Applied Clay Science 97-98 (2014) 241–245

Contents lists available at ScienceDirect

Applied Clay Science

journal homepage: www.elsevier.com/locate/clay

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The influence of disaggregation procedures on soil gravitational separation

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ARTICLE INFO

Article history:
Received 17 June 2013
Received in revised form 10 June 2014
Accepted 11 June 2014
Available online 26 June 2014

Keywords:
Soil
Particles
Separation
Dispersing agent

ABSTRACT

The use of dispersants in particle size analysis is a common practice, but this could cause bias on the gravitational separation of the different particle fractions in natural soil. The study highlights the results obtained in gravitational separation of silty and clayey fractions by using hydrogen peroxide (H_2O_2) and sodium hexametaphosphate ($(NaPO_3)_6$) in different combinations. The efficiency of the different treatments was verified by comparison against the results obtained on the same sediments without any treatment. The separation method is based on Stokes law to calculate the settling time of particles in deionized water under controlled temperature. This method was applied to three different agricultural soils of the Po River Plain. The sample treated with H_2O_2 and $(NaPO_3)_6$ at 4% showed the best results in terms of particle size degree of purity, ranging from 95% to 97% for clay and from 91% to 95% for silt. The degree of purity indicates the percentage of sediment with the particles of the provided grain sizes.

1. Introduction

Aggregates of soil particles, which are mainly formed as a result of precipitation processes of insoluble and soluble salts due to physical and chemical action of organisms and to electrochemical effects on clay particles, can significantly affect the results of the distribution particle size analysis (Matthews, 1991). For this reason using dispersants or disembedding agents is a common practice during the preparation of sediments for textural analysis (Evans et al., 2001; Psuty and Mureira, 2000). The most used chemical agents during soil or sediment samples pretreatment are H_2O_2 and $(NaPO_3)_6$ solution. H_2O_2 is mainly used to remove organic matter and stimulate deflocculation of particles, while $(NaPO_3)_6$ is used to facilitate the dispersion of electronegative colloids. The effect of particle disaggregation and deflocculation due to the addition of these chemical agents is useful both to improve the analytical response of grain size distribution analysis and to minimize the errors in the separation of silty and clayey fractions. This procedure can be very useful in studies regarding absorption/desorption of organic and inorganic contaminants or in studies of microbial degradation on separate particle fractions (Allen and Walker, 1987; Wang and Keller, 2009).

A wide range of techniques of separation are well known in the literature (ASTM, 2007; Day, 1965; de Jonge et al., 2000; Gee and Bauder, 1986; Huang et al., 1984). In addition, the separation of considerable amount of sediment (7% of dispersing phase) is possible and a high degree of purity of silty and clayey fractions can be achieved, by applying a gravitational separation method based on the Stokes law with a strict control of temperature (Salemi et al., 2010).

The aim of this study is to quantify the changes in particle size induced by sample pretreatment of the silty and clay fraction of agricultural top soils following the Salemi et al. (2010) procedure.

2. Materials and methods

The study was conducted on three different soils of the Po River lowland located in the Ferrara province (from here ITA, CCR and APO). These soil textures are very common in this area, overall they occupy an area greater than 50% of the province (Mastrocicco et al., 2010). According to the Wentworth classification (Wentworth, 1922) ITA is composed of 83% sand, 12% silt and 5% clay; CCR is 12% sand, 65% silt and 23% clay; and APO is 9% sand, 63% silt and 28% clay. The soil samples were collected in the first 10 cm of agricultural fields and their organic matter content, determined by applying LOI method (Heiri et al., 2001), is 2.7% for ITA soil, 4.5% for CCR, and 3.2% for APO. In order to proceed with the different treatments, the soil samples, after mixing and quartation, were divided into four samples (named A, B, C and D). For each sample triplicate analyses were performed. The A samples did not undergo any treatment; B samples were subjected only to oxygenation (H_2O_2 at 16 volumes) at room temperature until the complete disappearance of the effervescence; C samples were subjected to oxygenation and addition of $(NaPO_3)_6$ solution at a low concentration (0.5% in volume) to limit the cation exchange with the soil and minimize the chemical contamination; and D samples were subjected to oxygenation and addition of $(NaPO_3)_6$ at 4%, as suggested by ASTM (2007).

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http://dx.doi.org/10.1016/j.clay.2014.06.011
0169-1317/

The sand fraction (>63 μm) was separated from the rest of the sediment through wet sieving. The finer fraction, after the separation, was dried in an oven at 60 °C for at least 48 h in order to avoid any dimensional changes due to the extraction of water contained in the lattices of clay minerals. Then the fine fraction of samples A and B was rehydrated with 0.2 L of deionized Milli-Q water, while the fine fraction of samples C and D was rehydrated with deionized water and mixed with $(NaPO_3)_6$ at different concentrations. The complete dispersion of the sediment was ensured by stirring for 10 min using a magnetic stirrer. The dispersion was introduced into a 30 cm high borosilicate glass cylinder with an internal diameter of 8 cm, filled with 0.8 L of deionized water. The procedure was repeated three times, introducing the dispersion on the water surface using a borosilicate glass funnel. At each cycle, only the dispersion containing the clay particles (<4 μm) was siphoned respecting the settling time calculated by the Stokes law. The settling time was calculated taking into account the distance between the water surface and the bottom of the cylinder. The fine fraction density, fundamental in the settling time calculation, was determined by a Micromeritics AccuPyc 1330 pycnometer (2.62 ± 0.03 g/cm^3 for the soil ITA, 2.68 ± 0.02 g/cm^3 for CCR, and 2.66 ± 0.03 g/cm^3 for APO). The water viscosity was kept constant using a thermostatic bath made of a PVC tank (1 × 0.6 × 0.6 m) filled with water maintained at a constant temperature (20 ± 0.1 °C) by a heat exchanger (Resun CL450) and an electric pump (Eden 140). The particle size distribution and size limits of the separate fractions were carried out using a Micromeritics 5100 X-ray absorption Sedigraph (Artigas et al., 2005).

3. Results and discussion

The size fractions separated by gravitation were analyzed to determine the degree of grain size distribution purity. The results of the tests conducted on the treated samples (Fig. 1) show variable degrees of purity ranging from 85% to 97% in the clayey fraction and from 81% to 95% in the silty fraction. The untreated samples (A) after separation showed purities ranging from 87% to 95% for clay and 85% for silt. Despite the untreated samples (A) show high levels of grain size distribution purity, as discussed below, significant differences in textural frequency distributions appear evident when compared with the treated samples.

In particular, for the silty fraction, the adopted treatments show to be efficient only if the oxygenation is associated with the action of $(NaPO_3)_6$. For APO and ITA soils, treatment with only H_2O_2 leads to poorer results. This can be attributed to the refractory organic matter, which resists to treatment with H_2O_2. This is typical of soils rich in clay minerals and under long-term agricultural use (Leifeld and Kogel-Knabner, 2001). However, in all soils, it is evident that the use of $(NaPO_3)_6$ at a low concentration (0.5%) does not induce any significant effect. The highest values of dimensional purity are obtained only with $(NaPO_3)_6$ at a higher concentration added after oxygenation. These values do not significantly differ in the three soils analyzed. This means that the action of the dispersant is not influenced by the original textural nature of the soil. Similar observations can be made considering the degree of purity achieved in the clay fraction, for which, however, values are high also in the untreated samples A for APO and ITA soils. Also in this fraction the only oxygenation does not lead to significant improvements, while once again, the highest purity is achieved with the concomitant use of H_2O_2 and $(NaPO_3)_6$ at 4%. The effectiveness of this treatment is also supported by the low variability observed in the analyzed replicates. To better understand the action of the treatments in relation to the particle dispersion, the frequency grain-size distributions of the different samples separate fractions, obtained via X-ray absorption Sedigraph, can be considered.

Regarding the silty fraction (Fig. 2) the grain-size distribution appears to be very similar in the three analyzed soils. The untreated samples (A), the oxygenated one (B), and the oxygenated and treated with $(NaPO_3)_6$ at a low concentration (C) show very similar grain-size distributions, with an evident presence of particles with dimensions close to the dimensional limit of the lower class considered, or even lower.

For this reason, their purity percentage is lower compared to sample D. The latter shows a considerable decrease in frequency of particles with dimensions close to the lower limit and the almost total disappearance of smaller particles are observed. This can be explained by better dispersion of the particles and considerable decrease in electrochemical aggregates. Accordingly, the grain-size classes with higher frequency increase their relative importance. Similarly, analyzing the frequency distribution of the clayey fractions, a significant change is observed only in samples D, for which the decrease in coarser particles is associated with an increase in finer particles (Fig. 3).

Comparing size distribution changes of the two fractions can be thus assumed that the electrochemical aggregates, with an average size ranging from 7.5 to 9.5 phi and found both in the silty and clayey fractions, are realistically composed of clay particles smaller than 10.5 phi. The dispersion of these aggregates can be effectively achieved by using a dispersant with an adequate concentration. The dispersive effect of agents was also evaluated considering the textural parameters (Folk and Ward, 1957) of the analyzed fractions (Table 1). Regarding the silt, the samples B and C do not significantly differ from the reference untreated sample A, while sample D shows a considerable variation, with a shift of the mean diameter and the median toward coarser values and a slight improvement of the sorting. Similarly, in the clay fraction, the consequences of the treatments are evident only in samples D. In this case, they show a significant decrease in the mean diameters and

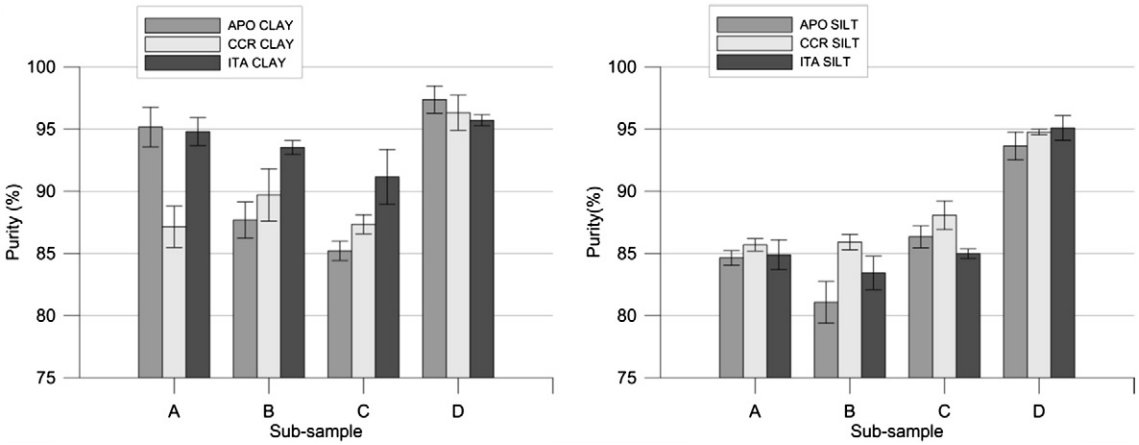

Fig. 1. Degree of purity (%) of the different fractions was analyzed. Error bars indicate the mean and ± the standard deviation of the triplicate measurements.