Smectite quantification in sediments and soils by thermogravimetric analyses

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Abstract

A new method is assayed, consisting in measuring the weight loss between 100 and 450 °C of samples solvated with ethylene glycol and previously saturated in Mg. This approach allows an approximation to be made of the dioctahedral Al-rich smectite content in samples. The proportion of expandable material in the sample is calculated according to the following equation, obtained from artificial mixtures prepared with variable proportions of smectite: \[ y = 3.96x - 4.05 \] \( (R^2 = 0.96) \), where \( x \) is the percentage of loss and \( y \) is the percentage of smectite in the sample. The slope of the equation agrees perfectly with the theoretical proportion of ethylene glycol in a solvated smectite, around 25%, and with the equivalent values determined on natural standards. In addition, the ordinate of the origin is also in agreement with the values of around 1% obtained in smectite-free samples. The method proposed is low-cost and easy to apply as a complement to X-ray diffraction determinations of expandable clay content in sediments and soils with the potential for landslides in civil engineering.

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1. Introduction

Smectite is the name for a group of clay minerals all of which display the startling property of being able to expand and contract their structures while maintaining crystallographic integrity (Moore and Reynolds, 1997). This property is the main control on the physical characteristics of natural materials in which smectite is present. Therefore, detecting the presence of smectite and determining its relative quantity in sediments and soils is very important in preventing many civil engineering problems. Engineering structures can suffer significant damage from seasonal shrinkage and swelling if not designed for the presence of smectite-rich soils (Nelson and Miller, 1997). Consequently, the geological setting of a construction site must always be described and characterized, which implies collecting and testing samples of rock and soils from the surface and below it.

In slow-moving landslides, one of the most important determining factors is the behaviour of high-plasticity soils submitted to high pore-water pressures. Smectite minerals are the main constituents of these soils and these minerals experience periodic swelling and shrinkage during alternating wet and dry seasons. Such cyclic swelling–shrinking movements of the ground are
critical to the stability of a natural slope specially with an increase in water content. Many authors have studied the effect and properties of smectite-dominated soils in landslides (Gibo et al., 1987 and Regea et al., 1997 in Japan; Meisina, 2004 in Italy; Yilmaz and Karacan, 2002 in Turkey, and others). In areas with a typical Mediterranean rainfall regime, these expansive soils are responsible for numerous slope instabilities that affect roads and highways (Yilmaz and Karacan, 2002).

Specifically, in SE Spain, smectite minerals were the main constituents of surface failure in two important landslides that produced a critical interruption of highways and roads (Ríogordo landslide and Diezma landslide; Chacón-Montero et al., 1988; Irigaray-Fernández and Chacón-Montero, 1991; Irigaray-Fernández et al., 1991; Azañón et al., in press).

Previous studies involving the role of smectite minerals in activating landslides have shown that these expansive-clay minerals greatly affect the strength performance of the soil (Meisina, 2004). Consequently, in slope stability studies, engineers need to know the smectite contents in order to predict how the material will perform. That is, the information on smectite proportions in potential landslide sites can be vital to the success of engineering projects. In fact, one of the properties used in engineering soil classification is the clay content (type and amount). Since the quantitative determination of minerals has always been a complex matter, the aforementioned research (Azañón et al., in press) carried out on SE Spain landslides has stimulated the search for a new, alternative method for the quantification of smectite in natural samples.

X-ray diffraction (XRD) methods have frequently been used for quantitative analyses, but many factors must be taken into account. For instance, factors related to the quantitative analyses of minerals such as orientation, crystallinity, homogeneity of the sample, effect of solid solution series, and calibration of the diffractometer are difficult to control. Other unsolved problems are peak interference and standard suitability. The most frequently used XRD methods for quantitative phase analyses are the Chung method (Chung, 1974a,b, 1975) and the Internal-Standard method (Snyder and Bish, 1989).

Although RockJock (Eberl, 2003) is one of the most powerful tools for quantitative analyses, this method unfortunately does not distinguish between dioctahedral micas and smectites, which is a fundamental distinction for the quantification of smectite in natural samples. In addition, Blum and Eberl (2004) described a method based on the uptake of polyvinylpyrrolidone (PVP) to measure the surface area of silicate minerals that can be used in combination with quantitative XRD to determine the abundance of smectitic minerals in a sample. Other sophisticated methods such as, for example, the Rietveld analysis are not easy to routinely apply to a large number of samples as it requires a complete determination of the crystal structure and chemical composition of all the major phases in the sample.

Therefore, after having tried the quantification of smectite in artificial mixtures by XRD techniques, we have explored the possibilities of thermogravimetric analyses, taking into account the facility of smectites to adsorb organic substances. The thermal behaviour of most smectites was established by Grim (1968) and reviewed by Earnst (1980, 1983). MacKenzie and Caillère (1975) emphasized that thermal methods play an important role in the qualitative and quantitative determination of clay minerals in soils. Nevertheless, attempts to quantify smectite proportions by thermal methods have failed until now due to the highly variable proportion of solvated water in smectites, which is affected by numerous factors. We propose the use of ethylene glycol instead of water for quantification. Ethylene glycol (EGC) treatment is one of the most widely used methods for identification of 2:1 clay minerals (McEwan and Wilson, 1980; Moore and Reynolds, 1997). As the emphasis of this paper is on the quantification of smectites in soils and sediments, we have focussed the study on dioctahedral Al-rich smectites, which are the major ones in such materials. The possible alternative presence of trioctahedral smectites can be easily checked and then measured by XRD methods (e.g., RockJock; see above).

2. Experimental

2.1. Samples

Eight samples have been prepared as mixtures in different known proportions (Table 1) of hydrothermal quartz, calcite, mica, and smectite.
In addition, to verify possible differences in swelling patterns related with the various compositions of dioctahedral Al-rich smectites, we have compared five standard smectite samples that cover a broad compositional range: SAz-1 (Ca-rich montmorillonite from Cheto Mine, Apache County, Arizona), SWy-2 (Na-rich montmorillonite from Crook County, Wyoming), SHCa-1 (hectorite from San Bernardino County, California), SCa-3 (montmorillonite “Otay” from San Diego County, California), and S-2 (K, Na-rich montmorillonite from Cabo de Gata, Spain). All of these samples are standards of the Clay Minerals Society Repository (see Chipera and Bish, 2001 and references therein for a complete description of samples) except for S-2 (Cuadros and Linares, 1996).

Examination of these samples by X-ray diffraction revealed certain impurities, as expected after the results of Chipera and Bish (2001). To minimize the effect of these impurities, the <1.4 μm fraction was extracted. A new collection of X-ray data showed that the impurities had been successfully reduced to a minimum, but were not completely absent. (The extraction was not successful for one of the initial standards, however, and it was therefore rejected).

In order to confirm the precision and applicability of the proposed method to natural samples representative of materials affected by landslide problems, two samples were selected from those known to have been affected by significant landslides that produced a critical interruption of highways and roads. Sample A-92 corresponds to a thin layer of sediments responsible for the Diezma landslide (Azañón et al., in press), which brought sediments and rocks onto the A-92 motorway (S Spain). Sample COL-09 is representative of the Ríogordo landslide (Irigaray-Fernández and Chacón-Montero, 1991), which displaced approximately 4,500,000 m³ of large carbonate blocks floating in smectite-rich clay a distance of several kilometres. The two samples were externally analysed in the E.R.M. laboratory (Poitiers, France) to determine their smectite proportions by a complex combination of methods including X-ray diffraction, chemical analysis and the Bernard calcimetric method to estimate carbonate concentrations and cationic exchange capacity (C.E.C.) according to Jackson’s (1964) methodology. Semi-quantitative estimation of mineralogical phases, previously identified, was made by a normative calculation based on chemical analyses of samples. This method is very similar to that of Calvert et al. (1989).

Sample G (Table 1) was also included in the analysis to check the quality of the results obtained in the E.R.M. laboratory, producing excellent results; the real composition of sample G is in the range provided by the laboratory for all four constituent minerals.

2.2. Quantification by X-ray diffraction

The Internal-Standard method has been applied in addition to the Chung method, the most common one for the majority of published results in geological literature, to the artificial mixtures. The Chung method (Chung, 1974a,b, 1975) is based on the determination of the Reference Intensity Ratios (RIR) of all the phases in the sample, which allows the intensity measurements to be normalized on the assumption that the sum of all phases in the sample is equal to 100%. The Internal-Standard method (Snyder and Bish, 1989) involves adding a known weight percent of a standard material, usually corundum, to the sample of unknown phase composition and subsequently measuring the intensities of both the sample and the reference phase.

2.3. Quantification by thermogravimetry

When a smectite sample is heated, the solvated H₂O is lost around 100–200 °C. Consequently, we initially explored the loss corresponding to the first dehydration reaction. However, there are two factors that strongly affect it: environmental humidity and the nature of the interlayer cation. According to Paterson and Swaffield (1987), the saturation of all samples with the same cation and equilibration at a fixed relative humidity are vital even in qualitative analyses.

Depending on humidity conditions, pure smectite (S-2) heated to 300 °C lost highly variable proportions, which ranged from 9.63 to 18.73% by weight.

To determine the effect of interlayer cations, we carried out a comparison of K as hydrophobic and Mg as hydrophilic cations. When the pure smectite was previously saturated in K using a KCl solution, weight loss was 7.04%; when the same treatment was carried out with MgCl₂, the loss was 13.07%.

The alternative use of the dehydroxilation reaction was ruled out due to the coincidence of its peak with that of mica.

2.3.1. Quantification by thermogravimetry of EGC solvated samples

Since H₂O is severely affected by a variety of factors, whereas EGC is able to normalize smectite behaviour and to facilitate its identification, we explored the possibility of quantifying smectite on the basis of EGC weight loss measured by thermal analyses.

Samples were twice saturated in Mg by dispersion of 1 g of solid in 1 M MgCl₂ solution. The solid was then washed with MgCl₂ solutions of decreasing concentration (0.1 and 0.01 M) and finally rinsed four times with Milli-Q water to eliminate any possible remains of MgCl₂.

The samples were dried and disaggregated before spreading them in Petri capsules. The preparations were then exposed to EGC vapours at 60 °C for 3 days to attain complete swelling and were finally analysed. It is recommended that the samples be analysed immediately after solvation to prevent any possible glycol loss. Notwithstanding, we repeated the determination some days later and the difference was minimal. Thermogravimetric analyses (TG) were carried out on ≈40 mg of sample in a Shimadzu TGA-50H at the Centro de Instrumentación Científica (C.I.C., University of Granada) in air at a heating time of 5 °C/min to 600 °C; however, in one of the samples (SAz-1) the heating experiment was maintained to 900 °C. The qualitative composition of gas loss during heating
was determined by means of a coupled Fourier transform infrared (FT-IR) spectrometer Nicolet Magna-IR 550 with a Nicolet TGA Interface.

3. Results

3.1. Quantification by X-ray diffraction

The results by the two methods are shown in Table 2. These data present a certain amount of error that is very evident for samples C, D, E, and G, for which the smectite quantities obtained by the Internal-Standard method are patently absurd.

3.2. Quantification by thermogravimetry of EGC solvated samples

3.2.1. Results obtained on selected dioctahedral-smectite standards

Table 3 presents the overall weight loss of the various standards between 100 and 450 °C. The losses were very similar for the different standards despite their different origins and chemical compositions. Some minor differences may be due in part to minor impurities in the standards, which were not completely avoided despite the use of the fine fraction. Some influence can also be expected from the different solid solutions present in Al-rich dioctahedral smectites, such as the initial nature of interlayer cations or octahedral compositions. Smectites deriving from Ca, for instance, seem to adsorb more EGC than those deriving from initial Na montmorillonite. Nevertheless, the differences remain quite small.

In order to determine the nature of the gas loss, their qualitative composition was determined by means of a coupled Fourier transform infrared (FT-IR) spectrometer (Fig. 1). The spectra obtained are very similar for the entire temperature range at which gas loss is significant. Fig. 1 shows the spectrum taken at 320 °C in which gas loss is at a maximum (Fig. 2). The spectrum contains peaks corresponding to water, carbon dioxide, and acetaldehyde as major phases, and a minor quantity of carbon monoxide. Fig. 2 shows the evolution of the gas loss as a function of temperature. With the exception of water, which has a small maximum around 100 °C, the maximum signal of all the gases is in the range of 250 to 420 °C. These data, together with differential thermogravimetric (DTG) and TG (Fig. 3) curves, allowed us to determine the optimum temperature range for quantifying the EGC weight loss.

According to the FT-IR results obtained (Fig. 1), EGC reacts with oxygen according to the reaction: 2OH–CH₂–CH₂–OH + 5O₂→4CO₂ + 6H₂O and undergoes dehydration at the same time: OH–CH₂–CH₂–OH→(CH₂═CH–OH ⇔ CH₃–CHO)+H₂O. The TG and DTG diagrams (Fig. 3) allowed us to quantify the loss of H₂O, CO₂, acetaldehyde (CH₃–CHO), and minor quantities of CO (Fig. 1) for a temperature range between 0–600 °C. The maximum loss of H₂O and CH₃–CHO was detected around 350 °C, whereas the maximum CO₂ loss was recorded around 380 °C (Fig. 2). Nevertheless, the loss of these gases from the samples actually occurs at lower temperatures, as is evident from the DTG and TG curves (Fig. 3); there exists some degree of hysteresis in the detection by FT-IR due to the time employed by the gas detector to trap them. The DTG curve (Fig. 3) shows two prominent maxima of weight loss, one at 194 °C and the other at 314 °C. The first peak is probably due to the evaporation of EGC, since the boiling point of EGC is 197 °C. The second maximum at 314 °C is probably due to the first step in the oxidation of organic matter.

Observe in Fig. 2 that H₂O, CO₂ and CH₃–CHO begin to be detected at 150 °C and that the greatest loss occurs before 300 °C, after which it tails off and is

Table 2
XRD results obtained by means of Internal-Standard and Chung methods for the artificial mixtures presented in Table 1

<table>
<thead>
<tr>
<th>Samples</th>
<th>Internal-Standard</th>
<th>Chung</th>
<th>Real proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>34</td>
<td>43</td>
<td>34</td>
</tr>
<tr>
<td>B</td>
<td>16</td>
<td>15</td>
<td>14</td>
</tr>
<tr>
<td>C</td>
<td>235</td>
<td>50</td>
<td>57</td>
</tr>
<tr>
<td>D</td>
<td>84</td>
<td>36</td>
<td>28</td>
</tr>
<tr>
<td>E</td>
<td>157</td>
<td>72</td>
<td>62</td>
</tr>
<tr>
<td>F</td>
<td>15</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>G</td>
<td>98</td>
<td>55</td>
<td>43</td>
</tr>
<tr>
<td>H</td>
<td>21</td>
<td>31</td>
<td>25</td>
</tr>
</tbody>
</table>

Table 3
Weight loss of smectite standards solvated with EGC between 100 and 450 °C

<table>
<thead>
<tr>
<th>Samples</th>
<th>Location</th>
<th>Smectite type</th>
<th>% loss</th>
<th>Corrected % lossa</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-2</td>
<td>Cabo de Gata</td>
<td>K, Na-rich montmorillonite</td>
<td>26.85</td>
<td>25.85</td>
</tr>
<tr>
<td>SAz-1</td>
<td>Cheto Mine, Apache County, AZ</td>
<td>Ca-rich montmorillonite</td>
<td>28.5</td>
<td>27.5</td>
</tr>
<tr>
<td>SWy-2</td>
<td>Crook County, WY</td>
<td>Na-rich montmorillonite</td>
<td>24.44</td>
<td>23.44</td>
</tr>
<tr>
<td>SHCa-1</td>
<td>San Bernardino County, CA</td>
<td>Hectorite</td>
<td>26.35</td>
<td>25.35</td>
</tr>
<tr>
<td>SSc-3</td>
<td>S. Diego County, CA</td>
<td>Montmorillonite</td>
<td>28.04</td>
<td>27.04</td>
</tr>
</tbody>
</table>

aAfter subtraction of EGC adsorbed on the surface according to the calculated equation (see Section 3.2.3).
practically complete by 450 °C. No other significant losses were detected up to 900 °C with the sole exception of water corresponding to dehydroxylation. Finally, the overall loss between 100 and 450 °C has been measured in TG curves (Fig. 3). Below 100 °C, the weight loss in thermal analyses corresponds to environmental H₂O. A minimum in the detection of water can be observed in the range between 100 °C and 150 °C. The upper thermal limit was chosen to be 450 °C as a compromise since, at this temperature, CO₂ loss is
nearly complete and other phyllosilicates such as kaolinite and micas begin to lose their hydroxyls.

3.2.2. Theoretical calculation of the ethylene glycol proportion in a Mg-saturated smectite

In addition to the thermogravimetric analyses on standard samples of smectite, the EGC proportion (wt. %) was theoretically calculated in two extreme cases of Mg-saturated smectites solvated with EGC: \((Si_{3.8}Al_{0.2})(Al_{1.5}Fe_{0.2}Mg_{0.3})Mg_{0.25}O_{10}(OH)_2\) and \(Si_4(Al_{1.8}Mg_{0.2})Mg_{0.1}O_{10}(OH)_2\), considering two molecules of EGC for each formula unit of smectite (McEwan and Wilson, 1980). The results were 25.05% and 25.51% of EGC by weight, respectively, which is in good accordance with the results obtained empirically (Table 3).

3.2.3. Application of the method to samples of known composition

In order to verify applicability and to determine error, this method has been applied to the eight artificial mixtures described above (Table 1). Table 4 shows the results of this experiment. The percentage of loss obtained experimentally has been represented vs. real smectite values (Fig. 4) to obtain the fitting equation 

\[ y = 3.96x - 4.05 \quad (R^2 = 0.96) \]

where \(x\) is the percentage of loss and \(y\) is the percentage of smectite. The results of applying this equation to our percentage of loss are in Table 4 (% determined column) and have allowed us to quantify the error of the method. The interpretation of the calculated equation is easier if it is expressed in another equivalent way,

\[ y = \left( x - 1.01 \right) / 0.25. \]

The subtraction of one unit from the percentage of loss \((x)\) is

**Table 4**

Smectite proportions obtained for artificial mixtures presented in Table 1 based on weight loss of samples solvated with EGC between 100 and 450 °C

<table>
<thead>
<tr>
<th>Samples</th>
<th>% loss ((x))</th>
<th>Determined %(^a)</th>
<th>Real % smectite</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>9.27</td>
<td>33</td>
<td>34</td>
<td>1</td>
</tr>
<tr>
<td>B</td>
<td>6.44</td>
<td>22</td>
<td>14</td>
<td>8</td>
</tr>
<tr>
<td>C</td>
<td>15.64</td>
<td>59</td>
<td>57</td>
<td>2</td>
</tr>
<tr>
<td>D</td>
<td>8.69</td>
<td>31</td>
<td>28</td>
<td>3</td>
</tr>
<tr>
<td>E</td>
<td>16.22</td>
<td>61</td>
<td>62</td>
<td>1</td>
</tr>
<tr>
<td>F</td>
<td>3.88</td>
<td>12</td>
<td>15</td>
<td>3</td>
</tr>
<tr>
<td>G</td>
<td>11.90</td>
<td>44</td>
<td>43</td>
<td>1</td>
</tr>
<tr>
<td>H</td>
<td>6.44</td>
<td>22</td>
<td>25</td>
<td>3</td>
</tr>
</tbody>
</table>

\(^a\) Values obtained from the application of the equation: \(y = 3.96x - 4.05\) (see Section 3.2.3 for explanation).
justified because there is a certain adsorption on the sample surfaces that does not contain smectite at all, as we have verified with a shale (Pb-4, Abad et al., 2001) and a pegmatitic mica. A correction for this adsorption is necessary so that only the EGC solvated by the interlayer cations of the smectites will be quantified. On the other hand, 0.25 represents the theoretical and empirically determined proportion of the loss by weight for 100%-smectite samples completely solvated in EGC (Table 3). Therefore, this equation is easy to interpret and coherent with the experimental data and theoretical calculations.

3.2.4. Application of the method to natural samples responsible for landslides

Table 5 presents the mineral composition of the two natural samples responsible for landslides determined by the E.R.M. laboratory and the smectite proportions determined by thermogravimetry of EGC solvated samples, as described in the present paper. A comparison of the results obtained by the two methods can be considered highly satisfactory. Our result for sample A-92 is clearly in the range provided by the E.R.M. laboratory and the smectite content obtained for sample COL-09 is only slightly lower than that provided by E.R.M. for the overall smectite-rich illite/smectite mixed-layer content of the sample.

### Table 5

<table>
<thead>
<tr>
<th>Samples</th>
<th>E.R.M. results</th>
<th>TGa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quartz</td>
<td>Mica</td>
</tr>
<tr>
<td>COL-09</td>
<td>9–14</td>
<td>4–9</td>
</tr>
</tbody>
</table>

*aValues obtained from the application of the equation: y=3.96x−4.05.

4. Discussion and summary

In this research, we propose a simple method to quantify smectites in natural samples. This method is based on thermogravimetric analyses as a complement to XRD methods, which provide excellent results for qualitative identification of smectites, but have severe limitations as regards quantifying smectite proportions in a sample. The most common XRD Internal-Standard and Chung quantitative methods have been applied to mixtures of known proportions (Table 1) and the results (Table 2) confirm that, when these methods are applied to samples with minerals of different sizes and morphologies, the findings are not very accurate. As Moore and Reynolds (1997) concluded, “Quantitative analyses of platy (clay) and nonplaty (nonclay) minerals in the same plate cannot be achieved by any of the XRD methods that have been discussed because these two classes of mineral morphologies cause them to have very different preferred orientations...”.

We propose an easy method that can be applied to large quantities of samples as a complement to verify traditional XRD results. When a smectite sample is exposed to EGC, H₂O is replaced by this organic substance. The EGC solvation normalizes smectite behaviour, which becomes independent of the humid environmental conditions and less dependent on the interlayer cation, factors which are vital even in qualitative analyses (Paterson and Swaffield, 1987). If samples are progressively heated, their weight loss should be proportional to the smectite content. As EGC is heavier than H₂O, the weight loss is greater and, as it is more closely linked to the structure, it occurs at higher temperatures (Fig. 3). Samples are previously homo-ionized with Mg, which is a hydrophilic cation, in order to normalize the EGC adsorption behaviour. The results in five pure smectite samples (Table 3) are very similar for a range between 100–450 °C and coherent with the theoretical value for a smectite completely solvated in EGC, which is a weight loss of around 25.2%. Therefore, smectite can be quantified in natural samples on the basis of ethylene-glycol weight loss measured by
thermal analyses by means of the equation \( y = 3.96x - 4.05 \) (\( R^2 = 0.96 \)), where \( x \) is the percentage of loss and \( y \) is the percentage of smectite in the sample. The application of the method to eight artificial mixtures prepared with various smectite proportions (Table 4) and to two externally analysed natural samples (Table 5) has produced coherent results within an acceptable margin of error.

In nature, there exist other expandable phases such as low-charge vermiculite or interstratified minerals containing smectite layers. Their influence in the weight loss is proportional to their swelling capacity. For example, an interstratified phyllosilicate having an \( X_s \) proportion of smectite layers will contribute to the final weight loss with \( X_s \) [interstratified quantity]. Since the importance of an expandable clay depends on the content in smectite layers (\( X_s \)), the contribution to the final result is acceptable and even desirable. To sum up, the method measures the total proportion of swelling layers in the samples, independently of their specific physical nature.

Natural materials are rarely homogeneous or continuous and this is an important factor to be taken into account during all the sampling and preparation processes, as the quantity of sample used in thermogravimetric measurements is very small (around 40 mg). Therefore, this could be a significant source of error and hence possible selective enrichment of the various minerals due to contrasting physical properties should be carefully avoided during the entire process.

The presence of expandable clays is a very significant problem in engineering projects such as the design of a railway or highway. The proposed technique to quantify smectites is easy to apply (it only requires a clay lab and a TG), of relatively low analysis and sampling cost, and provides an admissible quantification method of expandable clays that is independent of other mineral phases or amorphous materials and consequently does not accumulate errors related with the quantification of the varied set of materials usually present in soils and sediments.

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