm³ capacity. Then 30 cm³ of 0.1 mol dm⁻³ barium chloride solution was added to the soil. Extraction was carried out by 1 h on the rotary shaker. The solid and liquid phases were separated by centrifugation at 3000 g for 10 min. This operation was repeated twice and three supernatants were transferred to a 100 cm³ volumetric flask each time. The volumetric flask was filled up to the mark with barium chloride. The exchangeable cation contents (Al³⁺, Fe²⁺, Ca²⁺, Mg²⁺, Mn²⁺, K⁺ and Na⁺) were determined in the extract by ICP-AES (see The hexaamminecobalt(III) chloride method). After equilibrating the soil with 30 cm³ of 0.0025 mol dm⁻³ barium chloride solution, the solid phase was extracted overnight with 30 cm³ of 0.02 mol dm⁻³ magnesium sulfate. The magnesium concentration in extract was determined by ICP-AES. This concentration was subtracted from initial magnesium concentration (about 480 mg dm⁻³) and the difference gave the CEC value.

The sodium acetate method

The 4 g of medium- or fine-textured soil or 6 g of coarse-textured of air-dried soil was weighted and transferred to a tightly stoppered polyethylene centrifuge tube of about 50 cm³ capacity. Then 33 cm³ of 1 mol dm⁻³ sodium acetate solution was added to the soil and suspension was shaken in a mechanical shaker for 5 min. The solid and liquid phases were separated by centrifugation at 3000 g for 10 min, until the supernatant liquid is clear, and liquid was decanted. This operation was repeated three more times. Then 33 cm³ of 99% isopropyl alcohol was added and suspension was shaken in a mechanical shaker for 5 min, and was centrifuged at 3000 g for 10 min. This procedure was repeated two more times. In the next stage 33 cm³ of 1 mol dm⁻³ anmonium acetate solution was added to the soil and suspension was shaken in a mechanical shaker for 5 min, and was centrifuged at 3000 g for 10 min. This procedure was repeated two more times and the combined supernatants were diluted to the 100-cm³ mark with ammonium acetate solution. The solium concentration in extract was determined by ICP-AES. The CEC was calculated on the basis of the difference between initial concentration of sodium in extraction solution and the quantity remaining in extract.

The Kappen's method

The hydrolytic acidity values and the sum of exchangeable alkaline cations were determined according to Kappen [7].

The hydrolytic acidity

The 40 g of air-dried soil sample was treated with 100 cm³ of 0.5 mol dm⁻³ calcium acetate solution adjusted to pH = 8.2 (in the ratio w:v of 1:2.5). The suspension was shaken in polyethylene bottle of about 250 cm³ capacity, at rotary shaker (40 rpm), at room temperature for one hour, then filtrated. The filtrates were titrated with 0.1 mol dm⁻³ sodium hydroxide solution in presence of phenolphthalein indicator and the hydrolytic acidity values were calculated from the amount of sodium hydroxide solution consumed.

The sum of exchangeable alkaline cations

The 20 g of air-dried soil sample was treated with 100 cm³ of 0.1 mol dm⁻³ hydrochloric acid solution (in the ratio w:v of 1:5). The suspension was shaken in polyethylene bottle of about 250 cm³ capacity, at rotary shaker (40 rpm), at room temperature for one hour, then filtrated. The filtrates were titrated with 0.1 mol dm⁻³ sodium hydroxide solution in presence of phenolphthalein indicator and the sum of exchangeable alkaline cations were calculated from the amount of sodium hydroxide solution used.

A comparison between methods

The linear regression analysis (linear least square LLS) was used to compare the results of methods and to find the equation, which improve the relation between hexaammine(III) chloride and sodium acetate methods. Calculations were done using Statistica 9.0 PL software (Statsoft, Tulsa, USA).

Results and discussion

The cation exchange capacity (CEC) of soil obtained by hexaamminecobalt(III) method ranged from 19 to 215 mmol(+) kg⁻¹ (mean: 109 mmol(+) kg⁻¹; standard deviation: 53 mmol(+) kg⁻¹). The values of sum of exchangeable cations (S) were close to CEC (Fig. 1), and varied from 16 to 223 mmol(+) kg⁻¹ (mean: 105 mmol(+) kg⁻¹; standard deviation: 54 mmol(+) kg⁻¹). The exchangeable alkaline cations content can be arranged in series of decreasing values: Ca>>Mg>K. The content of exchangeable calcium ranged from 56 to 96% of S. Exchangeable magnesium and potassium accounted for 6 to 32% of S and 0.3 to 12% of S respectively. The cation exchange capacity (CEC) was significantly correlated with clay content (Table 2). It is worth noting that CEC was negatively correlated with organic carbon content. Reason for this is that the rich in organic matter ochric horizons contained relatively small amount of clay.

Table 2

The values of correlation	coefficients between	cation exchange	capacity (CEC) of soi	ls obtained with
hexaamminecobalt(II	I) chloride (CEC _{Cohex}) and basic physi	cochemical properties	s of the soils

	CEC _{Cohex}	Clay [%]	pH _{KCl}	pH _{H2O}	Corg	Cinorg
CEC _{Cohex}	1					
Clay [%]	0.91**	1				
pH _{KCl}	0.31	0.31	1			
pH _{H2O}	0.39	0.38	0.97**	1		
Corg	-0.45*	-0.54**	-0.34	-0.45*	1	
Cinorg	0.00	0.15	0.75**	0.71**	-0.27	1

* significant at 0.05 probability level, ** significant at 0.01 probability level



Fig. 1. The relationship between cation exchange capacity (CEC_{Cohex}) and sum of exchangeable cations obtained with hexaamminecobalt(III) chloride (S_{Cohex})

A comparison of the hexaamminecobalt(III) chloride and barium chloride methods

The results of cation exchange capacity of soils determination obtained by hexaamminecobalt(III) chloride and barium chloride methods were comparable, which was confirmed by linear regression equation (the slope of the least squares regression line equal to 1) and high value of determination coefficient ($R^2 = 0.97$) (Fig. 2).



Fig. 2. Relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and barium chloride (CEC_{BaCl2})

Both reagents extract similar amounts of exchangeable cations (Fig. 3). Only in the case of genetic horizons with high calcium carbonate content, barium chloride extracts

larger amounts of these cations. This observation can be explained by the dissolution of larger amount of this salt during the extraction procedure. This is because the hexaamminecobalt(III) method is a single extraction and barium chloride procedure includes three successive extractions with larger volume of used reagent. This hypothesis is confirmed by comparison of results of exchangeable calcium content obtained by two methods, which are similar, in contrast to magnesium and potassium content (Fig. 4).



Fig. 3. Relationship between sum of exchangeable cations (S) obtained with hexaamminecobalt(III) chloride (S_{Cohex}) and barium chloride (S_{BaCl2}); • $C_{inorg} > 80$ g kg⁻¹, $\circ C_{inorg} < 20$ g kg⁻¹





Fig. 4. Relationship between exchangeable calcium, magnesium and potassium obtained with hexaamminecobalt(III) chloride (Ca_{Cohex}, Mg_{Cohex}, K_{Cohex}) and barium chloride (Ca_{BaCl2}, Mg_{BaCl2}, K_{BaCl2}); • C_{inorg} > 80 g kg⁻¹, \circ C_{inorg} < 20 g kg⁻¹

A comparison of the hexaamminecobalt(III) chloride and sodium acetate methods

The values of CEC obtained by hexaamminecobalt(III) chloride and sodium acetate methods were less comparable than hexaamminecobalt(III) chloride and barium chloride methods ($R^2 = 0.69$) (Fig. 5).



Fig. 5. Relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and sodium acetate (CEC_{CH3COONa});
● C_{inorg} > 5 g kg⁻¹, ○ C_{inorg} < 5 g kg⁻¹



Fig. 6. Relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and sodium acetate predicted by linear regression model (CEC_{CH3COONa} predicted)

Generally, the first method gave higher values of CEC (the slope of the least squares regression line less than 1) with reference to the all soil horizons studied. This is because the hexaamminecobalt(III) ions have a higher capability of exchangeable cations exchange than the sodium ions. An inverse relationship was found for acidic soil horizons acidic with relatively high organic matter content. Reason for this is the fact that buffering capacity of sodium acetate solution (pH = 8.2) influences on the pH of the suspensions and consequently on variable charges of soil. In the case of soil horizons with low pH and high

organic carbon content (C_{org}) increases the negatively charged sites of organic matter and consequently cation exchange capacity. The relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and sodium acetate (CEC_{CH3COONa}) can be improved by linear regression equation with three factors: percent of clay (clay %), pH in potassium chloride solution (pH_{KCl}) and organic carbon content expressed in g kg⁻¹ (C_{org}). To calculate the CEC according to hexaamminecobalt(III) chloride method (CEC_{Cohex}) from the CEC given by sodium acetate method (CEC_{CH3COONa}) this equation is:

$$CEC_{CH3COONa} = CEC_{Cohex} - 1.72 clay\% + 3.05 pH_{KCl} + 3.09C_{org}$$
 ($R^2 = 0.90$)

The value of determination coefficient is 0.90 and is much higher than if the data are not corrected. The relationships between CEC_{Cohex} and sodium acetate predicted by linear regression model ($CEC_{CH3COONa}$ predicted) is presented in Figure 6.

A comparison of the hexaamminecobalt(III) chloride and sodium acetate methods

The hexaamminecobalt(III) chloride and Kappen's methods are not equivalent with reference to the all soil horizons studied. The CEC values obtained by these methods weren't correlated (Fig. 7).



Fig. 7. Relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and Kappen's methods (T): a) with b) without calcareous horizons; \bullet C_{inorg} > 80 g kg⁻¹, \circ C_{inorg} < 20 g kg⁻¹

The Kappen's method is not suitable for soil horizons with high carbonate or others acid soluble salts content, because in their case is obtained extremely overestimated values of CEC. After elimination of calcareous horizons from analyzed data correlation coefficient value was improved, but the CEC values were not well related ($R^2 = 0.14$). The differences were due the exchange ability and the quantity of index cation used, experimental conditions and relatively large analytical error in Kappen's method.

Conclusions

1. The cation exchange capacity (CEC) values and quantity of exchangeable cations (S) extracted depend on the precise experimental conditions such as physicochemical properties of soils studied, the exchange ability and quantity of index cation used,

extraction conditions (number and time of extraction steps, pH at which extractions are carried out) and analytical method used to determine the CEC.

- 2. Among the methods used in this work, the hexaamminecobalt(III) chloride and barium chloride methods are comparable. In both of these methods measurements of CEC carried out in not-buffered medium at soil pH and obtained values are more correspond to the actual sorption capacity of soils.
- 3. The barium chloride method gives higher values of sum of exchangeable cations than hexaamminecobalt(III) chloride method in the case of genetic horizons with high calcium carbonate content. This is due the dissolution of larger amount of this salt during successive extractions.
- 4. The values of CEC obtained by hexaamminecobalt(III) chloride and sodium acetate methods are less comparable than hexaamminecobalt(III) chloride and barium chloride methods. The first method gave higher values of CEC, but inverse relationship is valid for acidic soil horizons acidic with relatively high organic matter content. The relationship between cation exchange capacity (CEC) of soils obtained with hexaamminecobalt(III) chloride (CEC_{Cohex}) and sodium acetate (CEC_{CH3COONa}) can be improved by linear regression equation with three factors: percent of clay; pH in potassium chloride solution and organic carbon content.
- 5. The hexaamminecobalt(III) chloride and Kappen's methods are not equivalent. The Kappen's method is not suitable for soil horizons with high carbonate or others acid soluble salts content, because in their case is obtained extremely overestimated values of CEC.

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PORÓWNANIE METOD OZNACZANIA POJEMNOŚCI WYMIANY KATIONÓW I SUMY KATIONÓW WYMIENNYCH W GLEBACH

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Abstrakt: Celem niniejszej pracy było porównanie wyników oznaczania czterema niezależnymi metodami pojemności wymiany kationów (CEC) i ilości zasadowych kationów wymiennych (Ca, Mg, K) w glebach. W badaniach zastosowano tradycyjną zmodyfikowaną metodę Kappena oraz trzy standardowe metody, w których wykorzystuje się różne odczynniki ekstrakcyjne: octan amonu (pH = 7), chlorek baru i chlorek heksaaminokobaltu(III). Wartości pojemności wymiany kationów, uzyskane z zastosowaniem jonów baru (Ba²⁺) i jonów heksaaminokobaltu(III) (Co(NH₃)₆³⁺), są powtarzalne i porównywalne; te dwie metody można uznać za ekwiwalentne. Zmodyfikowana metoda Kappena daje zawyżone wyniki, szczególnie dla gleb kwaśnych, zasobnych w materię organiczną, oraz gleb zasadowych, zawizone wyniki, szczególnie dla gleb kwaśnych, zasobnych miejsc zdolnych do sorpcji, szczególnie powstałych w wyniku dysocjacji kwaśnych grup materii organicznej, wskutek zwiększenia wartości pH do pH odczynnika ekstrakcyjnego. Powoduje to zawyżenie pojemności wymiany kationów głównie dla gleb kwaśnych. Błąd ten można zredukować poprzez równanie regresji uwzględniające pH gleby i zawartość węgla w związkach organicznych.

Słowa kluczowe: pojemność wymiany kationów, kationy wymienne, jony baru, jony heksaaminokobaltu(III)