Application of a novel method for soil aggregate stability measurement by laser granulometry with sonication

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Summary

Aggregate stability is an important physical indicator of soil quality, and so methods are required to measure it rapidly and cost-effectively so that sufficient data can be collected to detect change with adequate statistical power. The standard methods to measure water stable aggregates (WSA) in soil involve sieving, but these have limitations that could be overcome if the aggregates were measured with a laser granulometer (LG) instrument. We present a novel method in which a LG is used to make two measurements of the continuous size distribution (<2000 µm) of a sample of aggregates. The first measurement is made on the WSA after these have been added to circulating water (initial air-dried aggregate size range 1000 to 2000 µm). The second measurement is made on the disaggregated material (DM) after the circulating aggregates have been disrupted with ultrasound (sonication). We then compute the difference between the mean weight diameters (MWD) of these two size distributions; we refer to this value as the disaggregation reduction (DR; µm). Soils with more stable aggregates, which are resistant to both slaking and mechanical breakdown by the hydrodynamic forces during circulation, have larger values of DR. We applied this method to 6 and 10 sub-samples, respectively, of soil aggregates (each *ca.* 0.3 g) from bulk soil material from two contrasting soil types from England, both under conventional tillage (CT). The mean DR values were, respectively, 178.4 and 30 µm, with coefficients of variation of 12.1 and 19% suggesting the DR value is reproducible for the small mass of soil used. We attribute the larger DR values to the greater abundance of micaceous clay minerals in one of the soils. The DR values computed for each Blackwater Drain (BD) sample after removal of organic matter (with hydrogen peroxide) were comparable to those subject to sonication suggesting that most of the aggregate structure is removed by sonication. We used aggregates (1000 to 2000 µm) from soil samples collected at 30 locations under CT (median soil organic carbon (SOC) =1.4%) across two types of parent material in the Blackwater drain sub-catchments of the Wensum catchment (Norfolk, England). These soils had no coarse WSA, so we rescaled the size distributions to estimate DR for particle diameters <500 µm. Dithionite extractable iron concentration, plus a minor contribution from parent material class, accounted for 64% of the variation in rescaled DR highlighting the importance of crystalline iron oxyhydroxides for aggregate stability in this region where long-term arable production has reduced top-soil SOC concentrations. We discuss how this technique could be developed to monitor aggregate stability as a soil physical indicator.

Introduction

Soil aggregation is a fundamental property of soils and is a primary control of aeration, hydrological properties such as water-holding capacity and the storage of organic carbon (Bronick & Lal, 2005). The stability of soil aggregates is also important because it influences how these properties change with time, and the susceptibility of soils to erosion by both wind and water. In this paper our focus is on the stability of soil aggregates in water. There is evidence that the stability of soil aggregates in temperate climates may decline in soils with organic content below some critical threshold (Webb & Loveland, 2003). To detect long-term trends in soil aggregate stability (AS), a key soil physical indicator, regulatory authorities require sensitive, rapid and cost-effective techniques which can be applied to samples collected from soil monitoring networks (Merrington *et al.*, 2006).

A wide range of methods for measuring water stable aggregates (WSA) has been developed and applied (Le Bissonais, 1996; Amezketa, 1999). A framework for assessing AS was presented by Le Bissonais (1996) incorporating both fast and slow-wetting of aggregates, the latter typically reducing the effects of slaking relative to the other aggregate breakdown mechanisms. In most of these methods, aggregates are passed through a set of sieves of particular mesh size. The limitations of sieve-based methods to measure the stability of soil macro-aggregates include: (i) the mass of stable aggregates is measured for only a few, discrete, sieve size fractions, (ii) no account is taken of the particle size distribution of the sub-sampled material and (iii) they are labour intensive.

With modification, these limitations could be overcome by undertaking AS measurements with a Laser Granulometer (LG) instrument, but this technology has not been widely applied to the quantification of AS of soils. We have developed a novel method to quantify the stability of macro-aggregates (1–2 mm) in circulating water of low ionic strength. We used a restricted size range because analyses have shown that results are more reproducible when this is the case (Kay & Dexter, 1990). In our method, soil aggregates suspended in water are circulated from a vessel through an LG analytical cell. Hydrodynamic forces in the circulating water lead to the breakdown of unstable aggregates; the particle size distribution (psd) of WSA is then determined by the LG instrument. The suspension remains in the vessel connected to the LG instrument, and the particles are thoroughly disaggregated by ultrasound (sonication), and the psd of the disaggregated material is measured. A major advantage of this technique is that we use the difference in the continuous size distribution of WSA and the disaggregated material (DM) to quantify the magnitude of aggregated material. We do so by computing the difference in mean weight diameter MWD (µm) between these two continuous distributions which we refer to as disaggregation reduction (DR - the reduction in MWD on disaggregation by sonication). This method accounts for both slaking and the mechanical breakdown of aggregates, the latter often associated with raindrop impact, but not the physico-chemical dispersion influenced by the electrolyte concentration of the soil solution.

A number of studies have used a LG to investigate the impact of specific mineral phases or organic matter (OM) on changes in soil aggregates (Buurman *et al.*, 1997; Muggler *et al.*, 1999) and more specifically, aggregate stability (Bieganowski *et al.* 2010; Mason et al., 2011; Fristensky & Grismer, 2008). In a comparison between soil aggregate disintegration (stability) measured by continuous water circulation through a LG and a wet-sieve based method, Bieganowlski *et al.* (2010) concluded that the former gave similar results to the latter for three different soil types. However, in contrast to the approach that we adopt in our study, the aggregate structure of the samples analysed by LG by Bieganowlski *et al.* was not destroyed and so the psd of the WSA and the DM could not be compared. Aggregates of varying stability were observed in the same size class for LG-based analyses of several aeolian sediments studied by Mason *et al.* (2011). The energy required to disrupt aggregates has been studied by the application of sonication to disaggregate soil material, and the size distribution measured using a combination of wet-sieving and pipette methods (Zhu *et al.* 2009), electrical sensing approaches (Coulter principle) for the finer (2-50 µm) fractions and also LG (Fristensky & Grismer, 2008). An effective technique should be able to measure small differences in AS between soil samples to determine those soil properties which confer greater stability. Previous studies have identified a range of soil properties which influence the stability of aggregates in water including OM (Haynes & Swift, 1990), iron oxyhydroxide concentration and crystallinity (Duiker *et al.*, 2003), exchangeable sodium percentage (Emerson, 1967), and clay mineralogy (Emerson, 1964).

We applied our LG-based technique to determine the magnitude of sub-sampling plus analytical variation of DR by analyses of numerous sub-samples of two, bulk soil samples. We then compared the magnitude of DR for two contrasting topsoils under conventional tillage to determine whether these differed with respect to the sizes of water-stable aggregates, and whether these differences were consistent with their soil properties. Finally, we measured DR for a set of 30 top-soil samples from across a set of four, small arable catchments (Blackwater drain; BD) part of the larger Wensum catchment of Norfolk where the transport of fine sediments to watercourses frequently leads to deterioration of water clarity (Coombes *et al.*, 1999). Median top-soil organic carbon (SOC) concentrations from these catchments (1.4%) are below the average SOC concentrations for soils in arable land for this region (2.5%; unpublished data from the British Geological Survey). We wished to determine whether specific soil properties in soils from these catchments, the quantity of soil sesquioxides, inorganic and organic carbon, could account for quantitative differences in AS.

Materials and Methods

Study sites and soil sampling

The locations of the two study sites are shown in Figure 1. The soils of the BD catchments are dominantly classed as Cambisols (IUSS Working Group WRB, 2006) and their texture class varies from sandy loam to clay based on the Soil Survey of England and Wales classification (Hodgson, 1974). More than 90% of the land across the catchments is cultivated by conventional tillage (CT). Across the BD catchments top-soil samples were collected from fifty locations; twenty-five soil sampling sites were selected independently and at random from within each of the two dominant parent materials (glacial till and sands and gravels: see Figure 1). Mean annual rainfall is around 620 mm. The soil sample locations were recorded with a kinematic differential GPS (2 cm accuracy). At each sampling location, a Dutch auger was used to collect top-soil (between 0 and 15 cm depth from the soil surface) at the corners and centre of a square of side length 2 metres to form a composite sample. The soil samples were collected during February 2011. The soils at the Bunny farm (BF) site have developed over a mudstone parent material and the soils are dominantly classed as Luvisols (IUSS Working Group WRB, 2006) with a soil texture class of clay loam or clay. The dominant crops grown at this and the BD site are cereals and oilseed rape, also under CT. Mean annual rainfall is around 600 mm. The top-soil sample from BF was a composite of material from four cores collected from locations separated by 1 metre along a transect; the soil was collected at depths between 2.5 and 7.5 cm from the soil surface during August 2011.

For each soil sample around 1 kg of topsoil was collected and returned to the laboratory in plastic bags and was immediately air-dried at room temperature, then sieved to pass 2 mm. The samples were then coned and quartered to retrieve a 50-g sub-sample and this material was ball-milled and used for a range of chemical analyses. The remainder of the <2-mm size sub-sample was retained to measure AS and mineralogy.

Total Organic Carbon

For the BD soil samples, a mass of 0.2 g of milled sample was weighed out and placed in a crucible. Cold 10% hydrochloric acid (HCl) was then added dropwise to each sample until it was wet, and a vacuum bath on which the crucibles were standing was turned on. More 10% HCl was added until the mixture ceased to react. This process was then repeated using 10% HCl at 95°C, followed by concentrated HCl at 85°C. Each aliquot of HCl was allowed to drain through the crucible prior to addition of the next aliquot. The purpose of this was to remove all inorganic carbon from the sample in order to obtain the TOC content. Each sample was then washed with hot (95°C) distilled water and placed in an oven at 105°C for at least two hours to dry. The samples were then cooled in a dessicator for at least thirty minutes, re-weighed and TOC determined with an ELTRA CS800 analyser (Eltra GmbH, Neuss, Germany). An internal reference material with a known quantity of organic carbon was also analysed five times throughout the analyses to assess accuracy and precision; the coefficient of variation was 1%.

For the BF sample, TOC was measured by loss on ignition. Samples were initially dried at 105°C to remove any residual water. Each soil was weighed prior to, and after, heating, the decrease in weight was calculated as a proportion of the initial weight and expressed as a percentage weight loss. Samples were heated in a furnace at 450°C for four hours and allowed to cool in a dessicator prior to weighing to produce loss on ignition data, which was used as a measure of the organic carbon content of the sample. It has been reported that the average carbon content of OM is approximately 58% (Broadbent, 1953), therefore an estimate of carbon content was calculated by multiplying the OM content (%) by 0.58.

Calcium carbonate content

The calcium carbonate ($CaCO_3$) content of the BD samples was determined by acidifying the soil and back-titration with alkali using an indicator. Ten grammes (±0.01 g) of air-dried soil sample was weighed into a 250-ml conical flask and 20 ml of 2N volumetric hydrochloric acid was added by pipette. The flask was transferred to a hotplate and gently heated before boiling for ten minutes. After cooling, the suspension was transferred quantitatively into a 100-ml volumetric flask via a filter funnel and Whatman No.1 filter paper and made up to volume. Ten ml of the resulting solution was pipetted into a 250-ml conical flask. Approximately 50 ml of deionised water and a few drops of phenolphthalein indicator were added. The solution was titrated with 0.2N NaOH until a permanent pink colouration remained. The endpoint of NaOH titration was used to calculate the mass of calcium carbonate in each sample and expressed on a dry mass basis. Repeated analyses of sub-samples showed the reported values were reproducible.

Dithionite and oxalate extractable iron and aluminium

These analyses were undertaken for the top-soil samples from the BD catchments. For the dithionite extraction, 1 g ground soil was weighed into a 30-ml centrifuge tube and 20 ml of 25% (w/v) sodium citrate ($Na_3C_6H_5O_7.2H_2O$) was added. A further 5 ml of 10% (w/v) sodium dithionite ($Na_2S_2O_4$) was added and the suspension was shaken overnight. The samples were then centrifuged at 1370 **g** for four minutes. A 10-ml aliquot was then removed by pipette. For the oxalate extraction, 1.5 g of ground soil was weighed into a centrifuge tube and 25 ml of ammonium oxalate (28.4 g l^{-1} = 0.2M) and oxalic acid (15.76 g l^{-1}) was added. The sample was shaken in darkness for two hours, and the supernatant filtered through a 0.45-µm filter membrane. The concentrations of Fe and Al in each of the two extracts was determined by ICP-AES and the results were expressed on a dry mass basis (mg kg^{-1}) as the concentration of dithionite extractable iron (Fe_d) and aluminium (Al_d), and oxalate extractable iron (Fe_o) and aluminiun (Al_o), respectively. No duplicate extractions were undertaken on subsamples.

Particle size distribution of soil material

The psd of both the soil aggregates (between 1 and 2 mm diameter) and the full size range of soil material (0 to 2000 µm range) was also measured for each of the BD and BF samples. We placed 1 g of soil in a test tube and added 10 ml H_2O_2 (30% in water). The mixture was allowed to stand for one hour, then heated at 60 °C for an hour and finally boiled for one hour while the volume was maintained at 10 ml by addition of water. After the mixture had stood for one hour a further 5 ml H_2O_2 was added and the mixture heated as above for two hours. The mixtures were then boiled for a further hour whilst the volumes were maintained at 10 ml, and boiling was continued as necessary until frothing ceased. Finally, 25 ml of calgon solution (35 g $(NaPO_3)_6$

plus 7 g $NaCO_3$ disolved in 1 litre of distilled water) was added to the residue and the mixture was shaken and then allowed to stand for 30 minutes after which the psd was determined using LG specifying an an 8µm threshold between the clay and silt size fractions (Konert & Vandenberghe, 1997).

Soil mineralogy

The BF sample and a single sample from the set BD soils were selected to determine their mineralogy. Repeated sub-samples of this material were those also subject to AS analysis (see below). Around 100 g of <2-mm soil was passed through a 63-µm sieve and this material was used in the XRD analysis. This was carried out using a PANalytical X'Pert Pro series diffractometer (PANalytical, Almelo, The Netherlands) equipped with a cobalt-target tube, X'Celerator detector and operated at 45kV and 40 mA. The samples were scanned from 4.5–85°2ϕ at 2.76°2ϕ per minute. The diffraction data were then initially analysed using PANalytical X'Pert Pro software coupled to the latest version (2009) of the International Centre for Diffraction Data (ICDD) database. Following identification of the mineral species present in the samples, mineral quantification was achieved using the Rietveld refinement technique (Snyder and Bish, 1989) using PANalytical HighScore Plus software.

Preparation of samples for aggregate stability

The samples from BD and BF had been stored in plastic bags (air-dried) for nine and six months respectively prior to assessing their AS. A mass of around 500 g of each soil sample was sieved to select aggregates in the size range between 1 and 2 mm; a subsample of around 10 g of aggregates was then transferred to a petri dish and examined under a strong light source. Any single mineral fragments (typically large quartz or flint grains) were removed with tweezers and stored separately to exclude them from AS measurement. Examples of single mineral fragments and fragments with partial mineral associations (between 1 and 2 mm diameter) removed from the BD soil samples are shown in Figure 2.

Aggregate stability test

Our aggregate stability measure is the difference between two measurements of MWD for a soil specimen. The first MWD measurement is made after the soil has been subject to circulation in water and the resulting mild disruptive forces (water stable aggregates). The second measurement is made after applying a sonication treatment which subjects soil aggregates to strong disruptive forces. The difference between the first and second MWD is called the disaggregation reduction or DR. If two soils differ with respect to the stability of their aggregates in water then we would expect the soil with the more stable aggregates to have the larger DR.

Note that the soil after applying sonication is regarded as a baseline state against which to compare the MWD of the water-stable aggregates. This is an operationally defined baseline state, which depends on the particular way in which the sonication treatment is applied which must therefore be standardized for any study in which results are to be compared between soils. The advantage of this procedure is that it is feasible to make both MWD measurements on a single soil specimen, as described later. This removes the potential source of sampling error that would be introduced if the two MWD measurements were made on different sub-samples of the soil. An alternative baseline state would be the soil particles after removal of organic carbon, as is done in a standard particle size analysis (Gee & Or, 2002). However, it is not practically feasible to undertake the removal of organic carbon from the same soil material used to determine the MWD of the water-stable aggregates since this would require the extraction of all the soil material from the LG and the separation from the bulk suspension, after rinsing of the LG vessel, of all the particles without preferential loss of finer material. We regard the operationally defined baseline state, sonicated material, as suitable for our purposes. This is because practical interest in aggregate stability of the soil is generally concerned with the soil's ability to maintain key functions when subject to mechanical stressors such as traffic, poaching by livestock and the impact of raindrops. It does mean, however, that the DR does not account for the presence of extremely resistant aggregates that are stabilized by organic carbon. As part of this study we compared the MWD of stable aggregates from sub-samples of soil specimens with that of subsamples of the same specimens after removal of organic carbon. This allowed us to evaluate the difference between the DR calculated for the two baseline states: material after applying sonication and material after removal of carbon. It also allowed us to evaluate the effect of using separate subsamples of soil material to determine the MWD of the water-stable aggregates and the baseline material as is necessary if the latter requires that carbon has been removed.

In the following paragraphs we describe in detail the procedures followed to determine the two MWD values needed to compute the DR.

The measurements of MWD to compute the DR were made with a LG instrument (Beckman Coulter LS 13320, Brea, CA (USA)) connected to an aqueous module; a calibrated temperature probe and sonicator probe were inserted into the aqueous module. The instrument measures the volume proportion (%) in 117 size classes from <0.04 µm to <2000 µm (the number of the fixed size classes is greater in the finer size range). The finest size material (<1 µm) was measured with an additional instrument component measuring particle scattering intensity difference between vertically and horizontally polarized light (PIDS; Xu, 2008). This technology overcomes the problems typically associated with sizing finer particles (<1 µm) by laser diffraction. The instrument was switched on at least two hours prior to analysis to ensure that it was at its operating temperature. The arrangement of the LG instrument and the aqueous module to which aggregates are added is shown in Figure 3.

First, the aqueous module, which is connected in a continuous loop with the LG analytical cell *via* two pipes (total fluid volume 1250 ml), was thoroughly rinsed with tap water and then flushed twice with reverse osmosis (RO; 16–17 MΩ) water which was at a consistent temperature (between 19 and 21°C). The water vessel was then filled with RO water and the water temperature recorded. The RO water contains very little excess CO_2 which can de-gas from mains water forming bubbles that can cause measurement errors. The speed of the pump, which circulates water through the loop between the aqueous module and the LG, was increased to the fast flow rate (73 ml s^{-1}) for 30 seconds and then decreased to purge any bubbles from the system. The LG detectors were then aligned and the background scatter determined across the full range of detector angles.

Soil aggregates (total mass between 0.2 and 0.4 g) were then transferred from a sample container to the RO water in the aqueous vessel until around 3% light obscuration was reported by the LG. The pump speed was then immediately increased to the fast rate (73 ml s^{-1}) to ensure all aggregates were in circulation. Preliminary tests showed that at slower flow rates, larger aggregates remained at the base of the aqueous vessel which leads to biased measurements of psd. After five seconds had elapsed the psd of the aggregates was measured continuously for 90 seconds by the LG; the temperature of the suspension and light obscuration (reported by the LG) at the start and end of this period were recorded. The pump speed was then reduced to the slow rate (14 ml s^{-1}) and the sonicator in the aqueous vessel (power rating 18 W) was switched on for ten minutes. Tests showed (results not presented) that this was sufficient time to break apart any aggregates which still remained; applying further sonication did not lead to a significant reduction in particle size. The temperature of the solution was measured again so the power used in heating could be calculated (North, 1976):

$$P_h = (m_w c_w + m_s c_s + m_v c_v) \frac{\Delta T}{\Delta t}, \tag{1}$$

where m_w is the mass of water, c_w is the specific heat of water (4.18 J g^{-1} at 20°C, m_s is the mass of oven-dried soil, c_s is the specific heat of the soil, m_v is the mass of the container, c_v is the specific heat of the container and ΔT is the temperature change in the suspension over the time period (Δt - ten minutes of sonication).

The pump speed was then increased to the fast rate once more and the psd measured for a further 90 seconds. Light obscuration (%) at the end of each analysis was also recorded. On a few occasions, when too much sample had been added initially to the aqueous vessel, light obscuration after sonication exceeded 18% which can reduce the accuracy of psd measurement. In such cases the sample was flushed from the system and the analysis repeated with a smaller sample mass to ensure obscuration after sonication was $< 18\%$. Between each analysis the aqueous cell was then rinsed thoroughly with mains water before the next analysis was undertaken. In common with other aggregate stability methods (Zhu *et al.*, 2009), we cannot quantify the mechanical energy leading to the breakdown of the initial aggregates (between 1 and 2 mm in diameter). In our method, this energy comprises: (i) the hydrodynamic forces of the circulating water, (ii) collisions between soil aggregates and the surfaces of the circulating system, and (iii) particle–particle collisions during circulation.

Two standard particle size materials (supplied by Beckman Coulter; mean diameter 32 and 500 µm) were used throughout the series of analysis to check for accuracy and precision of the psd measurements by the LG instrument. The psd analyses for each of the two measurements on each sample (WSA and DM) were reported as the volume proportion (%) for the size classes.

Calculation of disaggregation reduction

For each psd measurement the mean weight diameter was calculated as:

$$\mathrm{MWD} = \sum_{i=1}^{n} \bar{x}_i w_i, \tag{2}$$

where $\bar{x}_i$ is the mean diameter of each size fraction (µm), and w_i is the volume proportion (expressed as a decimal proportion) of the sample corresponding to that size fraction. We calculated the difference between the MWD of water-stable aggregates and MWD of the DM and refer to this as the disaggregation reduction (DR; µm) in other words the reduction in MWD on disaggregation by applying sonication. Soil types with more stable aggregates have larger values of DR since the stable aggregates are resistant to both slaking and mechanical breakdown by the hydrodynamic forces during circulation, and are disrupted only by applying sonication.

Preliminary measurements of AS using soil samples from the BD showed that few, if any, of the aggregates from these soils at sizes greater than 500 µm were stable in water, but in some cases there were notable changes in stability between 400 and 500 µm. When the data for volume percentage against particle size for both water stable and sonicated material were overlain in exploratory plots the two lines were almost coincident at sizes >500 µm. During LG analyses we noted that for samples with a substantial quantity of coarse material (diameter >500 µm) the variation in passage of these particles through the analytical cell caused large differences in estimates of MWD over the 90 seconds of measurement. This could lead to a biased estimate of MWD. To overcome this potential bias when comparing values of DR for the BD samples, we re-scaled the volume proportions in all size ranges <500 µm to sum to 100%, and re-calculated the MWD for both distributions on the re-scaled data, and DR by their difference.

Measurements of aggregate stability

To assess sub-sampling and analytical variation of the AS method, we used ten separate sub-samples of aggregate material from a single soil sample from the BD catchments and analysed each using our LG-based method. We also analysed six sub-samples from the BF soil sample. In each case we calculated the coefficient of variation (%CV) of DR and the standard error of the mean for different numbers of sub-samples. We also measured DR for two sub-samples of soil material from each of another 29 soil samples from the BD catchments (total n=30). In each case we calculated the DR for each sub-sample, and the mean DR of each sample using the values for the two sub-samples. We produced scatterplots for DR plotted against the other soil properties, which could account for aggregate stability, and investigated whether they explained a significant proportion of the variation in DR by fitting ordinary least squares linear regression models. We also included the parent material classification (tills or sands and gravels) as a categorical predictor variable.

As we noted earlier, our aggregate stability measure treats the sonicated soil material as an operationally-defined standard state against which to compare the MWD of the WSA. We argued above that this standard state allows us to measure both MWD on the same subsample of soil which has practical advantages, and that it represents a meaningful baseline state because many of the threats to soil aggregation, at least in the short to medium term, are mechanical disruption. Ideally one might prefer to treat disaggregated soil from which all organic carbon has been removed as the standard state, since otherwise the DR does not represent very resistant aggregates that are stabilized by organic carbon. As noted above, we do not think that it is feasible to make determinations of MWD on WSA and soil after removal of organic carbon for the same sub-sample. If one makes the comparison between different sub-samples, then sub-sampling error will inflate the error variance of the measurements of DR. To indicate the likely difference between our operationally-defined standard state and disaggregated soil with organic carbon removed, and to evaluate the effect of the additional source of sample error, we took an additional ten sub-samples from the single BD soil specimen that we had previously sub-sampled to evaluate the variability of DR measurements (previous section) and an additional sub-samples from the BF specimen. Each sub-sample was then treated separately. Organic carbon was removed by the procedure described earlier and MWD was determined for each. We then obtained 10 determinations of DR against a baseline state with organic carbon removed (DR-OMR) by matching each sub-sample from which the organic carbon was removed with one subsample of the same specimen for which the MWD of water-stable aggregates had been determined. This matching was done at random. The statistics of DR-OMR from ten replicates from the BD soil and six replicates from the BF soil were then computed.

Results and their interpretation

Sub-sampling plus analytical variation of DR

The overall mean DR from ten replicate measurements from one specimen of soil from BD was 30.0 µm and the standard deviation was 5.7 µm, CV was 19%. For the BF sample for which six sub-samples were measured repeatedly the overall mean DR was substantially larger (178.4 µm), the standard deviation was 21.7 µm, CV was 12%. The standard error of the mean DR estimated from the analysis of two samples would be 4.0 µm and 15.3 µm for the BD and BF soils, respectively, and we considered that this would be an appropriate rate of replication, given the time required to analyse the subsamples.

Comparison of DR with DR-OMR

The overall mean DR-OMR from ten replicate measurements from one specimen of soil from BD was 54.6 µm with a standard deviation of 27.7 µm, CV was 51%. For the BF sample for which six subsamples were repeatedly measured the overall mean DR was 240.8 µm, the standard deviation was 28.4 µm, CV was 12%. Note that in both cases the mean DR-OMR is larger than the mean DR, which is to be expected because removal of organic carbon will lead to the disaggregation of aggregates which are resistant to the ultrasound treatment. The CV for DR-OMR in the case of BD was more than twice that of DR, but in the case of the BF soil that CV was the same for DR and DR-OMR. For both soils, the application of sonication removed most of the aggregate structure because the MWD of the disaggregated material was far smaller than that of the WSA.

Differences in aggregate stability between soil types

Using measurements of the temperature of the suspension before and after sonication in Equation 1, we calculated that the mean power used in heating all samples was 19.3 W (1 J s^{-1}), with a minimum of 12.8 W and a maximum of 25.2 W.

The proportion of particle diameters for the WSA and DM (after sonication) for the two soil types are shown in Figures 4a and 4b; there are several noteworthy differences between the samples. First, note the small difference between the WSA and disaggregated material (DM) above approximately 300 µm diameter in the BD soil (Figure 4a) by comparison to much greater differences in the BF soil (Figure 4b).

There are very few WSA >300 µm diameter in the BD sample, but a considerable proportion in the BF sample which also includes those of sizes >1 mm. The size range over which the volume proportion of WSA is greater than DM highlights a net loss of material; this size range is much wider (70-1500 µm) for the BF soil by comparison with the range (48-310µm) for the BD soil. The size at which there is the greatest difference between WSA and DM is larger (275 µm for the BF sample than for BD sample (90 µm)). These features all show that the BF soil had a larger proportion of WSA by comparison with the BD soil. This is supported by the mean DR values for the two samples; 30.0 µm for the BD by comparison to 178.4 µm for BF. We attribute much of this difference to the contrasting mineralogy of the soils; the BF soil has a considerably larger (31%) quantity of micaceous clay minerals (illite and smectite) than that (16%) reported for the BD soil (Table 1). The micaceous clay minerals are effective in binding together soil particles because of their large surface area and are less subject to slaking (Emerson, 1964). The SOC concentration of the BF sample was 2.5% larger than the BD sample (1.4%); this may in part also account for the greater DR value of the former as SOC can enhance aggregate stability.

Variation in BD soil properties and aggregate stability

A summary of the soil properties for the BD samples, all fifty sites and the sub-set of thirty sites for which the AS test was applied, are presented in Table 2. The median SOC concentrations are generally small (1.4%) by comparison with regional top-soil SOC concentrations from unpublished data based on sampling and analysis covering all of East Anglia (2.2%, see Figure 1). The calcium carbonate concentrations (median 2%) are consistent with the estimated values of inorganic carbon concentrations for this region presented by Rawlins *et al.* (2011) using a regression model using measurements of calcium and aluminium concentrations in topsoil. There is an approximate two or three-fold variation in the concentrations of oxalate and dithionite extractable Fe and Al for the soils across the BD catchment; the Fe_d values have the largest concentrations with a median concentration of 6232 mg kg^{-1}.

The psd of the subset of 30 samples (size range 0 to 2000 µm; organic matter removed) is shown in Figure 5; the size distribution of the DM (post sonication; initial aggregate size 1000 to 2000µm) for the same samples are also shown for comparison. The soil texture classes of the soils extend from sandy clay loam, to clay loam to clay. By selecting only large aggregates, this particulate material has substantially greater proportions of sand material and also slightly more silt content by comparison with the size range of the bulk soil.

Exploratory scatterplots showed a strong linear correlation between the concentration of Fe_d and mean DR values (rescaled from the $< 500\mu$m size range) for the subset of 30 soil samples. An ordinary linear regression model fitted with Fe_d and parent material code as predictors (Figure 6) accounted for 64% of the variation in DR (adjusted R^2). The significance of the parent material classification was marginal (P=0.11), but it was retained in the regression model because the classification had been used to stratify the region prior to sampling. The residuals of the regression model were approximately normally distributed (skewness coefficient=1.0). None of the other quantitative soil properties (SOC, calcium carbonate, Fe_o, Al_d and Al_o) were strongly correlated with DR so we considered that their role in determining AS must be secondary.

Discussion

Soils from the region of the BD catchments have total silicon concentrations in the top decile of all soils across England and Wales (Rawlins *et al.*, 2012) and are therefore likely to be among the most quartz-($\mathrm{SiO_2}$) rich samples at national scale. We think it likely that the large proportion of quartz-dominated aggregates (Figure 2) in our BD sample explains why we needed to re-scale our DR values to the sub-500-µm range. It may not be necessary to re-scale data for other samples at a national scale because they are likely to contain smaller proportions of large quartz particles. It is often not clear from sieve-based methods how the presence of abundant large, single particle mineral fragments are dealt with. What may be considered aggregated material at the start of a wet-sieve analysis may comprise a large, single mineral grain, 1 mm in diameter, for example, coated by a few, small mineral fragments. After removal of these mineral coatings during the AS test, the large grain would be trapped by the 1 mm sieve aperture and be classed as a stable aggregate, although it is not an aggregate but a single particle. In our LG method, by contrast, we can quantify the presence of such mineral fragments by comparing the size distribution of the WSA and the DM. The quantity DR accounts for the size distribution of the DM, so the presence of large mineral fragments cannot bias this measurement as in wet-sieve approaches.

The concentration of SOC across the BD catchments (median 1.4%) is likely to have been reduced during the last 50 years or more by long-term arable production under CT. Given that SOC content is one of the dominant controls on AS (Haynes & Swift, 1990), it is likely that the latter has also declined over this period. Our results suggest that the current dominant control on AS across the BD catchments is the concentration of iron oxyhydroxides. The spatial variation of iron oxyhydroxide concentrations across the BD catchments, where soils are prone to losses of fine sediment, is likely to be far greater than any temporal change. It may be possible to estimate and map iron oxyhydroxide concentrations, as a predictor of AS, cost-effectively using diffuse reflectance infra-red spectroscopy, as demonstrated for sediments from other parts of England (Rawlins, 2011). Exploratory analyses using the data from the 50 sample sites across the BD catchments show that iron oxyhydroxide concentrations exhibit substantial autocorrelation (correlated variance 87%) at moderate length scales (variogram range=4500m) which suggests that sampling intensities of 1 sample every few square kilometres would capture much of the spatial variation.

One of the advantages of the LG-based AS method is the speed with which analyses can be undertaken; there is no requirement to dry and weigh aggregates trapped on sieves of differing mesh size, or for cleaning sieves between measurements. With just two sub-samples, we estimate that a single operator could apply the test to ten independent samples per working day; or twenty samples per day if only a single sub-sample is analysed, with a small associated increase in the error of DR. Given the speed of the analysis combined with an accurate, quantitative measure of aggregate stability, we consider that this procedure could be used to measure AS as a soil physical indicator (Merrington *et al.*, 2006) in future soil monitoring programmes which will likely require analyses of many hundreds of samples. Our method needs to be tested on soils which have been subject to a range of land-management practices which are likely to have led to changes in AS to determine whether significant differences in DR can be detected. If this can be demonstrated successfully, it suggests our technique might also be able to detect small changes associated with temporal monitoring.

Our additional analyses to compute DR-OMR, the DR measure of aggregation relative to a baseline state in which the soil organic carbon is removed, showed, as expected, that some aggregates in the soil are resistant to the ultrasound treatment but are disrupted by the removal of organic matter. This is a limitation of our method, because it does not account for such aggregates. However, it is not feasible to make measurements of MWD on water stable aggregates and on the same subsample after removal of organic carbon, as we discuss below. And for one of our sites we found, as expected, that determining DR-OMR by comparing the MWD of water-stable aggregates and material after removal of organic carbon from two random sub-samples introduces a substantial additional source of uncertainty. These practical considerations are strong reasons for the use of the DR measure where the baseline state is soil disaggregated by the ultrasound treatment. As we note above, for many practical purposes, this is a relevant baseline state because the threats to soil structure are mechanical: traffic, poaching, impact of raindrops, shear-forces created by surface flow and so on. However, we acknowledge that methodological developments that allowed the MWD of water stable aggregates and material after removal of organic carbon to be determined for the same sub-sample would be advantageous.

If our method, or a modified version, were to be applied more widely, a standard, aggregated material would be required to ensure that the results of its application were sufficiently reproducible. Such an aggregated material would need to respond consistently to the disrupting forces in the circulating suspension. The field-based sampling, storage and distribution of a bulk soil reference material is unlikely to have consistent disaggregation properties because AS is known to increase with storage time (Blake & Gilman, 1970) and also vary according to sampling period according to season (Blackman, 1992) most likely because of variations in antecedent soil moisture conditions (drying-wetting cycles). One effective solution might be the establishment of a procedure for creating artificial soil aggregates from widely available materials; some combination of geological, mineral components and fresh organic matter. A group of laboratories could generate their own soil aggregates and exchange them with others in the group to assess whether the results from application of the LG-based method were sufficiently reproducible for its deployment in soil monitoring.

By using a solution of low ionic strength in our AS test, we cannot account for the effects of variations in soil solution electrolyte concentrations, and specifically exchangeable sodium which can have a significant effect on aggregate stability in certain soils (Barzegar *et al.*, 1994). Further work is required to investigate whether a solution of specific composition would be needed for soil types with large quantities of exchangeable sodium. It would also be possible use a slow aggregate wetting procedure (using, for example, a tension table; Le Bissonnais, 1996) to investigate any differences in AS with the fast-wetting procedure described in our method.

There is currently no established method for measuring aggregate bond energy using sonication whilst simultaneously measuring a continuous aggregate/particle size distribution where the latter does not contribute disruptive energy to the former. For example, by using a LG for such measurements, energy associated with circulating the soil suspension (typically water) through the LG instrument also contributes to aggregate dispersion. To overcome this impediment, we suggest it may be possible to use a dense (1.8 g cm^{-3}) liquid such as sodium polytungstate (SPT) in which micro-aggregates (< 250 µm) are suspended. Disruptive energy would be applied using sonication with a feedback mechanism (see Zhu *et al.*, 2009). The SPT will prevent the disruption of particles which typically sink rapidly in water and are subject to disruptive collisions through circulation in lower density, aqueous solutions. The suspension would be circulated at minimal velocity so that only minimal amount of energy is imparted to the micro-aggregates during analysis. It would be a simple matter to test such an approach by measuring the MWD of micro-aggregates circulated through a LG in an SPT suspension to determine the extent of any disaggregation.

Conclusions

The main conclusions from our study are as follows:

1. We presented a novel method for measuring aggregate stability, based on the measurement of size distributions of aggregated material by a laser granulometer, before and after sonication. We refer to the difference in MWD (µm) of these two size distributions as disaggregation reduction, a quantitative estimate of aggregate stability. This method has several advantages by comparison with previous sieve-based methods.

2. The coefficient of variation of DR from the analysis of six and ten sub-samples of two different soil materials was 12.1 and 19%, respectively, so the method is reproducible. The method is also rapid. We estimate that if two sub-samples of each soil material are analysed, a single operator could complete ten analyses per working day, or twenty analyses if only a single sample were analysed.

3. The DR that we determine is based on treating sonicated soil material as a baseline disaggregated state. We showed that the MWD of soil material from which organic carbon has been removed can be substantially smaller in contrast to material which has subject to sonication, which indicates that our measurement of DR does not account for very resistant aggregates stabilized by organic matter.

4. Although we acknowledge (3) above, it is not practically feasible to treat material

with organic matter removed as the baseline state within this proposed method because the MWD of water-stable aggregates and of material with organic matter removed cannot be determined from the same sub-sample of material. If different sub-samples are used then the CV of the resulting determination of the DR may be much larger than for our proposed method on a single sub-sample, and we found this to be the case at one of our two study sites.

5. The two different soils we studied, both from arable sites in England under CT, had substantially different values of DR; 178.4 and 30 µm, respectively. We attribute this to the former having substantially larger quantities of micaceous clay minerals (<63µm fraction) compared with the latter.

6. We measured DR for soils from 30 sites across the Blackwater drain catchments, part of the larger Wensum catchment in Norfolk, England. These soils had small SOC concentrations (median 1.4%) which is caused by long-term cultivation. The iron oxyhydroxide content of these soils, with a small contribution from two classes of parent material, accounted for 64% of the variation in DR. This suggests that the iron oxyhydroxide content of these soils is currently the dominant control on AS. Other quantitative soil properties (calcium carbonate, organic carbon, aluminium oxides) were not strongly correlated with AS, as measured by DR.

7. Soils from the Blackwater drain catchments also contained substantial proportions of coarse, quartz fragments which we identified by comparing the size distribution of the WSA and the DM. The quantity DR accounts for the size distribution of the DM, so the presence of large mineral fragments, which are not aggregates, cannot bias the AS measurement, which can be the case in wet-sieve approaches.

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Figure captions

Figure 1 Location of the study sites: 1. Bunny farm (Nottinghamshire). 2. Blackwater drain catchments (Norfolk) where 50 soil samples were collected over two parent material types: glacial till (dark grey) and sands and gravels (pale grey). The paler shaded area on the national map shows the region of East Anglia (see text). Coordinates are metres of the British National Grid.

Figure 2 Components of soil material (1-2 mm diameter) from the Blackwater drain soil samples which were removed after examination prior to aggregate stability tests: a) single mineral fragments, b) mineral fragments with partial covering of finer material.

Figure 3 Arrangement of apparatus used for the measurement of aggregate stability (not to scale). Arrows depict the circulation of the suspension between the aqueous vessel and the laser granulometer.

Figure 4 Size distribution (log scale) of aggregates and sonicated disaggregated material for two topsoils (with differing parent materials) : a) Blackwater drain (tills or sand and gravels), b) Bunny farm (mudstone). The original distributions in a) have been re-scaled by using the distribution of sizes <500µm. Areas are highlighted where disaggregation leads to a net loss of coarser material which accumulates at finer sizes (shown as net gain) after sonication.

Figure 5 Ternary diagram showing particle size distribution for paired sub-samples of material from 30 locations: i) filled discs with material of size range 0 to 2000µm for which organic matter had been removed, ii) open discs for sub-samples subject to aggregate stability tests and sonication (initial aggregate size range 1000 to 2000 µm without removal of organic matter). The boundaries between soil texture classes are those of the Soil Survey of England and Wales (Hodgson, 1974).

Figure 6 Scatterplot of total dithionite extractable iron concentrations (Fe_d) plotted against disaggregation reduction for 30 soil samples collected across the Wensum catchment. The solid line is the linear regression model with Fe_d as a predictor for samples over glacial till, whilst the dashed line is for the sands and gravels which has a different intercept value.

Table 1 The proportion of mineral phases in the $< 63\mu$m size fraction of a topsoil sample from each of the two study areas by x-ray diffraction analysis).

Site	albite	calcite	chlorite	dolomite	hematite	[a]kaolin	K-feldspar	[b]mica	quartz
Bunny farm	8.8	nd	1.7	nd	0.5	1.4	7.3	30.7	49.6
Blackwater drain	6.9	<0.5	0.9	0.7	<0.5	1.3	7.4	16	66.3

nd=not detected

[a] undifferentiated kaolin group minerals possibly including kaolinite, halloysite etc)

[b] undifferentiated mica species including muscovite, biotite, illite and illite/smectite

Table 2 Summary statistics for measurements on fifty top-soil samples from the Blackwater drain catchments and a sub-set of thirty samples for which aggregate stability measurements were undertaken. Of the subset of 30 samples, 17 sites were over glacial till and 13 over sands and gravel parent materials.

	all soils	sub-set
Calcium carbonate (%)		
min	1.00	1.00
max	4.90	4.90
mean	2.16	2.28
median	2.00	2.05
sd	0.88	0.95
skew	1.22	0.92
Organic carbon (%)		
min	0.49	0.49
max	13.10	13.10
mean	1.72	1.89
median	1.40	1.38
sd	1.75	2.25
skew	5.56	4.15
Oxalate extractable Al (mg kg^{-1})		
min	910	910
max	2293	1981
mean	1470	1483
median	1435	1470
sd	276.4	265.5
skew	0.59	0.03
Oxalate extractable Fe (mg kg^{-1})		
min	1229	1229
max	3902	3902
mean	2326	2450
median	2321	2399
sd	528.7	579.5
skew	0.54	0.27
Dithionite extractable Al (mg kg^{-1})		
min	1143	1179
max	2340	2269
mean	1604	1632
median	1613	1643
sd	292.2	293.5
skew	0.34	0.02
Dithionite extractable Fe (mg kg^{-1})		
min	3854	3854
max	10569	10569
mean	6760	6883
median	6232	6785
sd	1765	1883
skew	0.47	0.34

Table 3 Regression coefficients of the ordinary least squares model between dithionite extractable iron and parent material class (predictors) and disaggregation reduction (predictand) from analysis of soil samples for 30 locations across the Blackwater drain catchments.

	Estimate	Std error	t	P
Intercept	30.4	11.5	-2.64	0.01
Fe_d	0.0094	0.0014	6.6	44.2×10^{-8}
^{a}SG	8.84	5.31	1.66	0.11

^{a} intercept coefficient for the SG (sands and gravels) parent material

Figure 1:

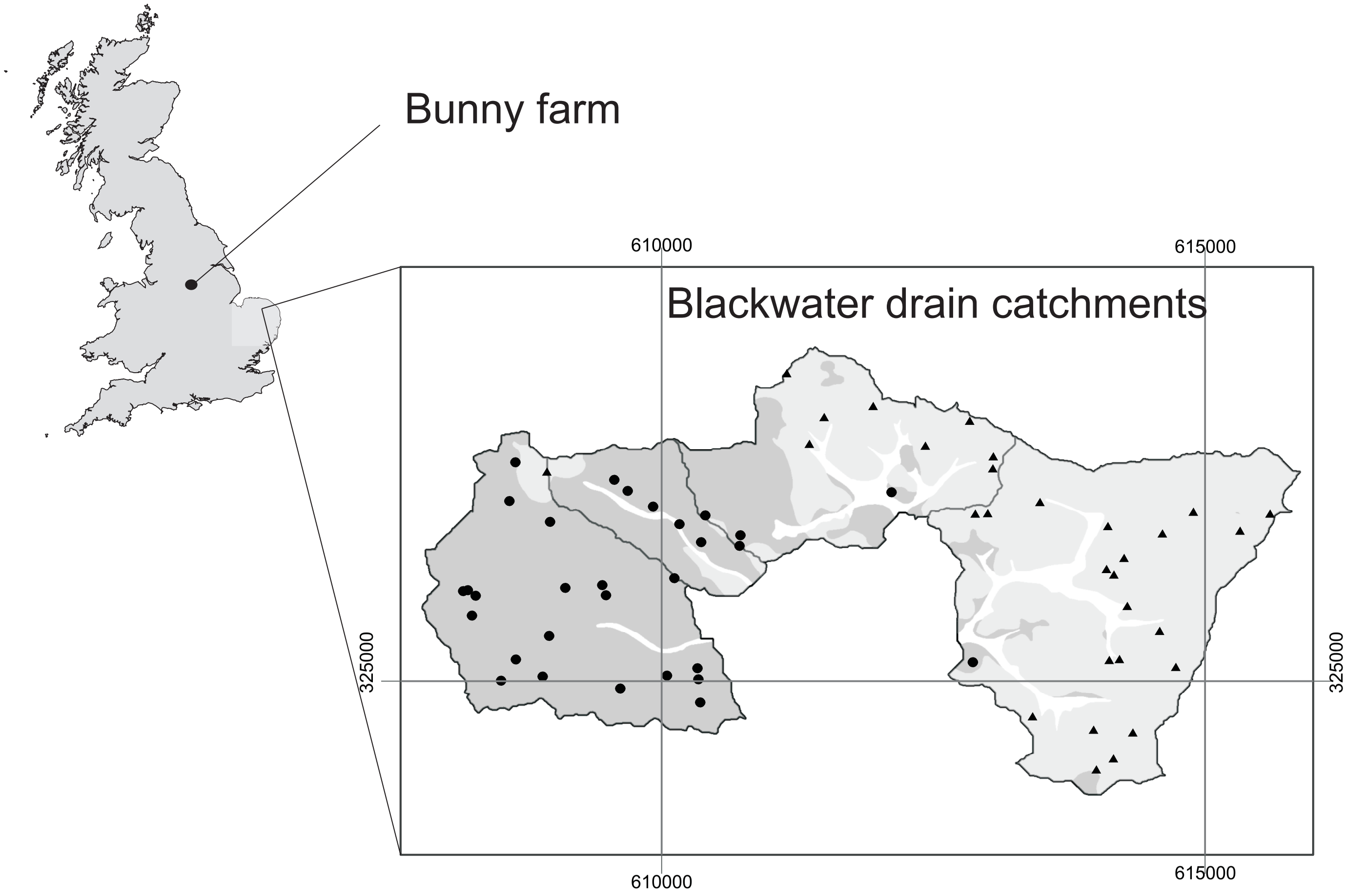

Bunny farm
Blackwater drain catchments
610000
615000
325000
325000
610000
615000

Figure 2:

Figure 3:

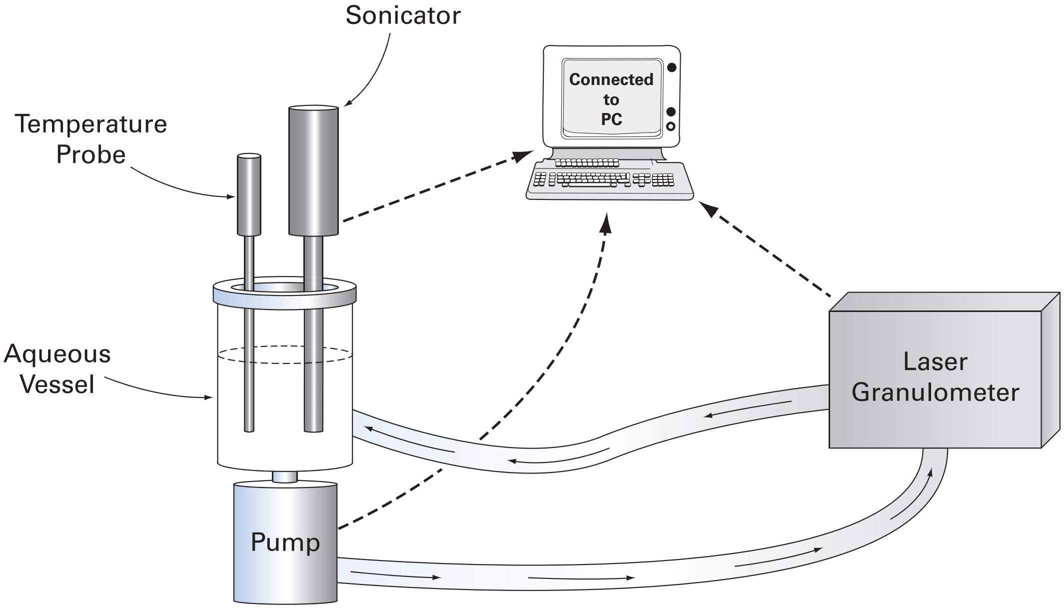

Sonicator
Connected to PC
Temperature Probe
Aqueous Vessel
Laser Granulometer
Pump

Figure 4:

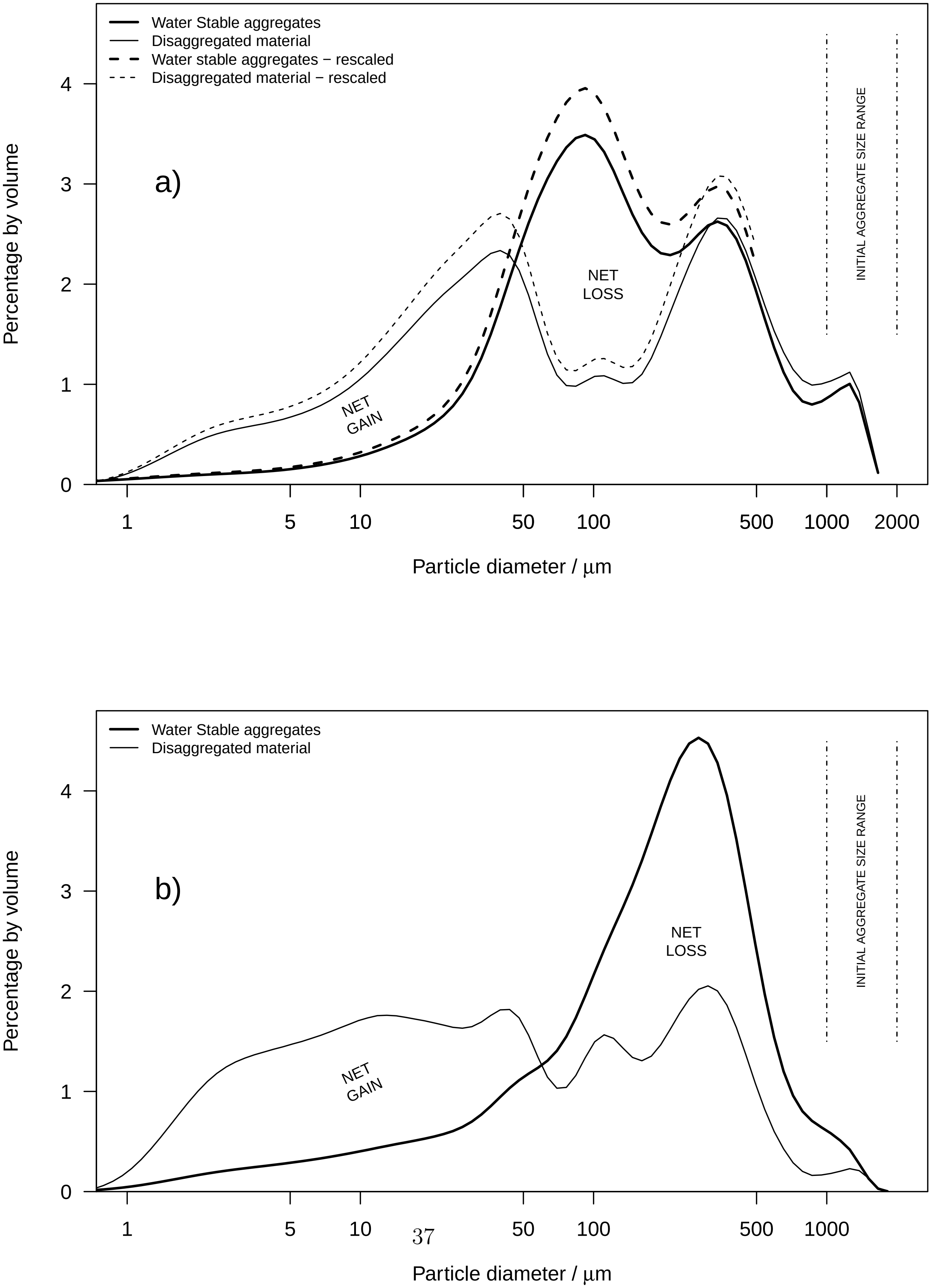

Water Stable aggregates
Disaggregated material
Water stable aggregates − rescaled
Disaggregated material − rescaled
a)
NET GAIN
NET LOSS
INITIAL AGGREGATE SIZE RANGE
Percentage by volume
0
1
2
3
4
1
5
10
50
100
500
1000
2000
Particle diameter / µm
Water Stable aggregates
Disaggregated material
b)
NET GAIN
NET LOSS
INITIAL AGGREGATE SIZE RANGE
Percentage by volume
Particle diameter / µm

Figure 5:

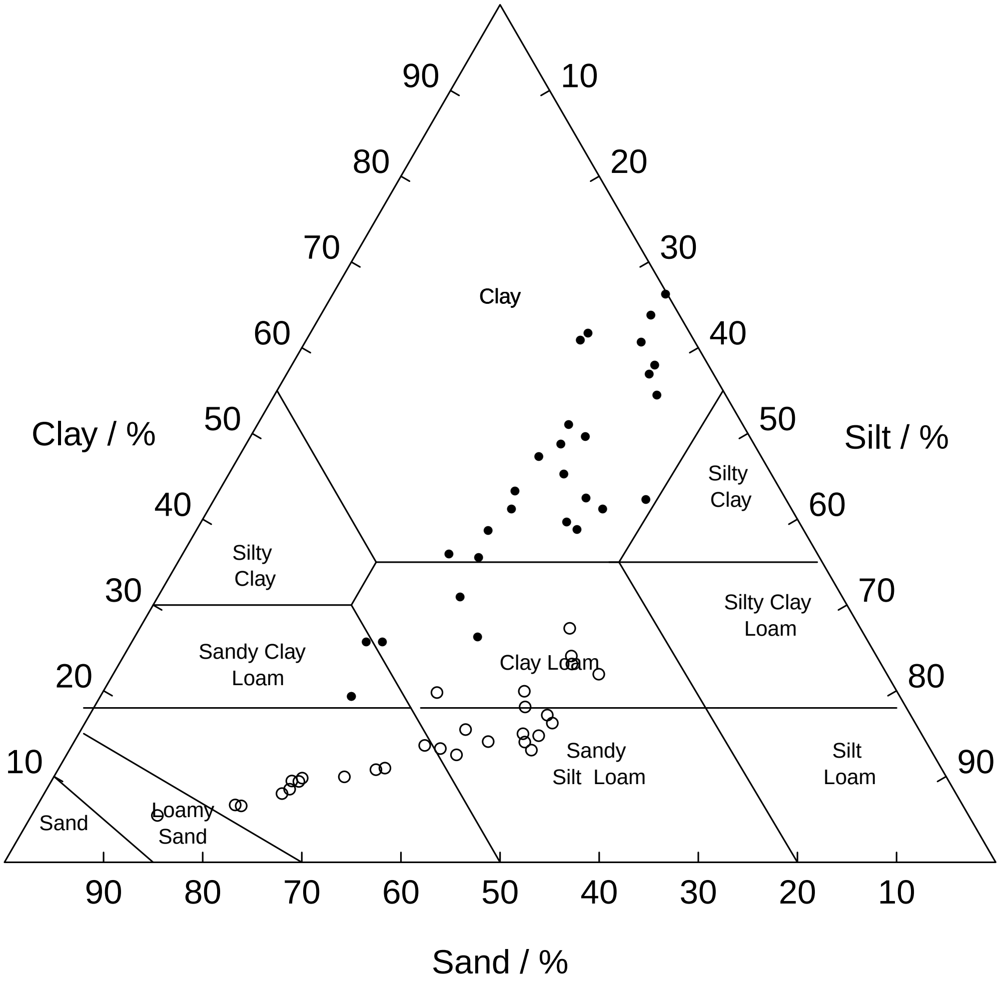
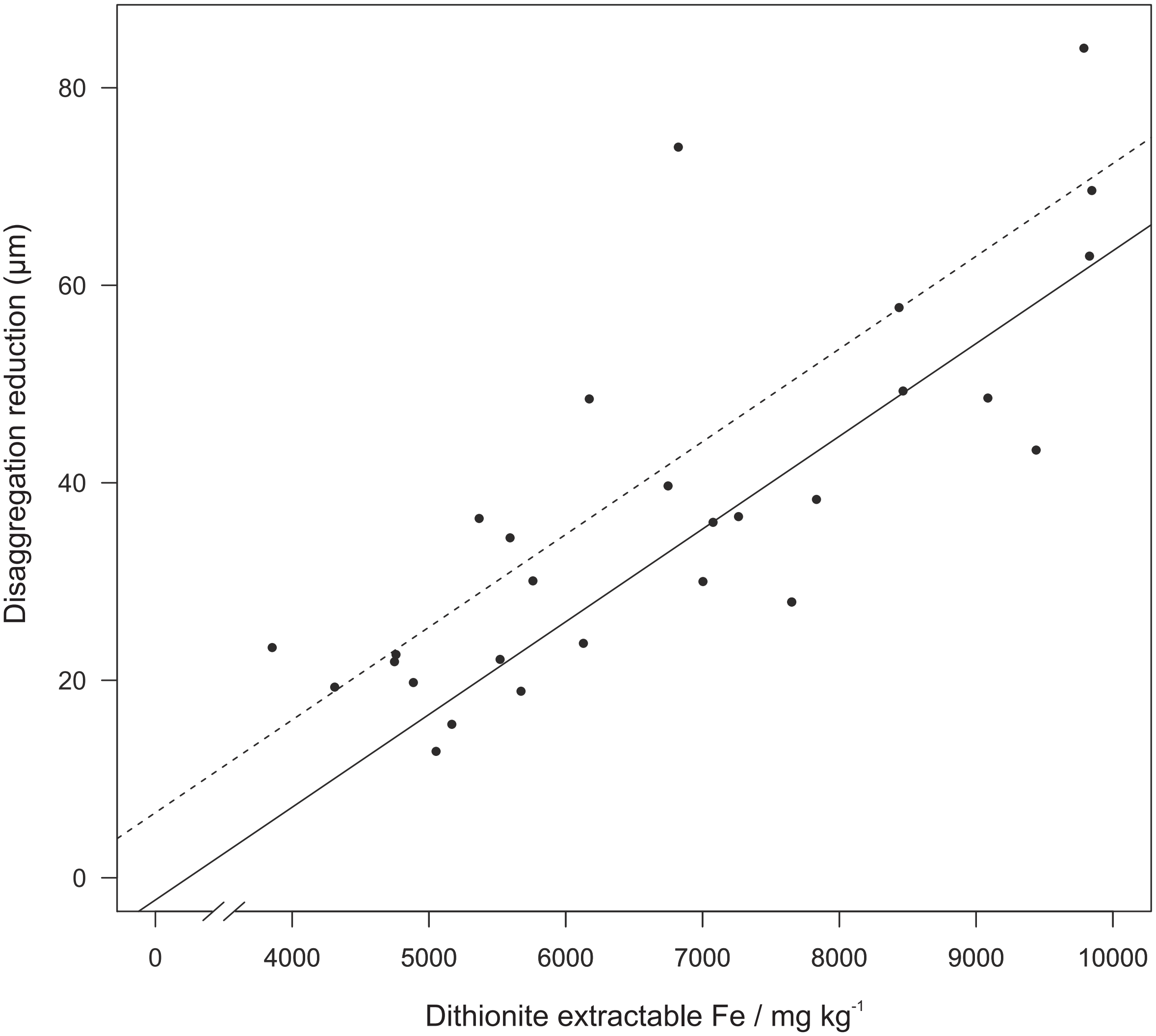

Clay / %
Silt / %
Sand / %
90
80
70
60
50
40
30
20
10
10
20
30
40
50
60
70
80
90
90
80
70
60
50
40
30
20
10
Clay
Silty Clay
Silty Clay
Sandy Clay Loam
Clay Loam
Silty Clay Loam
Sandy Silt Loam
Silt Loam
Sand
Loamy Sand

Figure 6:


Disaggregation reduction (µm)
0
20
40
60
80
0
4000
5000
6000
7000
8000
9000
10000
Dithionite extractable Fe / mg kg-1