

**Universität für Bodenkultur Wien**

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# **Methodological approaches for the determination of soil physical and physico-chemical parameters**

Master Thesis

submitted by  
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## Part B: Selected Publications

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- i MENTLER, A., STRAUSS, P., SCHOMAKERS, J., HANN, S., KÖLLENSBERGER, G., OTTNER, F. (2009): Organophilic clays as a tracer to determine erosion processes. Geophysical Research Abstracts 11
- ii SCHOMAKERS, J., MENTLER, A., STEURER, T., KLIK, A., MAYER, H. (2011): Characterisation of soil aggregate stability using low intensity ultrasonic vibrations. Int. Agrophysics. 25(2), 165-172
- iii SCHOMAKERS, J., MENTLER, A., DEGISCHER, N., BLUM, W.E.H., MAYER, H. (accepted, in review): Measurement of soil aggregate stability using low intensity ultrasonic vibration. Spanish J. Soil Sci. [publication late 2011]

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Part A

Outline Paper

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# 1 Introduction

Soil science has always had strong ties with agriculture and the science-based knowledge has very much contributed to the increase in agricultural production (Hartemink and McBratney, 2008). During the last century, agriculture as well as soil science were perceived as less important but with the rising demand for food, fodder and fuel, agriculture proves to be still fundamental to economic growth, poverty alleviation and environmental sustainability. The renewed interest in agriculture gave rise to an upturn in soil science (Hartemink and McBratney, 2008; Deininger *et al.*, 2011 ).

Between 1961-2007, the area of cultivated land had expanded at some 3.8 million ha per year globally which culminated in a total area of 1 554 million ha in 2007. An increase was listed for developing countries while industrialised and transitional ones registered a slight decline (Deininger *et al.*, 2011 ). Advances in productivity, especially the development of land-saving technology, prevented more ground from being cultivated. Therefore 70 percent of the increase in crop production between 1961 and 2005 was due to yield increases, 23 percent to the expansion of farmland and eight percent to the intensification of tillage (Bruinsma, 2009).

While agriculture has been the initial driver of the discipline of soil science, soil science nowadays also deals with research questions on climate change, environmental regulation and ecosystem services. Studies showed that agriculture leads to changes in soil properties such as nutrient status, pH, organic matter content and physical characteristics (e.g. Powlson *et al.*, 2011). The increasing demand on soil resources threatens not only agricultural productivity but also soil fertility and the problem is tightened by soil erosion. An estimated 35 ( $\pm 10$ ) billion tons of soil are eroded annually around the world. Of those,  $28 \cdot 10^9 \text{ t a}^{-1}$  of soil are mobilized by water,  $5 \cdot 10^9 \text{ t a}^{-1}$  by tillage operations and  $2 \cdot 10^9 \text{ t a}^{-1}$  of sediment are mobilized by wind (Quinton *et al.*). When erosion on agricultural land occurs, the runoff removes topsoil, nutrients, pesticides and organic materials and may carry them to water bodies where they become pollutants. The increasing demand on the finite resource soil and the decreasing per capita arable land area in densely populated regions of the world made soil erosion a global issue with regard to its on and offsite impacts (Lal *et al.*, 2007).

It has to be noted that soil erosion is a natural process caused by wind, water and ice that wear away the material on the land surface very slowly. But the rate of erosion depends on a variety of factors, including climate, lithology, topography, soil type and vegetation cover. It can be greatly exacerbated by a wide variety of human activities, including poor farming or grazing methods, deforestation and urbanization. The average rate of soil loss from global farmlands was estimated at  $6 \text{ t ha}^{-1} \text{ a}^{-1}$  (Wilkinson and McElroy, 2007), compared to a regeneration rate of  $10^{-2}$  to  $10^0 \text{ t ha}^{-1} \text{ a}^{-1}$  (Scheffer and Schachtschabel, 2010)<sup>1</sup>, which shows that soil erosion accelerates well beyond the pace of soil production.

Accelerated erosion is listed as one of the causes of soil degradation, other threats are carbon loss and nutrient depletion, soil compaction, acidification, pollution and salinisation. Studies by the UN and other international organisations showed soil erosion, nutrient depletion and pollution are key issues in regard to environmental degradation, climate change and world-food production. Research into soil degradation has hence had the biggest impact of all facets of soil science in both the popular press as well as the scientific community (Hartemink and McBratney, 2008).

Soil erosion is the severest problem of soil degradation on the European continent with an estimated cost of € 38 billion per year. It affects about 157 million ha of arable land in Europe

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<sup>1</sup> For granular soils (Lockergestein) the average regeneration rate is  $10^{-1}$  to  $10^0 \text{ t ha}^{-1} \text{ a}^{-1}$  and for hard rock (Festgestein) the average regeneration rate is  $10^{-2}$  to  $10^{-1} \text{ t ha}^{-1} \text{ a}^{-1}$ .

i.e. 16 % of the total arable area (Klik, 2003). In Austria, 1.37 million ha are under agricultural use and an estimated 450.000 ha soil are at risk of erosion (Rosner and Klik, 2005).

Maintaining sustainable soil resources or preserving soil resources for future generations is receiving more and global attention in scientific communities, by policymakers, and even in the agricultural community (Amézketa, 1999). Scientists have mainly focused on technical, “applied” studies with a clearly defined focus on a specific aspect of the soil. Fewer efforts are made to contribute to the general knowledge of soils, their components and relationships. Although soil science is more and more incorporated in multidisciplinary questions dealing with i.a. climate change or environmental degradation, the development of fundamental research “*What is soil?*” is of vital importance (Hartemink and McBratney, 2008; Churchman, 2010). Churchman (2010) showed that there are three aspects of soil science, which are unique to the discipline of soil science: i) the formation and properties of soil horizons, (ii) the occurrence and properties of aggregates in soil, and (iii) the occurrence and behaviour of soil colloids.

The aim of my work was to develop reliable methodologies to a) measure soil aggregate stability and b) to follow erosion-determined particle transport at soil surface level. The scientific approach differed greatly, as well as the respective application; still the research spectrum is indirectly connected, as aggregate stability is an influential factor governing soil erodibility. A decrease in aggregation and aggregate strength increases soil’s vulnerability to erosion, and therefore soil erodibility (Mahboubi and Lal, 1998).

Emphasis was put on method development, but the papers collected in this treatise can document some of my work on application with a focus on agricultural soils.

I like to contribute to efforts made to better understand changes in soil’s physico-chemical properties. On the one hand, I aim to appreciate the processes involved, on the other hand to offer methods to measure, meliorate or estimate human-induced changes in soil.

With this introductory chapter, I intend to accomplish two things: First, I would like to give an overview over the status quo of alternative tillage systems in Europe and research advances focusing on soil aggregates. Second, and concurrently, I would like to introduce recent studies on tracing techniques to evaluate soil translocation. For each of the discussed topics I set out how my own work links into the research advances and what its innovative contributions are.<sup>2</sup>

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<sup>2</sup> As chapter 2 is structured along different methods, I will not provide paper-by paper summaries of my writings but I will rather report on the relevant interesting findings at suitable points. The articles and the abstract compiled in Part B are referenced by Roman numerals (i.e., Abstract I, Articles I & II). Abstract I will be presented in greater detail than the articles to provide a more comprehensive picture of my work.

## 2 Adopting conservation tillage in Europe: a review

Economic and environmental pressures have led to tillage systems that minimise the costs associated with cultivation and ensure the timely sowing of crops. Conservation, i.e. non-inversion, reduced (RT), minimal or no tillage (NT) includes systems that involve fewer passes than conventional tillage (CT). The main objectives for conservation tillage are erosion control (Holland, 2004) and the limitation of mechanical disturbance of the soil to that required for seed placement (Morris *et al.*, 2010).

Today, Argentina is the leading country in adopting conservation agriculture with almost 20 million ha under no tillage. An increase in knowledge but especially the availability of machinery and herbicides accelerated the distribution of the new management system. In 2009, no-tillage farming was adopted on about 116 million ha worldwide (Tab. 1), corresponding to a growth rate of six million ha per year (Derpsch *et al.*, 2010).

Tab.1: Area under no-tillage by continent (Derpsch *et al.*, 2010)

Continent	Area (ha)	Percent of total (%)
South America	49 579 000	46.8
North America	40 074 000	37.8
Australia & New Zealand	17 162 000	11.5
Asia	2 530 000	2.3
Europe	1 150 000	1.1
Africa	368 000	0.3
World total	115 863 000	100

Europe ranges at the lower end of the table with only about one percent of the total area being under NT-management (Tab. 1). The main reason for the reluctance to adopt conservation agriculture is the fear of declining yields (Derpsch *et al.*, 2010). Results in literature proved to be contradictory though. Jones *et al.* (2006) concluded that crops established under non-inversion tillage yield slightly lower than those established after ploughing. Cannell (1985) argued that with adequate weed control, reduced tillage is as good as conventional tillage at the traditional depth on a wide range of European soil types. In northern Europe however, inversion tillage is often the most appropriate cultivation technique since it allows the infiltration of rainfall in autumn (Holland, 2004). Derpsch (2008) compared NT and CT systems in Brazil and Paraguay and found a considerable yield increase for the NT systems over a period of ten to fifteen years.

Another frequently voiced European objection against non inversion tillage systems is the reliance and the increased use of herbicides and other chemicals for disease and pest control during conservation management (Basch, 2005). Recent EU policy directives, e.g. the Water Framework Directive, raise concerns of particular herbicides becoming increasingly widespread in water drinking supplies (Morris *et al.*, 2010). Yet, Holland (2004) alludes to the lack of adequate research into environmental implications of conservation tillage in Europe. Derpsch (2008) who studied tillage in South America found an average decline in the use of herbicides of 30-50 % in the conservation systems of Paraguay over a period of ten years.

In Europe, the environmental consequences of farm management have indeed been widely ignored since on site impact is low compared to the off site problems like air and water pollution. In 2002, a thematic strategy on the protection of soil was started and adopted by the European commission in 2006. It aims to give soil the same importance as water and air but has failed to be adopted by the European Parliament so far (European Commission, 2010). In addition, Holland (2004) showed that farmers are less likely to consider soil erosion if it is less noticeably. This again coincides with results from the Austrian research project

GERAST<sup>3</sup> which provided funds for abstract I (cp. pages 5f.). Farmers were offered a closure premium for riparian agricultural zones to prevent farmland erosion affecting water bodies. Only 0.08 percent of eligible farms in three Austrian provinces took part. Of those who participated, over 50 percent did so because of environmental concerns (WPA, 2009).

Another reason for the slow adoption of conservation management is the moderate climate in the majority of European countries. Natural disasters are less frequent and do not demand urgent action in the agricultural sector. Also, the economic pressure on European farmers is less severe compared to their South American counterparts since EU policies support farmers with direct payments and subsidies for certain commodities. In consequence cost saving and adoption of alternative cultivation methods are rather discouraged. In addition, there are influential lobbying groups opposing the introduction (Derpsch *et al.*, 2010). Derpsch *et al.* (2010) showed that it is to some extent difficult for European farmers to obtain machinery for conservation management as opposed to equipment used in conventional farming.

### Agri-environmental programme ÖPUL in Austria

In 2007, the Austrian Federal Ministry of Agriculture, Forestry, Environment and Water Management responded to EU-regulation (EC) No. 1698/2005 and initiated the Agri-Environmental Programme ÖPUL<sup>4</sup>, a successor of ÖPUL 2000. The initials are short for the Austrian programme for the promotion of agriculture that is extensive, appropriate to the environment and protective of nature. The programme is embedded in the Rural Development Programme 2007-2013. The objective is “to ensure that environmental concerns of domestic agriculture are taken account of. Priorities are above all the protection of soil and waters as well as the maintenance of biological diversity.” Farmers participate voluntarily in the 30 measures and are compensated for deficit harvests or emerging expenses. In 2006, 75.2 % of all farms with agriculturally used land participated in the programme (Lebensministerium, 2008).

The ÖPUL measure “Protection and enhancement of ecologically valuable zones which are significant for water protection”<sup>5</sup> promotes the set-aside of agricultural land and the near to nature management of fields endangered by erosion. The measure promotes, for example, the adoption of vegetative buffer zones alongside agriculturally used land to protect adjacent water bodies from farmland pollution.

### Vegetative buffer strips

Vegetative buffers are strips of grass, shrubs and trees planted at the edge of fields or along streams, ditches, wetlands or other water bodies. They are designed to slow terrestrial inputs of water, trap sediments, filter nutrients and pesticides and provide habitat and corridors for fish and wildlife. Riparian buffers (fig. 1) between agricultural fields and streams play an important role in controlling the impacts of land use management on water quality and aquatic ecosystems (Yuan *et al.*, 2009). Klik (2003) called sediment depositions in streams due to soil erosion the gravest setback for water quality. Yuan *et al.* (2009) conducted a literature research and concluded that buffer strips significantly reduce sediment loading in surface runoff from agricultural fields although their effectiveness varied widely. Buffer width,

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<sup>3</sup> Effektivität von Gewässerrandstreifen zum Schutz von Oberflächengewässern

<sup>4</sup> Österreichisches Programm zur Förderung einer umweltgerechten, extensiven und den natürlichen Lebensraum schützenden Landwirtschaft

<sup>5</sup> Erhaltung und Entwicklung naturschutzfachlich wertvoller oder gewässerschutzfachlich bedeutsamer Flächen



vegetation type, density and spacing, sediment particle size, slope gradient and length, and flow convergence define primarily the trapping efficiency.

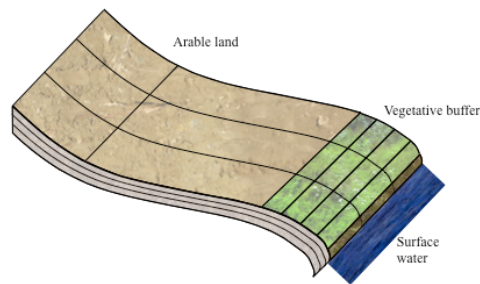


Fig. 1: Schematic display of the position of a riparian buffer zone alongside an agricultural field  
(Source: WPA, unpublished)

In general, wider buffer trap more sediment. Slope is an important factor, but findings about the overall relationship were not consistent. Grass and forest buffer strips are more or less equally efficient.

The work of abstract I was part of the two-year running research project “GERAST-Efficiency of vegetative buffer zones for the protection of surface water.” GERAST investigated the effectiveness of vegetative buffer zones in regard to necessary dimension, possible retention capacity and the acceptance of stakeholders on selected sites in the Austrian provinces of Styria, Lower and Upper Austria. The joint research project was conducted by

- i) the University of Natural Resources and Life Sciences - Institute for Soil Science,
- ii) WPA Beratende Ingenieure GmbH and
- iii) the Federal Agency for Water Management- Institute for Land & Water Management Research.

It was financed by the Ministry of Agriculture, Forestry, Environment and Water Management as well as the three provinces involved (WPA, 2009). Next to established erosion indicators and tools to model soil erosion, the organic tracer method was developed as a novel tool for the evaluation of the impact of vegetative buffer zones on farmland erosion.

### 3 Assessing soil aggregate stability

A priority for soil research is to provide the basis for agricultural management practices that avoid irrevocable damage to soil and its functions (Powlson *et al.*, 2011). Soil organic matter (SOM) dynamics and nutrient cycling need to be better understood and subsequently managed for achieving long-term sustainability and reduction of environmental costs of agricultural ecosystems (Six *et al.*, 2004). SOM consists of organic material, living organisms and humus. It is constantly built up and decomposed. Yet, it has long been shown that organic matter erodes from arable land to rivers disproportionately to its availability (Walling, 1990). The amount of SOM returned to soil has also declined in recent years, primarily as a consequence of more intensive tillage, the removal of crops residues, the replacement of organic manures with inorganic fertilizers, and the loss of ley from rotation (Holland, 2004).

Aggregate measurements are often used as surrogates of the complex soil matrix in an effort to advance the understanding of the dynamic soil properties. Aggregates physically protect SOM, influence microbial community structures, limit oxygen diffusion, regulate water flow, determine nutrient adsorption and desorption and reduce runoff and soil erosion. All of these processes have profound effect on SOM dynamics and nutrient cycling (Six *et al.*, 2004).

Number and size of aggregates in soil change as a result of natural and anthropogenic forces that affect the build up and breakdown of these structural units. This fact has been confirmed by numerous studies of the influence of temperature, moisture, soil organisms, tillage, traffic, organic matter, lime, fertilization, raindrop impact, plant roots and cropping systems on the number, size and stability of soil aggregates (e.g. Tisdall and Oades, 1982; Carter, 1992; Angers and Carter, 1996; Daraghmeh *et al.*, 2009).

The formation of soil aggregates is attended by different binding agents, which act at different hierarchical stages. Microaggregates (20-250  $\mu\text{m}$ ) are bound together by persistent binding agents (i.e. humified organic matter), oxides and highly disordered aluminosilicates. Macroaggregates (> 250  $\mu\text{m}$ ) consist of stable microaggregates, bonded together by temporary (i.e. fungal hyphae) and transient (i.e. microbial-derived polysaccharides) binding agents. These temporary agents form the nucleus for microaggregate formation in the centre of macroaggregates (Six *et al.*, 2004). Larger aggregates (> 250  $\mu\text{m}$ ) have greater porosity than smaller aggregates and in consequence macroaggregates composed of microaggregates can be more easily fractured along wider pores and cracks (Dexter, 1988).

Macroaggregation shows great seasonal dynamics (Mahboubi and Lal, 1998; Scheffer and Schachtschabel, 2010) and the mean residence time of macroaggregates has been estimated to average 27 days (Plante *et al.*, 2002). Six *et al.* (2004) called the aggregate hierarchy concept proposed by Tisdall and Oades (1982) and later modified by Oades (1984) the probably most significant theoretical advancement in the understanding of aggregate - SOM interactions<sup>6</sup>. Because of the hierarchical order of aggregates and their binding agents, microaggregate stability is higher and less dependent on agricultural management, such as crop rotation, than macroaggregate stability.

According to the hierarchy theory, articles I & II investigate macroaggregate stability of a Cambisol and a Chernozem soil under the influence of no tillage, reduced tillage and conventional mouldboard tillage.

Macro- and microaggregates are released by mechanical fractionation. Form and level of energy input has a significant influence on the properties and size distribution of the aggregates (Ashman *et al.*, 2009). In general, soil aggregate stability (SAS) can be determined by various established destruction mechanisms: 1) slaking of air-dried soil

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<sup>6</sup> The aggregate theory is applied for soils with 2:1 minerals where SOM forms bridges between negatively charged clay minerals, while in oxide dominated soils, electrostatic interactions between clay minerals and oxides dominate (Oades and Waters, 1991; Six *et al.*, 2004).

followed by wet-sieving (water stability tests), 2) wet-sieving of capillary re-wetted or field-moist soil, 3) dry sieving of soil, 4) end-over-end shaking followed by wet-sieving and 5) ultrasonic dispersion. Advantages and disadvantages of the various methods have been discussed widely (e.g. Amézketa, 1999; Ashman *et al.*, 2009). One main obstacle of imposing different, often arbitrary, forms of energy inputs to soil, is trying to compare results in literature. These can be contradictory or merely impossible to weigh against each other. Articles II & III evaluated the stability of aggregates using the USAS (ultrasound) method after Mayer *et al.* (2002) and the SAS (wet sieving) method after Kemper and Rosenau (1986). The two studies discuss the advantages and further the use of ultrasonic dispersion for aggregate measurements and add to previous and ongoing research at the University of Natural Resources and Life Sciences, Vienna in that particular field (e.g. Mayer *et al.*, 2002; Spiegel and Mentler, 2003; Mentler *et al.*, 2004; Kasper *et al.*, 2009; An *et al.*, 2010).

Ultrasonic dispersion allows for exactly quantifying the applied energy. The breakdown of aggregates during sonication is caused by cavitation, which occurs mainly on and between the surfaces of aggregates (Tippkötter, 1994). Since Esche (1952) hardly any investigations were made to locate the minimum acoustic pressure levels, which cause cavitation in water. Article I describes the steps taken in quantifying the cavitation threshold. Thereafter, it was possible to work at very low energy levels to still cause fragmentation of aggregates and to also show differences in the dispersion behaviour of the investigated agricultural soils. This proved to be especially valuable for weakly aggregated soils where the USAS method allowed for quantifying differences in aggregate stability for the three tillage systems. Conventional tillage reduced aggregate stability, notably especially in comparison to no tillage system samples. This decrease can be linked to a decrease in soil organic matter (Tisdall and Oades, 1980). A greater SOM content in conservation tillage systems is assumed to be the reason for greater aggregate stability, which can better withstand rapid wetting and mechanical forces and hence protect the soil from erosion forces (Kasper *et al.*, 2009).

It has been shown that arable soils require far lower energy inputs than grassland soils to cause disaggregation, suggesting lower stability (Ashman *et al.*, 2009). Articles I and II show accordingly that the macroaggregates of the investigated agricultural soils disperse quickly at low energy levels ( $1-2 \text{ J ml}^{-1}$ ) whereas for the extraction of dissolved organic carbon (DOC) the applied energy increased to levels above  $300 \text{ J ml}^{-1}$  (article I). DOC results from microbial processes and decomposition processes contribute to the loss of DOC (Kaiser *et al.*, 2001).

The relation between aggregation and organic carbon was shown early by Oades and Waters (1991) who stated that the binding effects for macroaggregates will disappear at the same rate as the decomposition of the root system. Article I evaluates the influence of no tillage, reduced tillage and conventional tillage on the carbon pool of the two soil types. Both soil samples, Pixendorf Chernozem and Tulln Cambisol, show overall lower total organic carbon (TOC) values for the ploughed tillage variants compared to the NT samples. The influence of ploughing on carbon pools can also be seen from DOC results, which support the conclusion that intensive tillage has a negative effect on soil organic matter dynamics. But results also reveal that the differences in TOC diminished with decreasing aggregate size. This again confirms conclusion of the aggregate hierarchy theory that small aggregates are independent of management.

Managing soil organic carbon is important because SOM influences numerous soil properties relevant to ecosystem functioning and crop growth. Even small changes in total C content can have disproportionately large impacts on key soil physical properties. Practices to encourage maintenance of soil C are important for ensuring sustainability of all soil functions (Powlson *et al.*, 2011). Articles I & II support other studies (Mahboubi and Lal, 1998; Spiegel and Mentler, 2003; Holland, 2004; Kasper *et al.*, 2009) that intensive tillage reduces aggregate stability and the amount of carbon that contribute to soil quality and the long-term sustainability of agriculture.

## 4 Organophilic clays as tracers to determine soil erosion processes

Sediment delivery from erosion plots has been well monitored. These data are essential in developing erosion prediction models, understanding erosion principles, assessing the on- and offsite impacts and deriving best management practices to combat soil erosion. But for the validation of physically based erosion models, e.g. Water Erosion Prediction Project WEPP or Erosion 3D, and for understanding soil erosion dynamics at process level, it is necessary to gather information on soil redistribution patterns within an erosion plot (Zhang *et al.*, 2001). Laveuf and Cornu (2009), for example, traced pedogenic processes in order to quantify the respective contribution of the successive processes on pedogenesis.

To obtain spatially distributed erosion data various types of tracers are in use which all have advantages and setbacks (Zhang *et al.*, 2001). The combination of several physical and chemical properties of sediment is called the fingerprinting technique to improve the reliability of estimates of sediment source areas:

Atomic bomb fallout radionuclide  $^{137}\text{Cs}$  (half life of 30 years) is one of the most widely used tracer for estimating long term erosion rates and erosion patterns (normally 30 years). But indeterminate input functions and indefinite spatial and depth distributions of the original deposition interfere with provision of reliable quantitative estimates. Moreover it is nearly impossible to trace the sediment source since uniform spatial distribution is always assumed.

Naturally occurring radionuclide  $^{210}\text{Pb}$  (half life of 20.2 years),  $^7\text{Be}$  (half life of 53 days) and  $^{234}\text{Th}$  (half life of 24.1 days) enter the soil via precipitation.  $^{210}\text{Pb}$  and  $^7\text{Be}$  have different depth distributions and hence a potential to identify predominant processes such as interill or rill erosion. But the continuous input as well as the unknown distribution pattern do not allow for reliable estimates, either.

To overcome these drawbacks, other radioactive ions, such as  $^{56}\text{Fe}$  (stable) or  $^{60}\text{Co}$  (half life 5.3 years) have been deliberately introduced to study surface movement. On the downside, the researcher has to hazard the consequences of distributing radioactive material into the environment.

Another, time-consuming option are noble metals-labelled natural particles. Only during measurement the metals e.g. Au, Ag or Ir are activated by neutron irradiation i.e. heating. The heating allows, on the one hand, the labelling elements to diffuse into sediment crystalline lattice. On the other hand it may alter soil chemical compositions.

Exotic particles such as glass beads, fluorescent dye coated particles, magnetic plastic beads or steel nuts overcome certain drawbacks of other tracers mentioned but morphology, distribution, transport mechanisms and analysis differ fundamentally from soil particles and to draw the line to soil erosion rates and patterns is rather difficult if not impossible.

Ideal tracers occur in soils in very low background concentration, bind strongly with soil particles or be easily incorporated into soil aggregates, do not interfere with sediment transportation, have great sensitivity in analysis and are easy and inexpensive to measure. The adsorption of the tracer by a plant must be of a very low order of magnitude and the tracer should not be toxic to biota (Zhang *et al.*, 2001).

Rare Earth Elements (REEs) are members of the group IIIB in the periodic table and defined as the 14 naturally occurring elements of the lanthanide series (La to Lu). They occur in most primary and secondary minerals in the soil and have similar chemical properties. In addition they are deposited through the atmosphere or anthropogenic activities i.e. waste, fertilizers. REEs have a high affinity towards P-compounds and make P-fertilizers a relevant

anthropogenic source of REE in soils (Laveuf and Cornu, 2009) and REE-oxides have been successfully used as multi-sediment tracer (Zhang *et al.*, 2003; Laveuf and Cornu, 2009; Polyakov *et al.*, 2009). REE-oxides are incorporated in soil aggregates (> 53 µm) and are relatively easy and accurately detected with inductively coupled plasma mass spectrometry (ICP-MS). Jarvis (1989) worked with a detection limit better than 1 µg kg<sup>-1</sup>.

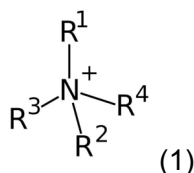
Abstract I offers a new method for tracing pedogenic movements. The organic tracer is a commercially available organophilic bentonite, Tixogel VZ (Southern Clay Products, Inc., Gonzales, TX 78629). For the production of the Tixogel VZ, the inorganic cations of natural bentonites are substituted by quaternary ammonium cations (quats) of different chain length. The quats compounds are strongly fixed between the clay layers.

Clay particles are easy to incorporate to soils and they move with soil aggregates without interference with the sediment transport and without plant uptake. The n-alkyl ammonium compounds have no background concentration in soil and are easily and accurately detected with ICP-MS with a detection limit of 0.3 µg L<sup>-1</sup>. Environmental concerns of pure quats do exist (Uhl *et al.*, 2005) but there are no restrictions on the use of bentonites according to REACH<sup>7</sup> Regulation EG 1907/2006, Regulation EG 1272/2008 and Regulation (EU) 453/2010 (EUBA, 2010).

## 4.1 Experimental

In the following the experimental steps to identify the quats within the bentonite's layers are retraced and summed up to give a brief overview over the methodology applied and the results gained. It was necessary to develop this methodology since the production company refused to disclose the composition of the ammonium compounds. Terminatory, the indoor rainfall experiment will be outlined.

### Materials



Quaternary ammonium compounds are organic ammonium compounds; all four valences are organically bound to a nitrogen atom. Quats are cationic tensides with surface-active substances. The molecule consists of a hydrophobic alkyl chain (C<sub>16</sub>-C<sub>18</sub>) and a hydrophilic group. The general chemical formula is: C<sub>n</sub>H<sub>2n+1</sub>NH<sub>3</sub><sup>+</sup>.

### Desorption procedure of alkyl ammonium cations

At first a liquid extraction method had been developed to extract the n-alkyl ammonium compounds from the inter crystal layers of the bentonite. The organic extraction solvent used was methanol and 0.1M barium chloride (90:10). The decrease of the alkyl ammonium compounds was measured through the reduction of carbon and nitrogen. The results were verified by X-ray diffraction and resulted in a recovery rate of about 85 % ammonium compound (Schomakers, 2008).

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<sup>7</sup> Registration, Evaluation, Authorization and Restrictions of Chemicals

### External standard

10 g Tixogel VZ was extracted (methanol and 0.1M barium chloride, ratio 90:10), centrifuged and filtered. The solution was evaporated using a rotavapor (40°C, -400 mbar). The flask was cooled down to -20°C and the residue was removed and freeze-dried for 24 hours (-50°C, 0.04mbar). The mass of the external standard was corrected with the mass of BaCl<sub>2</sub>, which had not been removed during evaporation. The external standard, dissolved in acetonitrile, was used for quantifying the four different alkyl ammonium cations from the Tixogel VZ.

### TOF-MS and HPLC-QQQ-MS analysis

Two different mass spectrometry (MS) systems were used for method optimization tests: Agilent 6220 TOF (time-of-flight) MS and Agilent 6410 Triple Quad (QQQ) LC/MS both coupled with High Performance Liquid Chromatography, Agilent 1200 SL HPLC. The advantages of TOF-MS are high ion-mass resolution and a very high admission speed of up to 10 000 amu per seconds. The high ion-transmission leads to a high limit of detection and TOF-MS can quickly generate full mass spectra. HPLC-MS, on the other hand, is routinely in use and allows for accurate identification and quantification of organic substances.

HPLC-TOF-MS was performed using a C<sub>18</sub> reversed phase column (Zorbax, 20x2mm). HPLC instrument settings included a flow rate of 0.5 mL·min<sup>-1</sup> (isocratic flow) with 90 % acetonitrile and 10% 0.1 % formic acid in water and a run time of seven minutes. The linear working range was 25 to 1000 µg·L<sup>-1</sup> with an injection volume of 1 µl.

HPLC triple quad-MS was performed using a C<sub>8</sub> reversed phase column (Zorbax, 20x2mm). HPLC instrument settings were as above. Source parameters of the QQQ-MS included a gas temperature of 350°C, a gas flow rate 10 l min<sup>-1</sup> and the nebulizer set at 40 psi. Voltage at the capillary was 4000 V, for fragmentation 100 V and within the collision cell 21-30 V.

All chemicals and solvent used were HPLC or higher grade. All measurements were done with 5 replications.

Results showed n-alkyl ammonium ions with different molecular masses: 304.3 (fig. 3), 332.3 (fig. 4), 360.3 (fig. 5) and 388.3 (fig. 6). The four substances (fig. 2) could be separated and identified.

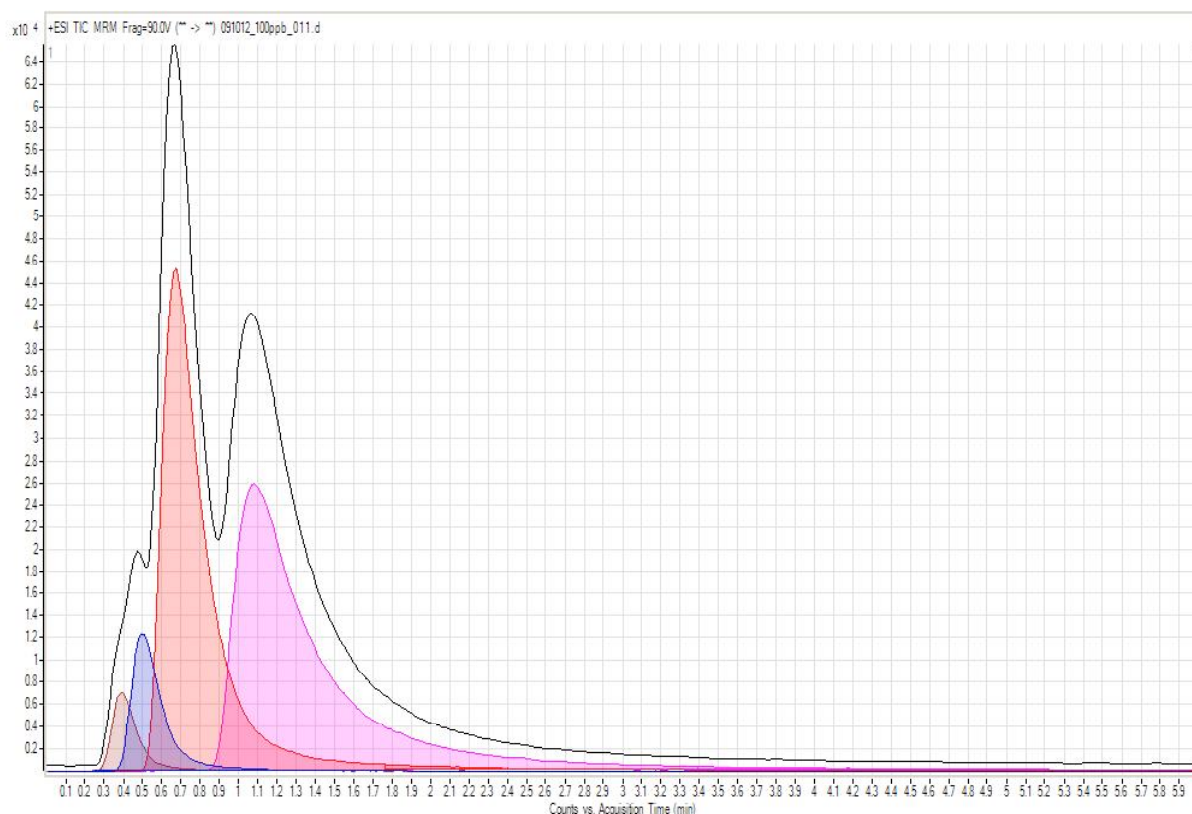


Fig. 2: Total ion current at mass detector (black line) and specific mass fragments of detected mass components (coloured lines) for a 100  $\mu\text{g/l}$  standard alkyl ammonium extracted from Tixogel VZ. The ordinate shows counts (-) the abscissa time (min)

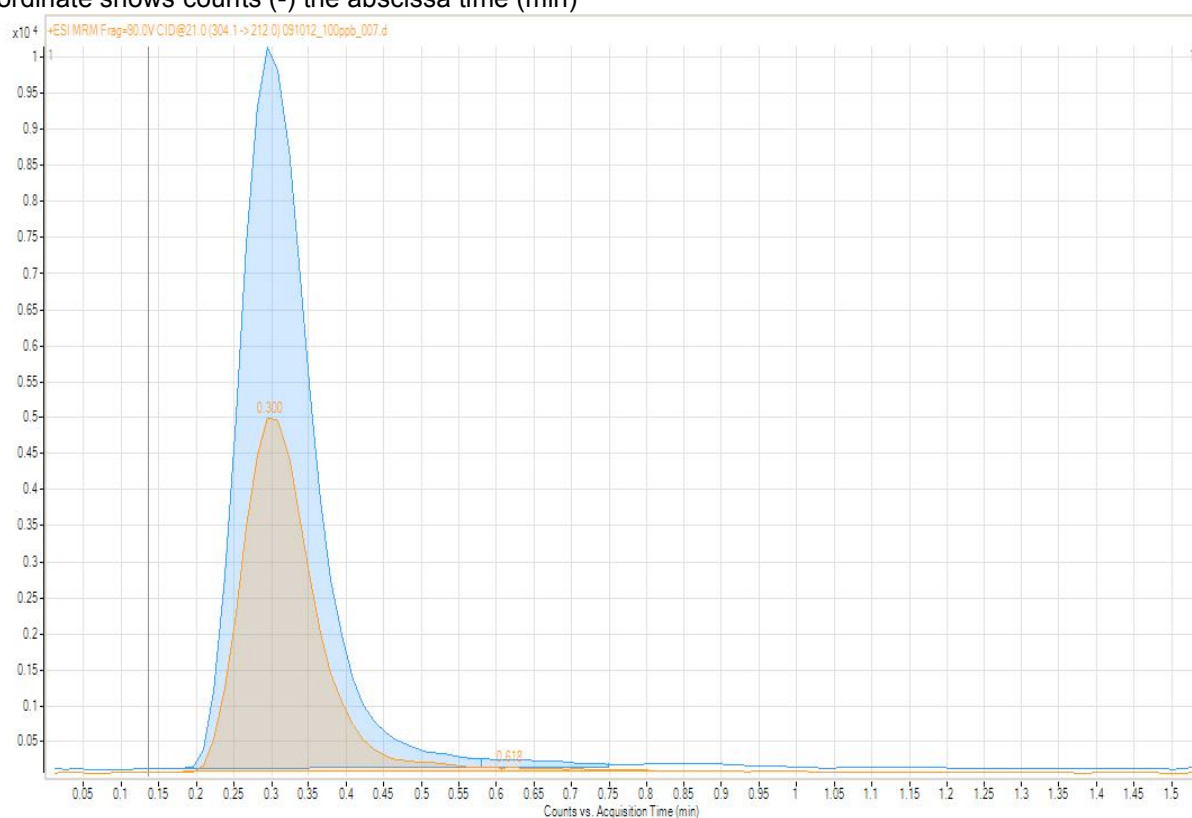


Fig. 3: Total ion current at mass detector (blue line) and specific mass fragments of detected mass components (yellow lines) for a 100  $\mu\text{g/l}$  standard alkyl ammonium extracted from Tixogel VZ. Retention time 0.61min; Precursor Ion 304.1; Product Ion 212.0. The ordinate shows counts (-) the abscissa time (min).

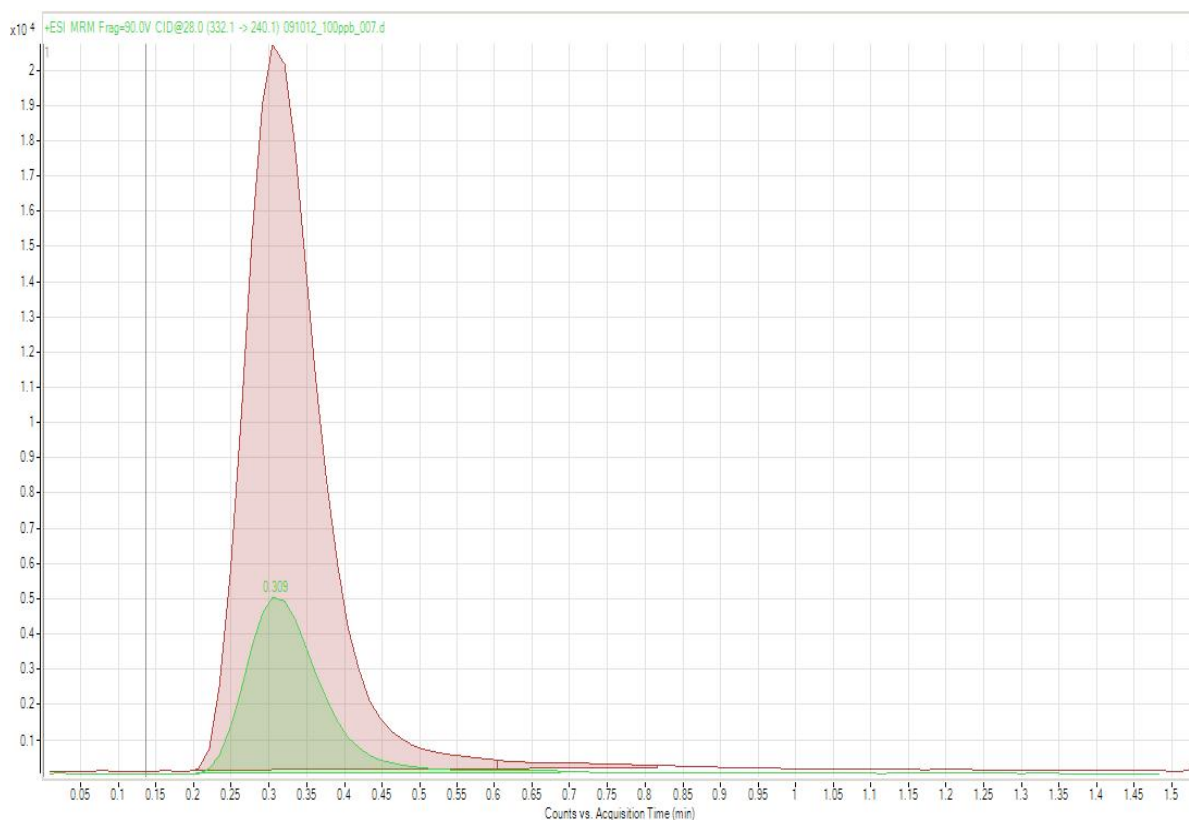


Fig. 4: Total ion current at mass detector (blue line) and specific mass fragments of detected mass components (yellow lines) for a 100  $\mu\text{g/l}$  standard alkyl ammonium extracted from Tixogel VZ. Retention time 0.53min; Precursor Ion 332.1; Product Ion 240.1. The ordinate shows counts (-) the abscissa time (min).

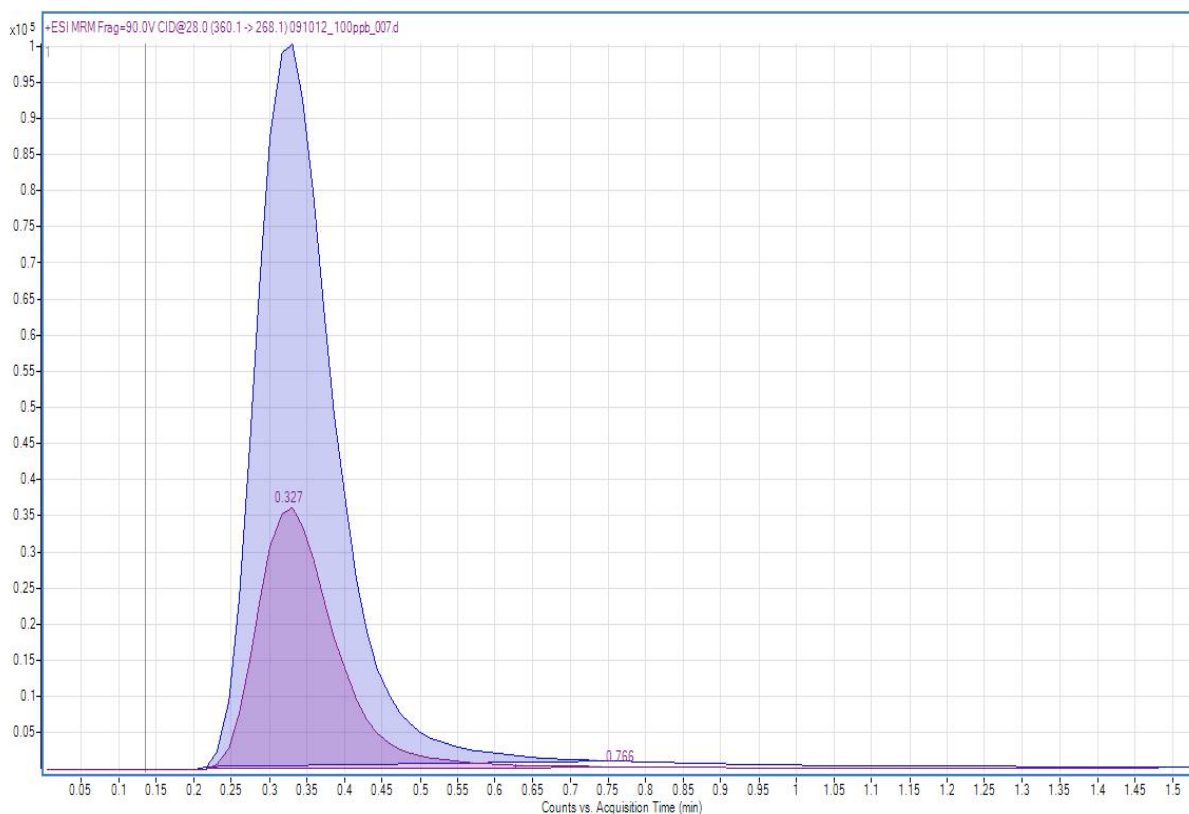


Fig. 5: Total ion current at mass detector (blue line) and specific mass fragments of detected mass components (yellow lines) for a 100  $\mu\text{g/l}$  standard alkyl ammonium extracted from Tixogel VZ. Retention time 0.6 min; Precursor Ion 360.1; Product Ion 268.1. The ordinate shows counts (-) the abscissa time (min).



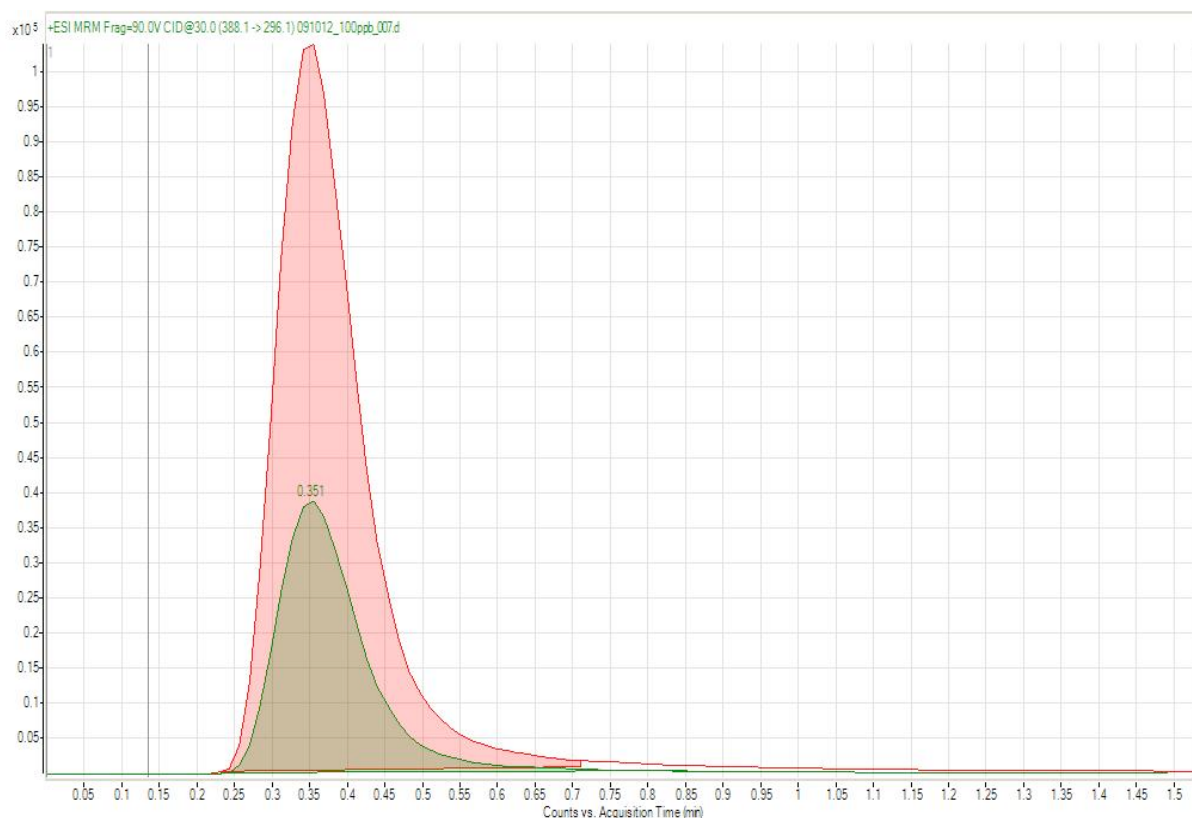
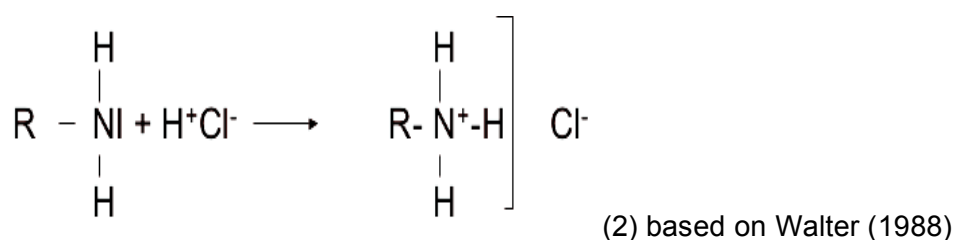


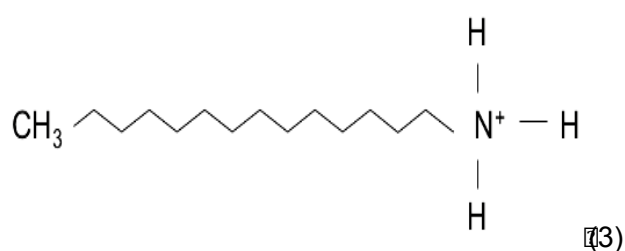
Fig. 6: Total ion current at mass detector (blue line) and specific mass fragments of detected mass components (yellow lines) for a 100 µg/l standard alkyl ammonium extracted from Tixogel VZ. Retention time 0.65 min; Precursor Ion 388.1; Product Ion 296.0. The ordinate shows counts (-) the abscissa time (min).

### Internal standard

As part of quality control, a synthesized C<sub>14</sub>-alkyl-ammonium molecule served as internal standard reference material (3). The standard was synthesised through alkylation of ammonia according to Ruehlicke and Kohler (1981). The generated primary alkylamine was subsequently mixed with HCl to gain alkyl ammonium chloride:



The mass of the synthesized alkyl- ammonium molecule (3) is 214 (C<sub>14</sub>H<sub>29</sub>NH<sub>3</sub><sup>+</sup>):



## HPLC- QQQ- MS

HPLC instrument settings were the same as in the previous experiments. The source parameters of the QQQ-MS differed in nebulizer settings (50 psi), fragmentation voltage (5 V) and collision cell energy (15 V). The other parameters (temperature, gas flow and capillary voltage) stayed unchanged.

3  $\mu$ l of the internal alkyl ammonium standard were injected. The mass spectrometer worked at positive ion mode i.e. the impressed voltage at the capillary, which transfers the HPLC eluate into the electrospray interface (ESI) was positive. The substance was subsequently protonated. The precursor ion was set at 214.1 according to the molecule mass. A precursor ion scan which monitored for specific loss from the precursor ion showed peaks for 85.1 (m/z) and 71.1 (m/z) with peak heights of  $3 \times 10^3$  and  $7 \times 10^3$ , respectively, for the determination of the product ion. To increase the specificity of detection of the known ammonium molecule the MRM (multiple reaction monitoring) mode was used (fig. 7).

The linear working range of the method was 62.5 to 1000  $\mu$ g L<sup>-1</sup> with  $y = 112.17x - 1445.59$  and  $r^2 = 0.9997$  (fig. 8). Relative standard deviations of repeated measurements of the internal standard were generally within 5%.

The characterisation of the alkyl ammonium compounds was achieved through a combination of different HPLC MS systems. In the experimental design it was possible to separate four molecule masses and with the internal standard possible interferences during measurement can be evaluated. Since the method had been completed, the tracer was successfully tested i.a. in an indoor rainfall simulation experiment.

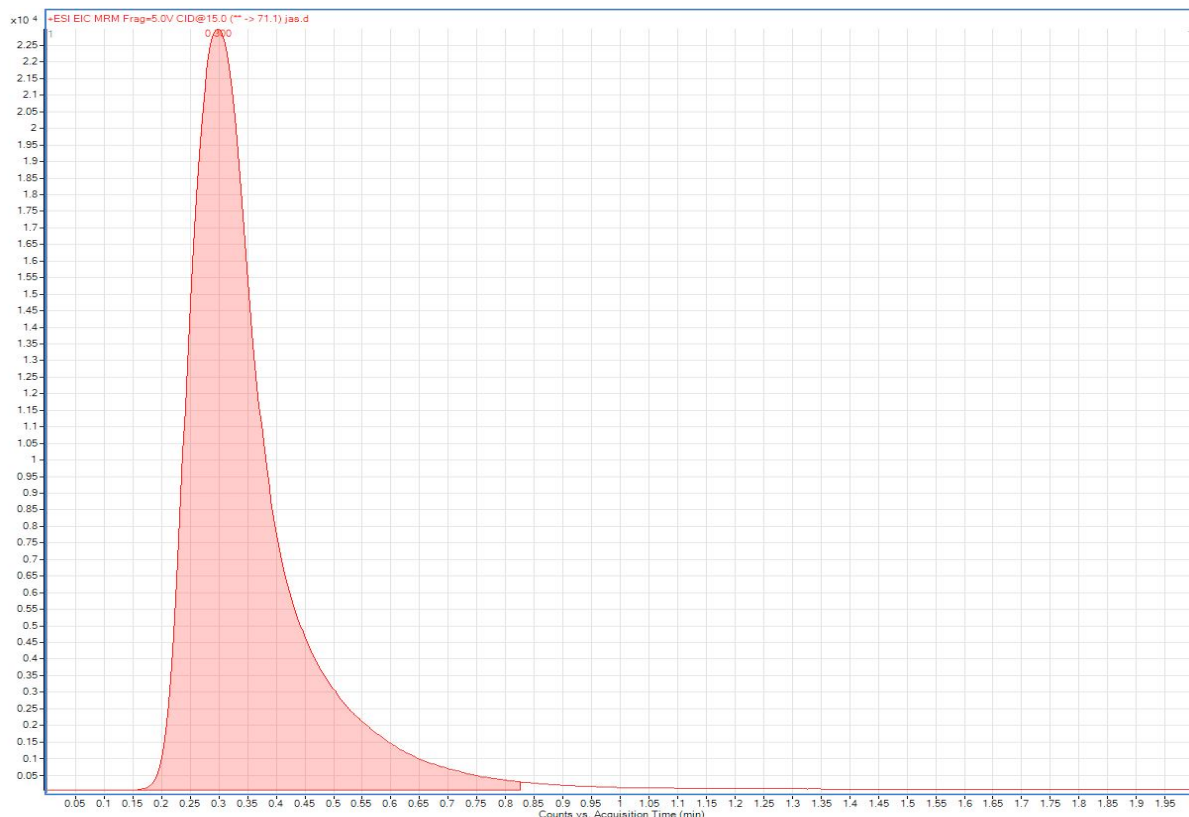


Fig. 7: Specific mass fragments of detected mass components for a C<sub>14</sub> internal standard alkyl ammonium. Retention time 1 min; Precursor Ion 214.1; Product Ion 71.1. The ordinate shows counts (-) the abscissa time (min).

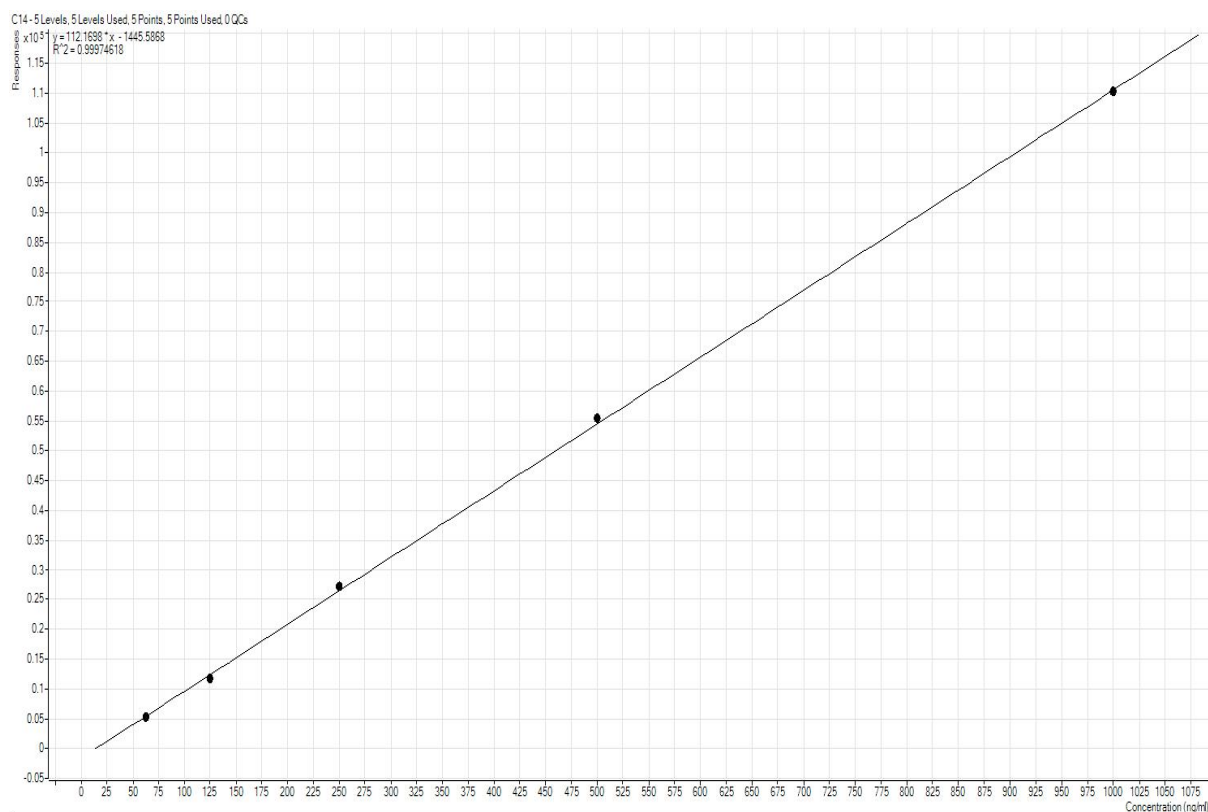


Fig. 8: Calibration curve for the internal C<sub>14</sub> – standard.

### Rainfall simulation experiment

The application of the organophilic clays in an indoor rainfall simulation experiment was conducted at the Institute for Land & Water Management Research of the Federal Agency for Water Management at the facility in Petzenkirchen/ Lower Austria. An arable gleyic Luvisol was used. To ensure complete mixing of the soil, serial dilution was applied. 10 g of soil was mixed with a predetermined amount of Tixogel VZ in a head over head shaker for two hours. Further 1 kg, 10 kg and 70 kg of soil were added and mixed for two hours each in a bulk mixer. This procedure was repeated several times to obtain enough soil tracer mixture. The ratio was 100 g tracer/70 kg soil.

During the rainfall simulation experiment, described in WPA (2009) and Strauss *et al.* (unpublished), runoff and sediment were collected in buckets, weighted to obtain runoff and air dried to obtain sediment. 1g of sediment was extracted for further chemical analysis.

The samples (1g) were shaken with a barium chloride and methanol solvent for the extraction of alkyl ammonium and subsequently sonicated to increase the extraction efficiency. The samples were centrifuged at 3450 g, filtered through 0.45 µm nylon membrane filters and transferred to 1.5 ml glass HPLC vials. High recovery of all four investigated tracers (Target mass: 304.3, 332.3, 360.3, 388.3; Ultrasonic amplitude: 23 µm, Absorbed ultrasonic energy: 78 J/ml; recovery: 77%, 78%, 92% and 77%) could be measured.

Strauss *et al.* (unpublished) concluded from the rainfall simulation experiment that there are indications that the bentonite can be transported not only as clay suspension but much more as microaggregates associated with other clay and silt particles. The results of the rainfall simulation experiment will be published in near future (Strauss *et al.*, unpublished).

## 5 Concluding remarks and outlook

This introductory chapter and the collection of publications deal with methods to measure processes of soil degradation, namely loss of soil structure and soil translocation. I tried to introduce the agronomic impact on soil degradation and the surrounding conditions of implementing alternative agricultural practices in Europe.

Measuring soil aggregate stability can only serve as one method to gain a better understanding of the complex soil matrix. Still it can serve, in conjunction with other soil parameters, as a tool to evaluate changes in soil's physical processes.

This introductory chapter also served to introduce my own work in developing a method for tracing pedogenic movements. The method evolved while working on my bachelor thesis and has been a constant case for improvement in the years of my master studies.

At the recent European Geosciences Union (EGU) conference in Vienna, Guzmán *et al.* (2011) presented results using the tracer in an indoor furrow shoulder system. Preliminary results indicated that the organophilic clay is able to provide insight into the spatial distribution of erosion at small scale and identify the source of sediment in furrow-shoulder systems.

In the near future, the Institute of Soil Science plans, in cooperation with the Institute of Hydraulics and Rural Water Management, to advance the use of quat tracers and conduct erosion experiments on fields in Lower Austria.

Currently, I work on a method to characterise natural organic matter in different arable and forest soil samples. I make use of HPLC-size exclusion chromatography (HP-SEC), coupled with on-line detection of UV absorbance and fluorescence. The characterisation is essential in understanding ecological processes like transport and fate of carbon, nutrients or toxic substances.

Churchman (2010) argued that soil science and soils should be considered holistically rather than via separate sub-discipline, such as soil physics or mineralogy, as it has been done in the past. For now, I am concentrating on the sub-disciplines soil physics and soil chemistry but I hope that, as estimated from Hartemink and McBratney (2008), the soil science community will benefit from the current upsurge in soil science. That new techniques and methodologies will take centre stage to address the pressing environmental questions.

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## Part B

### Selected Publications

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Abstract I:

MENTLER, A., STRAUSS, P., SCHOMAKERS, J., HANN, S., KÖLLENSBERGER, G. AND OTTNER, F. (2009): Organophilic clays as a tracer to determine erosion processes.



## Organophilic clays as a tracer to determine Erosion processes

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In recent years the use of new tracing techniques to measure soil erosion has gained attention. Beside long time existing isotopic methods the use of rare earth elements has been reported. We wanted to contribute to the efforts of obtaining better methods for determination surface soil movement and tested a novel method using organophilic clays as a tracer for erosion related studies. At present tests to extract organophilic clays from soil have been performed successfully using an Industrial produced organophilic bentonite (Tixogel TVZ, Süd-Chemie) treated with quaternary ammonium surfactants. A liquid extraction method with barium ions ( $\text{Ba}^{2+}$ ) and methanol was used to extract the n-alkyl ammonium compounds from the inter crystal layers of the modified Bentonite. To increase extraction efficiency, an ultrasound device was used (UW 2200 Bandelin, 10.000 cycles per second, vibration amplitude 54  $\mu\text{m}$ , sonification time of one minute). This procedure lead to a recovery rate of about 85% for the organophilic bentonite. This was clearly superior to alternative extraction methods such as acetonitrile in different mixing ratios. Quantification of the extracted surfactants was performed via high performance liquid chromatography – mass spectrometry (HPLC-MS, Agilent 1200 SL HPLC and 6220 time-of-flight MS). The mass spectra of this industrial produced organophilic clay mineral showed four different molecular masses ( $\text{M}+\text{H}^+$  of 304.30, 332.33, 360.36 and 388.39). The four substances could be separated by HPLC (20 x 2 mm Zorbax C18 reversed phase column, 0.5 mL/min isocratic flow with 90% acetonitrile and 0.1% formic acid in water, run time of 7 minutes). The linear working range of the method was 5 to 1000  $\mu\text{g/L}$ , with a limit of quantification of 1  $\mu\text{g/L}$  n-alkyl ammonium compound. All four compounds of the Tixogel were extracted with identical extraction efficiencies and are hence suitable for accurate quantification procedures. Next steps of the methodology to develop are the application of the organophilic clays in an indoor rainfall simulation experiment at a small scale of 2  $\text{m}^2$ . At present the methodology has been tested only for one particular soil. Future tests will be performed to see if the chosen methodology needs soil specific treatment when applied to more soils of different textural composition.

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Article I:

SCHOMAKERS, J., MENTLER, A., STEURER, T., KLIK, A., MAYER, H. (2011):  
Characterisation of soil aggregate stability using low intensity ultrasonic  
vibrations.

Int. Agrophysics. 25(2)

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## Characterization of soil aggregate stability using low intensity ultrasonic vibrations

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Received June 29, 2010; accepted October 13, 2010

**A b s t r a c t.** Ultrasonic dispersion equipment has been developed to investigate soil aggregate stability. An ultrasonic probe was vibrating in water containing soil aggregates. Constant vibration amplitudes of 0.5, 1 and 2  $\mu\text{m}$  were used in ultrasonic soil aggregate stability measurements. Ultrasonic power determined with an energy balance method was 0.7, 2.7 and 8.9 W for the three amplitudes. Vibration amplitudes were close to the cavitation limit of gas saturated deionized water, which was found at 0.5-0.6  $\mu\text{m}$  measuring absorbance with diode array spectrophotometer. Cambisol and Chernozem from a long-term tillage experiment in Lower Austria were investigated. The treatments were conventional, reduced and no tillage. Dissolved organic carbon measured with ultrasonic extraction was higher in Cambisol than in Chernozem. Measuring soil aggregate stability according to DIN norm showed greater stability of Cambisol than of Chernozem, however, can hardly show influences of tillage. Ultrasonic soil aggregate stability showed greater stability of Cambisol. Additionally, the ultrasonic soil aggregate stability method can demonstrate significant influences of soil tillage on aggregate stability *ie* stability decreased from no tillage to reduced tillage and conventional tillage for both soils. Ultrasonic soil aggregate stability measurements correlated with total organic carbon content in aggregates. Total organic carbon content was greater in no tillage than in conventional tillage and larger in Cambisol than in highly degraded Chernozem.

**K e y w o r d s:** soil aggregate stability, ultrasonic dispersion, cavitation limit, carbon distribution, tillage

### INTRODUCTION

Ultrasonic dispersion is a widely used method to disaggregate and disperse soil aggregates. Aggregates and particles of different size may be fractioned and used for further physical or chemical analysis without prior use of chemical agents (Ashman *et al.*, 2009; Zhu *et al.*, 2010). Ultrasonic

waves are emitted into water containing the soil aggregates, and cavitation bubbles are generated. High pressures occur when cavitation bubbles collapse, which promotes disaggregation and dispersion of soil aggregates.

Disaggregation is closely related to the power of the used ultrasonic equipment and to the time the water-soil mixture is subjected to ultrasonic vibrations. The absorbed energy (North, 1976) or absorbed energy per unit volume of fluid (Morra *et al.*, 1991) may be correlated to the process of soil particle disaggregation and dispersion and may serve to quantify the stability of soil particles. Several models for the aggregation of soil assume hierarchical order (Bronick and Lal, 2005; Fristensky and Grismer, 2008). In general these models divide soil particles into micro- and macroaggregates being larger or smaller than 250  $\mu\text{m}$ . The hierarchical nature of soil structure indicates that different aggregation mechanisms operate for different size classes of aggregates; consequently, aggregates of different size classes will have different stability (Amézketa, 1999).

One main benefit of the ultrasonic method is the ability to quantify several physical parameters of the experiment. The energy used to disaggregate and disperse soil particles, for example, may be used to quantify and compare the results of different ultrasonic stability tests (Fristensky and Grismer, 2008; North, 1976). Nonetheless, problems arise evaluating and comparing ultrasonic dispersion results of different laboratories due to several reasons:

- Different ultrasonic procedures: Probe- and tank-type ultrasonic devices are used. Magnitude of ultrasonic vibration is often quantified using the ultrasonic power. However, power displayed on the instruments may differ

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- from the actual power output depending on equipment, insertion depth and geometry of the ultrasonic probe (Oorts *et al.*, 2005; Schmidt *et al.*, 1999; Zhu *et al.*, 2009a).
- Different soil treatment: Experimental procedures may vary considerably and are often not well defined. Results strongly depend on pretreatment and soil water content (Raine and So, 1994). Treatment time and soil/water ratio influence the effectiveness of the ultrasonic dispersion (Zhu *et al.*, 2010).
- Different evaluation: Classification of soil particle sizes and presentation of results are not well standardized. The terms macro-, meso- and microaggregates, for example, refer to different particle sizes in the range from 8 mm to 2  $\mu\text{m}$ , and different numbers of classes serve to quantify the frequency of different aggregate fractions (Amézketa, 1999). This makes it necessary to interpret the results within the context of the used methodology (Daraghmech *et al.*, 2009).

The aim of this work is to further develop the ultrasonic method for soil science applications.

#### MATERIAL AND METHOD

Topsoil samples of Cambisol and Chernozem (0–5 cm) were taken from two sites in Lower Austria in fall 2009 (before harvest) that have been under continuous investigation in long-term tillage and erosion experiments. The two sites, Tulln and Pixendorf, situated North-West of Vienna, exhibit experimental fields with altered crop rotation and three different tillage systems:

1. conventional tillage (CT) with mechanical weed control,
2. reduced tillage (RT) with cultivator and cover cropping in winter,
3. no tillage (NT) with inter cropping in winter.

The soil management practices at each site started in the year 1999. The three different tillages were applied to a loamy clay Cambisol and a loamy silt Chernozem (Table 1). The average annual precipitation at both sites is 685 mm with a mean annual temperature of 9.4°C. Tulln has a gradient of 0–2% whereas gradient of Pixendorf site is 5–6%. The experimental set-up varies in allotment length only. The site Pixendorf features an allotment length of 100 m with an allotment width of six metres. In Tulln, the allotment length is 25 m. Samples were air-dried and sieved, and aggregates with diameters between 1 and 2 mm were obtained. Soil aggregate stability was performed in two ways:

- soil aggregate stability (SAS) according to DIN-Norm 19683-16,
- ultrasonic soil aggregate stability (USAS) according to Mentler *et al.* (2004), soil aggregates were analyzed after three different sonification procedures: vibration amplitude 0.5  $\mu\text{m}$  for 60 s, vibration amplitude 1  $\mu\text{m}$  for 30 s and vibration amplitude 2  $\mu\text{m}$  for 15 s.

Total organic carbon content (TOC) was measured with a CNS Analysator (Carlo Erba 1500) with flash combustion technique (ÖNORM L-1080-89). For the destruction of carbonates, the samples were pretreated with 15  $\mu\text{l}$  concentrated HCl and subsequently dried.

Extraction of dissolved organic carbon (DOC) from soil samples was conducted with the ultrasonic technique using Bandelin Sonoplus HD 2200 equipment. The ultrasonic probe had a cylindrical shape with a diameter of 12.7 mm in the extraction experiments. Extraction was performed with vibration amplitude 23  $\mu\text{m}$  and an extraction ratio of 1:50 and using high ultrasonic energy (specific energy greater than 312 J  $\text{ml}^{-1}$ ). With HP 8452A Diode Array Spectrophotometer at 254 nm the release of DOC ( $\text{mg l}^{-1}$ ) from the soil samples was determined, according to (Brandstetter *et al.*, 1996).

Commercially available ultrasonic equipment have several shortcomings, that limit their application in soil science: Control parameter is the ultrasonic power, their lowest power setting is rather high and their accuracy is low. In the present study, self-developed ultrasonic soil dispersion equipment is used, which is an adapted ultrasonic material testing system (Mayer, 2006). It is a probe type system, where one end of the cylindrical ultrasonic probe is dipped in water containing the soil aggregates. The probe performs resonance vibrations at ultrasonic frequency (approximately 20 kHz) leading to the emission of pressure waves into the fluid. Vibration amplitude of the cylindrical ultrasonic probe is measured with an induction coil. In a closed-loop electronic circuit, the amplitude is controlled and kept constant with very high accuracy. Vibration amplitude and duration of sonification are selected prior to the dispersion experiment. Compared with commercially available ultrasonic soil dispersion equipment, the self-built system has three main advantages:

- It uses the vibration amplitude rather than the power to control the magnitude of loading in the dispersion experiment. Under well-defined geometrical conditions, the vibration amplitude strongly correlates with the magnitude of the acoustic pressure waves emitted into the fluid, that cause dispersion of soil particles (Kuttruff, 1988; Millner, 1987). Measurement of the vibration amplitude of an ultrasonic probe can be performed accurately with numerous standard measuring methods including induction coils used here, optical methods *eg* with laser vibrometer or indirect methods performing strain measurements (Mayer *et al.*, 2002). In contrast, the actual power of

**Table 1.** Characterization of soil samples from Cambisol and Chernozem

Sample	Sand	Silt	Clay	N <sub>tot</sub>	TOC	CaCO <sub>3</sub>	C/N
	(%)						
Cambisol	11.13	39.87	51.0	0.25	3.33	1.4	13.1
Chernozem	23.61	64.89	11.5	0.14	1.86	14.9	13.5

ultrasonic equipment cannot be measured directly but it is derived from voltage and current signals, the efficiency of the ultrasonic transducer and other electrical and mechanical parameters of the system (Ratoarinoro *et al.*, 1995; Zhu *et al.*, 2009b). This is prone to errors and can lead to large differences between the displayed power and the actual power output (Oorts *et al.*, 2005; Schmidt *et al.*, 1999). Additionally, at low acoustic pressures close to the cavitation limit, the ultrasonic power fluctuates considerably due to the statistically varying water volume subjected to cavitation.

- It is very accurate. Closed-loop control of vibration amplitude and resonance frequency guarantee, that the maximum deviation of pre-selected and actual vibration amplitude is maximum  $\pm 1\%$ . In contrast, control of ultrasonic power is less accurate and prone to errors. Ultrasonic power may vary during the experiment and is influenced by the process of soil dispersion, for example. Detailed recent study shows that ultrasonic energy can be quantified within 10-20% accuracy only (Zhu *et al.*, 2009b).
- It allows performing experiments at high and very low vibration amplitudes. It is possible, for example to perform experiments below the cavitation limit. Weakly aggregated and degraded soils, for example, can be characterized in this way.

The ultrasonic soil dispersion equipment works at a frequency close to 20 kHz. Cylindrical ultrasonic probe with diameter 30 mm is used. This is larger than diameters of probes used in commercially available equipment, which are typically between 10 and 19 mm (Schmidt *et al.*, 1999). The larger diameter of the ultrasonic probe leads to a better homogeneity of the pressure field. Additionally, emission of acoustic energy increases about linearly with the area of the ultrasonic probe *ie* with the square of the diameter. Using probes with large diameters, certain ultrasonic power can be reached at lower vibration amplitudes and lower acoustic pressures.

Ultrasonic experiments were performed using 80 ml water in a beaker with diameter 44 mm made of PMMA glass. The insertion depth was about 4 mm in all experiments, which means that the distance from the lower end of the ultrasonic probe to the bottom of the beaker was about 50 mm. This length was chosen to avoid resonance of the acoustic waves in the water (half wavelength of sound waves of 20 kHz frequency in water is 37 mm and one wavelength is 74 mm). 4 g soil were inserted in 80 ml deionized water shortly before the dispersion experiment started. The solution was stirred with a magnetic device (2 Hz, cylindrical shape with length 25 mm and thickness 8 mm). Stirring started simultaneously with the ultrasonic vibration and was continued during the experiments to obtain homogeneous soil distribution in the solution.

Power of ultrasonic equipment used for soil science applications can be calibrated with calorimetric methods. North (1976) suggested a procedure, which was later used and adapted by several investigators (Morra *et al.*, 1991;

Oorts *et al.*, 2005; Raine and So, 1994; Roscoe *et al.*, 2000). North uses an energy balance criteria to determine the ultrasonic power *ie* when ultrasonic waves are emitted into water, power delivered by the equipment is balanced by the increase of thermal energies of water and beaker per second plus the loss of heat into the environment per second. The method suggested by North (1976) may be successfully used for commercially available ultrasonic equipment. Power output of such equipment is relatively large, and diameters of ultrasonic probes are relatively small, which causes a relatively small rate of heat loss. In the present investigation however, experiments with very low ultrasonic power close to the cavitation limit are performed. Additionally, the diameter of the ultrasonic probe and its mass are large which leads to large heat capacity and considerable heat transport due to thermal conduction. Due to these reasons, we use the following caloric calibration procedure to determine the power of the ultrasonic equipment. The vibration amplitude of ultrasonic probe end is kept constant during the experiment. The ultrasonic probe is inserted in water (mass of water is  $m_w$  and specific heat capacity is  $c_w$ ), and the increase of water temperature,  $\Delta T$  during the time period  $\Delta t$  is measured. Changes of thermal energy of water are caused by the ultrasonic vibrations and by the heat exchange with ultrasonic probe, beaker and laboratory air. With the ultrasonic power,  $P_{US}$  and the heat exchange per second,  $\Delta Q_{Exchange}/\Delta t$ , change of thermal energy of water per second,  $m_w c_w \Delta T/\Delta t$  is given by Eq. (1):

$$m_w c_w \frac{\Delta T}{\Delta t} = P_{US} + \frac{\Delta Q_{Exchange}}{\Delta t}. \quad (1)$$

Heat flow between water and environment due to convection or thermal conduction is driven by a temperature gradient. Heat flow from water to beaker, ultrasonic probe or laboratory air is proportional to the respective temperature difference. At the beginning of ultrasonic power measurements, all mechanical components of the system including ultrasonic probe and beaker are at ambient temperature. Water with temperature below ambient temperature is used at the beginning of the experiment.  $\Delta Q_{Exchange}/\Delta t$  is greater zero and increase of thermal energy of water per unit time is the sum of ultrasonic power and heat flow into the water. Water temperature is measured and increases during sonification. When water temperature is above ambient temperature,  $\Delta Q_{Exchange}/\Delta t$  becomes less than zero. When the temperature of water and environment coincide,  $\Delta Q_{Exchange}/\Delta t$  becomes zero and the ultrasonic power is directly correlated to the increase of water temperature.

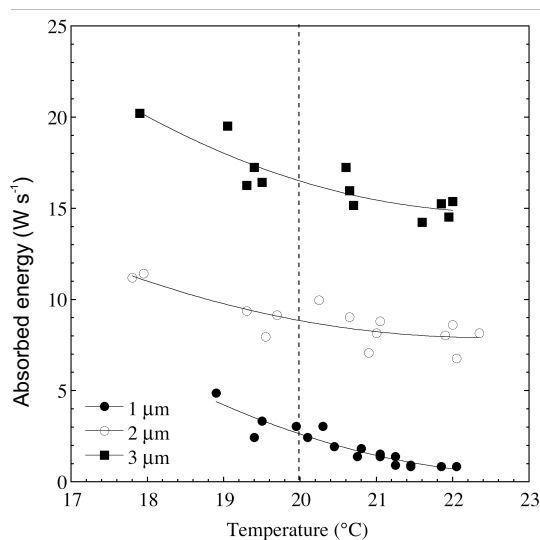
Spectroscopic experiments were conducted using a HP 8452A Diode Array Spectrophotometer. The tip of the ultrasonic probe is inserted into a beaker containing gas saturated de-ionized water. The beaker is mounted in the optical path of the photometer with the light source on one side and the diode array detector on the other.

A wavelength range from 300 to 800 nm was chosen with a bandwidth of 2 nm and an integration time of one second. The ultrasonic probe vibrates at different previously selected vibration amplitudes, and the absorption coefficient is measured. Cavitation leads to bubbles in the water that cause scatter and reflection and thus attenuation of the transmitted light. Therefore, the absorption coefficient abruptly increases when cavitation occurs, which serves to detect the occurrence of cavitation. The experiments were repeated five times with constant temperature 22°C in the absorption cell.

SPSS Version 8 (Bühl and Zöfel, 1999) was used to calculate means and standard deviations of all data and one-way ANOVA followed by the Duncan Test ( $p < 0.05$ ) to compare means.

### RESULTS AND DISCUSSION

Ultrasonic power is measured at different vibration amplitudes of the ultrasonic probe. Temperature increase of water is measured and the absorbed thermal energy per second is calculated. Figure 1 shows the results of measurements for vibration amplitudes 1, 2, and 3  $\mu\text{m}$ . Each measurement is repeated four times. The abscissa shows the water temperature. Ultrasonic probe and beaker have ambient temperature of 20°C, whereas water temperature is below ambient at the beginning of the experiments. Therefore, heat flows into the water from ultrasonic probe, beaker and laboratory air at the beginning. With increasing water temperature heat flow decreases and vanishes, when water temperature is 20°C. At higher temperature, heat flows from water to beaker, ultrasonic probe and laboratory air. Absorbed energy per second when water temperature is 20°C is the ultrasonic power.



**Fig. 1.** Increase of thermal energy of water per second for vibration amplitudes of 1  $\mu\text{m}$  (closed circles), 2  $\mu\text{m}$  (open circles), and 3  $\mu\text{m}$  (closed squares).

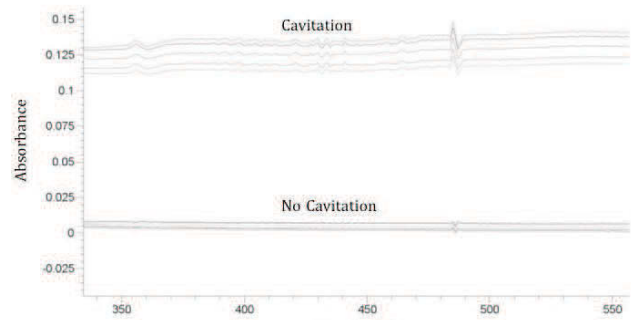
Ultrasonic power at vibration amplitude 1  $\mu\text{m}$  determined in this way is by mean 2.7 W. At vibration amplitude 2  $\mu\text{m}$ , the mean ultrasonic power is 8.9 W. Mean power at 3  $\mu\text{m}$  is 16.5 W, and at vibration amplitude 0.5  $\mu\text{m}$  mean power is 0.7 W (not shown in Fig. 1). Data scatter is relatively large, which is caused by the statistical occurrence and magnitude of cavitation close to the cavitation limit. Consequently, the absorbed energy varies in the different experiments.

Absorbance determined with HP 8452A Diode Array Spectrophotometer are shown for light with wavelengths between approximately 340 and 550 nm in Fig. 2. Wavelengths are indicated on the abscissa and absorption coefficients on the ordinate. If cavitation occurs, absorbance increases significantly compared to no cavitation. Cavitation bubbles in the light path of the spectrophotometer cause scattering and reflections, which increases absorbance. Increase of absorption coefficient is visible within the whole range of investigated wavelengths from 300 to 800 nm.

Experiments with different vibration amplitudes of the ultrasonic probe in the range between 0.3 and 0.8  $\mu\text{m}$  are performed to determine the cavitation limit. Figure 3 shows the absorption coefficient for different vibration amplitudes of the ultrasonic probe. Between 0.5 and 0.6  $\mu\text{m}$  there is sharp increase in absorbance. Thus minimum vibration amplitude necessary to cause cavitation in gas saturated deionized water is 0.5-0.6  $\mu\text{m}$ .

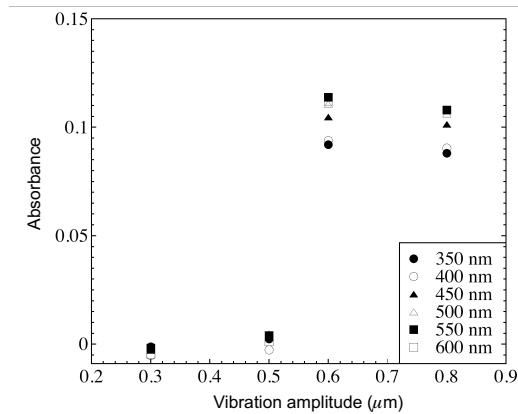
Few experimental investigations on the cavitation limit in water can be found in the literature. For frequency 15 kHz, the minimum acoustic pressure amplitudes necessary to cause cavitation in tap water are found in the range from 0.5 to 2 bar (Esche, 1952). With the vibration frequency,  $f$ , the vibration amplitude,  $u$ , the mass density,  $\rho$ , and the sound velocity,  $c$ , the acoustic pressure,  $p_a$ , can be calculated using Eq. (2) (Kuttruff, 1988; Millner, 1987):

$$p_a = 2\mu f u \rho c. \quad (2)$$



**Fig. 2.** Absorption coefficients determined with HP 8452A Diode Array Spectrophotometer for wavelengths between 340 and 550 nm without cavitation (lower lines) and with cavitation (upper lines).





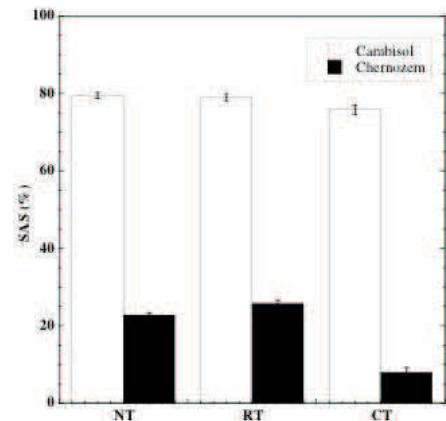
**Fig. 3.** Absorption of light with different wavelengths in water subjected to ultrasonic vibrations with amplitudes between 0.3 and 0.8  $\mu\text{m}$ .

Considering the mass density of water ( $998 \text{ kg m}^{-3}$  at  $20^\circ\text{C}$ ) and the sound velocity in water ( $1483 \text{ m s}^{-1}$  at  $20^\circ\text{C}$ ), the acoustic pressure amplitudes are 0.9 bar for vibration amplitude 0.5  $\mu\text{m}$  and 1.1 bar for 0.6  $\mu\text{m}$ . This means that the minimum acoustic pressure necessary to cause cavitation in gas saturated deionized water at 20 kHz frequency is about atmospheric pressure.

Additional experiments have been performed with deionized and degassed water. Cavitation limit under these conditions is at higher vibration amplitudes in the range between 1 and 1.5  $\mu\text{m}$ , which corresponds to acoustic pressures between 1.9 and 2.8 bar. This supports studies reported in the literature, that degassing of water raises the cavitation limit (Raine and So, 1994). In the present investigation, ultrasonic soil disaggregation experiments are performed using vibration amplitudes of 0.5, 1, and 2  $\mu\text{m}$ , which is close to or slightly above the cavitation limit.

Figure 4 shows mean values and standard deviation (five repetitions) of SAS measurements according to DIN-Norm 19683-16. Samples of Cambisol show overall higher stability than samples of Chernozem due to the higher content of organic carbon and clay. Differences in SAS between the three tillage systems proofed to be not significant for Cambisol. Soil aggregates of Chernozem lost more than 50% of their stability through CT in comparison to RT and NT. Differences of Chernozem in SAS between RT and NT proofed to be not significant. The hillside location increases the loss of clay and organic matter and reduces soil aggregate stability.

Figure 5 shows the results of ultrasonic soil aggregate stability measurements after sonification with three different vibration amplitudes and sonification times. Vibration amplitude 0.5  $\mu\text{m}$  for 60 s (Fig. 5a): with the ultrasonic power of 0.7 W the absorbed ultrasonic energy in 80 ml water and 4 g soil is 42 J and the specific absorbed ultrasonic energy is  $0.5 \text{ J g}^{-1}$ . Figure 5a shows that the implementation of different tillage practices had a wide influence on the water stable aggregate fractions, characterized by USAS



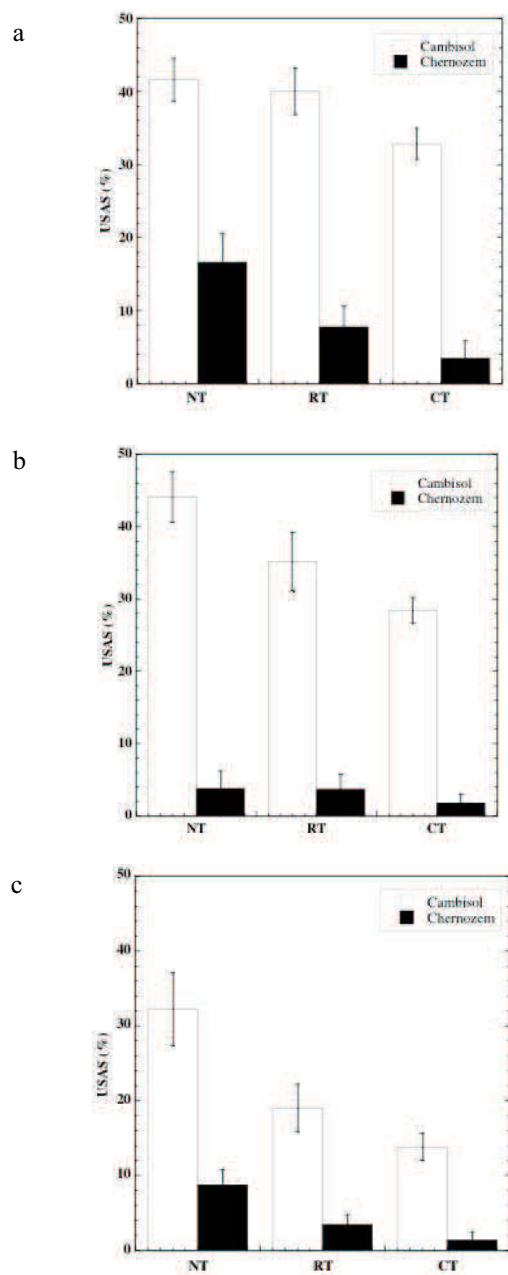
**Fig. 4.** Soil aggregate stability (SAS, %) of two soils (Cambisol and Chernozem) and three tillage systems (CT, RT, and NT).

with vibration amplitude 0.5  $\mu\text{m}$ . The largest decrease in aggregate stability can be observed in Chernozem (clay content 11%). Ten years of CT resulted in the loss of nearly 80% aggregate stability (USAS). In the same time period Cambisol (clay content 49%) was more resistant to conventional tillage systems and shows a reduction in USAS of only 20%. Vibration amplitude 1  $\mu\text{m}$  for 30 s (Fig. 5b): with the ultrasonic power of 2.7 W the absorbed ultrasonic energy is 81 J and the specific absorbed ultrasonic energy is about  $1 \text{ J g}^{-1}$ . Figure 5b shows a significant difference in USAS between CT and the conservation oriented tillage practices for Cambisol. Treatments NT and RT of Cambisol show no significant differences. Tillage systems of Chernozem, on the other hand, cannot be distinguished. Vibration amplitude 2  $\mu\text{m}$  for 15 s (Fig. 5c): with the ultrasonic power of 8.9 W the absorbed ultrasonic energy is 134 J and the specific absorbed ultrasonic energy is about  $1.6 \text{ J g}^{-1}$ . Figure 5c shows that the vibration amplitude 2  $\mu\text{m}$  delivers useful results for both soil types. Differences in USAS between NT and CT are significant for Cambisol and Chernozem. Cambisol lost nearly 60% of its stability, Chernozem more than 80%.

All USAS measurements after different sonification amplitudes and times show that Cambisol is more stable than Chernozem (Fig. 5a-c), which coincides with the results of the SAS method. In contrast to the SAS method, however, the USAS method using vibration amplitudes close to the cavitation limit can additionally detect differences in soil aggregate stability after different tillage. For both investigated sites, soils show a higher aggregate stability, if no tillage is practiced. RT decreases aggregate stability characterized by USAS and CT shows the lowest aggregate stability.

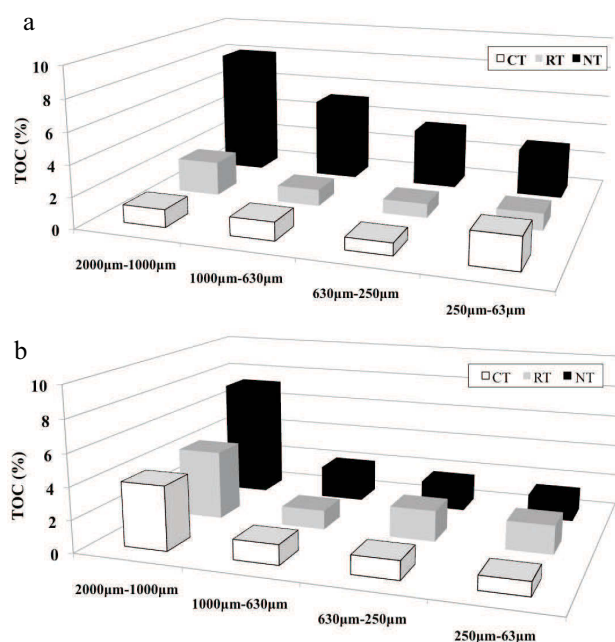
The distribution of total organic carbon (TOC) in water stables aggregates of Cambisol is shown in Fig. 6a. A relatively large content of TOC is visible in NT aggregates. TOC content is significantly larger in NT than in RT or CT aggregates for aggregates greater than 250  $\mu\text{m}$ . Figure 6b shows TOC in water stables aggregates of Chernozem from Pixendorf. TOC is higher in all NT aggregate fractions





**Fig. 5.** Ultrasonic soil aggregate stability (USAS, %) of Cambisol and Chernozem for three tillage systems (CT, RT and NT); USAS after sonification with amplitude: a – 0.5  $\mu$ m for 60 s, b – 1  $\mu$ m for 30 s, c – 2  $\mu$ m for 15 s.

compared to CT aggregate fractions. TOC in NT and CT mesoaggregates (>630  $\mu$ m) differs significantly, whereas differences in RT and CT fractions are not significant. Comparing all aggregate fractions, highest TOC content is measured in the NT fractions of Cambisol (up to 8%). TOC in aggregates greater than 250  $\mu$ m from Cambisol with CT and RT is lower compared with the same aggregate classes of Chernozem.



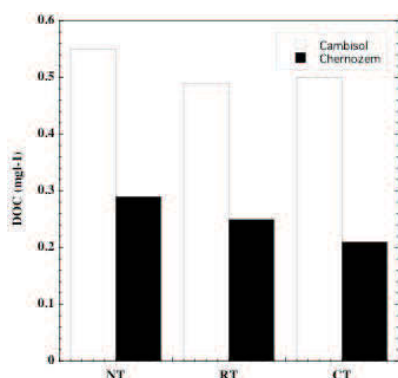
**Fig. 6.** Distribution of total organic carbon (TOC, %) in water stable aggregates for: a – Cambisol and b – Chernozem.

The DOC release from aggregates of different tillage systems at Tulln and Pixendorf using the ultrasonic extraction method is shown in Fig. 7. Tulln displays a significantly higher release of easily water-soluble organic substances. The DOC release for Chernozem displays differences among the management systems. For both soil types, the release of DOC in NT soils is higher than in CT soils.

The impact of common and conservation-oriented tillage systems on physical and chemical properties of soil can be observed in long-term tillage experiments. Soil management influences the stability of aggregates and noticeably the carbon storage and distribution within the fractions (Kasper *et al.*, 2009; Sohi *et al.*, 2010). The maintenance of a 'good' soil structure is critical for agricultural sustainability, and depends on the stability of the aggregates (Amézketa, 1999).

Both aggregate stability measurements, USAS and SAS, show a significant difference between the stability of Cambisol from Tulln and the Chernozem from Pixendorf. In addition, the USAS method using low vibration amplitudes close to the cavitation limit shows that tillage accelerates the disaggregation process for both soil types.

Agricultural management affects the stability of macroaggregates while microaggregates are independent of it (Amézketa, 1999; Fuller *et al.*, 1995). Cambisol contains more water stable macroaggregates, which show prolonged resistance towards disruption compared to the Chernozem from Pixendorf. A change in aggregate stability occurs for both soils comparing conservation oriented to conventional tillage systems, which is best demonstrated in ultrasonic experiments with vibration amplitude above the cavitation limit.



**Fig. 7.** Release of dissolved organic carbon (DOC, mg l<sup>-1</sup>) of Cambisol and Chernozem using ultrasonic extraction technique.

Analysis of the soil show that the energy of draft forces of tillage tools during cultivation separates macroaggregates and increases the disaggregation process. The reduction of soil aggregate stability goes along with a decrease in the amount of organic carbon and nitrogen (Kasper *et al.*, 2009; Sohi *et al.*, 2010). Tillage does not only reduce soil organic matter but influences the distribution of organic carbon in the aggregate fractions, which determines the surface-orientated processes *eg* carbon release and in particular water soluble carbon (DOC). The solubility depends on the surface of the aggregates, carbon content and molecular composition.

It is shown, that the chosen vibration amplitudes, sonification times and setup of experiments (ultrasonic probe, soil water ratio, *etc.*) is appropriate to monitor slight differences in soil aggregate stability. Key experimental parameters, like sonification time and vibration amplitude, can be adapted using the ultrasonic method whereas sieving methods are known to provide only relative estimates of the investigated parameters (Bieganowski *et al.*, 2010; Daraghmeah *et al.*, 2009).

## CONCLUSIONS

1. Ultrasonic soil aggregate stability (USAS) measurements using vibration amplitudes close to the cavitation limit can be successfully used to characterize low and highly aggregated soils. USAS can also demonstrate influences of tillage on soil aggregate stability, which can hardly be found using the conventional Soil aggregate stability (SAS) sieving method. Results of USAS measurements correlate with total organic carbon (TOC) content in aggregates.

2. Several parameters of the ultrasonic dispersion experiment need to be standardized to obtain reproducible results. It is suggested to use the vibration amplitude of the ultrasonic probe to control the experiment. Details of the experimental procedure, including determination of ultrasonic power, time of sonification, mass of water and soil aggregates, diameter of the ultrasonic probe, insertion depth *etc.* are suggested in the present work.

3. Ultrasonic dispersion equipment may be further used to quantify available carbon pools. Extraction of dissolved organic carbon (DOC) from aggregates is performed applying high ultrasonic energy to the soil water mixture.

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Article II:

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# Measurement of soil aggregate stability using low intensity ultrasonic vibration

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## ABSTRACT

The objective of this study was to analyse the influence of tillage on soil aggregate stability using the ultrasonic dispersion method with different levels of energy. Experiments are performed with self-developed equipment at low vibration amplitudes of 0.5  $\mu\text{m}$ , 2  $\mu\text{m}$  and 5  $\mu\text{m}$ . Ultrasonic power is 0.7 W, 8.9 W and 22.3 W for the three amplitudes. Samples of aggregates 2 000 - 250  $\mu\text{m}$  of a degraded loamy silt Chernozem, a loamy clay Cambisol and a loamy sand Cambisol from eastern Austria were collected under different tillage regimes: conventional tillage (CT) with mechanical weed control and no tillage (NT) with inter cropping in winter. Measuring Soil Aggregate Stability (SAS) according to DIN norm showed that the stability decreased in the sequence loamy clay Cambisol > loamy sand Cambisol > loamy silt Chernozem. Influences of tillage could be observed with SAS for the last two samples mentioned.

Ultrasonic dispersion tests at vibration amplitude 0.5  $\mu\text{m}$  and 2  $\mu\text{m}$  show higher stability of the Cambisol soils. Significant influences of soil tillage on aggregate stability for the loamy clay Cambisol and the degraded Chernozem were observed after short ultrasonic dispersion times and absorbed energies of 1 - 1.7 J  $\text{mL}^{-1}$ . The measured aggregate stability decreased in the following order: Cambisol NT > Cambisol CT > Chernozem NT > Chernozem CT. Differences in loamy sand Cambisol could not be detected with the ultrasonic method due to a low aggregation level of the macro aggregates. This study highlights the importance of quantifying the energy output of the ultrasonic equipment when analysing changes in soil aggregate stability, for the evaluation of tillage treatments. A more comprehensive analysis of aggregate stability can be obtained when using both, the wet-sieving SAS method and ultrasonic dispersion at low energy levels.

## KEYWORDS

Ultrasonic dispersion, soil aggregate stability, soil tillage, soil disaggregation

## Medición de la estabilidad de agregados de suelo, utilizando vibración ultrasónica de baja intensidad

## RESUMEN

EL objetivo del trabajo fue analizar la influencia del laboreo sobre la estabilidad de los agregados del suelo (soil aggregate stability – SAS) utilizando un método de dispersión ultrasónica con diferentes niveles de energía. Se hicieron ensayos con un equipo de dispersión ultrasónica desarrollado en la universidad de \*\*\*\*. Este equipo facilita la aplicación de amplitudes de baja vibración, de 0.5  $\mu\text{m}$ , 2  $\mu\text{m}$  y 5  $\mu\text{m}$ . La potencia ultrasónica determinada por un método de balance de energía es de 0.7 W, 8.9 W y 22.3 W para cada amplitud. Se tomaron muestras de agregados de 2 000 a 250  $\mu\text{m}$ , en tres tipos de suelo del este de Austria: un suelo del tipo Chernozem franco limoso degradado, un Cambisol franco arcilloso y un Cambisol franco arenoso, y sometidos a diferentes tipos de laboreo: labranza convencional (CT – conventional tillage), laboreo con eliminación mecánica de malas hierbas y sin labranza (NT – no tillage), con siembra intercalada en invierno. Los resultados de la estabilidad de los agregados del suelo obtenidos según la norma DIN (Deutsches Institut für Normung) mostraron que la estabilidad disminuía en el siguiente orden: Cambisol franco arcilloso > Cambisol franco arenoso > Chernozem franco limoso. La influencia del tipo de laboreo se observó en el segundo y tercer tipo de los suelos mencionados.

La dispersión ultrasónica por amplitudes de baja vibración mostró una estabilidad más alta en suelos del tipo Cambisol. Se observó una influencia significativa del tipo de laboreo sobre la estabilidad de los agregados del suelo (entre 1 y 1.7 J ml<sup>-1</sup>) en el Cambisol franco arcilloso y el Chernozem degradado, resultando una disminución de la estabilidad de los agregados en el orden siguiente: Cambisol NT > Cambisol CT > Chernozem NT > Chernozem CT. No se observaron diferencias en el Cambisol franco arenoso, porque el nivel de agregación de los macroagregados fue demasiado débil. Este trabajo destaca la importancia de cuantificar la energía aplicada por el equipo de ultrasonidos para el análisis de variaciones en la estabilidad de los agregados del suelo con respecto a diferentes tratamientos de labranza. La combinación de los métodos de análisis de estabilidad de agregados, por tamizado en condiciones de humedad (SAS) y por dispersión ultrasónica a niveles bajos de energía, permite un análisis más preciso de la estabilidad de los agregados del suelo.

#### **PALABRAS CLAVE**

dispersión ultrasónica, estabilidad de agregados de suelo, cultivo del suelo, desagregación del suelo

## **INTRODUCTION**

The breakdown of soil into smaller aggregates and particles can be accomplished using ultrasonic equipment. According to the aggregate hierarchy theory, breakdown occurs when sufficient mechanical stresses are applied to overcome the attractive forces within the aggregates (Raine 1998). Ultrasonic dispersion of aggregated soils is usually conducted using high-intensity ultrasonic instruments (Edwards and Bremner 1967; Field et al. 2006; Zhu et al. 2009b). Acoustic pressure waves are emitted into the soil water solution, which cause cavitation, stressing of soil aggregates and breaking of aggregate bonds. At higher vibration amplitudes, higher pressure waves occur and particle disruption is accelerated (Mentler et al. 2004).

The focus of this paper is the stability of the soil macro aggregate fraction in the range of 2 000 - 250 µm, coarse and medium sand included. The physical properties of these aggregates allow for an optimum of gas exchange and water availability. Roots and hyphae stabilize the aggregates, inhibit erosion and are affected by soil utilisation (Tisdall and Oades 1982; Tippkötter 1994). For sustaining agricultural productivity, a good soil structure is needed which in turn depends on the presence of stable soil aggregates (Amézketa 1999). Several authors (Amelung and Zech 1999; Field and Minasny 1999; Mentler et al. 2004) consider that large aggregates (> 250 µm) disrupt at relatively low absorbed specific energies. Roscoe et al. (2000) determined a critical energy of 260 - 275 J ml<sup>-1</sup> which was sufficient to disrupt most of the 2 000 - 100 µm fraction of the investigated Latosols. Carolino De Sá et al. (2002) examined the aggregate stability of Latosols under different land uses applying different levels of ultrasonic energy. High sensitivity and small coefficients of variations were observed when ultrasonic energies between 30 and 90 J ml<sup>-1</sup> were used, and the best energy level to determine differences in aggregate stability caused by different land uses was 36.3 J ml<sup>-1</sup>.

Traditional techniques (e.g. wet sieving) used in the characterisation of soil aggregate stability typically use only a single stability measurement taken after the application of an arbitrary energy for an arbitrary period of time. The results are highly dependent on the nature of the mechanical energy applied (Raine 1998).

One of the main advantages of the ultrasonic method to determine soil aggregate stability is the possibility to exactly measure the applied energy (North 1976; Raine and So 1993). However, ultrasonic power used in most equipment is too large to differentiate stability, especially of weakly aggregated soils. It has been shown that arable soils require far lower energy inputs than grassland soil to cause fragmentation (Ashman et al. 2009). This paper aims to investigate soil aggregate stability applying very small ultrasonic vibration amplitudes. The process of soil aggregate disruption is studied at low ultrasonic power after sonication with low ultrasonic energies. The used ultrasonic vibration amplitudes are about one decade smaller than those of commercially available ultrasonic equipment employed in soil dispersion experiments. This relatively gentle disruption of aggregates is applied to

characterise small differences in aggregate stability that are caused by different tillage of the same soil.

## MATERIAL AND METHOD

### Study site

Topsoil samples (0-10 cm) were taken from three sites in eastern Austria at the end of the growing season in fall 2009 that have been under continuous investigation in long-term tillage and erosion experiments. The soils have been classified according to WRB (IUSS 2007).

- i) A loamy clay Cambisol from Tulln in Lower Austria at 48° 18' N and 16° 02' E
- ii) A loamy sand Cambisol from Kirchberg (am Walde) in Styria at 48° 16' N and 15° 58' E
- iii) A degraded loamy silt Chernozem from Pixendorf in Lower Austria at 48° 16' N and 15° 58' E

The Ap horizon of the Pixendorf Chernozem is severely eroded due to its hillside location and therefore the soil type is addressed as physically degraded.

All sites are experimental fields with altered crop rotation and different tillage systems. Of these, two systems have been chosen to investigate soil aggregate disruption:

- a) Conventional tillage (CT, tillage depth 23 cm) with mechanical weed control and
- b) No tillage (NT, operation depth 2-7 cm) with inter cropping in winter.

The soil management practices in Lower Austria started in 1999, in Kirchberg in 2007. The mean annual precipitation at the sites in Lower Austria is 685 mm, in Kirchberg 730 mm with mean annual temperatures of 9.4°C and 9.1°C, respectively. Tulln has a slope of 0-2 %, Pixendorf 5-6 % and Kirchberg 12-15 %. The investigated soils are characterized in Table 1.

### Soil sampling and analysis

Sampling spots were evenly distributed over the whole field and taken representatively. Sample collection followed ISO standards (ISO 10381-2). Samples were taken from 0-10 cm soil depth in order to characterise soil surface macro aggregation. Kasper et al. (2009) found statistically significant differences in organic carbon ( $C_{org}$ ) and total nitrogen ( $N_{tot}$ ) for different tillage treatments especially in this surface layer. Each sample was air-dried, homogenously mixed and sieved to pass a 2 mm sieve.

Table 1: Site conditions at the experimental fields (sample depth 0 - 300 mm)

	<b>Sand</b> %	<b>Silt</b> %	<b>Clay</b> %	<b>N<sub>tot</sub></b> %	<b>C<sub>org</sub></b> %	<b>CaCO<sub>3</sub></b> %	<b>SOM</b> %	<b>C/N</b>
<b>Tulln Cambisol</b>	11.13	39.87	51.00	0.25	3.33	1.40	5.74	13.10
<b>Kirchberg Cambisol</b>	52.78	33.22	14.00	0.11	1.73	<0.50	2.98	13.30
<b>Pixendorf</b>								
<b>Chernozem</b>	23.61	64.89	11.50	0.14	1.86	14.90	3.21	13.50

Data represent mean values of six measurements during the growing season of 2009 (May to September). Differences between tillage systems were not significant (n=5).

All textural analyses were made with the pipette method using  $Na_4P_2O_7$  for dispersion and  $H_2O_2$  to destroy organic matter (ISO 11277). Carbon and nitrogen were measured using dry combustion with a Carlo Erba NA 1500 (ISO 10694; ISO 13878). SOM was calculated:  $C_{org} \times 1.724$ , the assumed average C-concentration of the organic matter of 58 % (Scheffer and Schachtschabel 2010). Determination of calcium carbonate was in accordance with the Scheibler volumetric method (ISO 10693).

### Soil aggregate stability (SAS)

Soil aggregate stability (%) of the investigated soils was calculated using the wet sieving method described in DIN-Norm 19683-16. The method is modified after Kemper and Rosenau (1986) and the SAS samples were analysed with three repetitions from each plot and tillage treatment ( $n = 18$ ). 4 g of air-dried soil aggregates of diameter of 2 000 - 1 000  $\mu\text{m}$  (*EW*) were dipped on a sieving machine equipped with sieves of 250  $\mu\text{m}$  mesh. The percentage of water stable aggregates (Eq. 1) was calculated with the obtained parameters:

$$\% \text{ SAS} = \frac{m_K - m_A}{EW - m_A} \cdot 100 \quad (1)$$

with  $m_K$ , the weight of the stable aggregates in the fraction  $> 250 \mu\text{m}$  after dipping and  $m_A$ , the mass of sand after chemical dispersion of the remaining aggregates.

### Ultrasonic dispersion

Ultrasonic dispersion experiments were performed with a self-developed ultrasonic dispersion equipment which is an adapted ultrasonic material testing system (Mayer 2006). It is a probe type system, where one end of the cylindrical ultrasonic probe is dipped in water containing the soil aggregates. The probe with a diameter of 30 mm performs resonance vibrations at ultrasonic frequency (approximately 20 kHz) leading to the emission of pressure waves into the soil water solution.

Vibration amplitude of the cylindrical ultrasonic probe is measured with an induction coil. In a closed-loop electronic circuit, the amplitude, rather than the power, is controlled and kept constant with very high accuracy. Vibration amplitude and duration of sonication are selected prior to the dispersion experiment. The same ultrasonic probe was used in all experiments.

During the ultrasonic experiments, the soil-water suspension was placed in a Plexiglas beaker with a diameter of 44 mm. The insertion depth of the ultrasonic probe was about 4 mm in all experiments, which means that the distance from the lower end of the ultrasonic probe to the bottom of the beaker was about 50 mm. This is  $\frac{3}{4}$  of the wavelength of acoustic waves in water at the used ultrasonic frequency of 20 kHz. Therefore no resonance may be attended in the soil water solution, which would cause undefined increases of acoustic pressure.

Dispersion experiments were performed with 4 g samples of air-dried aggregates (2 000 - 1 000  $\mu\text{m}$ ) in 80 ml de-ionised water. The soil was inserted shortly before the ultrasonic treatment and the solution was stirred with a magnetic device (2 Hz, cylindrical shape with length 25 mm and thickness 8 mm) to obtain homogeneous soil distribution. Stirring started simultaneously with the ultrasonic vibration and was continued during the experiments. The soil samples were analysed with five repetitions from each plot and tillage variant ( $n = 30$ ).

Ultrasonic dispersion was used to study the process of soil aggregate disruption after different applied specific energies. The soil macro aggregate fraction 2 000 - 250  $\mu\text{m}$  was investigated to describe aggregate breakdown curves for each soil type and tillage variant after the application of ultrasonic agitation at 0.5  $\mu\text{m}$ , 2  $\mu\text{m}$  and 5  $\mu\text{m}$  for different time periods (15 s, 30 s, 45 s, 60 s, 120 s, 240 s and 480 s). Ultrasonic power determined with an energy balance described in Schomakers et al. (in press) is 0.7 W, 8.9 W and 22.3 W, respectively. 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 sonication times and the mass fractions were correlated to the absorbed ultrasonic energy per millilitre.

### Soil mass fractionation

Mass fractions in the ultrasonic experiments were determined by wet sieving after the various ultrasonic treatments. Immediately after the sonication, the soil-water suspension samples



were placed in the sieving tower (Fritsch Analysette 3 Pro) with vibration amplitude set at 0.1  $\mu\text{m}$  and frequency at 50 Hz. A standardized sieve of aperture 250  $\mu\text{m}$  was used. Sieving lasted one minute with 700 ml water. The remaining soil fraction 2 000 - 250  $\mu\text{m}$  was transferred from the sieve to a porcelain cup with distilled water, placed in an oven and dried at 105 °C for 24 h. The final mass fraction was determined with an accuracy of 0.01 g.

### Statistical evaluation

SPSS Version 8 (Bühl and Zöfel 1999) was used to calculate means and standard deviations of all data and one-way ANOVA followed by the Duncan Test ( $p < 0.05$ ) to compare means.

## **RESULTS AND DISCUSSION**

### 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 1. The most stable soil is Tulln NT with 76 % stable aggregates. Differences in SAS between the tillage systems of Tulln are not significant. The overall higher stability of these two samples is due to the higher content of organic carbon and clay. Kirchberg NT has 42 % stable aggregates followed by Kirchberg CT (33 %) and Pixendorf NT (23 %). The least stable sample according to this method is Pixendorf CT, with 8 % stable aggregates. Pixendorf is a severely degraded Chernozem. The hillside location increases the loss of clay and organic matter. Through erosion of the topsoil, calcium carbonate accumulates on the surface and forms crusts (Scheffer and Schachtschabel 2010). Root growth is aggravated, and aggregate stability lowers. In addition, it has been shown that the soil structure deteriorates through tillage (Holland 2004; Lal et al. 2007). As roots and hyphae are decomposed and not replaced by intensive tillage, the organic matter content lowers and the stability of aggregates  $> 250 \mu\text{m}$  decreases (Tisdall and Oades 1982).

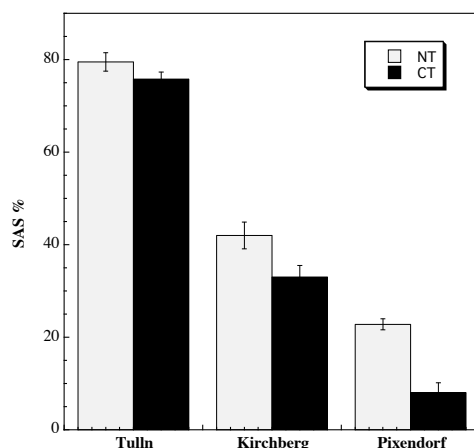


Fig.1 Soil aggregate stability (SAS) of the three investigated soils and tillage treatments (NT: no tillage; CT: conventional tillage) according to DIN-norm 19683-16. Error bars represent the standard deviation.

### Influence of absorbed specific energy on dispersion

Mass fractions of soil particles at different absorbed ultrasonic energies per millilitre and vibration amplitude of 0.5  $\mu\text{m}$  are shown in Fig 2a-c. All investigated soils show an overall decrease of the 2000 - 250  $\mu\text{m}$  aggregate fraction with increasing absorbed specific energy. At 0.5  $\mu\text{m}$ , Tulln is the most stable soil, followed by Kirchberg, and the least stable soil is

Pixendorf. This coincides with the SAS method (Fig. 1). The sand-size fractions of Tulln (Fig. 2a) steadily decrease with prolonged ultrasonic treatment. Initially the NT treatment (58 %) is significantly more stable than the CT treatment (38 %) but the difference progressively decreases and at absorbed specific energy of  $1 \text{ J ml}^{-1}$ , i.e. after 2 min of sonication, the difference is not significant.

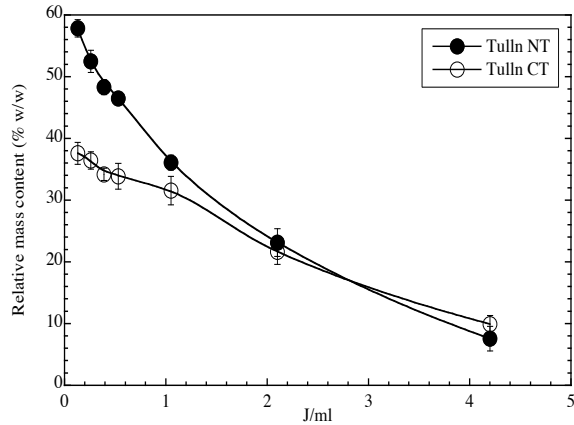


Fig. 2a

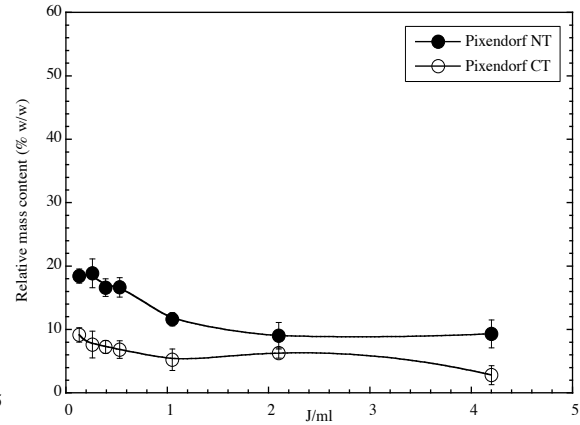


Fig. 2b

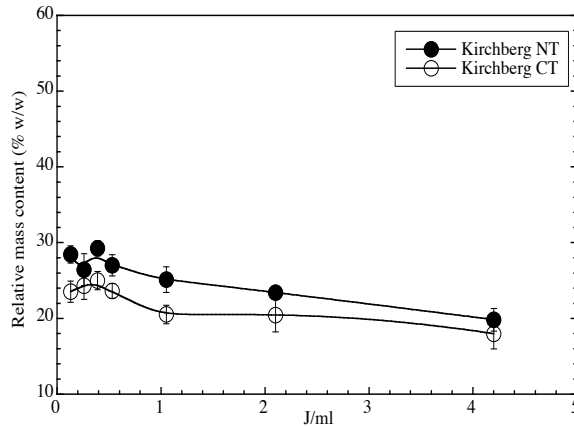


Fig. 2c

Fig. 2a-c Relative mass content of coarse and medium sand-size fractions (2 000 - 250  $\mu\text{m}$ ) at different absorbed specific energies and vibration amplitude 0.5  $\mu\text{m}$ . Data of Tulln (2a), Pixendorf (2b) and Kirchberg (2c) are shown for no tillage, NT (closed circles) and conventional tillage, CT (open circles). Error bars represent the standard deviation.

Both, the CT and NT fractions of Pixendorf (Fig. 2b) and Kirchberg (Fig. 2c) are disaggregated gradually but the CT fractions are less stable than the NT fractions throughout the treatment. The higher stability of the NT aggregates is probably caused by the higher content of organic matter, which enables the soil surface aggregates to withstand rapid wetting and mechanical forces. Differences between the tillage systems are more pronounced for Pixendorf especially up to  $1 \text{ J ml}^{-1}$ , the difference is significant. Hence, the stability of both soils, Tulln and Pixendorf can be well characterized at low vibration amplitude of 0.5  $\mu\text{m}$  up to a sonication time of 60 seconds. CT and NT fractions of Kirchberg, on the other hand, cannot be significantly distinguished at 0.7 W.

Figure 3a - c show the influence of a vibration amplitude of 2  $\mu\text{m}$  on the dispersion of the three soils. The rapid decrease of Tulln sand-size fractions  $> 250 \mu\text{m}$  (Fig. 3a) can be observed and abates at about  $6.7 \text{ J ml}^{-1}$ , i.e. after 1 min of sonication at 2  $\mu\text{m}$ . The most significant difference between the tillage variants can be observed at  $5 \text{ J ml}^{-1}$ . It is not possible to discriminate between NT and CT thereafter and up to the investigated energy of  $134 \text{ J ml}^{-1}$  (data not shown).

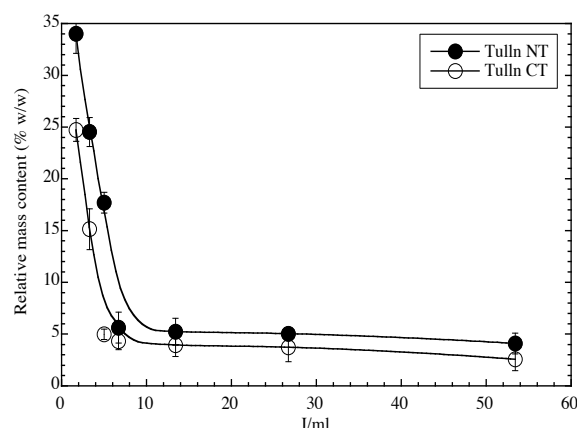


Fig. 3a

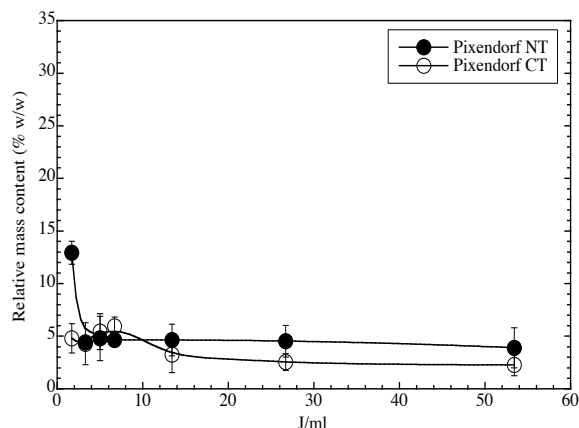


Fig. 3b

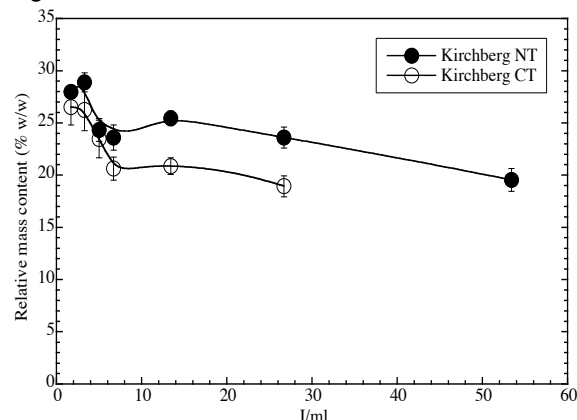


Fig 3c

Fig. 3a-c Relative mass content of coarse and medium sand-size fraction (2 000 - 250  $\mu\text{m}$ ) at different absorbed specific energies and vibration amplitude 2  $\mu\text{m}$ . Data of Tulln (3a), Pixendorf (3b) and Kirchberg (3c) are shown for no tillage, NT (closed circles) and conventional tillage, CT (open circles). Error bars represent the standard deviation.

Disaggregation is more rapid and affects more stable aggregates at higher vibration amplitude of 2  $\mu\text{m}$  compared to 0.5  $\mu\text{m}$ . The accelerated particle disruption at higher vibration amplitudes is a consequence of higher acoustic pressure emitted into the soil water solution. After 1 min of sonication at 2  $\mu\text{m}$ , the fractions of Tulln disperse almost completely to the measured final sand content (> 250  $\mu\text{m}$ ) of about 5 % at very high-absorbed energies (134  $\text{J ml}^{-1}$ , not shown).

Fig. 3b shows a significant difference between Pixendorf CT (4.8 %) and NT (13.0 %) at 1.7  $\text{J ml}^{-1}$ , only. With an ultrasonic power of 8.9 W it is therefore not possible to differentiate between the tillage variants for the degraded Chernozem with an increase of energy. This is consistent with findings of similar experiments. With an ultrasonic energy output of 8.92  $\text{J ml}^{-1}$ , Kasper et al. (2009) found no significant differences in soil aggregate stability between conventional, reduced and minimal tillage of the investigated sandy loam Chernozem.

The increase of applied energy and the change in mass distribution within the fractions were correlated and adjusted according to the regression equation in the statistical analysis. Adapting linear regression for Tulln and Pixendorf, the aggregate size fraction 2 000 - 250  $\mu\text{m}$  of Pixendorf NT correlates significantly negatively ( $r = -0.886$ ;  $p < 0.01$ ;  $n = 16$ ) by increasing the applied energy. This coincides with the same aggregate fraction from Tulln NT ( $r = -0.996$ ;  $p < 0.01$ ;  $n = 16$ ).

The 2 000 - 250  $\mu\text{m}$  fraction of the Kirchberg samples (Fig. 3c) progressively decreases with prolonged ultrasonic treatment. More aggregates of 2 000 - 250  $\mu\text{m}$ , which cannot be dispersed using 2  $\mu\text{m}$  ultrasonic treatment are present in this soil than in Tulln and Pixendorf. This can be explained by the overall high sand content in the Kirchberg samples (Tab. 1). Mass contents at high ultrasonic energy (134  $\text{J ml}^{-1}$ ) disperse to about 17 % (data not shown). Again, it is not possible to significantly differentiate between the tillage variants nor to see a substantial decline of the sand fraction 2 000 - 250  $\mu\text{m}$  at low vibration amplitude of 2  $\mu\text{m}$ . Dispersion of Kirchberg sand fractions indicates that the aggregation level is low.

Moreover the tillage experiment started in 2007, only. Differences in aggregate stability between the tillage variants might be less pronounced within the investigated triennium.

Dispersion experiments at 5  $\mu\text{m}$ , in the range of 16.7 J ml<sup>-1</sup> to 134 J ml<sup>-1</sup> (not shown) do not allow for a differentiation between soil aggregate stability nor tillage variant. The mass fractions 2 000 - 250  $\mu\text{m}$  decrease rapidly indicating that the aggregates are too weakly bonded to withstand acoustic pressure amplitudes at ultrasonic power of 22.3 W.

It is shown that the dynamic of soil dispersion depends on the absorbed ultrasonic energy and the amplitude. Influences of vibration amplitudes on soil dispersion and aggregate stability assessment have already been studied (Mayer et al. 2002; Mentler et al. 2004; Zhu et al. 2009a). Fristensky and Grismer (2008) observed declining aggregate stability with an increase in aggregate size and the relatively rapid disruption of the 2 000 - 1 000  $\mu\text{m}$  aggregates with the applied energies between 0 and 5 800 J g<sup>-1</sup>. The present results demonstrate the influence of low vibration amplitude on the dispersion of the macro aggregate fraction 2 000 - 250  $\mu\text{m}$  of two soil types under agricultural use. While the Chernozem and the loamy clay Cambisol can both be clearly characterized at energy levels up to 1 J ml<sup>-1</sup> (at 0.5  $\mu\text{m}$ ) and up to 1.7 J ml<sup>-1</sup> (at 2  $\mu\text{m}$ ), tillage systems for the loamy sand Cambisol cannot be distinguished.

## CONCLUSIONS

Ultrasonic dispersion experiments at low vibration amplitudes can show differences in aggregate stability of soils under different tillage systems (conventional tillage and no tillage). Further analysis of the soil fraction 2 000 - 250  $\mu\text{m}$  reveals different dispersion behaviour between the investigated soils with an increase in applied energy. Compared to 2  $\mu\text{m}$ , 0.5  $\mu\text{m}$  has a low impact on soil aggregates. Forces exerted on aggregates and particles at 5  $\mu\text{m}$  were too large to allow for any differentiation between the soils or tillage treatments.

Commercial equipment typically works at much higher vibration amplitudes, acoustic pressures and ultrasonic power (Roscoe et al. 2000; Zhu et al. 2009b) than that used in the present study. This limits a comprehensive characterisation of soil aggregation, where gentle stressing of less stable aggregates is preferable. Ashman et al. (2009) investigated soil stabilisation by biological processes and compared slaking, shaking and ultrasound procedures for step-wise aggregate fragmentation. The applied ultrasonic power of 100 W was not suited for biological studies since it aggressively disturbed the microbial community. In future, experiments at low vibration amplitude could be an alternative fractionation method in biological studies but more research is required.

This study, however, supports the conclusion of others (Holland 2004; Lal et al. 2007; Kasper et al. 2009) that conventional soil cultivation decreases aggregate stability. With experiments at 0.5  $\mu\text{m}$  it is possible to demonstrate influences of tillage on soil aggregate stability of relatively stable arable soils as well as of unstable soils. The results suggest similar ranking of the soils concerning their stability compared to the wet-sieving method (SAS), and indicate that both, the wet-sieving method and the dispersion at low vibration amplitude are important for evaluating changes in soil structure parameters.

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