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Liquid and plastic limits of clayey, organic C-rich mountain soils: role of organic matter and mineralogy.

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Abstract

The soil liquid (LL) and plastic limit (PL), and their difference PI (plasticity index) are physical properties related with the soil mechanical behavior, determined on the <0.425 mm soil fraction, and used in different fields of earth and soil sciences. They are known to depend on soil organic matter (SOM) and/or clay content, but these soil properties are generally determined on the <2 mm fraction (fine earth). Furthermore, these dependencies can be affected by many qualitative aspects, such as the specific surface area (SSA) and the equivalent basal spacing (EBS) of clay minerals, and clay aggregation. The aims of our research were therefore i) to investigate the role of clay and SOM contents in a set of clay- and organic C-rich mountain soils, considering both the <2 mm and <0.425 mm fractions; ii) to assess the effect of the mineralogical composition (type of minerals, EBS and SSA) and clay aggregation in the <0.425 mm fraction. The relationships observed between Atterberg limits and the clay and organic C amounts evidenced the importance of considering the <0.425 mm soil fraction instead of the fine earth. The relative role of clay and organic C contents in the <0.425 mm fraction was comparable for LL, while in the case of PL the most relevant variable was TOC. Therefore, in a first stage of increasing water content, the role of SOM is fundamental (i.e. in the semisolid state and plastic interval), then the interactions among clay particles dominate. No clear relationship was instead observed with the mineralogical composition, although SSA and EBS were both correlated positively with LL and PI. The aggregated clay amount was positively related with LL and PI, and EBS. This, together with the relationships with EBS and SSA, evidenced that a role of clay exists in determining the soil mechanical behavior, but it seems to be more related to the interactions among clay particles occurring in the soil, than to the simple mineralogical composition. The findings suggested that, while PL seems to be more controlled by the organic fraction, LL and PI are more influenced by SOM and clay minerals and their interactions.

Keywords: Atterberg, specific surface area, soil mineralogy, equivalent basal spacing, Ligurian Alps

List of abbreviations: LL (liquid limit), PL (plastic limit), PI (plasticity index), SOM (soil organic matter), MIF (mineral intensity factor), SSA (specific surface area), EBS (equivalent basal spacing), BS (base saturation) TC (total C), TOC (Total organic C), SEE (standard error of the estimate), VIF (variance inflactionary factor).

1. Introduction

In earth sciences, the plastic limit (PL) and liquid limit (LL) were defined by Atterberg (1911) as the soil gravimetric water contents (%) needed for the transition of the < 0.425 mm fraction from the solid to plastic state, and from the plastic to liquid state, respectively. The detailed methods description is provided by ASTM and BSI standard methods (BSI, 1990; ASTMD 4318–10e1, 2010), and by McBride (2002).

Atterberg limits are relevant for a wide range of purposes such as soil classification in engineering (Atterberg, 1911; Casagrande, 1932), soil mechanics (Haigh 2012; Haigh et al., 2013; Zbik et al., 2015), agricultural soil management and plant growth (Keller and Dexter, 2012; Abbaspour-Gilandeh and Sedghi, 2015, Zolfaghari et al., 2015a), and natural hazard assessment (Yalcin, 2007; Ramezanpour et al., 2010; Diaz-Hernandez et al., 2015), with a particular focus on sloping areas (Summa et al., 2010; Stanchi et al., 2013, 2015).

In agricultural and forest soils, LL and PL have been often related with soil organic matter (SOM) and clay contents (e.g. De La Rosa, 1979: De Jong et al., 1990; Mbagwu and Abeh, 1998; Schmitz et al., 2004). However, as pointed out by Blanco-Canqui et al. (2006) and Zolfaghari (2015b), the studies on Atterberg limits and related soil properties often gave contrasting results; dependencies were not always observed and, when present, they could be affected by a large number of additional characteristics such as SOM type, hence land use, and soil mineralogy. Moreover, soil databases normally report the chemical properties of the fine earth fraction (i.e. <2 mm), which is the standard fraction for analytical purposes (e.g. van Reeuwijk, 2002; Soil Survey Staff, 2014a).

Stanchi et al. (2015) found no correlation between LL, PL and the clay fraction content, while they observed a positive relationship with organic C on a dataset of mountain forest soils in the Aosta Valley (N-W Italy), particularly in SOM-enriched soil horizons such as A, Bs, and Bh. They therefore concluded that SOM was the dominating property controlling LL and PL in the mountain topsoils of the study area, and that its effect in deeper soil horizons was visible only when the organic C amount was above a certain threshold (around 20 g kg^{-1}). This predominance of SOM seems reasonable, considering that the clay fraction of the studied soils was relatively scarce (always less than 20%), as often observed for alpine soils and it showed a small inter-sample variation. Keller and Dexter (2012) found that LL and PL were well related with both SOM and texture in a dataset of agricultural topsoils (i.e. tilled layer) from EU and USA, where the correlation with clay was stronger for LL than PL, even if the reasons were not clear. In addition, they concluded that PL was not affected by clay contents lower than 35%, confirming that a linear correlation cannot always be foreseen.

Mineralogical characteristics may play some role in determining the soil mechanical properties. Depending on the charge density of exchangeable cations that provide strong hydrophilic sites for solvent sorption (Schoonheydt and Johnston, 2006), several water layers may be incorporated in the interlayer of phyllosilicates with low layer charge, such as smectites. When instead the layer charge is higher, as in illites,

or the interlayer is occupied by Mg-octahedral sheets or stable Al polymers, as in lithogenic or pedogenic chlorites (Tolpeshta et al., 2010), the amount of water that can be sorbed is much lower. As layer charge affects the interlayer distance, an index that may help understanding the effects of mineralogy on soil mechanical properties is the equivalent basal spacing (EBS), a global evaluation of the interlayer distance suggested by Schmitz et al. (2004). Badia et al. (2015) studied the relationships between Atterberg limits and soil mineralogy in illite-rich soils using either semi-quantitative mineralogical analysis or EBS, and found that the least plastic soils had abundant quartz and carbonates. Hajjaji et al. (2010) observed a good correlation between LL, PL and EBS for sediments taken from stratigraphic sequences in Tunisia. Furthermore, different clay minerals have a different specific surface area (SSA), which may vary depending on the formation pathway and environment, globally affecting the colloidal behavior and the water sorption capacity. Dolinar (2012) observed a direct relationship between the Atterberg limits (LL, PL) and the SSA. Keller and Dexter (2012) speculated that the mechanical behavior of soils at water contents higher than the liquid limit might be more controlled by SSA than the one in the plastic range. Among the qualitative aspects related with the clay fraction, Keller and Dexter (2012) highlighted the role of the aggregated clay particles that can actually behave as silt-sized grains in soils, hence reducing its plasticity. Some indirect effect of soil aggregation was observed also by Blanco-Canqui (2006), who explained differences in soil consistency with variations in SOM amount and the effects it had on structure. Also Zolfaghari et al. (2015b) remarked that clay aggregation and SOM quality might need further investigation with respect to Atterberg limits, and this might be particularly true for the surface soil horizons where the influence of the organic component is more pronounced.

Therefore, despite some general consensus on the influence of the clay fraction and organic matter on the soil liquid and plastic limits, some points still need to be addressed by research. First, while LL and PL are determined on the <0.425 mm soil fraction, the other soil properties (e.g. texture, SOM content) generally refer to the fine earth soil fraction (i.e. <2 mm). In addition, the role of clay mineralogy in organic matter-rich soil horizons such as non-agricultural soils (e.g. mountain forest topsoils), potentially prone to natural hazards, is still poorly studied, as a large part of the research focuses on agricultural soils, sediments or stratigraphic sections.

In particular, little is known about mid-altitude mountain soils located in geographical regions where even small changes in temperature and precipitation largely affect snowfall frequency and amount, thus altering the precipitation regime (Terzago et al., 2013) with potential effects on natural hazards frequencies. We therefore investigated a set of forest soils (Ligurian Alps, NW Italy), that are relatively rich in both the clay fraction and SOM, with the aim of evaluating the relationships among LL, PL, and these soil properties from both the quantitative and the qualitative point of view. In particular we investigated:

1) the relative effect of clay and organic matter. We hypothesized that LL and PL might be better correlated with the properties of the <0.425 mm soil fraction than those of the fine earth;

2) the relationships between the mineralogical composition and aggregation of the clay fraction, and LL and PL in the <0.425 mm size range.

2. Materials and Methods

2.1. Description of the study area

The study area is located in the Ligurian Alps (NW-Italy, Figure 1) at elevation ranging from 800 to 1600 m a.s.l., at the boundary between Piemonte and Liguria Regions, 30 km far from the Mediterranean Sea. It is particularly interesting for the combination of environmental factors, as it is located at the convergence of the Mediterranean, European, and Alpine biogeographic regions (Cornara et al., 2014).

The average annual temperature ranges between 4 and 7.5 °C, decreasing with altitude and with local variability with slope aspect. The annual precipitation is around 1000-1200 mm, with spring and fall maxima, and summer minima (Biancotti et al., 1998). The soil moisture regime is always udic, while the temperature regime varies from mesic (below 1300 m a.s.l.) to frigid (above 1300 m a.s.l.). Forests cover a large part of the territory and are dominated by beech (*Fagus sylvatica* L.), chestnut (*Castanea sativa* Mill.), larch (*Larix decidua* L.), fir (*Abies alba* Mill.), and Scots pine (*Pinus sylvestris* L.). The soils of the area develop on a wide range of parent materials, from pelitic and metapelitic sedimentary (or weakly metamorphosed) rocks to hard and coarse grained quartzite. Pelitic and metapelitic rocks allow the development of relatively clay-rich soils that are therefore characterized by both high SOM contents, which are typical of forest soils, and fine texture (with both lithogenic and pedogenic layer silicates). Quartzite instead supports the development of coarser textured soils. A detailed description of soils, vegetation cover, and geology of the area is provided by Catoni et al. (2016). We selected 11 soil samples on varying parent material (Table 1): 2 were on quartzite or metamorphic porphiroids, 3 on limestone-dolostone, and 6 on the Helminthoides Flysch unit formed by both calcareous and non-calcareous weakly metamorphosed siltstones and claystones discontinuously interbedded by sandstones. Topsoils (0-10 cm) were collected, then air dried and sieved at 2 mm (fine earth fraction).

2.2. Chemical and physical analyses

On the <2 mm fraction, the total C (TC) contents were determined by dry combustion with an elemental analyzer (NA2100 CE Instruments, Rodano, Italy). The content of inorganic C was measured with the TIC-solid module of a TOC analyzer (Vario TOC132 Elementar, Hanau, Germany). The total organic C (TOC) content was computed as the difference between C measured by dry combustion and carbonate-C. Soil particle distribution was determined by the pipette method with Na-hexametaphosphate after pre-treatment for SOM oxidation with H_2O_2 (Gee and Bauder, 1986). As TOC is linearly related to SOM, it was considered as an indicator for SOM content in further statistical analyses. The exchangeable amounts of elements (Ca, Mg, K and Na) were determined by AAS after extraction with NH_4^+-acetate at pH 7.0 and the CEC was determined spectrophotometrically after back-exchange of NH_4^+ with NaCl (Soil Survey Staff,

2014a). Only the base saturation (BS, sum of measured exchangeable cations/CEC*100) and the exchangeable Ca^{2+} contents are reported.

The fine earth fraction was then dry-sieved at 0.425 mm and we determined LL and PL according to the standard methods (ASTMD 4318–10e1, 2010), i.e. the automatic cone penetrometer (cone angle 30°, total mass 80 g, penetration depth 20 mm) and the thread rolling method. The plastic index (PI) was computed as the difference between LL and PL. TOC content was determined on the <0.425 mm fraction as already done for the <2 mm fraction. We plotted all our samples on the plasticity chart, ideated by Casagrande (1932; and then modified by Howard, 1984). The meaning of the chart has been deeply discussed by Polidori (2003) and Polidori et al. (2004). It is the scatterplot of PI vs. LL, used in geotechnical engineering to represent graphically the mechanic behavior of clays. In particular, the A-line in the chart allows separating materials with different mechanical behavior, i.e. a different relationship between PI and LL. The U-line instead represents an upper envelope for most of the world soils (Zolfaghari et al., 2015b).

The clay of the <0.425 mm size fraction, to be used for mineralogical analyses, was separated with two dispersion methods, i.e. only water dispersion ($clay_{<0.425mmw}$) and Na-hexametaphosphate dispersion after H_2O_2 pre-treatment ($clay_{<0.425mmPER}$) as reported in Stanchi et al. (2008). The difference between the two clay contents in each soil sample ($clay_{<0.425mmPER}$ - $clay_{<0.425mmw}$) was computed, and interpreted as a measure of the amount of aggregated clay present in the <0.425 mm size range.

The SSA of the clay separated from <0.425 mm soil fraction was estimated using the methylene blue absorption method (spot test) as described in ASTM (1984), which allows to consider both the external and internal surface areas of the clay minerals.

The soil specific surface area (SSA, expressed in $m^2\ g^{-1}$) was computed as:

$$SSA = \left(\frac{1}{320}\right)\left(\frac{10}{1000}\right)(vol)Av\left(\frac{A_{mb}}{m}\right) \qquad (1)$$

where 320 g mol^{-1} is the molar weight of methylene blue, 10/1000 is the concentration of the methylene blue solution (10 g l^{-1}), vol is the amount of methylene blue solution added (ml), Av is Avogadro number (molecules mol^{-1}), A_{mb} is the covering area of methylene blue ($130 \cdot 10^{-20}$ m^2 $molecule^{-1}$), m is the mass of dry soil (g). The methylene blue determination was first carried out for water-dispersed clay (W) and then repeated on H_2O_2-pretreated samples (PER).

All chemical and physical analyses were made in duplicate and then averaged.

2.3. Mineralogical analyses

The mineralogical composition (<0.425 mm fraction) was evaluated through X-ray diffraction using a PW1810 diffractometer (40 kV and 20 mA, Co Kα, graphite monochromator) on the clay fraction (both after water dispersion and after Na-hexametaphosphate dispersion plus SOM oxidation). The clay fraction was saturated with $MgCl_2$ and analyzed as air-dried (AD), ethylene glycol solvated (EG), and heated (550°C) oriented mounts. Scans were made from 3 to 35 º2θ at a speed of 1 º2θ min^{-1}.

Chlorite was identified by the presence of a 1.4-nm peak after 550°C heating, illite by the 1.0-nm peak in

175 the AD diffraction pattern, swelling clay minerals by the presence of a peak or shoulder at 1.6–1.7 nm after
176 EG treatment. Interstratified minerals were recognized by the presence of a diffraction peak or band
177 exhibiting a behavior intermediate to that of the single components. Associated minerals (i.e. quartz,
178 feldspars) were identified by peak positions according to the data reported by Brown (1980).
179 A semi-quantitative evaluation of mineral abundance was performed by taking into account the areas of
180 the peaks and the MIFs (mineral intensity factors) reported by Islam and Lotse (1986). For the analysis, the
181 background was subtracted and the peak intensities and positions were calculated using the second
182 derivative option of the PowderX software (Dong, 1999).
183 EBS (Equivalent basal spacing) was computed as in Schmitz et al. (2004) as:
184 $$EBS = TCf \sum(Cf_i \times BS_i^{(001EG)}) \qquad (2)$$
185 where TCf is the total amount of phyllosilicate clay in the sample (% weight fraction) and Cf_i is the
186 percentage abundance of the i^{th} mineral; Bs_i is the average basal spacing in nm (*001* or *002*) referred to the
187 ethylene glycol treatment, thus the highest is the index the highest the content of smectite and other
188 swelling minerals (e.g. swelling mixed layers or low charge vermiculite). Because of the abundance of
189 chlorite whose interlayer is filled with a brucitic or gibbsitic sheet that cannot sorb water, the EBS was
190 modified by attributing to chlorite a basal spacing of 0.7 nm (instead of 1.4 nm) that is equivalent to that of
191 kaolinite. EBS was computed both for water-extracted clay (EBS_W) and pre-treated clay (EBS_{PER}).
192 *2.4. Data treatment*
193 All the statistical analyses were carried out with SPSS software version 20.0. Multiple regression analysis
194 was performed in order to quantify the relative clay and SOM contribution to LL, PL and PI.
195 **3. Results**
196 The $TOC_{<2mm}$ content ranged from 18.5 to 84.3 g kg^{-1} (Table 1) and was positively correlated with $TOC_{<0.425mm}$
197 (r=0.78, p<0.01, Table 2). The studied soils had a varied clay content (Table 1) with values between 124 and
198 434 g kg^{-1} for the <2 mm fraction. The clay fraction contents of the <0.425 mm fraction were in the range
199 21-118 g kg^{-1} for $clay_{<0.425mmw}$, and 134-332 for $clay_{<0.425mmPER}$. These values were highly variable after each
200 extraction method, giving rise to differences in the range 61-272 g kg^{-1} (Table 1), and no correlation was
201 found between clay content in the two soil fractions (i.e., <2 and <0.425 mm) or between the two methods
202 of extraction in the <0.425 mm soil fraction (Table 2). Ca^{2+} always dominated among basic cations and the
203 base saturation was extremely variable depending on parent material (average 51%).
204 Soil LL (Table 1) ranged from 42% to 85%; PL ranged from 31 to 61% and could not be determined for
205 sample 7. LL and PL were significantly correlated with each other (r=0.87, p<0.01, Table 2).
206 PI ranged from 5 to 24% (Table 1), i.e. soils ranged from slightly to moderately plastic according to the
207 ASTM D 4318–10e1 (2010) classification. Considering Casagrande's chart plotted in Figure 2, all samples fell
208 below the U-line, and also fell on or below the A-line (mostly in the area representing "*inorganic silts with*
209 *high compressibility and organic clays*").

LL and $TOC_{<2mm}$ were not significantly correlated (Table 2), while a positive correlation was present between PL and $TOC_{<2mm}$ ($r=0.69$, $p<0.05$). Both LL and PL were better correlated with $TOC_{<0.425\ mm}$ ($r=0.71$, $p<0.01$; $r=0.85$, $p<0.01$, respectively). LL and PL were not correlated with $clay_{<2mmPER}$. No relationship was present with $clay_{<0.425mmW}$, while a positive correlation was visible between LL and $clay_{<0.425mmPER}$ ($r=0.64$, $p<0.05$), as well as between PI and $clay_{<0.425mmPER}$ ($r=0.72$, $p<0.01$). The difference between $clay_{<0.425mmPER}$ and $clay_{<0.425mmW}$ contents showed a strong relationship with LL ($p=0.73$, $p<0.05$) and PI ($r= 0.84$, $p<0.01$), as reported in Table 2. No relationship of Atterberg limits with base saturation and exchangeable Ca contents was observed (data not shown).

Stepwise multiple regression including clay and TOC contents was significant and evidenced a similar contribution of clay and TOC to the variability of LL (equation 3), while the determination coefficient was lower for PL and the only significant variable was TOC (equation 4).

$$LL = 0.687\ TOC_{<0.425mm} + 0.615\ Clay_{<0.425mmPER} \qquad adjR^2=0.85,\ SEE = 4.4 \qquad (3)$$

$$PL = 0.847\ TOC_{<0.425mm} \qquad adjR^2=0.68,\ SEE = 4.7 \qquad (4)$$

VIF (i.e. variance inflation factors), used as indicators of the goodness of the regression, were always around 1, so that a strong multicollinearity between variables could be excluded. Both regressions were significant ($p<0.01$).

The mineralogical composition of the clay fraction showed a complex suite of minerals, present both as single and mixed layer phases (Figure 3). The clay mineralogical composition of the studied samples (considering always the <0.425 mm fraction, two dispersion methods) is detailed in Table 3 and depended on the clay dispersion method. Illite was detected in all samples (range 16-50% for W, 8-87% for PER). Smectite in W dispersion was observed, though in limited amounts, only in samples 6 and 16, while traces were present in sample 17. Among PER samples, 1-4-7-16, and 17 showed some amount of the mineral, but in most cases the relative proportion was ≤2%. Chlorite was present in all W-dispersed samples in variable amounts (1-28%); in PER samples a comparable range was observed. Vermiculite was always present, and exceeded 50% in samples 1, 4, 12 (W). After PER pre-treatment, it was present in all samples except 17, and ranged from 10 to 49%. Interstratified minerals were sporadically present, even if in some cases they accounted for a relevant proportion of the total (e.g. W samples 7, 9, 11, 16, 17 Table 3). In PER samples the amount of interstratified minerals generally decreased, with the exception of illite-vermiculite in samples 7, 9, 11, 12. In the clay fraction, the amount of quartz was always <3% and feldspars were always below 10%. EBS never exceeded 0.145 nm for water dispersible clay minerals (W; Table 3) and increased after the removal of SOM (EBS for PER ranged from 0.158 to 0.388 nm). The SSA of the <0.425 mm soil fraction differed between treatments only for sample 17. The global range of SSAw was 12.2-85.6 $m^2\ g^{-1}$, without any clear trend with mineralogy.

No significant relationship was visible between Atterberg limits and EBS_w (Fig. 4a), nor mineral abundances, but EBS_{PER} was related to LL (Fig. 4b). EBS_{PER} was also strongly correlated with $clay_{<0.425mmPER}$-$clay_{<0.425mmW}$ ($r=0.843$, $p<0.01$, data not shown). SSA_w was related with LL and PI as visible in Figure 4c.

4. Discussion

LL and PL values (Table 1) were in the range reported for Italian mountain soils (e.g. Stanchi et al., 2012, 2015). However, the averages were slightly higher than reported from a similar number of topsoil samples from previous studies (e.g. Stanchi et al., 2012, PL=38, LL=49). This was likely due to the different environmental characteristics of this study area, i.e. often finer soil textures and higher organic matter contents, and was confirmed by higher PI values (average 16% vs. 10% of the previous research). The relatively high organic matter content was related with the good development of organic horizons in these forest soils, as described by Catoni et al (2016). High $clay_{<2mmPER}$ content was always measured, too, even if we excluded from this study the paleosols which are also abundant in the area (D'Amico et al., 2016).

In the <2 mm fraction a positive correlation was present only between $TOC_{<2mm}$ and PL. According to Keller and Dexter (2012) the plastic limit in topsoils is positively related to SOM, but in our dataset, the use of TOC and clay fraction contents determined on the <0.425 mm fraction considerably improved the relationship (Table 2) highlighting the importance of the fraction choice to properly understand the soil mechanical behavior. This suggests that the contrasting results previously reported in literature about the variables controlling LL and PL can be partially related to methodological issues, as the amounts of clay fraction in the <2 mm and <0.425 mm are not proportional.

Equation (3) and (4) obtained by regression of TOC and/or clay fraction contents in the finer fraction (<0.425 mm) showed a good predictability of LL and PL from these properties in terms of significance and R^2. While for LL the two considered factors showed a similar relevance (eq. (3)), only $TOC_{<0.425mm}$ was retained in the model for PL prediction and explained a relevant part of the variability, as visible from eq. (4). The importance of the considered factors was further confirmed by the correlation observed between the amount of aggregated clays in the <0.425 mm fraction (i.e. ($clay_{<0.425mmPER}$-$clay_{<0.425mmW}$)) and both LL and PI. As visible from the correlations reported in Table 2, the amount of aggregated clays was strongly related with the plastic index of the soils in the study area, i.e. soils with a wider range of plastic behavior, together with higher LL, also showed higher amounts of aggregated clays in the <0.425 mm fraction. This suggests that, in the studied environment, PL seemed to be more controlled by the amount of organic C, while LL seemed to be controlled by the clay fraction in terms of abundance and degree of aggregation promoted by SOM.

The conceptual stages of the clay/water interactions taking place for increasing water content can explain these findings. First, water fills the soil pores (at the solid/semisolid state). Once pores are filled, water is retained by SOM due to its intrinsic hydrophilic behavior (e.g. Yang et al., 2014); in such conditions the soil is at the semisolid or plastic state. Then water is adsorbed by clay minerals (transition from plastic towards

279 liquid state). For increasing water contents above the liquid state, the specific soil volume further increases
280 and the clay particles are dispersed. At the liquid state the separation distances between clay particles or
281 clay-sized aggregates are in equilibrium with the inter-particle forces (Pandian and Nagaraj, 1990).
282 Therefore, in a first stage of increasing water content, the role of SOM is fundamental (in particular, in the
283 semisolid state and plastic interval), then the interactions among clay particles become more relevant, up
284 to full colloidal repulsion (i.e. above LL).
285 Keller and Dexter (2012) speculated that the mechanical behavior of soils with water content exceeding LL
286 might be more controlled by SSA than the one in the plastic range. It is also generally assumed that SSA in
287 fine grained soils mainly depends on clays (Dolinar, 2012). However previous research (e.g Dolinar, 2012)
288 quantified SSA by N_2 adsorption, which allows to measure only the external surface area of minerals and is
289 little sensitive to the SSA of SOM (Catoni et al., 2014). Despite the differences in SSA determination
290 methods, our findings seem to support this hypothesis, as the soil SSAw was positively related with LL and
291 PI but not PL, which instead seemed to be more controlled by SOM, as also showed by the regressions
292 obtained. Some role of SOM was however evidenced by the methylene blue spot test for sample 17
293 (chestnut stand on quartzite), displaying a reduction of SSA after SOM oxidation (Table 2). This specific
294 behavior suggested a strong contribution of SOM to available surfaces, in line with forest environments
295 with SOM-rich topsoil. Some general considerations can be made on the base of soil profile morphology
296 and description. Sample 17 showed a loose structure, suggesting a weaker degree of organo-mineral
297 interactions. Moreover, this sample had a much higher C/N ratio than the others with the same vegetation
298 used in this work (17.3 vs an average of 13.7, data not shown), thus indicating a lower degree of
299 transformation of organic matter. Upon destruction of SOM, new mineral surfaces and pores become
300 available in case of mineral-organic interactions (Echeverrìa et al., 1999), while if organic matter and
301 minerals were present as poorly transformed separated phases, SOM oxidation would simply result in area
302 subtraction. No clear relationship was visible between LL, PL and mineralogy, i.e. no correlation was
303 present between LL, PL and the abundance of specific minerals, or with subsets of minerals (e.g.
304 interstratified or expandable). The positive relationship between EBS_{PER} and LL (Fig. 4) indicates that soils
305 with high EBS_{PER} require higher amounts of water to pass from plastic to liquid state, i.e. EBS somehow
306 affects clay-water interactions. This is in agreement with the positive correlation we found between
307 aggregated clay and EBS_{PER}, suggesting that the clay fraction tends to be more aggregated when it contains
308 higher amounts of 1.6 and 1.4 nm phyllosilicates (i.e. smectites and vermiculites), whose interlayer may
309 incorporate water molecules. A general trend towards a greater aggregation of more highly charged
310 minerals (such as, e.g., vermiculite vs illite) can be expected, but the aggregation phenomena in soils are
311 complicated by the presence of organic matter and by the charge of sand particles on which clay particles
312 react (Falsone et al., 2007, 2016). Another factor that may affect clay aggregation is the presence of
313 divalent cations, such as Ca^{2+}, on the cation exchange complex (e.g. Wuddivira and Camps-Roach, 2007)

that may act as a bridge between negatively charged components such as SOM and layer silicates. Despite the variability in exchangeable Ca contents of the soils, we did not find any correlation between Ca^{2+} and Atterberg limits or clay aggregation. The lack of correlation with PL may be related to the dominant effect of SOM, while in the case of LL, the high amounts of water added may induce clay dispersion even in the presence of flocculating cations with large hydration shell such as Ca^{2+} (Sposito, 1989). These complex interactions may justify the absence of a clear trend in increase or decrease of single specific minerals in the aggregated clay (Table 3) and the lack of correlations between limits and mineralogy that are often assumed to be present.

5. Conclusions

We investigated the relationship between the Atterberg limits (LL, PL) and clay fraction, and organic matter contents in forest topsoils from the Ligurian Alps. While LL did not show any correlation with TOC content of the <2 mm fraction, and PL only a weak one, strong positive correlations were instead visible between both Atterberg limits and the TOC content of the <0.425 mm fraction, i.e. the one used for Atterberg limits determination according to international standard methods. We also observed a relationship between LL and $clay_{<0.425mmPER}$. Therefore, we can conclude that the observed relationship are more evident when the <0.425 mm fraction is considered, as hypothesized in the aims, and contrasting results about the role of these variables in previous research might be partly due to methodological issues.

The role of the clay fraction and SOM in the <0.425 mm size fraction was investigated through regression analysis, evidencing a similar relevance of the two soil variables in the case of LL, while for PL only the TOC content was retained. This suggested that PL is more controlled by SOM while LL seems to be affected by both SOM and clay fraction. In addition, the amount of aggregated clay in the <0.425 mm fraction strongly affected LL and PI, i.e. the interval of soil plastic behavior, in the studied environment.

The semi-quantitative mineralogical composition did not add relevant information. However, the relationships between SSA, EBS and the Atterberg limits provided further insight on the mechanisms explaining the soil behavior in the plastic interval, again evidencing a qualitative role of clay fraction and its aggregation.

The findings helped a deeper understanding of the mechanical properties of the studied soils, with potential insights in soil protection and hazard prevention, i.e. soil resistance to large water inputs due to intense rainfall. This might be particularly relevant as mid-elevation areas at intermediate latitudes, such as the study sites, are very sensitive to climate change, and variations in the rainfall pattern might affect the topsoil degradation.

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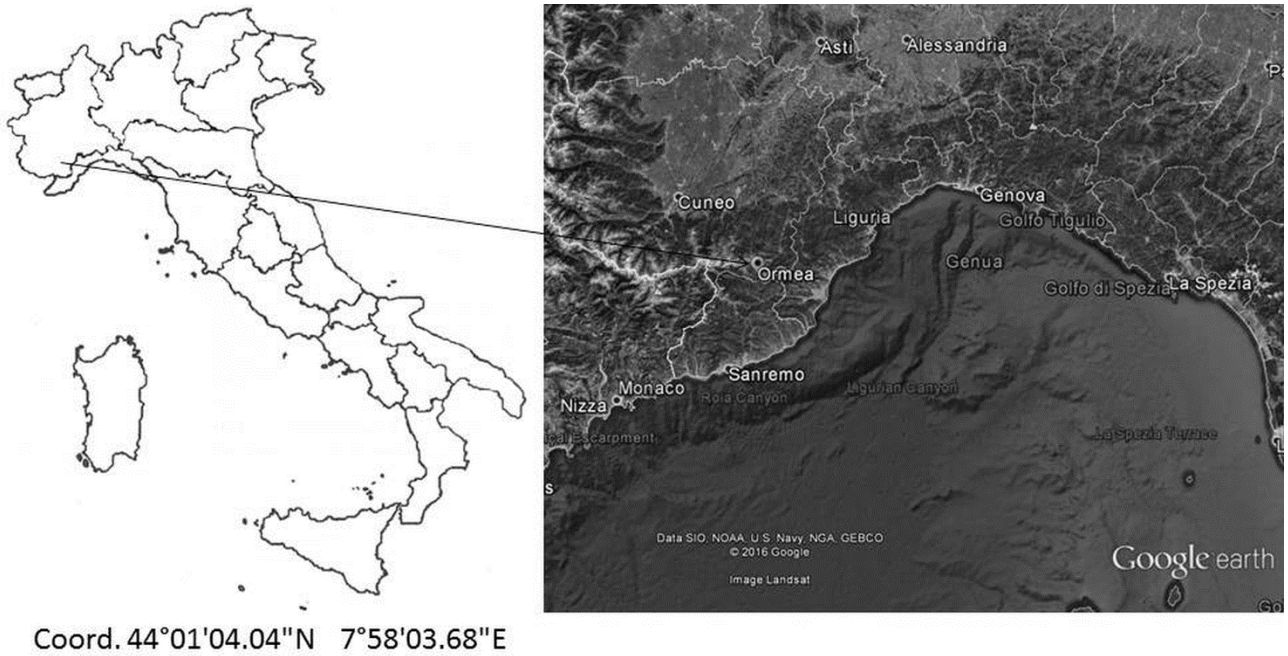
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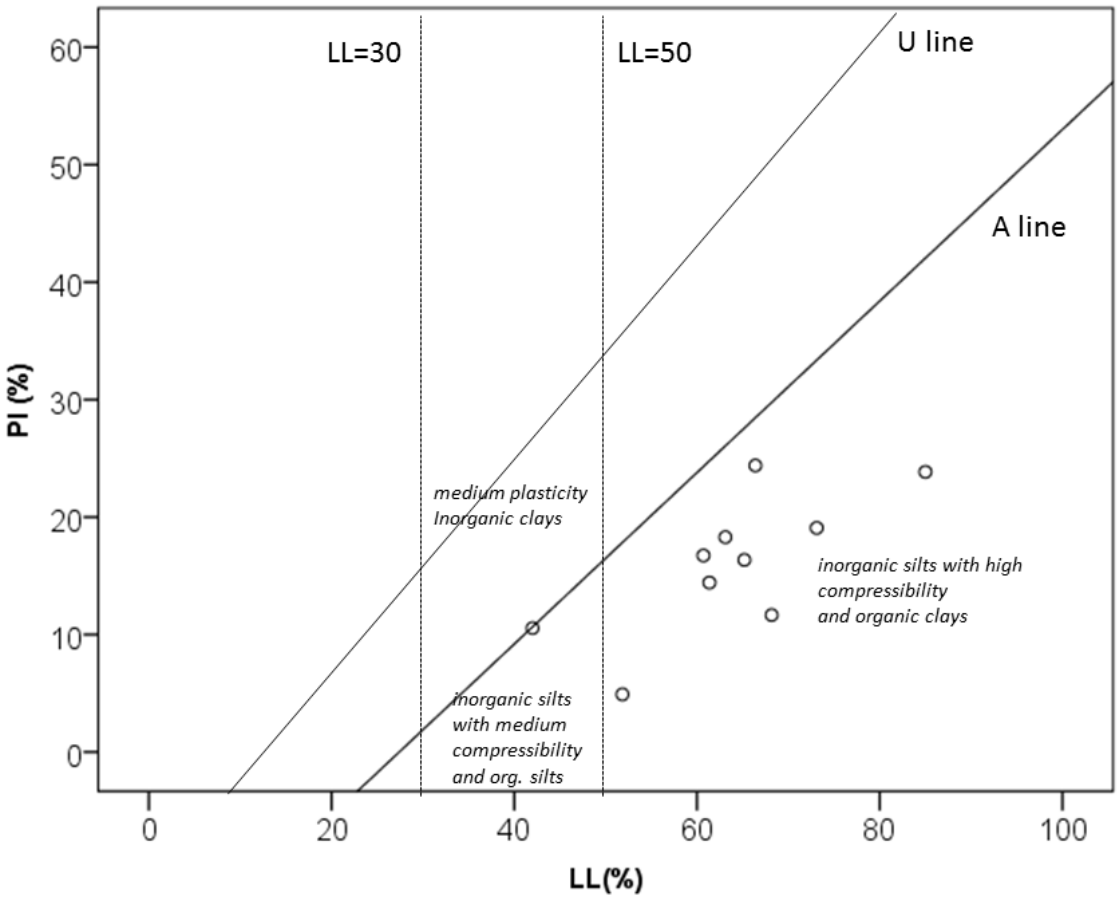
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466
467 **Figure captions**
468 **Figure 1: The study area. Coordinates refer to the town of Ormea, located at the centre of the study area.**

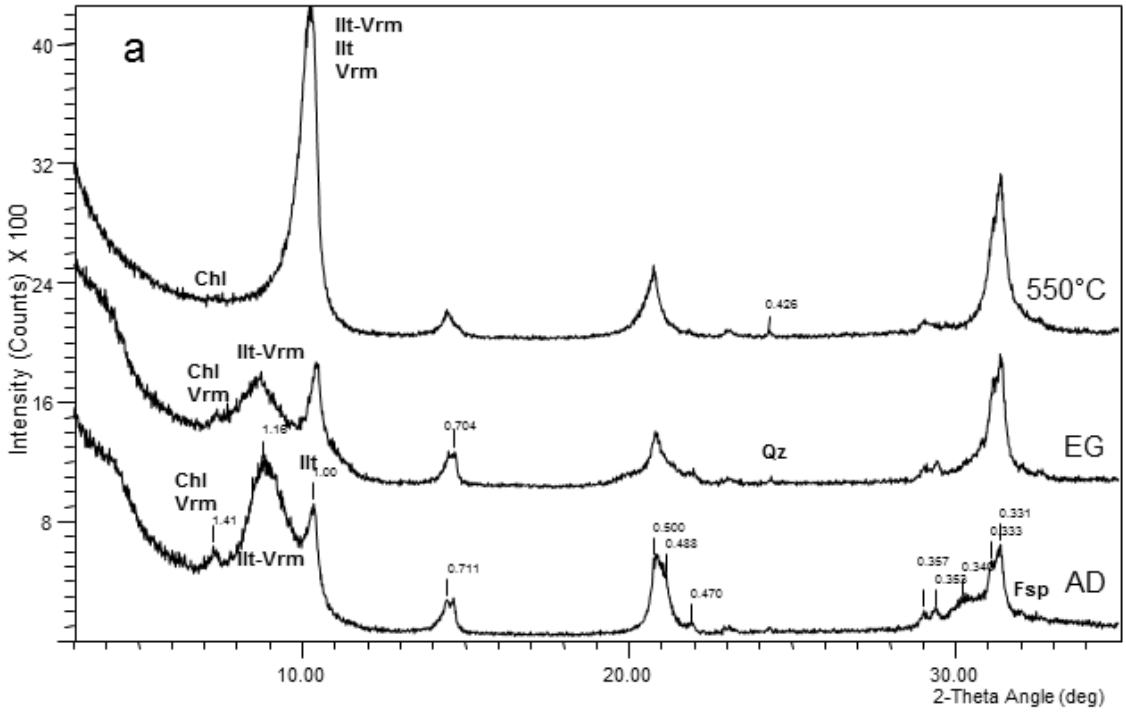


469
470 **Figure 2: Casagrande plasticity chart displaying the A-line and the U-line.**



471

472 **Figure 3: XRD patterns of the clay of sample 9 after water extraction (clay$_{<0.425mmW}$, a) and peroxide**
473 **oxidation followed by hexametaphosphate dispersion (clay$_{<0.425mmPER}$, b). AD, EG and 550°C indicate air-**
474 **dried, ethylene glycol solvated and heated sample, respectively. Peak marks are in nm and the minerals**
475 **contributing to the peaks are also reported Abbreviations for minerals are reported after Withney and**
476 **Evans (2010) (Ilt (illite), Sme (smectite), Vrm (vermiculite), Chl (chlorite), Qz (quartz), Fsp (feldspars).)**


a
Ilt-Vrm
Ilt
Vrm
Chl
Chl
Vrm
Ilt-Vrm
Chl
Vrm
Ilt
Ilt-Vrm
Qz
Fsp
550°C
EG
AD
Intensity (Counts) X 100
2-Theta Angle (deg)

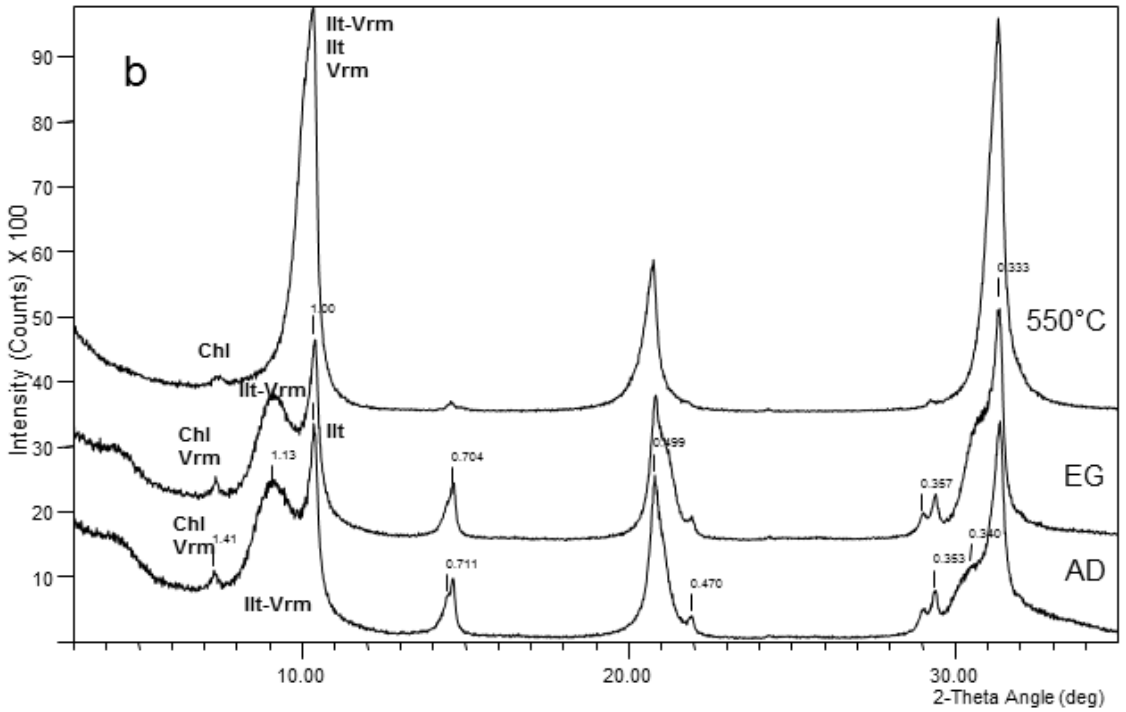

b
Ilt-Vrm
Ilt
Vrm
Chl
Ilt-Vrm
Chl
Vrm
Ilt
Chl
Vrm
Ilt-Vrm
550°C
EG
AD
Intensity (Counts) X 100
2-Theta Angle (deg)

Figure 4: Relationship between LL, PL, PI and EBS (a,b), SSA_w (c). Significant relationship are represented by a continuous line. The number of samples was 11 for LL, 10 for PL and PI.

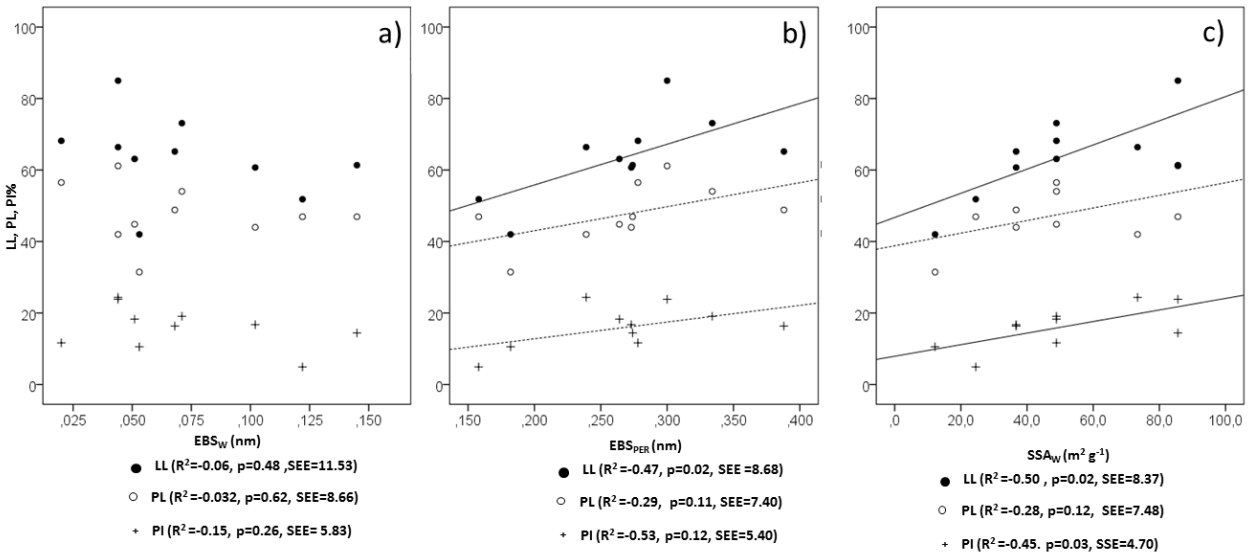


Table 1: Selected chemical and physical properties of the two soil fractions.

WRB classification USDA classification	Altitude (m asl)	PM	$TOC_{<2mm}$ (g kg^{-1})	$Clay_{<2mmPER}$ (g kg^{-1})	pH	$TOC_{<0.425mm}$ (g kg^{-1})	BS (%)	Ca_{exc} (cmol$_c$ kg^{-1})	LL (%)	PL (%)	PI (%)	$Clay_{<0.425mmw}$ (g kg^{-1})	$Clay_{<0.42}$ (g kg
keletic Alisol (Loamic, utanic, Differentic, ptic, Loaminovic) udalf, clayey-skeletal, mixed,	1321	FLY	32.7	132	5.0	21.5	35.7	3.13	52	47	5	99	160
tic Colluvic Protic Eutric oamic) rthent, fine-loamy, mixed, nacid, frigid	1609	FLY	32.0	315	7.0	30.6	90.2	15.93	61	47	14	118	258
letic Chernozem (Loamic, lyperhumic) iudoll, loamy-skeletal, , mesic	838	LIM	63.2	180	8.1	72.3	100	26.10	68	57	12	21	139
rthoskeletic Luvisol (Clayic, ypereutric, Hyperhumic,	1230	LIM	81.6	434	7.4	55.2	99.0	26.0	55	nd	nd	28	134

apludalf, clayey, mixed, e, nonacid, mesic.													
olluvic Protic Orthodystric Loamic, Humic)	1666	FLY	49.9	217	5.6	42.6	40.5	6.1	63	45	18	46	256
rthent, sandy-skeletal, mixed, igid													
yperdystric Cambisol Humic)	1234	QTZ	32.5	170	4.7	21.7	29.0	2.56	42	31	11	46	146
rudept, sandy-skeletal, d, mesic													
c Orthoeutric Regosol Humic)	1021	FLY	84.3	306	5.9	56.2	51.7	11.85	73	54	19	62	290
rthent, fine-loamy, mixed, e, nonacid, mesic													
etic Protic Hyperdistric Loamic, Humic)	1032	FLY	18.5	273	4.8	17.9	4.50	0.30	61	44	17	89	274
rthent, fine-loamy, mixed, d, mesic													
hodystric Regosol (Loamic, umic, Transportic)	875	FLY	20.1	247	5.5	26.0	25.1	3.49	66	42	24	41	251
rthent, fine-loamy, mixed, e, acid, mesic													
ypereutric Cambisol (Loamic,	1411	LIM	58.6	370	6.3	36.1	57.2	15.11	65	49	16	59	332
udept, fine-loamy, mixed, e, nonacid, frigid													
etic Hyperdystric Cambisol Escalic, Humic) over Skeletic erdystric Regosol (Loamic) rudept, loamy-skeletal, peractive, acid, frigid	970	QTZ	62.6	124	4.5	81.1	25.7	2.50	85	61	24	49	302
			48.7 (23.2)	252 (99)	5.9 (1.2)	41.9 (21.6)	50.7 (32.5)	10.3 (9.4)	63 (11)	48 (8)	16 (6)	60 (30)	231 (72)

*The horizons sequence does not include organic horizons.

nd: not determined

WRB classification (IUSS Working Group WRB, 2014); USDA classification (Soil Survey Staff, 2014b)

PM is the soil parent material (FLY: Helminthoides Flysch units; LIM: limestone; QTZ: quartzite)

LL: liquid limit; PL: plastic limit; PI: plastic index; for clay fraction subscript indicates the investigated soil fraction (< 2mm or <0.425 mm) and the clay dispersion method (simple water dispersion, W; pre-treatment for SOM oxidation with hydrogen peroxide, PER)

BS is the base saturation

Table 2: Correlation matrix for relevant soil properties. In brackets the p values.

LL (%)	PL (%)	PI (%)	$TOC_{<2\ mm}$ ($g\ kg^{-1}$)	$TOC_{<0.425\ mm}$ ($g\ kg^{-1}$)	$Clay_{<2mm,PER}$ ($g\ kg^{-1}$)	$Clay_{<0.42mm5,w}$ ($g\ kg^{-1}$)	$Clay_{<0.425,PER}$ ($g\ kg^{-1}$)	Cla C
	0.87 (0.001)	0.73 (0.009)	0.39 (0.238)	0.71 (0.015)	-0.09 (0.800)	-0.16 (0.644)	0.64 (0.035)	0.7
		0.29 (0.420)	0.69 (0.027)	0.85 (0.002)	-0.08 (0.830)	-0.13 (0.724)	0.33 (0.352)	0.3
			0.17 (0.647)	0.35 (0.317)	0.25 (0.495)	-0.33 (0.352)	0.72 (0.020)	0.8
				0.78 (0.005)	0.34 (0.300)	-0.49 (0.128)	-0.01 (0.970)	0.2
					-0.09 (0.800)	-0.55 (0.081)	0.03 (0.929)	0.0
						-0.01 (0.977)	0.17 (0.622)	0.1
							0.29 (0.380)	-0.1
								0.91

Table 3: Semi-quantitative mineralogical distribution (%) in the water-dispersed (W) and H_2O_2 pretreated (PER) soil fraction (<0.425mm).

ID	Ilt	Sme	Chl	Vrm	Ily-Sme	Ilt-Chl	Ilt-Vrm	Chl-Vrm	Qz	Fsp	EBS (nm)	SSA (m^2 g^{-1})
						W						
1	27	-	5	63	3	-	-	-	1	1	0.122	24.5
4	22	-	5	67	-	-	-	2	1	3	0.145	85.6
6	50	8	28	6	-	-	-	-	2	6	0.020	48.9
7	18	-	4	-	77	-	-	-	1	tr	0.034	48.9
8	31	-	12	27	-	-	27	-	1	2	0.051	48.9
9	20	-	1	6	-	-	71	-	1	1	0.053	12.2
11	19	-	6	23	-	-	49	-	tr	3	0.071	48.9
12	32	-	13	51	-	-	-	-	2	2	0.102	36.7
15	42	-	11	37	-	5	-	-	1	4	0.044	73.4
16	16	4	15	-	61	2	-	-	1	1	0.068	36.7
17	25	tr	12	5	-	57	-	-	1	tr	0.044	85.6
						PER						
1	55	2	17	13	4	-	-	3	2	4	0.158	24.5
4	42	1	7	35	-	-	-	5	tr	10	0.274	85.6
6	28	-	13	40	-	-	-	15	2	2	0.278	48.9
7	8	1	1	49	3	-	38	-	tr	tr	0.172	48.9
8	40	-	23	33	-	-	-	-	2	2	0.264	48.9
9	8	-	2	47	-	-	41	-	tr	2	0.182	12.2
11	17	-	11	38	-	-	31	-	tr	3	0.334	48.9
12	41	-	18	17	-	-	17	-	tr	7	0.273	36.7
15	54	-	21	10	-	-	-	10	1	4	0.239	73.4
16	26	7	13	46	2	1	-	-	3	2.1	0.388	36.7
17	87	2	2	-	-	-	7	-	-	2	0.300	48.9

Ilt (illite), Sme (smectite), Vrm (vermiculite), Chl (chlorite), Qz (quartz), Fsp (feldspars). Abbreviations for minerals after Withney and Evans (2010).

EBS (equivalent basal spacing), SSA (specific surface area). Tr indicates traces.