Characterisation of soil aggregate stability by ultrasonic dispersion

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A b s t r a c t. Ultrasonic soil aggregate stability (USAS) of 5 European soils was investigated in ultrasonic dispersion tests, studying mass fractions of macroaggregates at different absorbed specific energy levels. The parameter %USAS $(1.95\ \mathrm{J\ ml^{-1}})$ was calculated using the mass fraction of macroaggregates (2000-200 m) at absorbed specific energy $1.95\ \mathrm{J\ ml^{-1}}$, the respective mass fraction prior to sonification and the sand fraction greater then 200 m after chemical dispersion. Soil aggregate stability (SAS) determined by the method of wet sieving (%SAS) served for comparison with the ultrasonic results. The same ranking of stability of the 5 soils was found using %SAS and %USAS$(1.95\ \mathrm{J\ ml^{-1}})$. %USAS$(1.95\ \mathrm{J\ ml^{-1}})$ was found more sensitive than %SAS for dividing relatively stable soils and less sensitive for dividing relatively unstable soils. The vibration amplitude used in ultrasonic dispersion tests influences the dynamic of soil dispersion. At higher vibration amplitudes, the disaggregation of soil particles needs less absorbed specific energy.

K e y w o r d s: ultrasonic dispersion, soil aggregate stability, soil particle dispersion

INTRODUCTION

Ultrasonic dispersion is a powerful method to disrupt soil aggregates into smaller aggregates and particles without chemical dispersion agents [5,7,10,24,33]. An ultrasonic probe is inserted into a mixture of soil and water, and acoustic pressure waves are emitted from the free end of the probe. Turbulent flow and cavitation cause friction, stressing and rupture of the soil aggregates. Aggregates and particles of different size may be subsequently fractionated (e.g., by sedimentation, sieving or centrifugation) and may be used for further physical or chemical analysis without prior chemical contamination.

With continuance of the ultrasonic treatment, the amount of sand- and silt-size material progressively decreases and the clay fraction increases [7]. The size fractions after different sonification times are correlated with the absorbed specific ultrasonic energy, i.e., the ultrasonic energy absorbed per unit mass of dry soil or the absorbed energy per unit volume of solution [22]. At large absorbed specific energies, the size distribution of soil particles was found similar to that obtained by chemical dispersion for some soils [7,14,30]. Other soils containing organo-mineral complexes [21] or coal or other particles from coal industry [31] showed a greater quantity of fine particles after long ultrasonic treatment which led to recommendations to limit the specific ultrasonic energy in dispersion experiments. Clay fraction in very stable soils, however, could not be completely dispersed with ultrasonic treatment but with a combined ultrasonic and chemical treatment [2,3].

North [22] was the first to consider the absorbed ultrasonic energy necessary for aggregate breakdown and soil dispersion as a measure for the stability of soil. In an energy balance consideration, the ultrasonic energy emitted into the solution is balanced by the heat generated in the mixture of soil and water, the loss of heat into the environment and the energy to disperse the soil aggregates and particles. Macroaggregates (> 250 m) are easily disrupted at low energy. More energy and prolonged ultrasonic treatment is necessary to disperse microaggregates consisting of complexes of

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clay, polyvalent metal cation, and organic matter into basic clay-metal-organic particles of < 2 μm diameter. North [23] assumed the dispersion process as 'primary' disaggregation of microaggregates into sub-complexes of approximately clay size (< 2 μm) followed by 'secondary' disaggregation of these sub-complexes into particles. Considering the weight fraction of particles < 2 μm, North defined a specific stability index of soil necessary for 'primary' disaggregation until the percentage of weight fraction < 2 μm was almost constant for prolonged ultrasonic treatment. The stability index, however, did not reflect soil management, and soils with stability considered commonly as problematic had similar or higher stability index than rarely problematic soil.

The most important parameter to describe the degree of aggregate breakdown and particle dispersion during sonification is the specific ultrasonic energy absorbed by the soil-water-mixture, and models of dispersion kinetics are mainly based on this parameter. Moen and Richardson [20] were the first to propose an exponential decrease of the undispersed clay with increasing time of ultrasonic exposure (i.e., increasing ultrasonic energy). Fuller and Goh [11] used a two-parameter model to correlate the remaining not dispersed clay (A) to the total aggregated clay (A_{max}) and the absorbed ultrasonic energy per millilitre solution (E):

$$A = A_{\max} e^{-\frac{\ln(2)}{E_{1/2}}E}. \qquad (1)$$

The parameter $E_{1/2}$ is the specific energy necessary to release 50% clay from soil aggregates. The 'stability constant' $E_{1/2}$ was successfully used to characterise the stability of different soils [11] as well as the influence of long-term cultivation on clay dispersibility [12]. Similarly, Levy *et al.* [18] used the percentage of undispersed clay after different absorbed ultrasonic energies to classify stable, semi-stable and unstable soils.

Raine and So [25] include the soil spontaneously dispersed on immersion, (c) and two regression constants (a and b) to correlate the relative fraction of dispersed material (D) to the specific ultrasonic energy, (E):

$$D = c + b\left(1 - e^{-aE}\right). \qquad (2)$$

They used ultrasonic dispersion experiments and high precision calorimetric measurements to determine the energy consumption for soil dispersion and found, that the dispersive energy measured in ultrasonic tests is significantly affected by the investigated soil [25]. Additionally, they compared the energy necessary to obtain mass fractions < 20 μm and < 2 μm in ultrasonic and end-over-end shaking experiments. These energies were comparable in several soils, whereas end-over-end shaking was less effective than ultrasonic dispersion in other soils disrupting silt sized material into individual clay particles [25,27].

Several authors consider that large aggregates disrupt at relatively low absorbed specific energies, whereas prolonged ultrasonic treatment and higher energies are necessary to disperse microaggregates [7,22]. Amelung and Zech [1] divided between the dispersion of macroaggregates (2000-250 μm) and microaggregates (250-20 μm) and found that macroaggregates disperse at relatively low absorbed ultrasonic energies, whereas more energy was needed to disperse microaggregates. Roscoe *et al.* [29] considered stable aggregates (100-2 μm fraction) and unstable aggregates (2000-100 μm fraction) and determined a critical energy of 260-275 J ml^{-1}, which was sufficient to disrupt most of the unstable aggregates and leave the stable aggregates. Tippkötter [32] correlated the ultrasonic dispersion behaviour of aggregates of 2000-60 μm to the stability of differently used land and different parent soil. Field and Minasny [9] described the process of ultrasonic dispersion as a stepwise process, where the disruption of aggregates 2000-20 μm is followed by aggregate liberation and the formation of sub-complexes (20-2 μm) and subsequent dispersion into discrete clay particles (< 2 μm). Initial increase of the 20-2 μm mass fraction due to disruption of aggregates was followed by decline of this fraction at larger absorbed energies, and a maximum was observed at certain 'critical' absorbed energy. Two parameters served to fit the 20-2 μm mass fraction and to characterise aggregate stability.

Imeson and Vis [15] compare aggregate stability measured by ultrasonic dispersion and water drop impact test and found that both procedures were consistent. For highly erodible soils they recommend water drop impact tests to determine aggregate stability, whereas low power ultrasonic dispersion gave better resolution investigating more stable soils. Similarly, Cerda *et al.* [4] compared dispersion at different ultrasonic power and constant sonification times to drop impact tests. Certain minimum ultrasonic power was required to disperse different soils depending on their stability, and this threshold power was correlated to rainfall simulation experiments.

The present investigation serves to compare soil aggregate stability determined with ultrasonic dispersion experiments to a standard technique to measure soil stability. The dynamics of soil disaggregation in the ultrasonic experiments has been used to divide stable from unstable soils. The literature survey shows, that prolonged ultrasonic treatment may completely disperse clay sized particles, which does not occur in the drop impact tests or even with prolonged end-over-end shaking [6,27]. Higher pressure has been imposed on the soil aggregates and particles in the ultrasonic tests, and smaller soil particles were affected and disrupted. To correlate soil stability determined with ultrasonic and standard testing procedure, disaggregation of macroaggregates served to characterise soil stability rather than dispersion of microaggregates and clay fraction.

Fraction of macroaggregates determined after certain absorbed specific energies served as a measure for aggregate stability and was compared to the standard technique of wet sieving to determine soil aggregate stability (SAS) proposed by Kemper [16]. Five different soils were investigated with both methods. Additionally, the influence of acoustic pressure was considered for the studying of ultrasonic dispersion at different vibration amplitudes of the ultrasonic probe.

MATERIAL AND METHODS

Investigated soils

Dispersion experiments have been performed with the following soils: Eurosoil 7, Ritzlhof, Nagyhorvati, Somogybabod and Tettofrati. Characterisation of these soils is shown in Table 1. The soils were air-dried and sieved (maximum diameter 2 mm). Before dispersion testing, the soils were pre-wetted at 60 hPa matrix potential at 24°C for 24 h in a pressure chamber.

Soil aggregate stability (SAS) according to DIN-Norm 19683-16

Soil aggregate stability of the investigated soils was characterised using the method of wet sieving described in DIN-Norm 19683-16. With this method, soil aggregates of diameter of 2000-1000 m were dipped on a sieve of 250 m. The soil mass used in the experiment, EW was 4 g. The mass of stabile aggregates after dipping, m_K and the mass of sand after chemical dispersion of the remaining aggregates, m_A is determined. These quantities are used to calculate the percentage of stable aggregates (%SAS):

$$\%SAS = \frac{m_K - m_A}{EW - m_A} 100. \qquad (3)$$

Ultrasonic dispersion

Ultrasonic dispersion experiments were performed with a probe-type dispersion equipment. The experimental setup is shown in principle in Fig. 1. A titanium alloy probe was inserted into the soil-water mixture and vibrated at approximately 20 kHz. The ultrasonic probe had a cylindrical shape and circular cross section (diameter 12.7 mm). The same ultrasonic probe was used in all experiments, and the insertion depth was kept constant at 10 mm.

Dispersion experiments were performed with 10 g soil in 200 ml pure degassed water. The solution was stirred with a magnetic stirring device (2 Hz, cylindrical shape with length 25 mm and thickness 8 mm). Stirring started 10 s prior to the ultrasonic vibration and was continued during the ultrasonic experiments to obtain homogeneous soil distribution in the solution.

All soils were tested at a constant vibration amplitude of the ultrasonic probe of 23 m. The vibration amplitude was determined using an electromagnetic induction coil and strain gauges as described previously [19]. Under the experimental conditions investigated, the ultrasonic power emitted into the suspension at vibration amplitude of 23 m was 26 W, which was determined with calorimetric measurements. Particle size analysis at different absorbed ultrasonic energies per millilitre served to monitor the progress of disaggregation in the ultrasonic field. The dispersion experiments were stopped after certain sonification times and the mass fractions were correlated to the absorbed ultrasonic energy per millilitre. A cooling device was used to prevent temperature increase and to keep the solution temperature at 20±2°C.

A Somogybabod soil was used to investigate the influence of vibration amplitude on the process of ultrasonic soil dispersion. Beside a vibration amplitude of 23 m, this soil was tested at vibration amplitudes of 10 and 42 m (ultrasonic power 10.5 and 49 W), respectively. Ultrasonic vibration amplitudes of 23 and 42 m were obtained using commercially available ultrasonic dispersion equipment (Bandelin Sonoplus HD 2200). Such equipment, however, is limited to vibration amplitudes greater or equal to 23 m, and ultrasonic equipment designed for the fatigue testing of materials was adapted to investigate soil dispersion at a vibration amplitude 10 m. For this purpose, a ultrasonic probe rather than a fatigue testing specimen was attached to the load train and stimulated to resonance vibrations.

T a b l e 1. Characterisation of the soils investigated according to WRB (World Reference Base for Soil Resources, 1998) and USDA (Keys to Soil Taxonomy US Department of Agronomy)

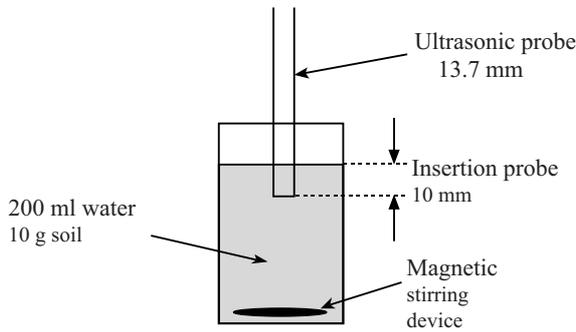| Country	Site	Soil type	Clay	Silt	Sand	$C_{org.}$	CEC $(cmol_Ckg^{-1})$
				(%, w/w)			
Austria	Eurosoil 7	Cambisol (WRB)	19	35	46	6.68	49.80
Austria	Ritzlhof	Cambisol (WRB)	23	60	17	1.27	20.78
Hungary	Nagyhorvati	Haplic Luvisol (WRB)	20	50	30	0.99	12.04
Hungary	Somogybabod	Calcaric Regosol (WRB)	45	45	10	0.87	18.40
Italy	Tettofrati	Typic Udifluvent (USDA)	12	61	27	1.10	12.45

Fig. 1. Principle of the probe-type ultrasonic dispersion experiments.

Particle size analysis

Mass fractions in the ultrasonic experiments are determined by wet sieving immediately after the various treatments. The entire sand fraction was analysed with standard sieves and classified in different aggregate and particle sizes: coarse sand (2000-630 m), medium sand (630-200 m), fine sand (200-63 m) and silt and clay (rest). Determination of mass fractions (accuracy 0.001 g) was performed after drying at 105°C for 24 h. All experiments were repeated 5 times.

A first series of experiments served to characterise soil particle distribution after the pre-treatment prior to sonification (pre-wetting and stirring for 10 s). Additionally, chemical dispersion experiments [13] were performed using 0.4 M tetra-sodium-diphosphate-decahydrate as a dispersion agent and head-over-head shaking (1 Hz, radius 40 mm) for 6 h at 20°C.

Ultrasonic soil aggregate stability (USAS) evaluation

Evaluation of aggregate stability in ultrasonic tests was performed using the mass fractions of coarse and medium sand (aggregates greater than 200 m), which was similar to the SAS method, where a sieve of 250 m was used. The fraction of stable aggregates at certain absorbed specific energy, E was used to calculate the percentage of stable aggregates, %USAS(E) similar to the SAS-method:

$$\% \, USAS(E) = \frac{m_U \, S(E) - m_S}{m_0 - m_S} \, 100 \, . \tag{4}$$

where: $m_{US}(E)$ is the fraction of coarse and medium sand at specific energy E, the mass fraction of coarse and medium sand determined by chemical dispersion is m_S, and m_0 is the mass fraction of aggregates > 200 m after the pre-treatment prior to starting the ultrasonic vibration.

Soil aggregate stability (SAS) according to DIN-Norm 19683-16

The results of the Soil Aggregate Stability (SAS) measurements according to DIN-Norm 19683-16 are presented in Fig. 2. The most stable soil is Eurosoil 7 with 37 % stable aggregates. Ritzlhof has 27% stable aggregates followed by the Hungarian soils Nagyhorvati (19%) and Somogybabod (18%). The least stable soil according to the SAS method is Tettofrati with 11% stable aggregates.
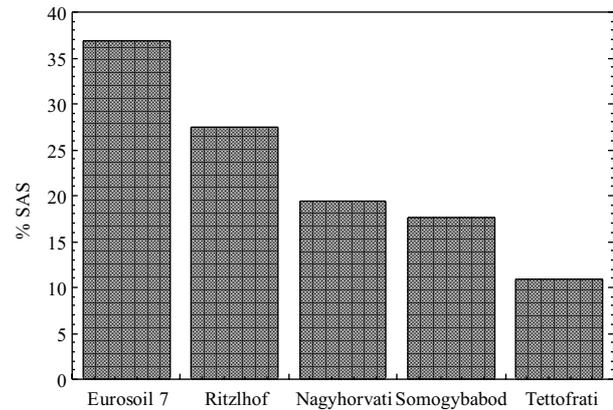

Fig. 2. Soil aggregate stability (SAS) according to DIN-norm 19683-16.

Influence of absorbed specific energy on dispersion

Mass fractions of soil particles at different absorbed ultrasonic energies per millilitre and vibration amplitude 23 m are shown in Fig. 3. All investigated soils showed a decrease of the entire sand fraction with an increasing absorbed specific energy. Considering the three sand fractions in more detail, the coarse sand fraction progressively decreases with a prolonged ultrasonic treatment in Eurosoil 7, which is the most stable soil according to the SAS-method. Coarse sand fraction in the other 4 soils are disaggregated already at absorbed specific energy of 0.65 J ml^{-1}, i.e., after 5 s of ultrasonic sonification. Medium sand is more stable, and decreasing mass fractions with increasing absorbed energies are found in all soils. Fine sand fraction of Eurosoil 7 increases at absorbed below approximately 30 J ml^{-1} and decreases at higher absorbed energies. This may result from aggregates of 2000-200 m which were disrupted into aggregates of fine sand size at low specific energy and disperse with prolonged ultrasonic treatment. In four less stable soils, disaggregation of fine sand fraction was observed already at low absorbed energy.

Figure 4 shows the influence of vibration amplitude on the Somogybabod soil dispersion. Sand fraction decreased with an increasing absorbed specific energy for all vibration
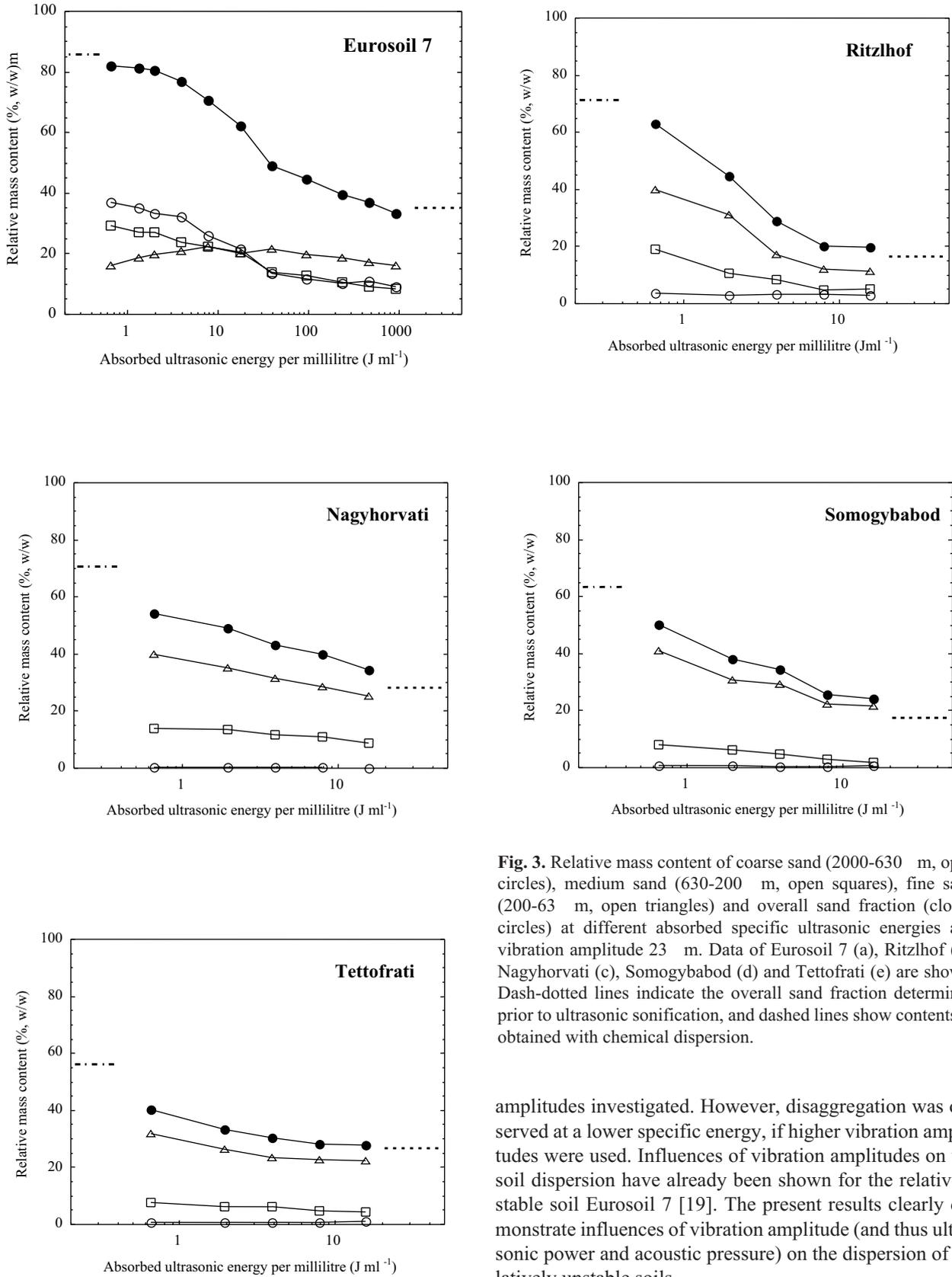
Fig. 3. Relative mass content of coarse sand (2000-630 m, open circles), medium sand (630-200 m, open squares), fine sand (200-63 m, open triangles) and overall sand fraction (closed circles) at different absorbed specific ultrasonic energies and vibration amplitude 23 m. Data of Eurosoil 7 (a), Ritzlhof (b), Nagyhorvati (c), Somogybabod (d) and Tettofrati (e) are shown. Dash-dotted lines indicate the overall sand fraction determined prior to ultrasonic sonification, and dashed lines show contents as obtained with chemical dispersion.

amplitudes investigated. However, disaggregation was observed at a lower specific energy, if higher vibration amplitudes were used. Influences of vibration amplitudes on the soil dispersion have already been shown for the relatively stable soil Eurosoil 7 [19]. The present results clearly demonstrate influences of vibration amplitude (and thus ultrasonic power and acoustic pressure) on the dispersion of relatively unstable soils.
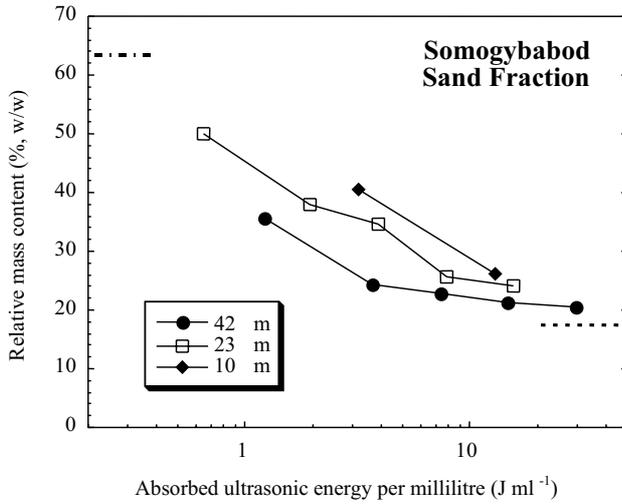
Fig. 4. Sand fraction (2000-20 m) of Somoygbabod soil at different absorbed specific ultrasonic energy at vibration amplitudes of 42, 23 and 10 m, respectively.

Accelerated particle disruption at higher vibration amplitudes is a consequence of higher acoustic pressure emitted into the soil water solution. Pressure waves cause cavitation, stressing of soil aggregates and breaking of aggregate bonds. Minimum acoustic pressure amplitude is necessary to stimulate cavitation, depending on the properties (temperature, purity, gas content, etc.) of the fluid [8, 28]. The acoustic pressure amplitude (p) depends on the density of the fluid, (ρ), the sound velocity in the fluid, (c) and the sound vibration velocity amplitude (v) [17]:

$$p = \rho \cdot c \cdot v. \qquad (5)$$

Using the vibration amplitude, (u) and the frequency of the ultrasonic vibration, (f), this may be rewritten:

$$p = 2\pi \cdot f \cdot \rho \cdot c \cdot u. \qquad (6)$$

Thus, the acoustic pressure increases linearly with the vibration amplitude. Forces on soil aggregates and particles are therefore greater at larger amplitudes. Disaggregation of soil particles is more rapid and affects more stabile aggregates at higher amplitudes, which may explain the observed influence of vibration amplitude on dispersion.

Ultrasonic soil aggregate Stability (USAS)

The ultrasonic soil aggregates stability determined at specific energy 1.95 J ml^{-1}, %USAS(1.95 J ml^{-1}) is shown in Fig. 5. The parameter %USAS(1.95 J ml^{-1}) well coincides to the SAS method showing that Eurosoil 7 is more stable than Ritzlhof being more stable than the Hungarian soils and the Italian soil. However, the parameter %USAS (1.95 J ml^{-1}) is less sensitive than the SAS method to divide relatively unstable soils. %SAS of Nagyhorvati and Somogybabod is 19 and 18%, and %SAS of Tettofrati is 11%. The parameter %USAS(1.95 J ml^{-1}) is 24, 22 and 18%, re-
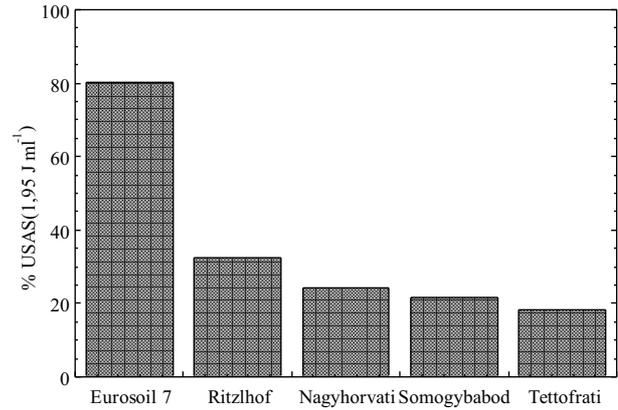

Fig. 5. Soil aggregate stability characterised with %USAS (1.95 J ml^{-1}).

spectively. This means that the SAS method clearly differentiates between the Italian and the two Hungarian soils, whereas %USAS(1.95 J ml^{-1}) suggests less difference in stability. On the other hand, %SAS of the relatively stable Eurosoil 7 and Ritzlhof is 37 and 27% whereas a more pronounced difference is found in the ultrasonic tests and %USAS (1.95 J ml^{-1}) is 80 and 33%, respectively.

The presented results show that %USAS (1.95 J ml^{-1}) is useful, in principle, to characterise soil aggregate stability. The evaluation is based on the absorbed specific energy of 1.95 J ml^{-1} which is used to diminish influences of possible slight differences in pre-treatment mainly affecting initial disruption of soil particles [26]. Higher absorbed specific energies decrease the mass fraction 2000-200 m which is used for the analysis. In the future work, further refinement of the experimental procedure is expected using smaller ultrasonic vibration amplitudes which reduce acoustic pressure and forces on soil aggregates and may lead to more pronounced differences characterising relatively unstable soils.

CONCLUSIONS

Soil aggregate stability can be characterised by the evaluating of the mass fraction of macroaggregates (2000 -200 m) in the ultrasonic dispersion tests. The parameter %USAS (1.95 J ml^{-1}) which is calculated using the mass fraction of non-disrupted aggregates at a specific energy of 1.95 J ml^{-1}, the mass fraction prior to sonification and the sand fraction greater than 200 m after chemical dispersion sug- gests similar ranking of the soils concerning their stability to the method of wet sieving (SAS). %USAS (1.95 J ml^{-1}) is more sensitive classifying relatively stable soils and less sensitive to characterise unstable soils than %SAS. An improved characterisation of the soil aggregate stability using ultrasonic dispersion technique may be attended varying the vibration amplitude and thus the forces acting on soil aggregates and particles.

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