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Determination of an Appropriate Method for Dispersion of Soil Samples in Laser Diffraction Particle Size Analyses

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Abstract: Laser diffraction method is a fast, reliable, repeatable and more precise method than the classical methods such as sieve, hydrometer and pipette. Laser diffraction method is applied to many materials such as sand, clay, abrasives, ceramics, cement, paints, foods, pharmaceuticals, cosmetics, emulsions and sprays for the determination of particle size distribution with particles in the size range of 0.02 to 2000 microns. Different sample preparation and dispersion method is required for each material because of its different inherent properties. Ultrasonic action and Calgon solution are widely used for dispersion of soil samples in laser diffraction analysis. Ultrasonic action is an application of high-frequency sonic energy to the suspension in order to disperse aggregates. Calgon is a trademark of sodium hexametaphosphate which is a chemical substance. There is no standard procedure for dispersing the soil samples in the laser diffraction analysis. The objective of this study was to develop an effective and reliable method for dispersing the soil samples. For this purpose a Malvern Master SizerX Long Bad laser diffraction instrument was used and 38 soil samples taken from various location of Turkey were analyzed. Three methods were attempted to determine the most appropriate method for dispersing the soil samples, these are; (*i*) using ultrasonic action only, (*ii*) adding 30 ml Calgon solution directly to the sample bath immediately before analysis and applying ultrasonic action during analysis, and (*iii*) soaking the soil samples in 30 ml Calgon solution for at least 12 h before analysis and then loading to the sample bath and applying ultrasonic action during analysis. Calgon solution which was used in the analysis was prepared according to standard procedure given in ASTM standard for the hydrometer method. Clay content as well as whole particle size distribution was evaluated for effectiveness of the dispersion methods. Results obtained from 38 soil samples showed that more effective method is soaking procedure in Calgon solution for 12 h before analysis.

1. Introduction

Particle-size distribution is a fundamental physical property of soils and is conventionally determined by sieve-hydrometer or sieve-pipette methods in soil mechanics applications. Particle-size distribution is commonly used for soil classification and for the estimation of some other useful physical properties of soils such as drainage, permeability, porosity, consolidation, shear strength and liquefaction potential in geotechnical engineering practices.

Over recent decades, various new methods for particle-size analysis have been developed. Some of these are electroresistance particle counting, time of transition, image analysis, X-ray absorption, electrical sensing and laser diffraction [1-5]. Of these methods, laser diffraction is the most widely used and preferred method because of its many advantages. These advantages can be listed as (*i*) it is covering a wide range of particle sizes (typically 0.02 to 2000 micron); (*ii*) it requires a small amount of sample (typically less than 1 g), (*iii*) it requires short time of analysis (typically 5–10 minutes per sample); (*iv*) it has a high repeatability and good reliability.

Laser diffraction method can be applied to many materials such as sand, clay, abrasives, ceramics, cement, paints, foods, pharmaceuticals, cosmetics, emulsions and sprays [6]. This method was developed in 1970's and is used also for the determination of particle size distribution of soils since 1980's. However this method is not a standard method for the determination of particle size distribution of soils. An international standard, ISO 13320, was introduced in 1999; however it is not addressed particle size measurement of any specific materials. This standard is only covered general principles, diffraction theories, validation of inversion procedures, terminology etc [7]. Hence, each discipline must determine the principles of particle size measurement of their own materials. In this context, since 1980's, many studies (e.g., [8 – 24]) have been conducted for the determination of particle size measurement of soils using laser diffraction method. Many uncertainties were cleared through these studies. However a universal method for dispersing the soil samples has not been established in geotechnical applications.

The objective of this study was set out to find a possible acceptable procedure for dispersing the soil samples. For this purpose 38 natural soil samples taken from various locations of Turkey were analyzed. The origins of these samples are mainly sedimentary, and they were obtained from borehole or trial pits. Detailed information about the samples used in this study and the proposed procedure are given in the following sections of this paper.

2. Materials and Methods

2.1 Materials

A Malvern Master Sizer X (long bed) laser diffraction instrument with a wavelength of 633 nm (He-Ne laser beam source) was used in the analyses. This instrument contains 31-element solid-state detector array. It measures particle size over the range of 0.1 to 2000 µm with four lenses that have different focal length and measurement limits. A reverse Fourier lens that has a measurement limit ranging from 0.1 to 80 µm and a focal length of 45 mm was used in the study. The software employed was Malvern version 1.2. Mie theory was used in the study because it is more appropriate for the analyses of soil samples, especially for the determination of the clay-size fraction [10, 12, 19, 20, 21, 23]. As suggested by Özer et al. (2010) [23], values of 1.55 for refractive index and 0.1 for absorption coefficient (imaginary part of refractive index) were assigned for soil samples in the analyses. A value of 1.33 for refractive index was selected for suspending medium (in this case water) [25].

An MSX 17 sample preparation unit was used for preparation of the samples for laser diffraction analyses. The unit has an approximately 1-liter sample bath and comprises a variable-power ultrasonic system with a nominal frequency of 40 kHz [26]. The unit also has a variable speed pump and a stirrer. Continuously stirring mixer maintains homogeneous the suspension during analysis. Sonication is used to facilitate dispersion of the soil samples by breaking the inter-particle forces. The pump was used to circulate the suspension from sample bath to the measurement cell during analysis.

Tap water was used as a suspending medium in the analyses. Calgon (sodium hexametaphosphate) solution was used for dispersion of the soil samples. This solution was prepared according to standard procedure given in ASTM D 422 (2007) [27] for the hydrometer method.

38 soil samples taken from various location of Turkey were used in the analyses. These samples were taken from trial pits or borehole. Liquid limit of these samples are within the range of 29 to 114 (%), plastic limit 15 to 44 (%) and plasticity index 8 to 78 (%). Three of the samples are non-plastic (NP). Location of the samples in the plasticity chart was given in Figure 1. USCS type of the samples are CH (16 samples), MH (8 samples), CL (8 samples), ML (2 samples), SC (2 samples), and SM (2 samples).

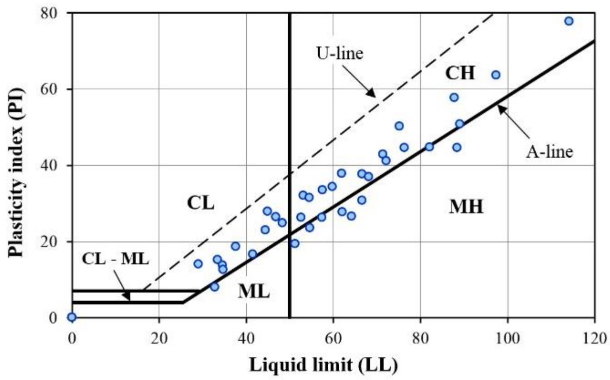

Figure 1. *Location of the samples in the plasticity chart*

2.2 Methods

Soil samples were dried in a 110 ± 5 ºC heated oven and crushed using a plastic hammer by avoiding particle breakage and then were sieved through No. 200 (0.075-mm) sieve, as described in ASTM D 422 (2007) [27]. Particles passing from No. 200 sieve were collected in a cylindrical container (typically 50 mm in diameter and 40 mm in deep) as suggested by Özer and Orhan (2007) [22]. An about 15-20 g of dry soils were collected passing from No. 200 sieve for laser diffraction analyses.

Flocculation or aggregation is occurs mainly in silt and clay particles. For this reason the samples retained in No. 200 sieve (sand and gravel fractions) were discarded from the analyses and only the samples passing from the No. 200 sieve where used in the study. Hence, it was aimed to determine the effectiveness of the dispersion methods on the silt and clay fractions.

The samples collected in cylindrical containers have not been dried again in the oven before analyses for two reasons: Dry weight of the samples is not necessary for the laser diffraction analyses because it uses volume of particles rather than weight and another reason is that the best sub-sample can be obtained from the clayey and silty soils in a damp state [10, 12, 22]. Silty and clayey soils can gain hygroscopic moisture from laboratory atmosphere during crushing and sieving procedures and this moisture content is generally sufficient for the damp state needed for sub-sampling. Before each sub-sampling from the cylindrical container, the samples with a damp state were mixed thoroughly with a small spatula in the container to obtain a homogeneous mixture, as suggested by Özer and Orhan (2007) [22]. Thereafter a small subsample was taken from the container for dispersion and thereafter for particle size analysis. The subsamples taken by this way can be sufficiently representing the whole sample in the container (Özer and Orhan, 2007) [22]. The following dispersion methods were applied to the samples before laser diffraction particle size analyses:

(i) Applying only ultrasonic action**:** Sample bath of the instrument was filled with tap water. Pump, stirrer and ultrasonic action was run and background measurement was taken. Soil sample to be analyzed (in a damp state) were mixed thoroughly with a small spatula in the cylindrical container, and then a small representative subsample was taken from the container as suggested by Özer and Orhan (2007) [22]. The subsample was poured to the sample bath little by little observing the obscuration ratio from the computer online. Amount of soil sample needed for analysis was determined by obscuration ratio and between 15-30 % was suggested by manufacturer of the instrument for lower and upper limits of this ratio [26]. Therefore, when obscuration ratio reached a value between 15-30 %, sample addition to the sample bath was stopped. Calgon solution was not added to the sample bath in this dispersion method. Thus, only ultrasonic action was used to disperse the soil samples. The duration of the ultrasonic action for proper dispersion varies from sample to sample. Sufficient time for proper dispersion can be assessed observing the inspection window of the software (v 1.2) during analysis as suggested by Özer (2006) [28]. The time between 3 to 5 min was sufficient for dispersion of the samples used in this study. After completion of the sample dispersion, analysis was run and measurement results was taken. Pump, stirrer and ultrasonic action was maintained in operation from start to completion of the analysis.

(ii) Using calgon solution without pre-treatment and applying ultrasonic action**:** Sample bath of the instrument was filled with tap water. Pump, stirrer and ultrasonic action was run and background measurement was taken. Soil sample to be analyzed (in a damp state) were mixed again thoroughly with a small spatula in the cylindrical container. A small representative subsample was taken from the cylindrical container and poured to the sample bath little by little observing the obscuration ratio from the computer. When obscuration ratio reached about 15 to 20 %, sample addition to the sample bath was stopped. Care was taken to not exceed 20% of the obscuration ratio because it may increases during dispersion process with time under the effect of ultrasonic action and calgon solution. Immediately after addition of the sample, 30 ml Calgon solution was poured to the sample bath. Pump, stirrer and ultrasonic action was kept in run from start to completion of the analysis. Therefore, dispersion of the sample was maintained with both Calgon solution and ultrasonic action in this dispersion method. Ultrasonic action time needed for dispersion of the sample was assessed by observing the inspection window of the software as suggested by Özer (2006) [28]. After completion of the sample dispersion, analysis was run and measurement results was taken.

(iii) Using calgon solution with pre-treatment and applying ultrasonic action**:** Soil sample to be analyzed (which is slightly damp state) were mixed again thoroughly with a small spatula in the cylindrical container. A small representative subsample was taken from the cylindrical container and placed to another smaller glass cylindrical container in a volume of about 50 ml. Amount of the subsample to maintain the obscuration ratio falling between 15 and 30% was predicted by experience. Covered the subsample with 30 ml Calgon solution and stirred with a small spoon or spatula until the subsample was thoroughly wetted. It was allowed to soak for at least 16 hours before analysis as suggested by ASTM D 422 (2007) [27] for hydrometer analysis. At the end of the soaking period, laser diffraction instrument was operated, sample bath of the instrument was filled with tap water and background measurement was taken after the pump, stirrer and ultrasonic action was run. Subsample was stirred again for a while to make a homogeneous solution and transferred to the sample bath. Any residue was washed from the glass container into the sample bath with water taken from sample bath by using a medium-size syringe.

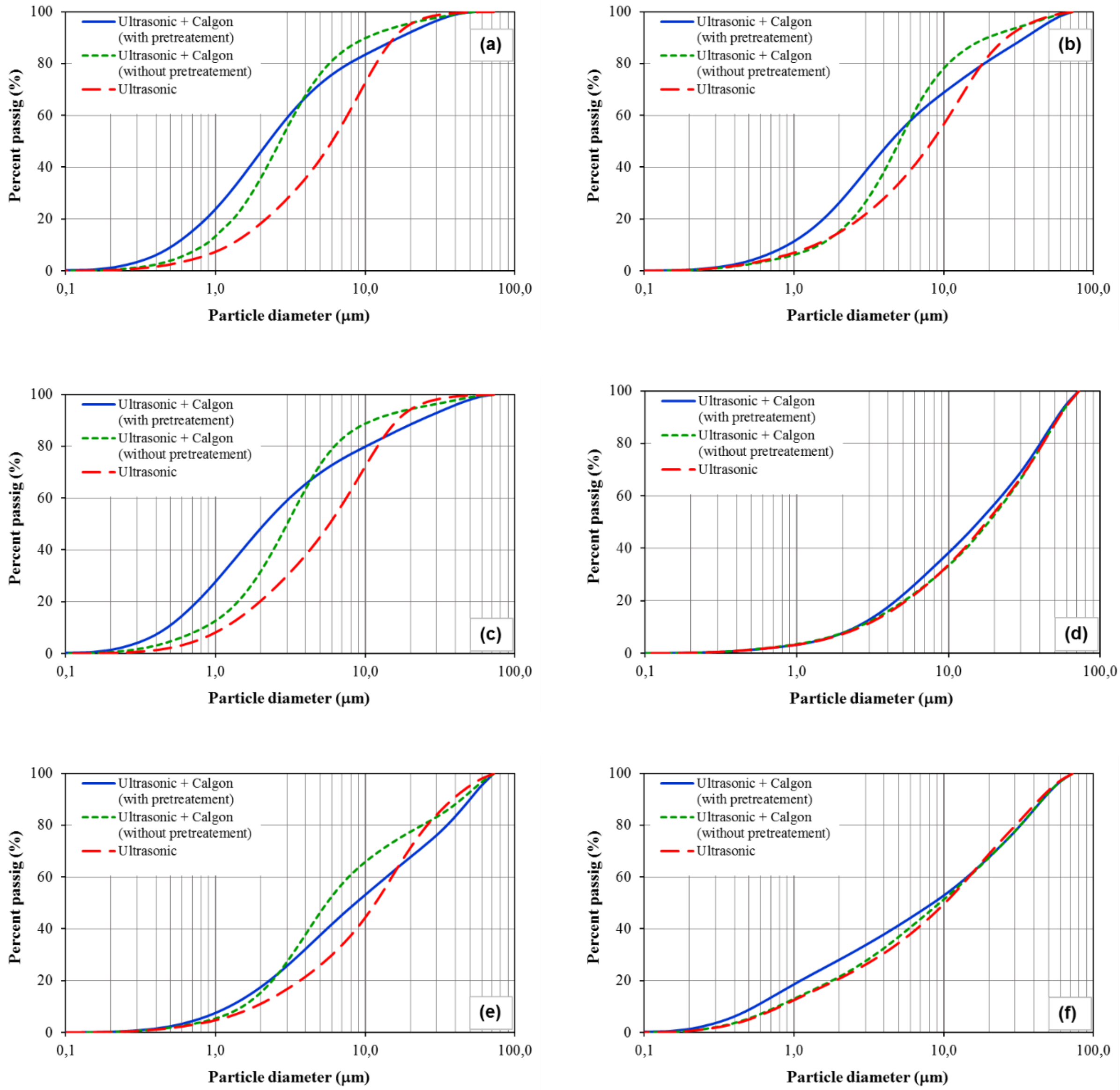

Figure 2. *Particle size distributions of a; (a) CH soil, (b) CL soil, (c) MH soil, (d) ML soil, (e) SC soil, and (f) SM soil*

Pump, stirrer and ultrasonic action was maintained in operation during analysis. Therefore also in these analysis, dispersion of the samples were maintained with both Calgon solution and ultrasonic action. After completion of the sample dispersion, analysis was run and measurement results was taken.

3. Results and Discussions

Because it is not possible to show the particle size distributions of all 38 samples here, one sample were selected from each USCS soil type and particle size distributions of them were presented in Fig.2. The results of the other remaining samples are in a form similar to those shown in Fig.2.

As can be shown in Fig.2, when using only ultrasonic action for dispersion (*method-i*), percent of the particles smaller than 15 to 20 μm are generally lower than those obtained from the other dispersion methods used in the study. For particles greater than about 15 to 20 μm, values of percent passing are higher than the other dispersion methods. When using calgon solution without pretreatment (*method-ii*) particle size distributions were shifted upward with some exceptions. This means that better dispersion was obtained from *method-ii* compared to *method-i*. Particle size distribution curves obtained by using ultrasonic action and calgon solution with pretreatment (*method-iii*) intersect with the curves obtained by *method-i* and *method-ii* at some points. The points of the intersection was vary from sample to sample. Upper part of the particle size distribution curves obtained by *method-iii* is located below the curves obtained by *method-i* and *method-ii*. On the other hand, lover part of the particle size distribution curves obtained by *method-iii* is located above the curves obtained by *method-i* and *method-ii*. This means that when calgon solution with pre-treatment and ultrasonic action is used, the volume of fine particles was increased and volume of coarse particles was decreased compared to *method-i* and *method-ii*. This is an indication of an effective dispersion. Namely; volume reduction of coarse particles may be caused by separation of fine particles from coarse particles. Consequently, fine particles leaving the coarse particles increasing the volume of fine particle while decreasing the volume of the coarse particles. They are the results of a suitable dispersion.

Another indication of an effective dispersion may be an increase in clay content. The clay contents (< 0,002 mm) obtained from three different dispersion methods is given in Fig. 3 for all of 38 soils samples used in the study.

As can be shown in Fig.3, the clay contents obtained using ultrasonic action and calgon solution with pretreatment (*method-iii*) are always higher than those obtained from the other dispersion methods (*method-i* and *method-ii*). The lowest clay contents were obtained in the case of using ultrasonic action only (*method-i*).

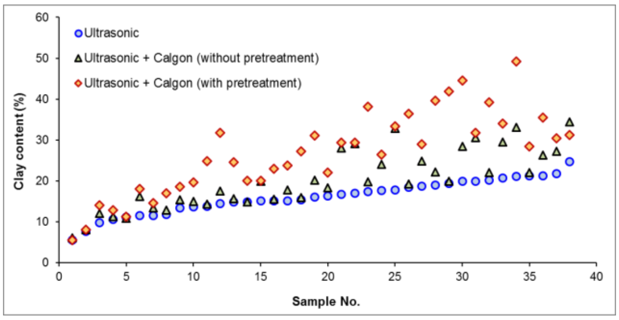

Figure 3. *Clay contents obtained from three different dispersion methods.*

4. Conclusions

Three methods were tested for dispersing the soil samples covering the broad range varying from CH to SC. These methods are (*i*) using ultrasonic action only, (*ii*) using ultrasonic action and calgon solution without pretreatment, and (*iii*) using ultrasonic action and calgon solution with pretreatment.

Of these methods, the best dispersion were obtained in the case of using ultrasonic action and calgon solution with pretreatment. As a result, it is suggested to soak the soil samples for at least 16 h in Calgon solution (typically 30 ml) prior to laser diffraction particle size analysis and to run the ultrasonic action during analysis.

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