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## Cation Exchange Capacity (CEC) determination from spectroscopy

P. C. KARIUKI\*

Division of Geological Survey, International Institute for Aerospace Survey and Earth Sciences (ITC), PO Box 6, 7500AA Enschede, The Netherlands;  
e-mail: kariuki@itc.nl

F. VAN DER MEER

International Institute for Aerospace Survey and Earth Sciences (ITC),  
PO Box 6, 7500AA Enschede, The Netherlands; e-mail: vdmeer@itc.nl, and  
Delft University of Technology. Faculty of Civil Engineering and Geosciences,  
Department of Applied Earth Sciences, Mijnbouwstraat 120, 2600 GA Delft,  
The Netherlands; e-mail: f.d.vandermeer@citg.tudelft.nl

and P. N. W. VERHOEF

Delft University of Technology, Faculty of Mining and Petroleum Engineering,  
Department of Applied Earth Sciences, Mijnbouwstraat 120, 2600 GA Delft,  
The Netherlands; e-mail: P.N.W.Verhoef@citg.tudelft.nl

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**Abstract.** Swelling soils are a major engineering problem and their detection is one of the most important site investigation undertakings in the construction industry. Methods of their detection have been developed over the years, most of which utilize the soil physical properties as the indices of their identification. Some of these have proved quite expensive and time-consuming. In this research an alternative, relatively faster and cheaper method is explored. The method involves the use of Methylene Blue Absorption (MBA) as the engineering index to estimate the Cation Exchange Capacity (CEC) and spectral absorption feature characteristics as the mineral identification method. The relationship between the MBA and the characteristics of the absorption features is used to explore the possibility of estimating CEC from spectral data.

### 1. Introduction

In civil engineering works, the swelling potential of soils ranks high among the engineering properties requiring attention. This is due to the great cost resulting from the damages caused to buildings where the soils are of high swelling potential, a hazard that has been described as second to none in Canada and the USA (Nelson and Miller 1992). The swelling potential depends on the composite effects of several interacting factors, among which are type and amount of clay particles present, type of adsorbed cation, clay particle size and organic matter content. The type and

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\*Corresponding author.

amount of clay mineral is the major contributing factor where the order of increase in influence is smectite > illite > kaolinite. Their identification and quantification is therefore critical in areas earmarked for development. This is traditionally done by use of indirect indicative indices such as Cation Exchange Capacity (CEC), Atterberg limits and colloid content tests (Mitchell 1993). These methods are, however, time-consuming and at times expensive. Spectroscopy offers a faster method for their identification.

Spectral information found in the shortwave-infrared (SWIR) range has been identified as a diagnostic tool for clay minerals (Chabrilat *et al.* 1997, Clark 1999). Dalal and Henry (1986), Ben-Dor and Banin (1990) and Ben-Dor *et al.* (1991) have all shown that it can also be used to predict some soil properties, among which are clay content, clay type and specific surface area. In this letter, an attempt is made to qualify presence of the three clay minerals based on recognition of their unique spectral characteristics and in turn use these characteristics to estimate the CEC. The extent by which the spectral data estimate the CEC is taken to show its potential to predict the swelling potential of engineering soils.

### 1.1. Study area

The study area (figure 1) is bounded by latitudes 1°00' to 1°30'S and longitudes 36°30' to 37°30'E. Temperatures range between 14–28°C and the rainfall is moderate and fairly uniform throughout the year at an annual average of between 762–1000 mm. This, coupled with weathering of mainly volcanic rocks and good drainage, has resulted in development of soils rich in kaolinite to the north-west and south-east of the study area. Poor drainage conditions prevailing in the middle of the area have resulted in black cotton soils (vertisols) high in smectite contents.

## 2. Materials and methods

A total of 47 samples collected at depths of 30 cm and representing a wide range of the soil types were used in the study. Their preparation for CEC estimation was as described by Reeuwijk (1995). Split samples were used for spectral data acquisitions.

### 2.1. CEC estimation [the Methylene Blue Absorption (MBA) test]

The MBA test is a method based on the principle of replacing cations at the external and internal structure of the clay mineral by an organic molecule. It is used to estimate the CEC of the clay minerals present in a soil. The amount of methylene blue adsorbed is exactly equal to the sum of the amounts adsorbed by each of the clay minerals. It is therefore an additive process representing the clay type ratios.

The procedure followed consisted of weighing 2 g of soils into Erlenmeyer flasks followed by addition of 30 ml of distilled water. The mixture was made uniform by thorough shaking, after which a simplified titration on the suspensions was carried out (for details see Verhoef 1992) to obtain the total amount of methylene blue required to complete the cation replacement.

Maximum absorption of methylene blue corresponded to complete exchange of the inorganic by the organic ions and was therefore used to estimate the CEC. Thus,

$$M_f(\text{CEC}) = (100 \times N \times p) / A [\text{meq}/100 \text{ g}] \quad (1)$$

where meq = milli-equivalents;  $p$  = amount of methylene blue adsorbed (ml);  $A$  = weight of soil used;  $N$  = normality of the methylene blue solution [meq/l] given

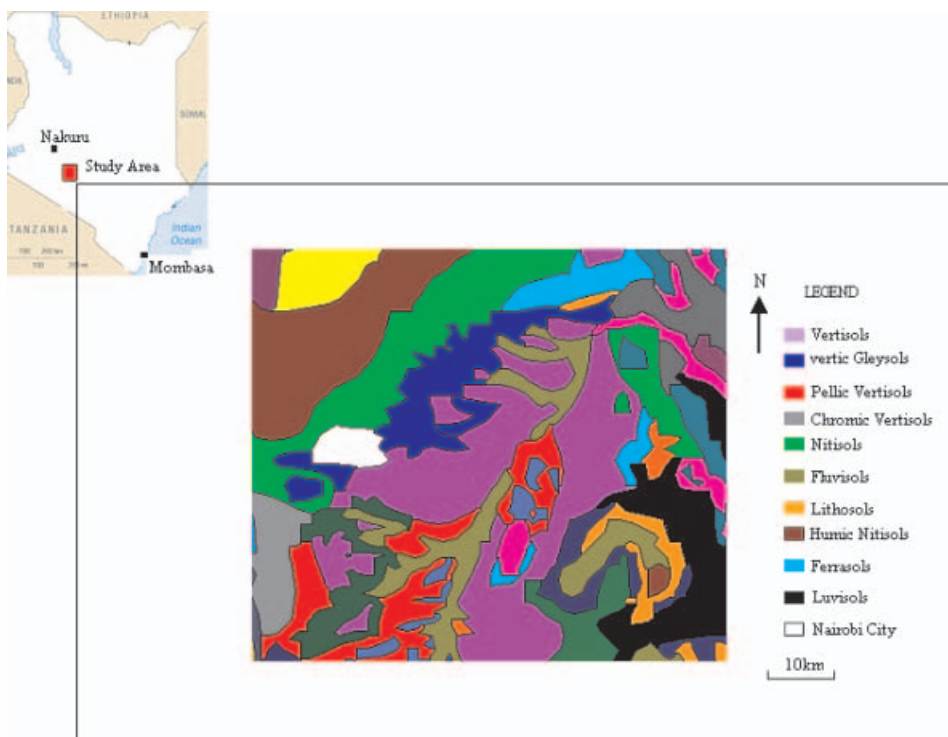


Figure 1. Soil Map of Study Area.

by  $(c \times 1000)/319.9 \times (100 - 12.34)/100$  [meq/l];  $c$  = concentration of methylene blue solution (g/l); and  $M_f$  = amount of methylene blue adsorbed per 100 g clay [meq/100 g].

The  $M_f$  value was then converted to soil activity by division with percentage clay in the soil. Table 1 gives the activity classes and the corresponding swell potential. Used soils were observed to represent all classes.

## 2.2. Spectroscopy

### 2.2.1. Spectral data acquisition

Spectra of oven-dried (at 105°C) split samples of the soils were measured using the Portable Infrared Mineral Analyser (PIMA) spectrometer. The spectrometer has an instantaneous field-of-view (IFOV) of approximately 2 mm × 10 mm, an average spectral resolution of 7–10 nm and sampling interval of 2 nm covering the spectral range 1300–2500 nm.

Table 1. Classification of soil based on the MBA test.

Class	MBA (g/100 g)	$M_f$ (CEC)(meq/100 g)	Swell potential
Inactive	<1	<2	None
Slightly active	1–2.5	2–5	Low
Normal soil	2.5–6	5–12	Moderate
Active soils	>6	>12	High

### 2.2.2. Spectral analysis

A feature extraction algorithm was used to obtain the absorption features in the spectra. The feature extraction involves a second differential analysis to find the absorption minima, followed by a quadratic interpolation that finds the exact wavelengths of the feature minima.

Absorption feature mapping as described by Mustard and Sunshine (1999) was then used for the interpretations where differences in the parameters of the absorption features, i.e. position, asymmetry, area and full width half maximum (FWHM), were used to group the samples into classes based on the interpreted clay mineral abundance.

## 3. Results

### 3.1. Feature mapping

The characteristics of the absorption feature parameters were used to differentiate between the soil samples.

#### 3.1.1. Feature position

The presence or absence of four absorption feature positions, i.e. 1400 nm, 1900 nm, 2200 nm and 2380 nm, provided important understanding of the differences between the soils in terms of clay mineralogy.

The feature at 1400 nm separated the soils into two groups based on shape and sharpness where kaolinitic samples gave deep and sharp features with a shoulder to the left, while the smectitic group had the feature deep and sharp but with the shoulder to the right (figure 2). The shape and precise location of the 1900 nm feature also varied and in smectitic samples was sharp with a shift towards 1908 nm, whereas in kaolinitic samples it was broad with a shift to values near 1915 nm. The position at 2200 nm had the kaolinitic group showing the characteristic doublet but mainly as a shoulder to the left of the absorption minima whereas the smectitic group had the feature shallow and broad. The feature at 2384 nm was found to be present only in kaolinitic samples.

#### 3.1.2. Asymmetry

Asymmetry is associated with order in the crystal structure (Hauff 2000). In the method used, it is the area to the left of the absorption minima divided by that to the right. At the 1400 nm and 2200 nm feature positions it was found to show differences between the smectitic and kaolinitic samples. Where kaolinite was abundant the asymmetry was to the left resulting in higher values, whereas in high smectite presence it was found to be to the right resulting in low asymmetry values.

#### 3.1.3. Depths

The order in which the depths at 1400 nm, 1900 nm and 2200 nm increased was found to differ, being  $2200 > 1400 > 1900$  nm for kaolinitic and  $1900 > 1400 > 2200$  nm for smectitic samples. This was attributed to strengthening of the lattice hydroxyl features at 1400 nm and 2200 nm in kaolinitic, and 1400 nm and 1900 nm by free water OH in smectitic samples.

### 3.2. Spectra as an indicator of soil activity

The results from both the absorption feature mapping and soil activity estimation were analysed statistically to obtain relationships between the surface charge of the clay minerals and their identifying parameters in absorption. Several significant relationships at 95% confidence level were obtained.

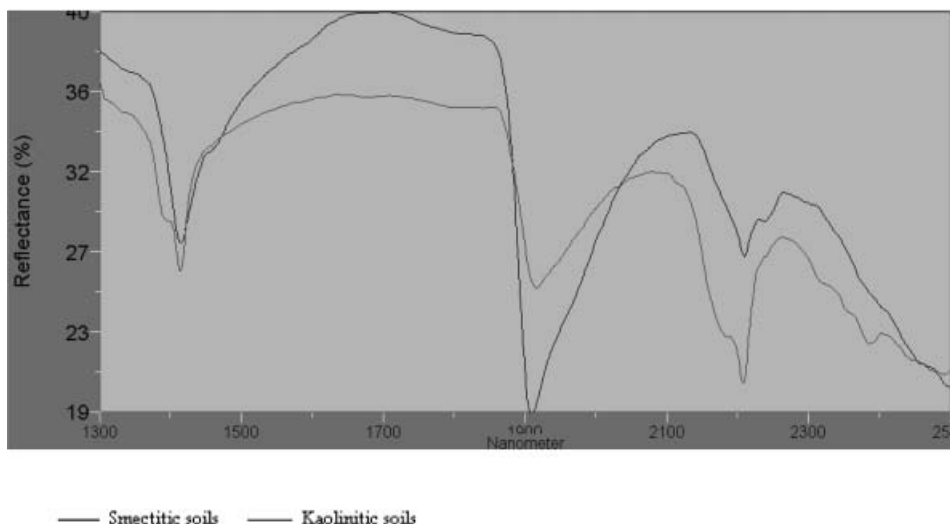


Figure 2. Spectra of soil samples rich in smectite and kaolinite showing characteristic differences used in the interpretation.

Asymmetry at the 1400 nm (figure 3) and 2200 nm (figure 4) feature positions gave exponential and linear negative coefficients respectively whereas the position at 1900 nm gave a negative and its depth positive linear correlations with the soil activity.

All the established significant relationships can be explained based on the physical properties of the soils in the presence of the two clay minerals in terms of their available surface area and are addressed in the discussion.

#### 4. Discussion

Smectite's small size and high levels of substitution lead to smectitic soils acquiring relatively high specific surface areas and charge, thus a high capacity to absorb water molecules (Ben-Dor *et al.* 1999). Kaolinite, on the other hand, gives kaolinitic soils

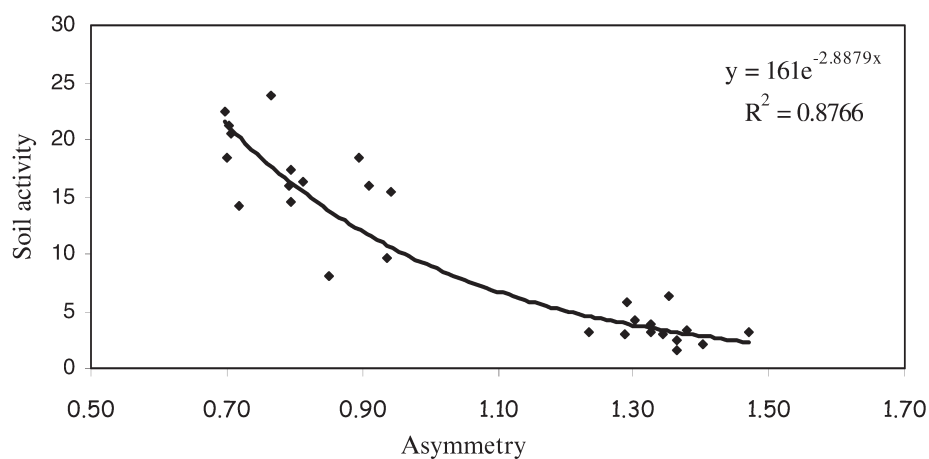


Figure 3. Decrease in asymmetry with increase in soil activity at 1400 nm.

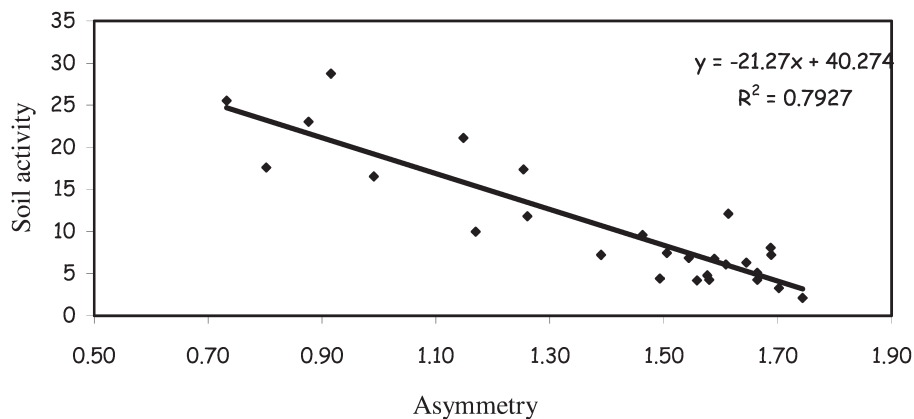


Figure 4. Decrease in asymmetry with increase in soil activity at 2200 nm.

smaller specific surface areas and little if any surface charge due to their larger particle size and lack of substitution. These properties, as observed in the results, are reflected in both activity and the spectral characteristics of these soils.

The present interlayer water in the smectitic soils gave them strong molecular water bands (1400 nm and 1900 nm), reflecting the large specific surface areas covered by this water. Kaolinitic soils, on the other hand, emphasized the lattice OH at 1400, 2200 and 2380 nm and weakened the 1900 nm feature, due to the higher fraction of the octahedral (Aluminium–OH) group, i.e. 1:1 (octahedral:tetrahedral), and small surface area. This is reflected by the intensity of these features in the presence of the two minerals and the resulting activity.

The decreasing order in structure at the multiple site fillings of hydroxyl (evidenced by the strength of the doublets at 1400 and 2200 nm), with increasing surface area is reflected by the negative correlation between asymmetries at the two positions and soil activity. The linear relationship at 2200 nm confirms linearity between molecular mixtures (clay mineral and interlayer water) from kaolinitic to smectitic soils while the exponential relationship at 1400 nm shows complexity of the feature due to its representation of both the free water OH and lattice OH.

Shifts associated with absorbed water within a mineral (Hauff 2000) are reflected by the wavelength position at 1900 nm negative correlation with soil activity. It reflects an increase in wavelength with decreasing influence of electrostatic forces acting on the water.

Presence of the feature at 2380 nm, diagnostic for kaolinite and usually described as subtle (Crowley and Vergo 1988) underscores the capability of spectroscopy as a strong tool for specific mineral identification even in complex mixtures such as soil.

## 5. Conclusions

Estimation of soil CEC has been shown to be possible through the use of absorption feature mapping in the spectral range 1300–2500 nm and the standardized CEC (soil activity). The characteristics of the absorption feature parameters found to possess significant relationships with the CEC can all be explained in physical terms where they represent the structural differences between clay minerals in the soils. Differences in structured and free water have also been established. The fact

that these structural differences are evident in a soil setting shows the applicability of spectroscopy to predict soil properties.

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