### **Manuscript Details**

Manuscript number GEODER\_2018\_52

Title Relating soil C and organic matter fractions to structural stability

Article type Research Paper

#### **Abstract**

Soil organic matter (SOM) is important for maintaining soil structural stability (SSS). The influence of soil organic carbon (SOC) and different organic matter components on various SSS measures were quantified. We used a silt loam soil with a wide range of SOC (0.0080-0.0427 kg kg-1 minerals) sampled in spring 2015 from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research. Four treatments were sampled: Bare fallow, continuous arable rotation, ley-arable rotation, and grass. Soils were tested for clay dispersibility (DispClay), clay-SOM disintegration (DI, the ratio between clay content without and with SOM removal) and dispersion of particles <20 µm. The SSS tests were related to SOC, permanganate oxidizable carbon (POXC), hot water-extractable carbon (HWC), mid-infrared photoacoustic spectroscopy (FTIR-PAS) and mineral fines/SOC ratio. SSS increased with increasing content of SOM components. The relationships between SOM components and SSS followed a broken-stick regression with a change point at ~0.0230 kg SOC kg-1 minerals (clay/SOC~10) coinciding with a change from the tilled treatments to the grass treatment. We found a greater influence of SOC, POXC and HWC on SSS at contents below the change point than above. A stronger linear relation between POXC and DispClay compared to SOC and HWC suggests that POXC was a better predictor of the variation in DispClay. POXC and HWC were less related to DI than SOC. The grass treatment had a very stable structure, shown in all SSS tests, probably due to the absence of tillage and large annual inputs of stabilizing agents. This suggests that a change in management from arable rotation to permanent grass is an effective tool for improving SSS.

**Keywords** soil structural stability; soil organic carbon; permanganate oxidizable carbon; hot

water-extractable carbon; soil management

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Suggested reviewers Mike Beare, Hugh Riley, Denis Angers, Cassio Antonio Tormena, Pascal Boivin

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January 18th, 2018

To the Editorial Board of *Geoderma*,

Please consider the attached manuscript entitled "Relating soil C and organic matter fractions to structural stability" by Jensen et al. for review and publication in *Geoderma*. It describes original research not published elsewhere and not submitted for publication in other journals.

Soil organic matter (SOM) is important for maintaining soil structural stability (SSS). The current study relates a number of SOM components to various SSS tests using contrasting treatments from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research (UK). This was done without confounding effects of soil type, soil texture, and climate. The results showed that the relationships between SOM components and SSS followed a broken-stick regression with a change point coinciding with a change from the tilled treatments to the grass treatment. The effect of increasing SOM components on SSS was greater below the change point, i.e. at low contents. The grass treatment had a very stable structure indicating that changing management from arable rotation to permanent grass is an effective tool for improving SSS.

If you have any further questions regarding our manuscript submission, please do not hesitate to contact me.

Sincerely, Johannes Lund Jensen

## Highlights

- Soil structural stability increased with an increase in SOM components
- The effect of increasing SOM components was greater at low contents
- The change point for soil structural stability was at a clay/SOC ratio close to 10
- Grassland soil has a very stable structure compared to tilled soil

Relating soil C and organic matter fractions to structural stability 1 2 Johannes L. Jensen\*1, Per Schjønning1, Christopher W. Watts2, Bent T. Christensen1, Clément 3 Peltre<sup>3</sup>, Lars J. Munkholm<sup>1</sup> 4 5 <sup>1</sup> Department of Agroecology, Aarhus University, Blichers Allé 20, 8830 Tjele, Denmark 6 7 <sup>2</sup> Department of Sustainable Agriculture Sciences, Rothamsted Research, Harpenden, Hertfordshire 8 AL5 2JQ, United Kingdom <sup>3</sup> Department of Plant and Environmental Sciences, University of Copenhagen, Thorvaldsensvej 40, 9 10 1821 Frederiksberg, Denmark \* Corresponding author 11 *E-mail address*: jlj@agro.au.dk (J. L. Jensen). 12 13 Type of Paper: Original research paper 14 Number of figures: 6 15 Number of tables: 3 16 Submitted to Geoderma the 18th of January 2018

#### **ABSTRACT**

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Soil organic matter (SOM) is important for maintaining soil structural stability (SSS). The influence of soil organic carbon (SOC) and different organic matter components on various SSS measures were quantified. We used a silt loam soil with a wide range of SOC (0.0080-0.0427 kg kg<sup>-1</sup> <sup>1</sup> minerals) sampled in spring 2015 from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research. Four treatments were sampled: Bare fallow, continuous arable rotation, levarable rotation, and grass. Soils were tested for clay dispersibility (DispClay), clay-SOM disintegration (DI, the ratio between clay content without and with SOM removal) and dispersion of particles <20 µm. The SSS tests were related to SOC, permanganate oxidizable carbon (POXC), hot water-extractable carbon (HWC), mid-infrared photoacoustic spectroscopy (FTIR-PAS) and mineral fines/SOC ratio. SSS increased with increasing content of SOM components. The relationships between SOM components and SSS followed a broken-stick regression with a change point at ~0.0230 kg SOC kg<sup>-1</sup> minerals (clay/SOC~10) coinciding with a change from the tilled treatments to the grass treatment. We found a greater influence of SOC, POXC and HWC on SSS at contents below the change point than above. A stronger linear relation between POXC and DispClay compared to SOC and HWC suggests that POXC was a better predictor of the variation in DispClay. POXC and HWC were less related to DI than SOC. The grass treatment had a very stable structure, shown in all SSS tests, probably due to the absence of tillage and large annual inputs of stabilizing agents. This suggests that a change in management from arable rotation to permanent grass is an effective tool for improving SSS.

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**Keywords**: soil structural stability; soil organic carbon; permanganate oxidizable carbon; hot waterextractable carbon; soil management

- 41 **Abbreviation**: A, Continuous arable rotation; BF, Bare fallow; CEC, Cation exchange capacity; DI,
- 42 Clay-SOM disintegration; DispClay, Clay dispersibility; DispFines20, Dispersion of particles <20
- 43 μm; Fines20, Mineral particles <20 μm; FTIR-PAS, Mid-infrared photoacoustic spectroscopy; G,
- 44 Grass; HWC, Hot water-extractable carbon; LA, Ley-arable rotation; LF-free-SOC, Light fraction-
- 45 free-SOC; LFSOC, Light fraction organic carbon; NTU, Nephelometric turbidity unit; PCA,
- 46 Principal component analysis; POXC, Permanganate oxidizable carbon; SSA, Specific surface area;
- 47 SSS, Soil structural stability.

#### 1. Introduction

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The importance of soil organic matter (SOM) on key soil properties and functions is wellknown (e.g., Johnston et al., 2009), and as a consequence loss of SOM is considered as a major threat to sustained soil functions (Amundson et al., 2015). One soil property affecting key soil functions is soil structure. Soil structure is the relative arrangement of particles and pores (Dexter, 1988), and the ability of soil structure to resist external stresses both mechanical and or from water is soil structural stability (SSS). Greater SSS is essential for minimizing the risk of downward transport of fine particles carrying pollutants to the water environment (de Jonge et al., 2004), soil erosion (Le Bissonnais, 1996), soil cementing and seedbeds with hard and non-friable aggregates (Kay and Munkholm, 2004). SOM content is an important factor affecting SSS (Bronick and Lal, 2005), and a range of studies have shown that an increase in SOM content increases SSS (e.g., Jensen et al., 2017a; Watts and Dexter, 1997). Soil organic carbon (SOC) is the main constituent of SOM and since an increase in SOC content generally increases SSS it may serve as a proxy for SSS. Labile organic compounds are considered readily decomposable, and are potentially better indicators for soil functions (Haynes, 2005). For example, permanganate oxidizable carbon (POXC) is considered a processed labile component of SOM and has been found to be more sensitive to differences in management than total SOC (Culman et al., 2012). POXC is easy and cheap to measure, and has been suggested as the best single predictor of soil health (Fine et al., 2017) and as a better predictor of crop productivity than total SOC (Hurisso et al., 2016). Similarly, hot water-extractable carbon (HWC) has been highlighted as a soil quality indicator more sensitive to management changes than total SOC (Ghani et al., 2003). HWC is considered a labile component of SOM consisting of microbial and plant derived material (Hirsch et al., 2017; Villada et al., 2016). Another measure, which potentially could better explain changes in SSS than total SOC could be to subtract light fraction organic

carbon (LFSOC) from total SOC since LFSOC is a fraction not closely associated to mineral particles (Gregorich et al., 2006). Mid-infrared photoacoustic spectroscopy (FTIR-PAS) can be used to assess differences in SOM quality (Peltre et al., 2014; Peltre et al., 2017), which potentially could improve the explanatory power in predicting SSS.

Increasing evidence suggest that soils exhibit a capacity factor for carbon sequestration also known as saturation state (Hassink, 1997; McNally et al., 2017). The saturation state of the soil has been found to influence the SSS measure, clay dispersibility, rather than SOC *per se* (Dexter et al., 2008). The saturation state has been assessed through the clay/SOC ratio, and a critical value close to 10 corresponding to a soil where the mineral particles are saturated with SOC has been found in several studies (Dexter et al., 2008; Getahun et al., 2016; Jensen et al., 2017a; Schjønning et al., 2012). Soils with clay/SOC>10 may thus be considered SOC-unsaturated, and for such soils SSS may be reduced. A similar threshold has been found for mineral particles <20 µm (Fines20) where the ratio of Fines20/SOC>20 indicates less SSS. Consequently, these mineral fines to SOC ratios may serve as soil type independent threshold values for SSS.

Previous studies often rely on samples retrieved from contrasting sites with different soil types and textures making quantification of the effect of SOM components on SSS dubious. This because the effects on SSS can be affected by other aggregate forming factors. More knowledge on the quantitative importance of SOM components on SSS using different pretreatments and energy input in the tests are needed.

The objective of this study was to quantify the influence of SOC on soil structural stability parameters determined in different ways and to test if knowledge about SOM characteristics could improve the predictive ability. A wide range of measures for the determination of SSS exists ranging in sample preparation, pretreatment, degree of disturbance and quantification (Le Bissonnais, 1996; Pojasok and Kay, 1990; Pulido Moncada et al., 2015). In this study, we applied

stability tests varying in pretreatment and ranging from low to very high degree of disturbance for a comprehensive evaluation of SOC effects on SSS. Soil was retrieved from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research (Highfield-LTE), a silt loam with a relatively homogeneous topsoil texture and a large gradient in SOC that has developed during at least 56 years of contrasting management practices without the confounding effects of soil type, soil texture and climate. Treatments were selected to obtain the widest possible gradient in SOC content, and thus ensuring major differences in SOM components.

#### 2. Materials and methods

#### 2.1 The Highfield-LTE and treatments

The Highfield-LTE was established in 1949 on a silt loam soil (Table 1) at Rothamsted Research, Harpenden, UK (51°80'N, 00°36'W) in a field that had been under permanent grass for centuries. The soil belongs to the Batcombe series, and the parent material include a relatively silty (loess-containing) superficial deposit overlying and mixed with clay-with-flints (Avery and Catt, 1995). The soil is classified as an Aquic Paludalf (USDA Soil Taxonomy System) and Chromic Luvisol (WRB) (Watts and Dexter, 1997). Average annual temperature and precipitation are 10.2°C (mean of 1992-2014) and 718 mm (mean of 1981-2010), respectively (Scott et al., 2014).

We selected three treatments in the ley-arable experiment:

Continuous arable rotation (A), winter cereals (winter wheat, *Triticum aestivum* L. and winter oats, *Avena sativa* L.) fertilized with 220 kg N ha<sup>-1</sup> y<sup>-1</sup> and maintained under standard Rothamsted farm practice with straw removed. Ley-arable rotation (LA), three-year grass/clover ley (meadow fescue, *Festuca pratensis* L.; timothy-grass, *Phleum pratense* L.; white clover, *Trifolium repens* L.) followed by three years arable (managed as A). The grass/clover ley received no N and was cut and

removed in early summer. The small amount of regrowth was topped in early autumn and left on the plots. Two of the sampled plots were drilled with winter cereals following three years of grass/clover, whereas the other two were drilled with grass/clover following three years of winter cereals. Grass (G), ploughed and reseeded to grass (predominantly rye grass, *Lolium perenne* L.) when the experiment was established (1949). The grass was managed as the grass/clover ley in LA.

We also selected the bare fallow (BF) treatment, which is not part of the original ley-arable experiment and located adjacent to the ley-arable experiment (denoted Highfield bare fallow and Geescroft bare fallow). The BF treatment has been maintained free of plants by regular tillage (ploughed or rotavated two to four times a year) since 1959. The ploughing depth in BF, A and LA was 0.23 m. The A, LA and G plots were fertilized with 65 kg P ha<sup>-1</sup> and 250 kg K ha<sup>-1</sup> every three years.

The A, LA and G treatments were part of a randomized block design with four field replicates, whereas the four BF plots were located at one end of the experiment (Fig. 1). The dimensions of the LA plots were 50 m x 7 m, whereas it was 10 m x 6 m for the other plots. The A, LA and G plots were smaller since they were part of a reversion experiment initiated in 2008. For more details see Johnston (1972) and the electronic Rothamsted Archive (www.era.rothamsted.ac.uk).

#### 2.2 Soil sampling

Soil was sampled in March 2015 at field capacity water conditions (corresponding approximately to a soil water potential of -100 hPa). Soil blocks (2750 cm<sup>3</sup>) were sampled from the 6-15-cm soil layer by careful use of a spade. Three sampling sites in each experimental plot were randomly chosen and labeled subplot. One of these blocks was extracted from each subplot adding up to three blocks per plot. The soil was kept in rigid containers to prevent soil disturbance during transport and stored in a field-moist condition at 2°C until required. Soil from the blocks at subplot

level were spread out in steel trays at room temperature, carefully fragmented by hand in several sittings along natural planes of weakness and finally left to air-dry.

#### 2.3 Basic chemical and physical analysis

Soil texture of air-dried bulk soil (crushed and passed through a 2-mm sieve) was determined by the hydrometer method for clay (<2 μm) and silt (2-20 μm) content and the sieve method for mineral particles >63 μm (Gee and Or, 2002). The soil was tested for CaCO<sub>3</sub> by adding a few droplets of 10% HCl, but none was found. SOM was removed by H<sub>2</sub>O<sub>2</sub> before estimation of clay and silt as described in Jensen et al. (2017b). The SOC content was determined on ball-milled subsamples using dry combustion (Thermo Flash 2000 NC Soil Analyzer, Thermo Fisher Scientific, Waltham Massachusetts, USA). Specific surface area (SSA) was determined by the ethylene glycol monoethyl ether method (Petersen et al., 1996), and cation exchange capacity (CEC) was determined after Kalra and Maynard (1991). Soil pH was determined in 0.01 M calcium chloride (CaCl<sub>2</sub>) solution (1:2.5, w/w). Clay, silt and SOC content were determined at subplot level, whereas the other properties were determined at plot level.

#### 2.4 Soil organic matter characteristics

Permanganate oxidizable carbon (POXC) was determined at subplot level following Culman et al. (2012). Air-dry 2-mm sieved soil equivalent to 2.5 g oven-dry weight was weighed into a 50 ml falcon tube, and was shaken in 18.0 ml of distilled water and 2.0 ml 0.2 M potassium permanganate (KMnO<sub>4</sub>) with pH 7.2 at 33 rpm for 2 min. After shaking, the soil was allowed to settle for 10 min after which 0.5 ml of the supernatant was transferred to falcon tubes containing 49.5 ml of water. The absorbance of the diluted solution was measured at 550 nm using a spectrophotometer (Thermo Electron Spectronic Helios Alpha Beta UV-visible). Absorbance of

four standard stock KMnO<sub>4</sub> solutions were measured to create a standard curve, and the absorbance of the samples were converted to POXC using the equation of Weil et al. (2003).

Hot water-extractable carbon (HWC) was determined at subplot level following Ghani et al. (2003). Briefly, air-dry 2-mm sieved soil equivalent to 3 g oven-dry weight was weighed into a 50 ml falcon tube, and was shaken in 30 ml distilled water at 33 rpm for 30 min, at 20°C. After centrifugation (3500 rpm, 20 min) the supernatant was decanted, soil resuspended and shaken for 16 h at 200 rpm, at 80°C. After centrifugation, the supernatant was transferred to 50 ml maxi-spin filter tubes equipped with a cellulose acetate membrane filter (0.45 μm pore size), filtered by centrifugation for 10 min at 3000 rpm and carbon determined by wet oxidation using a Shimadzu TOC-V analyzer.

Fractionation based on density was determined at subplot level using a modification of the method described by Sohi et al. (2001). Briefly, 10 g of air-dried 2-mm sieved soil was weighed into a 50 ml falcon tube, 35 mL of Nal solution with a density of 1.8 g cm<sup>-3</sup> was added, and the solution was shaken at 33 rpm for 2 h. The suspension was centrifuged for 30 min after which floating particles was transferred to a glass fiber filter (type GF/A, 110 mm diam., 1.6 µm retention, Whatman International, Kent, UK), and filtered under suction in a vacuum filtration unit (Büchner funnel). The light fraction organic matter (LFOM) retained on the filter was washed carefully and transferred to a crucible. To ensure a quantitative removal of LFOM the procedure was repeated. The remaining heavy fraction (HF) was washed three times and transferred to a large crucible. The oven-dry weight of the LF and HF were estimated by drying (105 °C for 24 h). The amount of OM recovered was estimated by loss-on-ignition (LOI; 500 °C for 4 h) both for the LFOM and HF. A 5 g air-dry bulk soil sample was dried to allow expressing results on an oven-dry basis after which LOI was determined. The LOI of bulk soil was used to make a model to predict the SOC of the HF

based on a multiple regression of SOC against LOI and clay (Model H2.1, Table 2 in Jensen et al.,
 2018):

SOC = 0.515 LOI (
$$P$$
<0.001) - 0.043 Clay ( $P$ <0.001), ( $n$ =48,  $R$ <sup>2</sup>= 0.990) (1)

Ten tests without soil (blind tests) were performed. The blind test estimate was subtracted from the LFOM estimate. The LFOM was converted to LFSOC by multiplying with 0.515 (Eq. 1), expressed as percentage of the sum of LFSOC and HFSOC, and normalized to the measured SOC content. Light fraction-free-SOC (LF-free-SOC) was calculated by subtracting LFSOC from SOC.

Fourier transform mid-infrared photoacoustic spectroscopy (FTIR-PAS) was determined at plot level following Peltre et al. (2014). Air-dry 2-mm sieved soil samples were ball-milled and packed in 10-mm diameter cups and functional groups of soil components were investigated using a Nicolet 6700 FTIR spectrometer (Thermo Scientific) equipped with a PA301 photoacoustic detector (Gasera Ltd. Turku, Finland). Spectra were recorded with an average of 32 scans within the range 4000-600 cm<sup>-1</sup> and with 2 cm<sup>-1</sup> intervals. A flow of helium was used as purge gas to remove noise produced by ambient moisture and CO<sub>2</sub> as well as moisture from the sample after insertion of the cup in the photoacoustic detector chamber. We focused on the 1700-1300 cm<sup>-1</sup> region to reduce overlapping from bands arising from soil minerals. The spectral peak area between 3000 and 2800 cm<sup>-1</sup> were integrated as described in Peltre et al. (2017), and reflects the amount of aliphatics in the soil (Leifeld, 2006).

#### 2.5 Soil structural stability

Clay dispersibility (DispClay) was determined at subplot level on 1-2 mm aggregates extracted from the air-dry 2-mm sieved soil. The aggregates were adjusted to a matric water potential of -100 hPa as described in Schjønning et al. (2012). In short, the aggregates were put on a

tension table at -100 hPa, gradually exposed to reduced suctions until -3 hPa, and finally equilibrated at -100 hPa by gradually increased suctions. The rewetting was done with great caution to avoid air explosion (slaking). Artificial rainwater was added to cylindrical plastic bottles containing 10 g of aggregates in order to obtain a soil:water ratio of 1:8 by weight. After end-overend rotation (33 rpm, 23-cm diameter rotation) for 2 min, the bottles were left to stand for 230 min, after which the upper 50 mm (60 ml) containing particles  $\leq$ 2  $\mu$ m was siphoned off. The weight of dispersed clay was determined after oven-drying (105 °C for 24 h) and corrected for particles >250  $\mu$ m isolated by chemical dispersion.

Dispersion of particles <20  $\mu$ m (DispFines20) was measured at different time steps at plot level on field-moist soil. Soil was retrieved from the minimally-disturbed soil cubes using a small corer (22-mm diameter) and gently crumbled by hand to pass an 8-mm sieve. Artificial rainwater was added to a cylindrical bottle containing soil equivalent to 1 g oven-dry weight to obtain a soil:water ratio of 1:100 by weight. The bottle was end-over-end rotated (33 rpm, 23-cm diameter rotation) for 2, 4, 8, 16, 32, 64 and 128 min. At each time step the bottle was left to stand for 67 sec, after which the upper 30 ml containing particles <20  $\mu$ m was siphoned off and turbidity of the suspension was measured on a Hach 2100AN turbidimeter (Hach, Loveland, CO). After the turbidity measurements taken at time steps 2-64 min the soil suspension was transferred back to the bottle. Thus, the measurements at the different time steps was done on the same sample. After the final measurement, the 30 ml was transferred to a beaker and bulked at treatment level. For each treatment, correlations between nephelometric turbidity unit (NTU) and particle concentration were made by doing dilution series. The calibration curves can be seen in Fig. S1 in Supplementary material. The results were corrected for particles > 250  $\mu$ m isolated by chemical dispersion.

Soil samples at subplot level were analyzed without H<sub>2</sub>O<sub>2</sub>-removal of SOM before estimation of clay as described in Jensen et al. (2017b), and clay-SOM disintegration (DI) was calculated as the ratio between clay content estimated without SOM removal and with removal. Soil with DI values <1 kg kg<sup>-1</sup> can be interpreted as extremely stable since they have resisted disintegration after end-over-end rotation for 18 h in sodium pyrophosphate.

#### 2.6 Calculations and statistics

The soil components measured in the paper are given as fractions of oven-dry weight (105°C for 24 h) of the SOM-free mineral fraction. This includes mineral particle size fractions, SOC, POXC, HWC, LF-free-SOC, SSA, CEC and DispClay. DispFines20 is given as a fraction of SOM-free mineral fraction <20 µm.

The statistical analysis and processing of spectral data applied the R-project software package Version 3.4.0 (R Foundation for Statistical Computing). Treatment effects for the comparison of A, LA and G were analyzed with a linear mixed model including block as a random effect. The criterion used for statistical significance of treatment effects was P < 0.05. When the treatment effect was significant, further analyses were made to isolate differences between treatments (pairwise comparisons) using the general linear hypotheses (glht) function implemented in the R multcomp package and the Kenward-Roger method to calculate degrees of freedom (Kenward and Roger, 2009). Treatment differences for the comparison of BF and the other treatments were calculated based on a pairwise t-test, acknowledging that this is a less robust test, and that the treatment differences could be due to soil variation since the BF treatment is not a part of the ley-arable experiment. Inverse transformation was performed on DispFines20 to stabilize the variance. The broken-stick model was fitted using the segmented package in R. A piece-wise linear model was used:

 $y = \beta_0 + \beta_1(x) + \beta_2(x-c)^+ + e$  (2)

where y is the dependent variable, x is the independent variable, c is the change point and e is the residual standard error (Toms and Lesperance, 2003). The  $^+$  sign indicates that the last term only is valid when x > c.

Spectral data processing included baseline correction, smoothing using a Savitzky-Golay filter calculated on three data points on each side with a zero-order polynomial, and normalization by the average absorbance on the whole spectra. Principal component analysis (PCA) on the FTIR-PAS spectra was performed using the *ade4* package in R.

#### 3. Results

#### 3.1 Basic soil characteristics

Differences in the contents of clay, silt and sand were in general not significant between treatments (Table 1) and the effect of the contrasting management practices could be investigated without confounding effects related to soil texture. SSA differed significantly following the same pattern as SOC. CEC was significantly higher for G compared to the BF treatment, and the amount of exchangeable Ca<sup>2+</sup> was significantly higher for G compared to the other treatments. Soil pH was not affected by the contrasting management practices.

#### 3.2 Soil organic matter characteristics

Concentration of SOC differed significantly in the order G>LA=A>BF (Table 2). POXC, HWC, LFSOC, and the aliphatic C-H peak area (3000-2800 cm<sup>-1</sup>) followed the treatment differences in SOC. The full spectral range as well as the SOM fingerprint region (1700-1300 cm<sup>-1</sup>) at plot level can be seen in Supplementary material, Figs. S2 and S3, respectively.

The aliphatic peak area normalized by the SOC content was higher in the BF than the G treatment indicating that SOM in G soil was more depleted in aliphatics. The POXC contributed to 1.7, 2.6, 2.8 and 2.5 % of total SOC, respectively, and the HWC in BF, A, LA and G contributed to 4.6, 4.5, 5.0 and 4.9 % of total SOC, respectively. The increase in POXC with an increase in SOC was different for the G treatment compared to A and LA (Fig. 2a). The narrow range in SOC content for BF did not allow an evaluation of the POXC-SOC relation within the SOC-depleted BF soil, but the first slope of the broken-stick model was similar to a linear regression with only A and LA (35.4 compared to 33.9 g kg<sup>-1</sup> SOC). This indicates that the level of POXC for BF was in line with the trend of the other tilled treatments (A and LA). The x-intercept of the broken-stick model in Fig. 2a was 0.00458 kg SOC kg<sup>-1</sup> minerals, and suggests that no POXC was oxidized at and below this SOC content. For the wide range in SOC in this study, HWC correlated linearly to SOC with an intercept value close to zero (Fig. 2b). Our data thus point to a concentration of ~0.05 kg HWC per kg SOC (~5%) irrespective of SOC level.

The PCA analysis based on the 1700-1300 cm<sup>-1</sup> region clearly separated the treatments on the first principal component (PC1) explaining 84.6% of the spectral variance (Fig. 3a). Field plots from the G and BF treatment were located on the left and right side of the PCA plane, respectively. Field plots from the A and LA treatments were in the center of the PCA plane and did not differ much. Examination of the loading of PC1 indicated that BF soils were relatively enriched in organic compounds vibrating in the range between 1700 and 1580 cm<sup>-1</sup> with a peak at 1625 cm<sup>-1</sup> (Fig. 3b). Absorption in this region is attributed to vibration of aromatics and carboxylate at 1600-1570 cm<sup>-1</sup>, amine at 1610 cm<sup>-1</sup>, clay-bound water at 1640 cm<sup>-1</sup>, aromatics at 1660-1600 cm<sup>-1</sup> and amides at 1670-1640 cm<sup>-1</sup> (Table 1 in Peltre et al., 2017). In contrast, G soils were relatively enriched in organic compounds vibrating in the range between 1580-1495 cm<sup>-1</sup> and 1475-1325 cm<sup>-1</sup> with peaks at 1550, 1510 and 1385 cm<sup>-1</sup> (Fig. 3b). These regions are attributed to vibration of nitrate at 1380

cm<sup>-1</sup>, carboxylate at 1390 cm<sup>-1</sup>, amide III at 1420 cm<sup>-1</sup>, carbonates at 1430 cm<sup>-1</sup>, aliphatic methyls at 1445-1350 cm<sup>-1</sup>, lignin rings at 1505-1515 cm<sup>-1</sup> and amide II at 1570-1540 cm<sup>-1</sup> (Table 1 in Peltre et al., 2017).

#### 3.3 Soil structural stability

The amount of dispersible clay differed significantly in the order BF>A>LA>G, and the disintegration of soil without SOM removal was significantly lower for the G treatment compared to the other treatments (Table 2). Linear, semi-logarithmic and broken-stick models were employed to describe the correlations of SOC, POXC and HWC to DispClay and DI (Table 3). The coefficient of determination (R<sup>2</sup>) was highest when DispClay and DI were related to SOC with a broken-stick model (see relation on Fig. 4a). Similar relationships were found when relating POXC and HWC to DispClay and DI (Figs. 4b and 4c).

Relating changes in DispClay to LF-free-SOC did not improve R<sup>2</sup> compared to SOC (Fig. 5a), whereas LF-free-SOC increased the explained variation in DI by 1 %-unit (Fig. 5b).

DispFines20 was significantly lower for the G treatment compared to the other treatments at all time steps (Fig. 6a), and the release-curve had a contrasting shape compared to the other treatments. DispFines20 was significantly lower for the LA than the A treatment after both 64 and 128 min. The release rate was markedly higher in the beginning for BF, A and LA compared to G (Fig. 6b). From approx. 24 min onwards, G had a higher release rate compared to the other treatments. At all time steps, DispFines20 was virtually constant across the four G treatment plots despite a range in SOC, while considerable variation was observed for the narrower SOC ranges of the other three treatments (Fig. S4 in Supplementary material).

#### 4. Discussion

4.1 Linking soil organic matter components to soil structural stability

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POXC has been promoted as a relatively processed or stabilized pool of active SOC (Culman et al., 2012), and as organic material to support biological functions (Idowu et al., 2008), while HWC has been promoted as an indicator of soil biochemical quality (Ghani et al., 2003). Both SOM fractions are considered labile and sensitive indicators for assessing management-induced changes (Culman et al., 2012; Ghani et al., 2003). Labile organic compounds are known to bond mineral particles together and thus stabilize them against mechanical damage (Degens, 1997). However, this mechanism would not be expected to play any role for the clay-SOM disintegration (DI) test that involves a rather extreme disruptive energy to soil structural units (end-over-end shaking for 18 h in sodium pyrophosphate solution). In accordance with this, we note a higher coefficient of determination in the broken-stick regression relating DI to SOC (R<sup>2</sup>=0.88) than for POXC and HWC ( $R^2=0.82$  and  $R^2=0.79$ , respectively). One may speculate that stable organo-mineral associations (i.e. at submicro-aggregate and primary particle scale) are causing the extreme stability at high SOC contents. The similar pattern observed for POXC and HWC (broken-stick) then relates to the near linear relations observed between total SOC and these two fractions (Fig. 2). For the DispClay SSS measure, we observe nearly identical coefficients of determination in the broken-stick models describing the data: R<sup>2</sup> equals 0.94, 0.93 and 0.91 with SOC, POXC and HWC as predictor (Table 3). We further note that the broken-stick is "less broken" especially when using POXC as predictor of trends in data (slope ratios, Table 3). This observation is supported by a higher ability of POXC to describe data in a linear model (R<sup>2</sup>=0.91) compared to HWC and SOC

Our study does not allow a definite clarification regarding which mechanisms are in play in SSS. The indication that POXC is superior in describing the variation in DispClay may be related to

(R<sup>2</sup>=0.82 and R<sup>2</sup>=0.84, respectively). Overall, this may indicate that POXC is superior to SOC and

HWC in describing the variation in DispClay.

a link to bonding agents such as polysaccharides, which are assumed predominantly active at microaggregate scale (Tisdall and Oades, 1982). However, the composition of POXC is unknown, which is related to the destruction of the fraction by oxidization. The lower predictive ability of HWC to explain SSS data and its close correlation to SOC may indicate that it is a too simplistic quality characteristic of SOC. Other studies have emphasized the need to focus on the carbohydrate-C contents in the hot water-extracts (e.g., Haynes, 2005), and studies have shown that hot water-extractable carbohydrate-C was a better predictor of SSS than SOC (Haynes and Swift, 1990).

Changes in DispClay and DI may be better explained by LF-free-SOC than total SOC. However, the difference in using LF-free-SOC compared to SOC was marginal (Fig. 5). The decrease in DispClay and DI when going from BF to G could be related to a general enrichment in aliphatics and lignin, and a decrease in carboxylic groups and amides (Table 2, Fig. 3). The higher amount of carboxyl-rich and amide-rich SOM suggest that the SOM in the BF soils were more oxidized being in agreement with the findings of Barré et al. (2016). Such compounds have been related to microbial processed and stable SOM in organo-mineral associations (Kleber et al., 2015). A higher proportion of aliphatics in SOM from the BF soils also support the presence of a more decomposed SOM. The results indicate that plant residues were decomposed rapidly in the BF soils leaving behind SOM enriched in microbial processed OM. In contrast, less oxidized and continually renewed compounds accumulated in the G soils.

#### 4.2 Management system effects on soil structural stability

The four treatments can be seen as three management systems comprising the BF treatment with intensive tillage, no plants or carbon input, the A and LA treatments with plants and tillage, and the G treatment with plants and absence of tillage. The G treatment differed from the other treatments by having a very stable structure and a better ability to resist increasing degrees of

disturbance. Consequently, the potential maximum SSS was only fully achieved for the long-term G treatment. This was reflected in the DispClay and DI tests where the change point of the brokenstick model for SOC content was confounded with treatment, and represented a change from the LA to G treatment (Fig. 4a). Also the results on DispFines20 support a change in stability between the G treatment and the other treatments illustrated by the contrasting curve, release rate and higher stability at all time steps (Fig. 6). Permanent grass stands out from the other treatments by having a greater input of above- and belowground plant residues. Hirsch et al. (2009, 2017) found a markedly greater number of roots and mesofauna in G compared to BF and A, and a larger abundance of fungi. Roots and fungal hyphae can act as binding agents, enmesh aggregates (Elmholt et al., 2008; Tisdall and Oades, 1982), and potentially increase SSS, and mesofauna can contribute to stability via stabilizing decomposition products (Oades, 1993). In addition, the effect of these stabilizing agents are persistent since they are continuously replaced, and remain undisturbed due to the absence of tillage. The limited effect of SOC on DispFines20 within the G treatment at all time steps (Fig. S4 in Supplementary material) may be related to the larger scale applied in the test, i.e. whole-soil samples not broken down to more than 8 mm. At a larger scale, management system drivers such as macro-aggregate stabilizing agents seemed to be more distinct, while SOC played a minor role. The greater stability of the G treatment may also be related to the absence of tillage shown to be detrimental to the preservation of stabilizing agents.

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The higher amount of soluble Ca<sup>2+</sup> ions in the G treatment increases the tendency of clay particles to flocculate (Le Bissonnais, 1996), and may also contribute to the higher stability. However, the importance of cations for aggregate stability is considered less important in soil high in clay or SOC (Bronick and Lal, 2005). Matthews et al. (2008) found a decrease in wettability for

G, whereas wettability was similar for BF, A and LA treatments. Thus, the very stable structure of the G soil may also be partially related to decreased wettability.

#### 4.3 Critical carbon levels

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Our SSS measures DI and DispClay showed a change in the relation to SOC at around 0.0230 kg kg<sup>-1</sup> minerals for this soil (broken-stick change point; Fig. 4a). The carbon saturation concept (Six et al., 2002; Stewart et al., 2007) implies the existence of a SOC concentration that for a given soil provides a full "coverage" of the surface of soil minerals with SOC. This potential carbon storage capacity (Ingram and Fernandes, 2001) was verified for a range of grassland soils assumed saturated with organic carbon (Hassink, 1997). A SOC concentration of ~0.0230 kg kg<sup>-1</sup> minerals found in this study may thus be hypothesized to reflect the potential storage capacity for this soil. The broken-stick pattern for DispClay indicates that SOC influences SSS more for soils with SOC below the change point than above (Fig. 4a). DispClay increases more with reduction in SOC when the soil is unsaturated with carbon (below the change point) than when it is saturated. This is in line with Jensen et al. (2017a), who found SOC to be important for SSS for SOM-depleted soil. Interestingly, the DI test with extreme energy input showed that all unsaturated soil samples behaved similarly and fully disintegrate (slope not significantly different from zero; Fig. 4a). The SOC threshold for a change in SOC effects on SSS differ from soil to soil, e.g. around 0.0110 kg SOC kg<sup>-1</sup> minerals for a sandy loam in Denmark (Jensen et al., 2017a). Thus, SOC critical to SSS seems soil type dependent. Dexter et al. (2008) and Jensen et al. (2017a) found an increasingly compromised SSS when the clay/SOC ratio was above 10. Schjønning et al. (2012) and Jensen et al. (2017a) found that a Fines20/SOC ratio of 20 serve as a similar critical threshold value. The clay/SOC and Fines20/SOC ratios for a change in DispClay and DI were calculated by dividing the average clay or Fines 20 content with the change point giving values of 11 and 23,

respectively. Thus, our results support the soil clay/SOC~10 or Fines20/SOC~20 as defining factors for SSS.

In this study the thresholds for changes in SOC (as well as POXC and HWC) effects on SSS is confounded with management (Fig. 4). Thus the calculated soil mineral fines/SOC thresholds may relate to a quantity of SOC as well as management system (as discussed in section 4.2). This was unavoidable since systems with a wide range in SOC often will require contrasting management. However, confounding effects derived from differences in soil type, soil texture and climate were eliminated.

#### 5. Conclusions

We exploited the unique range in SOM within Highfield, which has developed due to contrasting long-term management practices. Soil structural stability (SSS) increased with an increase in SOM components. However, the relationships followed a broken-stick regression with the greater effect occurring when SOM components were less. The SOM fractions permanganate oxidizable carbon (POXC) and hot water-extractable carbon (HWC) were less related to clay-SOM disintegration than SOC. However, POXC seemed superior in describing the variation in clay dispersibility compared to SOC and HWC. The permanent grass had a very stable structure - even when exposed to high degree of disturbance. This may be ascribed to the management system, which result in higher amount of stabilizing agents due to greater and annually renewed inputs of above- and belowground plant residues as well as absence of tillage. Both aspects promote a high abundance of soil microbiota and mesofauna. For this soil, management changes promoting SOM content increased SSS up to a specific threshold coinciding with a change to permanent grass.

rotation to permanent grass seem promising tools for improving SSS. Further, this study supports the existence of critical soil mineral fines/SOC ratios for SSS with change points at clay/SOC~10 and Fines20/SOC~20.

#### Acknowledgements

We gratefully acknowledge the technical assistance of Stig T. Rasmussen, Dept. Agroecology (Aarhus University), and the technical staff at Rothamsted Research. We thank Bodil B. Christensen, Palle Jørgensen, Karin Dyrberg, Kim M. Johansen, and Morgane Coulumbel for technical assistance. We thank Kristian Kristensen for statistical advice. The study was supported by the Green Development and Demonstration Programme (GUDP) of the Ministry of Environment and Food of Denmark through the "Cover crops for optimization of cereal based cropping systems" (Grant No. 3405-11-0225) and "Optimized soil tillage in cereal based cropping systems" (Grant No. 34009-12-0502) projects, and by the EU 7th Research Framework Programme, Distributed Infrastructure for Experimentation in Ecosystem Research (ExpeER) through the project "Identification of soil organic carbon thresholds for sustained soil functions in agroecosystems" (Grant No. 262060). The Rothamsted Long-term Experiments National Capability is supported by the UK Biotechnology and Biological Sciences Research Council (BBSRC) and the Lawes Agricultural Trust.

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## Figure captions

- Fig. 1. Distribution of plots in Highfield showing the arable (A), ley-arable (LA) and grass (G)
- treatments in blocks 1-4 of the ley-arable experiment, and the bare fallow (BF) treatment in blocks
- 592 1-3 of the bare fallow experiments.
- Fig. 2. (a) Permanganate oxidizable carbon (POXC) as a function of SOC and (b) hot water-
- extractable carbon (HWC) as a function of SOC for the four treatments at subplot level. The
- broken-stick and linear regression models are indicated.
- Fig. 3. Principal component analysis (PCA) based on FTIR-PAS spectra for the different
- treatments. The dots indicate the four plots of each treatment. For treatment abbreviations, see Fig.
- 1. (a) Scores plot in the plane defined by principal component 1 (PC1, explaining 84.6% of the
- variance) and principal component 2 (PC2, explaining 7.5% of the variance) of the PCA. (b)
- 600 Loadings of the PCA for PC1 and PC2.
- Fig. 4. Clay dispersibility of 1-2 mm aggregates rewetted to -100 hPa (solid lines) and
- disintegration (the ratio between clay content estimated without SOM removal and with removal)
- (dashed lines) as a function of (a) soil organic carbon (SOC), (b) permanganate oxidizable carbon
- 604 (POXC), and (c) hot water-extractable carbon (HWC) for the four treatments at subplot level. The
- broken-stick models (Table 3) are indicated. See Table 3 for equations and R<sup>2</sup>-values.
- Fig. 5. (a) Clay dispersibility and (b) Clay-SOM disintegration as a function of soil organic carbon
- 607 (SOC; black symbols) and LF-free-SOC (Light fraction-free-SOC; white symbols). The broken-
- stick models and R<sup>2</sup>-values are indicated.
- Fig. 6. (a) The ratio between dispersed particles <20 μm and the total content of particles <20 μm as
- a function of Log10(min) at treatment level. The standard error of the mean is indicated (n=4). The

polynomials are fitted to the four replicates time's seven data points per treatment. Letters denote statistical significance at P<0.05 for the comparison of A, LA and G. An asterisk (\*) indicates if BF is significantly different from A, LA and G based on a pairwise t-test. (b) Release rate (kg kg $^{-1}$  minerals dispersed min $^{-1}$ ) as a function of Log10(min) at treatment level. A stepwise, simple calculation of slope from time step to time step was employed to calculate the release rate, and a smoothed spline curve was added.

**Table 1** Soil characteristics. Within rows, letters denote statistical significance at P<0.05 for the comparison of A, LA and G. An asterisk (\*) indicates if BF is significantly different from A, LA and G based on a pairwise t-test. For treatment abbreviations, see Fig. 1.

	BF	A	LA	G
Texture <sup>a</sup>				
Clay <2 μm	0.270	0.264	0.255	0.261
Silt 2-20 μm	0.249	0.263	0.261	$0.272^{*}$
Silt 20-63 μm	0.335	0.318	0.324	0.319
Sand 63-2000 μm	0.146	0.155	0.160	0.148
Specific surface area (m <sup>2</sup> g <sup>-1</sup> minerals) <sup>b</sup>	56.7	67.9a*	$68.4^{a*}$	78.4 <sup>b*</sup>
Exchangeable cations and CEC				
Na <sup>+</sup> (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	0.4	$0.5^{a}$	$0.4^{a}$	$0.7^{b*}$
K <sup>+</sup> (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	3.3	6.3	17.7	5.8
Ca <sup>2+</sup> (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	93.7	113.3a	128.7a	155.6 <sup>b*</sup>
Mg <sup>2+</sup> (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	5.4	4.0	4.1	4.6
Sum of bases (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	102.9	107.1a	120.7ab	142.0 <sup>b*</sup>
CEC (mmol <sub>c</sub> kg <sup>-1</sup> minerals)	145.8	173.8	171.3	$209.9^{*}$
Base saturation (%)	72.8	65.5	74.8	74.4
pH (CaCl <sub>2</sub> )	5.7	5.1	5.1	5.4

akg kg<sup>-1</sup> of mineral fraction and based on oven-dry weight.

bClay is included as a co-variable since it is significant in itself and makes the treatment effect significant.

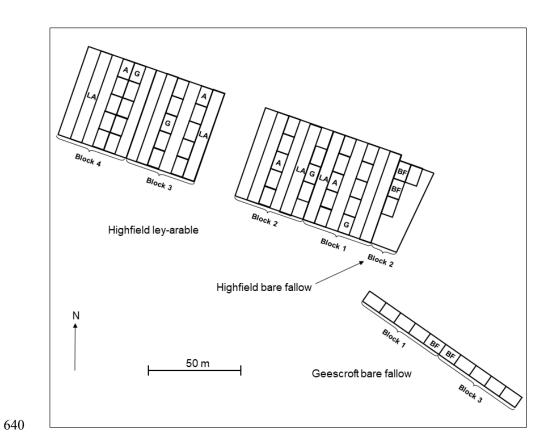
**Table 2** Soil organic matter characteristics, clay dispersibility of 1-2 mm aggregates rewetted to -100 hPa and clay-SOM disintegration (the ratio between clay content estimated without SOM removal and with removal). Within rows, letters denote statistical significance at P<0.05 for the comparison of A, LA and G. An asterisk (\*) indicates if BF is significantly different from A, LA and G based on a pairwise t-test. For treatment abbreviations, see Fig. 1.

	BF	A	LA	G		
Soil organic matter characteristics	Soil organic matter characteristics					
Soil organic carbon (SOC, kg kg <sup>-1</sup> minerals)	0.0090	0.0173a*	$0.0216^{a*}$	$0.0329^{b*}$		
Permanganate oxidizable carbon (POXC, g kg <sup>-1</sup> minerals)	0.161	$0.458^{a*}$	$0.600^{b*}$	0.818c*		
% of SOC	1.7	2.6ab*	2.8b*	$2.5a^*$		
Hot water-extractable carbon (HWC, g kg <sup>-1</sup> minerals)	0.437	$0.777^{a^*}$	1.082 <sup>b*</sup>	1.611 <sup>c*</sup>		
% of SOC	4.6	4.5a	5.0 <sup>b</sup>	4.9ab		
Light fraction carbon (LFSOC, g kg <sup>-1</sup> minerals)	0.167	1.285a*	1.732a*	2.579b*		
% of SOC	1.9	7.4*	$8.0^{*}$	$7.8^{*}$		
Aliphatic peak area	58	99a*	121a*	159 <sup>b*</sup>		
Aliphatic peak area/SOC	65	57	56	49*		
Soil structural stability						
Clay dispersibility (DispClay, kg kg <sup>-1</sup> minerals)	0.0115	$0.0074^{c*}$	$0.0051^{b*}$	$0.0034^{a*}$		
Clay-SOM disintegration (DI, kg kg <sup>-1</sup> minerals)	1.02	0.96 <sup>b</sup>	1.00 <sup>b</sup>	0.74 <sup>a*</sup>		

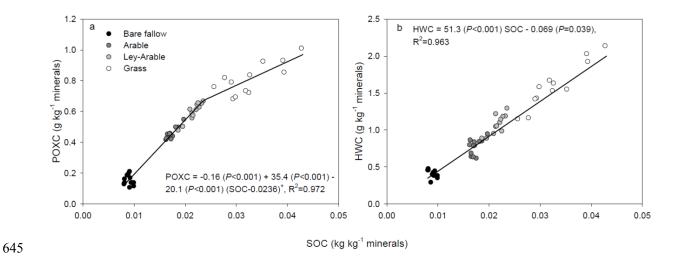
**Table 3** Parameters of the linear, semi-logarithmic and broken-stick models for clay dispersibility (DispClay; kg kg<sup>-1</sup> minerals) and clay-SOM disintegration (DI; kg kg<sup>-1</sup> minerals) as a function of soil organic carbon (SOC; kg kg<sup>-1</sup> minerals), permanganate oxidizable carbon (POXC; g kg<sup>-1</sup> minerals), and hot water-extractable carbon (HWC; g kg<sup>-1</sup> minerals). The change point of the broken-stick model and the corresponding 95% confidence interval is indicated. The relation between the first and second slope estimate of the broken-stick model (Slope1/Slope2) was calculated if both slopes were significant. The coefficient of determination (R<sup>2</sup>) is indicated.

Predictor	Model		Equation	Change point	Slope1/Slope2	R <sup>2</sup>
SOC	Linear	DispClay	0.0134***-0.32*** SOC			0.839
		DI	1.16***-11.6*** SOC			0.723
	Semi-log	DispClay	-0.0189*** -0.0148*** log(SOC)			0.930
		DI	0.16*** -0.442*** log(SOC)			0.555
	Broken-stick	DispClay	$0.0160^{***}$ - $0.49^{***}$ SOC + $0.39^{***}$ (SOC- $0.0235$ ) <sup>+</sup>	0.0235*** [0.0209:0.0260]	4.6	0.940
		DI	$1.03^{***}$ - $2.9^{NS}$ SOC $-18.0^{***}$ (SOC- $0.0225$ ) <sup>+</sup>	0.0225*** [0.0199:0.0251]		0.880
POXC	Linear	DispClay	0.0131***-0.0122*** POXC			0.907
		Di	1.11***-0.366*** POXC			0.550
	Semi-log	DispClay	0.0029*** -0.011*** log(POXC)			0.891
		DI	0.83*** -0.261*** log(POXC)			0.364
	Broken-stick	DispClay	0.0136***-0.0138*** POXC + 0.0084* (POXC-0.694)+	0.694* [0.564:0.824]	2.6	0.927
		DI	1.02***-0.087 <sup>NS</sup> POXC - 1.00*** (POXC-0.628)+	0.628*** [0.573:0.683]		0.819
HWC	Linear	DispClay	0.0128***-0.0061*** HWC			0.815
		DI	1.13***-0.21*** HWC			0.648
	Semi-log	DispClay	0.0059*** -0.0133*** log(HWC)			0.900
		DI	0.90***-0.378*** log(HWC)			0.490
	Broken-stick	DispClay	$0.0156^{***}$ - $0.0105^{***}$ HWC + $0.00760^{***}$ (HWC- $0.970$ ) <sup>+</sup>	0.970*** [0.833:1.107]	3.6	0.913
		DI	$1.02^{***}$ - $0.049^{NS}$ HWC $-0.341^{***}$ (HWC- $1.104$ ) <sup>+</sup>	1.104*** [0.921:1.288]		0.788

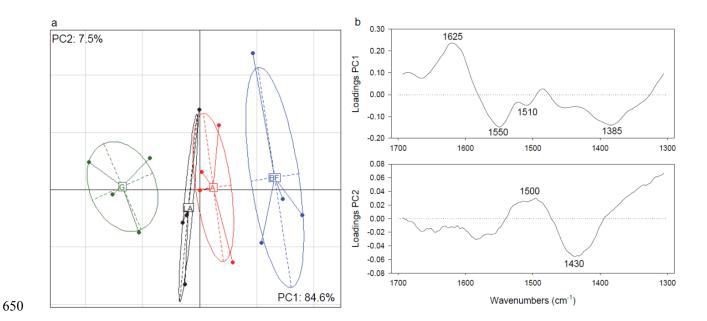
- \* and \*\*\* indicate significance level at P < 0.05 and P < 0.001, respectively.
- NS: Not significant.
- 639 +: Indicates that the last term is valid only when the content of SOC, POXC or HWC are larger than the change point.



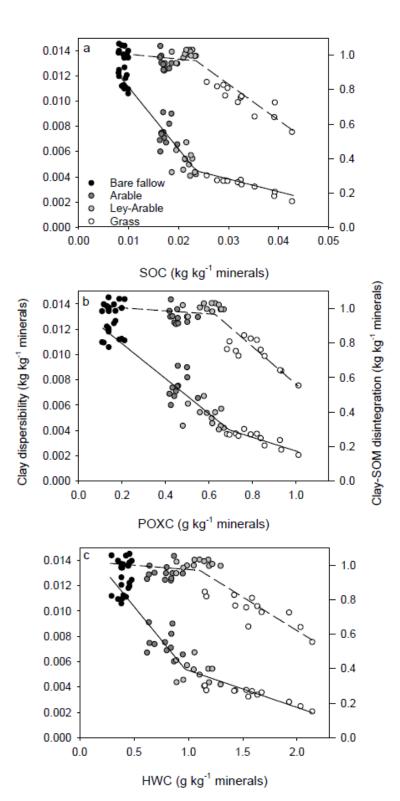
**Fig. 1.** Distribution of plots in Highfield showing the arable (A), ley-arable (LA) and grass (G) treatments in blocks 1-4 of the ley-arable experiment, and the bare fallow (BF) treatment in blocks 1-3 of the bare fallow experiments.



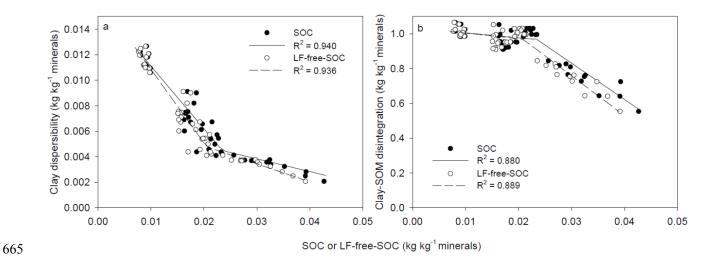
**Fig. 2.** (a) Permanganate oxidizable carbon (POXC) as a function of SOC and (b) hot water-extractable carbon (HWC) as a function of SOC for the four treatments at subplot level. The broken-stick and linear regression models are indicated.



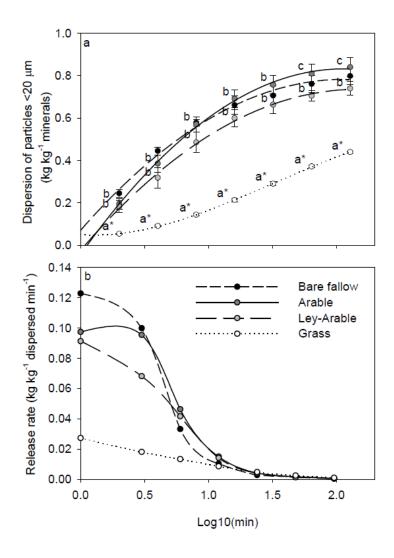
**Fig. 3.** Principal component analysis (PCA) based on FTIR-PAS spectra for the different treatments. The dots indicate the four plots of each treatment. For treatment abbreviations, see Fig. 1. (a) Scores plot in the plane defined by principal component 1 (PC1, explaining 84.6% of the variance) and principal component 2 (PC2, explaining 7.5% of the variance) of the PCA. (b) Loadings of the PCA for PC1 and PC2.



**Fig. 4.** Clay dispersibility of 1-2 mm aggregates rewetted to -100 hPa (solid lines) and clay-SOM disintegration (the ratio between clay content estimated without SOM removal and with removal) (dashed lines) as a function of (a) soil organic carbon (SOC), (b) permanganate oxidizable carbon (POXC), and (c) hot water-extractable carbon (HWC) for the four treatments at subplot level. The broken-stick models (Table 3) are indicated. See Table 3 for equations and R<sup>2</sup>-values.



**Fig. 5.** (a) Clay dispersibility of 1-2 mm aggregates rewetted to -100 hPa and (b) Clay-SOM disintegration (the ratio between clay content estimated without SOM removal and with removal) as a function of soil organic carbon (SOC; black symbols) and LF-free-SOC (Light fraction-free-SOC; white symbols). The broken-stick models and R<sup>2</sup>-values are indicated.



**Fig. 6.** (a) The ratio between dispersed particles <20 μm and the total content of particles <20 μm as a function of Log10(min) at treatment level. The standard error of the mean is indicated (n=4). The polynomials are fitted to the four replicates time's seven data points per treatment. Letters denote statistical significance at P<0.05 for the comparison of A, LA and G. An asterisk (\*) indicates if BF is significantly different from A, LA and G based on a pairwise t-test. (b) Release rate (kg kg<sup>-1</sup> minerals dispersed min<sup>-1</sup>) as a function of Log10(min) at treatment level. A stepwise, simple calculation of slope from time step to time step was employed to calculate the release rate, and a smoothed spline curve was added.

# Supplementary material for the article entitled: "Relating soil C and organic matter fractions to structural stability" by Jensen et al.

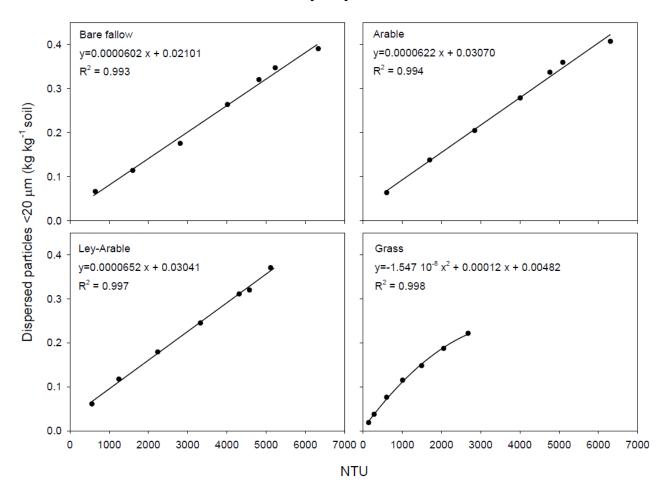
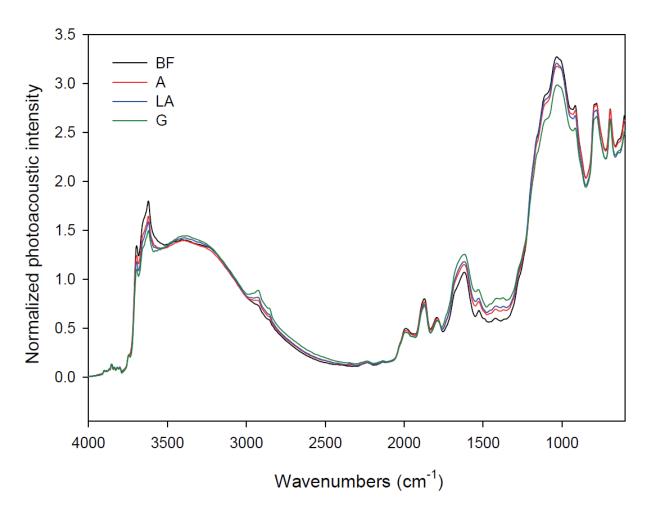
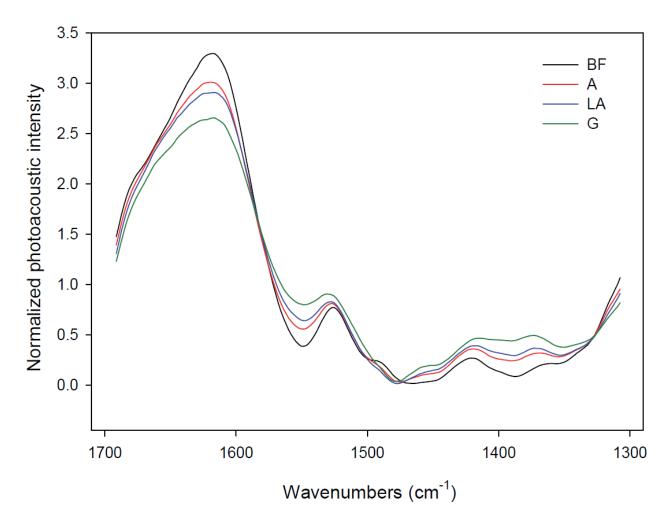


Fig. S1. The correlation between nephelometric turbidity (NTU) and dispersed particles  $<20~\mu m$  (kg kg<sup>-1</sup> soil) for the four different treatments.



**Fig. S2.** Spectra of the different treatments from Highfield over the selected FTIR region 4000-600 cm<sup>-1</sup>. The spectra are presented as the average of the spectra from the four field plots. BF, Bare fallow; A, Arable; LA, Ley-arable; G, Grass.



**Fig. S3.** Spectra of the different treatments from Highfield over the selected FTIR region 1700-1300 cm<sup>-1</sup>. The spectra are presented as the average of the spectra from the four field plots. BF, Bare fallow; A, Arable; LA, Ley-arable; G, Grass.

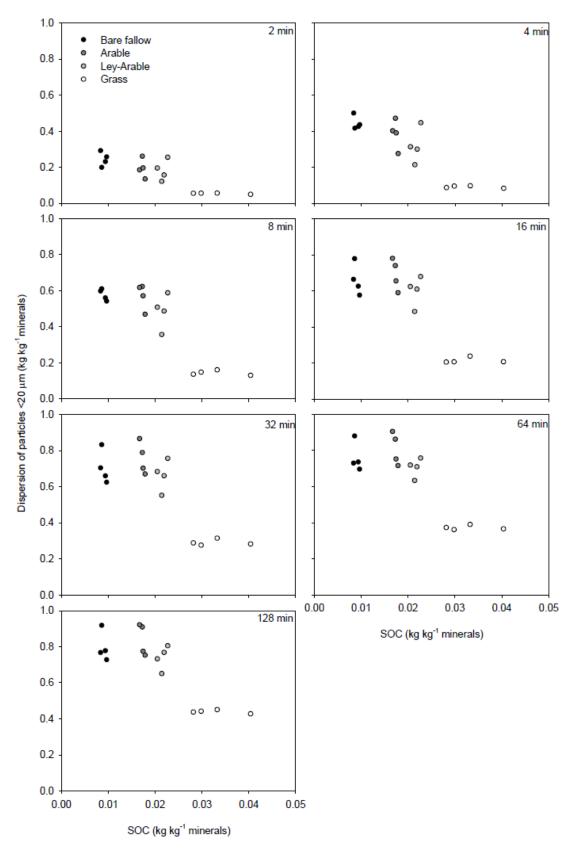


Fig. S4. The ratio between dispersed particles <20  $\mu$ m and the total content of particles <20  $\mu$ m as a function of soil organic carbon at plot level for the seven time steps.