Problems with determining the particle size distribution of chalk soil and some of their implications

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Abstract

Particle size distribution (psd) is one of the most important features of the soil because it affects many of its other properties, and it determines how soil should be managed. To understand the properties of chalk soil, psd analyses should be based on the original material (including carbonates), and not just the acid-resistant fraction. Laser-based methods rather than traditional sedimentation methods are being used increasingly to determine particle size to reduce the cost of analysis. We give an overview of both approaches and the problems associated with them for analyzing the psd of chalk soil. In particular, we show that it is not appropriate to use the widely adopted 8 µm boundary between the clay and silt size-fractions for samples determined by laser-based to estimate equivalent proportions of these size fractions to analyses based on sedimentation.

We present data from field and national-scale surveys of soil derived from chalk in England. Results from both types of survey showed that laser methods tend to over-estimate the clay-size fraction compared to sedimentation for the 8 µm clay/silt boundary, and we suggest reasons for this. For soil derived from chalk, either the sedimentation methods need to be modified or it would be more appropriate to use a 4 µm threshold as an interim solution for laser methods. Correlations between the proportions of sand- and clay-sized fractions, and other properties such as organic matter and volumetric water content, were the opposite of what one would expect for soil dominated by silicate minerals. For water content, this appeared to be due to the predominance of porous, chalk fragments in the sand-sized fraction rather than quartz grains, and the abundance of fine (<2 µm) calcite crystals rather than phyllosilicates in the clay-sized fraction. This was confirmed by scanning electron microscope (SEM) analyses.

"Of all the rocks with which I am acquainted, there is none whose formation seems to tax the ingenuity of theorists so severely, as the Chalk, in whatever respect we may think fit to consider it".

Thomas Allan, FRS Edinburgh 1823, Transactions of the Royal Society of Edinburgh.

Keywords: particle size distribution, chalk, sedimentation, laser methods

Introduction

The term soil texture relates specifically to the qualitative determination of the particle size fractions in the field by hand-texturing. When these fractions have been determined in the laboratory by sieving, sedimentation or laser methods, they are referred to as the particle size distribution (psd) of the soil. Published studies (Hunt *et al*., 2006; Lewis *et al*. 2003; http://www.cdms.net/ldat/ld6NS001.pdf) and DEFRA's RB209 'Fertiliser Recommendations for Agricultural and Horticultural Crops' (MAFF, 2000) indicate that psd should be taken into account in determining rates of fertilizer and pesticide application to the soil. Inaccurate determination of soil psd where soil has developed on chalk in England has clear implications for decisions in agricultural management, particularly in relation to the leaching of nitrate. In England chalk is the parent material for soil over 8825 km^2, about 7 % of the soilscape (Figure 1). Nitrate sensitive areas (NSAs) and nitrate vulnerable zones (NVZs) have been established in England to try to reduce the amount of nitrate that enters the surface and ground water supplies. The designation of these areas and zones has put pressure on farmers to use nitrate fertilizers more diligently (DEFRA, 2002).

Figure 1 near here

The sand-sized fraction of soil (63–2000 µm, Soil Survey of England Wales, Hodgson, 1974) is predominantly chemically inert quartz that has a low water-holding capacity. The clay-sized fraction (<2 µm) typically comprises mainly phyllosilicate clay minerals that readily absorb and adsorb water. Although it has been recommended that calcareous-rich soil should be decalcified before particle-size analysis (Francis and Aguilar, 1995), it is our contention that key soil properties, such as the water holding capacity and its implications for the loss of applied fertiliser, cannot be understood if the carbonate-fraction is removed.

The psd for precision farming is usually determined by laser diffraction methods from numerous soil samples taken within one field because sedimentation methods are more costly. Sampling and laboratory analysis account for the main cost of producing accurate soil maps. Identifying differences in soil textural class by hand texturing could reduce the cost of mapping soil texture, but this is not sufficiently refined to show how the sand silt and clay sized fractions vary across a field. The precision farming approach has been encouraged in NSAs and NVZs because it should lead to more efficient application of agro-chemicals and result in less pollution of ground and surface waters. Carbonate rich soil is particularly prone to the rapid transmission of agro-chemicals through the profile. Therefore, given the widespread use of laser-based methods to determine psd for precision farming, the reliability of the laser approach requires further investigation in the context of such soil.

Laser and sedimentation techniques define the size of a particle differently, and therefore measure different properties of the same material (Konert and Vandenberghe, 1997). To account for the predominance of non-spherical phyllosilicate clay minerals, laser methods generally require a different boundary between the clay and silt-sized fractions to be equivalent to sedimentation to deal with the predominance of non-spherical, phyllosilicate clay minerals. Konert and Vanenberghe (1997) suggested that the upper limit for the clay fraction determined by laser should be 8 µm for its percentage to be equivalent to that measured by sedimentation with a 2 µm limit for a range of psds. This paper has been cited extensively (136 times) since it was published and this ‘fudge factor’ has been widely used to express psd determined by laser. It is applied routinely by several agencies when they analyze soil psd commercially, and sometimes without informing the client. This is clearly as misleading for land management as assuming that the laser and sedimentation methods will give the same results. It is important that practitioners understand the different methods for psd and the assumptions upon which they are based.

We investigate whether the standard application of sedimentation and laser methods with this ‘fudge factor’ is appropriate to determine the soil’s psd when the parent material is calcite-rich Cretaceous Chalk. This rock is widespread in southern and eastern England, and northern France. Our aim here is to show that the discrepancies in psd measurements by sedimentation and laser diffraction methods are different for soil developed on chalk compared with that developed on other parent materials. Differences in the mineralogy of the size-fractions affects what we assume to be their typical behaviour in terms of water and nutrient retention, and this has important implications for fertilizer and pesticide recommendations which need to be understood better. The existing methods of sedimentation and laser diffraction might need to be amended for chalk soil to ensure that fertilizer and pesticide rates are appropriate in NSAs and NVZs.

Determination of particle size distribution (psd)

Sedimentation methods

The air-dry < 2mm soil fraction is usually pre-treated with hydrogen peroxide to destroy organic matter and reduce aggregation of the soil particles to determine the soil's psd. The soil is then dispersed using sodium hexametaphosphate, ultra sonic dispersal, or decalcification (NEN 5753, 1990). Differences in sample pre-treatment can affect the results of particle size analysis, particularly for chalk soil. For example, Hartwig and Loeppert (1991) showed that shaking and sonic dispersal before particle size analysis increased the proportion of clay-sized carbonates and decreased the proportions of sand- and silt- sized carbonates. They recommended an initial treatment with Na to disperse soil aggregates to avoid grinding carbonate particles when determining the psd for carbonate rich soil.

The pipette method (Avery and Bascomb, 1982) has become a standard method of determining psd. However, it is time-consuming as it is based on the settling velocities of different sized spherical particles derived from Stokes's Law for streamlined flow (Rowell, 1994):

$$v = 2gr^2(\rho_s - \rho_l) / 9\eta \; , \qquad (1)$$

where v is the sedimentation velocity (m s^{-1}), r the particle radius (m), g the gravitational force per unit mass (9.81 N kg^{-1}), ρ_s the density of the particle (2600 kg m^{-3} is the average density for soil particles), ρ_l is the density of the liquid (998 kg m^{-3} at 20˚C for water) and η the viscosity of the liquid (1.002 x 10^{-3} N s m^{-2} at 20˚C for water).

Sedimentation analysis relies on two assumptions (i) that particles are solid, smooth spheres, and (ii) their density is the same and equivalent to the average value of particle density (see above). Violation of the two assumptions associated with sedimentation methods (Konert and Vandenberghe, 1997) has important implications for the reliability and comparability of the results of sedimentation. Phyllosilicate clay minerals tend to have a platy shape and so settle slowly in liquid compared with other minerals with the same average diameter. Consequently, the clay-sized fraction can be over-estimated by sedimentation. The particle density also varies with mineralogy and this often varies with size fraction of the soil.

Laser Methods

Determination of psd by a laser granulometer cell depends on different degrees of diffraction of a laser beam by particles, which registers at photoreceptors for different sized particles. The intensity of light at each detector is measured and the psd is determined from a model, usually the Fraunhofer. Laser methods determine the size of the particles based on their diameter at the angle at which they intercept the laser beam. Laser granulometers have been shown to be consistent in results reported based on the analysis of standards materials (Konert and Vandenberghe, 1997). Nevertheless, there are three main assumptions that underpin laser methods for psd are: (1) transformation of the laser diffraction patterns into particle sizes assumes that the particles are spherical; (2) orientation of the particles is assumed to be random and (3) the theoretical basis for the chosen inversion procedure (Mie-theory or Fraunhofer model) is assumed to be correct and appropriate for the particles being investigated.

All of these assumptions might not be met for a given soil sample. The recorded diameter for non-spherical, platy, phyllosilicate clay minerals will vary markedly depending on the angle at which they intercept the laser beam. Also laser diffraction measurements often take place in continuous flow and so particles can become oriented according to shape, although this is not the case for the Coulter machine used in this study. The size classification of phyllosilicate particles is clearly problematic by whichever method is used to determine it. Finally, there are difficulties related to the models used to convert laser diffraction results into particle diameters.

The Fraunhofer model assumes that particles are opaque and non-porous, so the optical properties of the particles are not considered, only diffraction of the laser. Mie Theory considers diffusion and absorption of the laser by the particles in addition to diffraction, so knowledge of optical properties is required (http://www.cilasus.com/theory/index.html). Malvern Instruments (2000) note that the Fraunhofer became the default model for laser granulometers in the 1970s because of lack of computing power. They show that for particles smaller than 50 µm, the Mie theory gives more accurate estimates, but this was based on samples with a uni-modal distribution. Bayvel and Jones (1981) also illustrate that the Fraunhofer model is less appropriate than Mie Theory for particles smaller than 5 µm and Buurman et al. (2001) note that histograms of grain size distribution from the Fraunhofer model frequently show a dip in the grain-size distribution around 1 µm which is probably an artefact of the calculation method. However, despite these documented problems with the Fraunhofer model, the Mie Theory has not been widely adopted in soil science because the size fractions usually comprise a mixture of mineralogies for which it is difficult to determine the optical properties of. Konert and Vandenberghe (1997) also note that the Fraunhofer model is well-suited to non-spherical clay particles that make up much of the clay-sized fraction in soils and that the Mie-theory inversion procedure suppresses the amount of this type of fine material measured compared with the Fraunhofer model.

Several authors (Beuselinck, *et al.* 1998; Loizeau *et al.*, 1994; McCave *et al.*, 1986 and Pieri *et al.*, 2006) have observed that the clay-sized fraction is often under-estimated considerably by laser methods compared to sedimentation. The main reason suggested is the shape of phyllosilicate clay minerals. Pedocchi and Garcia (2006) investigated both methods simultaneously and identified the difference in light scattering by irregularly shaped particles compared to spheres as the main source of error in estimates of the settling velocity, but this assumes that the settling velocities for such particles are correct. Beuselinck *et al.* (1998) compared laser and sedimentation methods on milled quartz and samples of silty soil; the results for both methods were similar for milled quartz as particles are a regular and similar shape. The results for soil, however, are more difficult to interpret as they usually do not have a unimodal distribution or consistent mineralogy. Other authors have emphasized the contribution that particle density makes to differences between laser and sedimentation methods. Fedotov *et al.* (2007) indicated that over-estimation of the clay fraction by 1.5–5 times can be related to differences in particle density. Zobeck (2004) observed that correlation between laser and sedimentation methods varied with mineralogy and was stronger when non-calcareous soil samples were separated from calcareous ones.

Methods

Study region

Our study region comprises much of the area of lithomorphic chalk soil of England (Figure 1). The Chalk is subdivided into; the Grey and White Chalk Subgroups which outcrop in the northern, transitional and southern provinces (Figure 1). The composition of the chalk varies between provinces; the Grey Chalk contains most clay, whereas the White Chalk contains > 98 % calcium carbonate. The provinces have close stratigraphic correlations containing the same clay-rich marker beds suggesting that the broad mineral compositions are similar (Mortimore *et al.*, 2001). The clay mineral assemblage is dominated by smectite (Jeans, 2006) and soil on this bedrock is a Leptosol (IUSS Working Group WRB, 2006) or rendzina (Avery, 1980).

Sample collection and analysis

The soil samples analyzed for this study are from three surveys: a survey of three neighbouring fields on the Yattendon Estate (the southern province), the National Soil Inventory (NSI) (northern and transitional provinces) and the Geochemical Baseline Survey of the Environment (G-BASE) (northern and transitional provinces) (Figure 1).

Figure 2 near here

Yattendon

Topsoil samples (0–15 cm) were taken on a 30-m grid (308 points total) over three arable fields on the Yattendon Estate in Berkshire; fields 214, 215 and 217 (Figures 1 and 2). The fields were divided into landscape units (Figure 2). At each grid node, 6 cores of soil from 1 m^2 were bulked. Standard field observations were made at each grid node and standard laboratory analyses (Avery and Bascomb, 1982) were done on the air-dry <2 mm fraction of samples (Table 1). The same methods were applied at four field sites on different parent materials. The sites, Cricket Meadow (CM) and Football field (FF) were developed on the Lower Greensand, Underwood (UW) on Oxford Clay and Wallingford (Wall) on plateau gravels. These sites give a range of non-chalk soils for comparison with the Yattendon site.

Table 1 near here

Fifty samples were selected at random from each of the Yattendon fields for organic matter analysis by Tinsley's (1950) wet oxidation method. The equation of the line fitted to the relation between loss on ignition (LOI) and Tinsley organic matter values was used to convert all LOI data to Tinsley values; these were then divided by 1.724 (MAFF, 1986) to give organic carbon equivalents. Total carbon was determined using a LECO SC-444 carbon and sulphur analyser for 109 of the samples taken from all fields at Yattendon. This completely removes the carbon (organic and inorganic) fractions as the temperature reaches 950ºC. The percentage of organic carbon (Tinsley) was subtracted from the percentage of total carbon (LECO) to determine the percentage of inorganic carbon in each sample. Average percentages of inorganic carbon as a percentage of total carbon were determined for each landscape unit.

Three methods of particle size analysis were applied to the Yattendon samples. Hand-texturing of selected samples was done by a single observer by wetting the air-dry

<2 mm fraction of the soil and following the procedure of Nortcliff and Landon described in Rowell (1994). Sedimentation of selected samples was done by the pipette method of Avery and Bascomb (1982) following peroxide pre-treatment to remove organic matter. Laser psd was done by Natural Resource Management Limited (NRM – www.nrm.uk.com) with < 1 g of air dry <2 mm soil dispersed in 3.3 % sodium hexametaphosphate solution by a Coulter laser granulometer without prior ultrasonic dispersal. An 8 µm threshold was used for the upper limit of the clay-sized fraction and the Fraunhofer model was used.

For every batch of laboratory analyses, replicate, control and reference samples were included to ensure analytical variation was within acceptable limits.

National Soil Inventory (NSI)

Sampling for the NSI was done between 1978 and 1983. Almost 5700 samples were taken on a 5-km grid across England and Wales (McGrath and Loveland, 1992). The top-soil (0-15 cm, excluding litter) was sampled. Twenty-five cores were taken at the nodes of a 5-m grid within a 20-m square centred on the 5-km grid node and bulked. The air dry < 2 mm fraction was used to determine psd (2 µm for clay/silt boundary) by the pipette method following peroxide pre-treatment to remove organic matter (Avery and Bascomb, 1982).

Geochemical Baseline Survey of the Environment (G-BASE)

Soil was sampled randomly within every other square kilometre of the British National Grid across eastern England (see 1). At each site five cores of topsoil (0-15 cm, excluding litter) were taken from the centre and corners of a 20-m square and bulked. Particle size distribution was determined for 0.25 g of the air-dry <2 mm fraction of the soil which had been pre-treated with hydrogen peroxide and centrifugation to remove organic matter using a Coulter laser granulometer and the Fraunhofer model. Reference standards were measured to ensure accuracy. The sand/silt fraction boundary was 63 µm and for the silt/clay boundary values of 2 µm or 8 µm were used.

A 50 g sub-sample was finely ground and the concentration of calcium (expressed as its weight percent oxide; CaO) was determined by wavelength dispersive XRFS (X-Ray Fluorescence Spectrometry). The concentration of calcium oxide will generally be large for chalk compared to non-chalk parent materials and Ca increases with carbonate content.

For NSI and GBASE sample locations a parent material code (based on combinations of solid or superficial geology) was assigned. from 1:50,000 maps of bedrock geology and superficial deposits of England, part of DigiMap GB (British Geological Survey, 2006). Locations with chalk parent material were selected from both the NSI and G-BASE surveys. In addition, sampling locations with three other parent materials (blown sand, mudstone and tidal flats) were selected from the surveys so that differences in determining psd by laser granulometry and sedimentation on other parent materials could be compared to those observed for chalk. The distribution of samples derived from each of the four parent materials from the NSI and G-BASE surveys is shown in Figure 1.

Scanning electron microscope (SEM) analysis

One soil sample taken from the middle of the eastern part of field 215 (Figure 2) at Yattendon was examined by SEM. It was split into two sub-samples; A (pure chalk) and B (a mixture of chalk and other soil mineral fragments) for characterization. Sub-samples were mounted on aluminium stubs using Leit-C carbon cement and carbon adhesive tabs. The sub-samples were carbon-coated then observed with an LEO 435 VP digital scanning electron microscope (SEI). This was equipped with an Everhart-Thornley detector for secondary electron imaging and a KE Developments four-quadrant (4 diode-type) solid-state detector for backscattered electron imaging (BSEM). The SEM analysis was performed by SEI under conventional high vacuum conditions, using a beam accelerating voltage of 20 kV and a beam current of between 100-300 pA. Qualitative microchemical mineral composition was determined with an Oxford Instruments INCA energy-dispersive X-ray microanalysis system (EDXA).

Correlation Analysis

Pearson's correlation coefficients were calculated between various soil variables and soil particle size fractions for the Yattendon site as a whole (Yall) and for each of the 3 fields separately (Y214, Y215 and Y217). Correlation coefficients between the same soil variables were also determined for the other field sites where the soil has developed on different parent materials (CM, FF, UW and Wall). As all these data were on a grid they are not independent, and the correlation coefficients will be larger than would be expected if there was no spatial autocorrelation. Nevertheless, they provide a good indication of the relative strengths of the relations between variables. As sand, silt and clay are expressed as percentages of the air dry < 2 mm fraction of the soil they are subject to non-stochastic constraints because they must sum to 100%. This can lead to spurious correlations between compositional variables (Aitchison, 1986), and by implication, the correlation of these fractions with other soil properties might be affected. A log-ratio transform can be applied to compositional variables, such as psd, (Lark and Bishop, 2007) before computing the correlation coefficients.

The centred log-ratio (clr) transform of Aitchison (1986) was used here as it has been used successfully for compositional data in correlation analysis and principal components analysis (PCA), (Neff, 1994).

If there is a composition X of D elements:

$$\mathbf{X} = [x_1, x_2, \ldots, x_D],$$

such that

$$x_i > i = 1,2,\ldots,D,$$

and

$$\sum_{i=1}^{D} x_i = 1.$$

The centred log-ratio transform of an observation (x_i) is:

$$\mathrm{clr}(\mathbf{X}) = \ln((x_i)/g\{(x_i)\}) \qquad (2)$$

where g is the geometric mean of the elements of the composition:

$$g = \sqrt[D]{x_1 \times x_2 \ldots. \times x_D} \qquad (3)$$

Results

The Yattendon Survey

The soil at Yattendon has developed on the White Chalk. The soil on the plateau area and in the bottom of the valley is the darkest in colour (Figure 2) and that on the south-facing slopes is the lightest. The variation in soil colour at this site is related to soil depth; the shallower was the depth to chalk, the lighter the soil colour appeared. The colour of the soil in the aerial photograph (Figure 2) is also governed by the amount of chalk at the surface. The soil on the plateau has developed on clay with flints overlying the chalk, which has resulted in deeper and darker colour. The average inorganic carbon as a percentage of total carbon (Table 2) confirms the observations from the aerial photograph that field 214, which comprises the north-facing slope and most of the plateau area, has the least inorganic carbon or chalk in the topsoil (Figure 2). The topsoil of field 215, particularly that on the south-facing slope, has the most inorganic carbon and chalk.

Table 2 near here

Correlation analysis

The percentages of sand-, silt- and clay-sized particles at each grid node (Figure 2) determined by laser granulometry using an 8 µm threshold were used for this analysis.

There were 308 sampling points for the site; each individual field has about 100. Pearson correlation coefficients (*r*) >0.195 are significant at the 0.05 level (Ebdon, 1994) for each field. Table 3 gives the correlation coefficients between several soil properties and the raw clay, silt and sand percentages for the whole site (308 points). The values in bold emphasize the correlations between certain soil variables and the sand- and clay-sized fractions. Where the sand-sized fraction comprises mainly chemically inert quartz, its correlations with LOI, moisture correction factor (MCF) and volumetric water content (VWC) are typically negative. Table 3 shows that the reverse is generally true at Yattendon where the correlations are positive. When the clay-sized fraction comprises chemically reactive phyllosilicate clay minerals, its correlations are generally positive with LOI, MCF and VWC. Again the reverse is true at Yattendon where the correlations are negative ($-0.351 < r < -0.541$). The correlation coefficients between soil properties and the clr transformed clay, silt and sand percentages for Yattendon are given in Table 4. They are similar to those for the raw data (Table 3) in both strength and sign; they are not consistently stronger or weaker. This indicates that the compositional nature of these data is not the cause of the correlations being the reverse of what we would expect. The correlations between raw sand, silt and clay data and other variables and the clr transformed sand, silt and clay data and other variables were similar in sign and strength to those for the individual fields at Yattendon. Therefore, only the correlations for the clr transformed data are given for each field.

Tables 3 and 4 near here

Tables 5–7 give the correlations between the clr transformed sand, silt and clay data and the other soil properties separately for each field at Yattendon. The correlations between clr sand and clay and the other soil properties are weak and insignificant in field 214 (Table 5), and there is no pattern of positive or negative correlations. This field, which comprises the north-facing slope and most of the plateau area (Figure 2), has the smallest proportions of inorganic carbon of the fields studied at Yattendon (Table 2). Figure 2 shows that this field is also markedly darker than the other two fields, which suggests that the topsoil contains fewer chalk fragments. The depth of soil to chalk on the plateau is between 70 and 120 cm, and the parent material is clay with flints.

Tables 5-7 near here

The correlations between clr sand and LOI, MCF and VWC are positive and moderate, and those between clr clay and these properties are negative and moderate in field 215 (Table 6). This field contains the south-facing slope, which has the largest proportion of inorganic carbon (15 % greater than the other fields, Table 2) and a small part of the plateau area (Figure 2). Figure 2 shows that this field is also markedly lighter in colour than field 214.

The correlations between clr sand and LOI, MCF and VWC are positive and those between clr clay and these properties are negative in field 217 (Table 7). The correlations for this field are slightly weaker than those for field 215 (Tables 6 and 7). The valley in field 217 (Figure 2) has the second largest proportion of inorganic carbon at the Yattendon site (Table 2). The south-facing slope in this field is white (Figure 2), but this area was not sampled, whereas the valley which was sampled is darker, but not as dark as field 214.

Tables 5–7 also show that for all fields there is a positive correlation between Munsell value and clr clay, and a negative one with clr sand. Lighter soil has larger Munsell values and vice versa. Darker coloured soil is often associated with large clay and organic matter contents, but again these typical relationships are the reverse here.

The correlation results suggest that as the proportion of inorganic carbon increases the soil becomes paler, and more chalk fragments are present. Furthermore, the correlations between clr sand and LOI, MCF and VWC become more strongly positive and those between clr clay and these variables become more strongly negative. We suggest that this is so where soil has developed on chalk because a considerable proportion of the sand-sized fraction comprises porous chalk fragments which are chemically reactive and hold water unlike quartz grains that are usually typical of this fraction. In addition, we suggest that a significant proportion of the clay-sized fraction comprises calcite crystals that are chemically unreactive, do not readily hold water and do not have the platy shape of phyllosilicate clay minerals. As the 8 μm threshold for the boundary between the clay and silt-sized fractions was used here, some fine quartz grains from the silt-sized fraction could have been included in the clay-sized fraction. The different mineralogy of the sand- and clay-sized fractions where soil has developed on chalk could account for the differences in the correlations between the particle size fractions and other soil properties. The correlation results between clay and LOI, MCF and VWC, and sand and LOI, MCF and VWC in Tables 3–7 are compared with those from the non-chalk sites in Table 8. They show that the relationships of LOI, MCF and

VWC with clay are all positive for the non-chalk sites and all negative for the chalk sites (except for clay and MCF for Y214), and the reverse is generally true for the relationships of these variables with sand. Indeed, Kruskall Wallis H tests showed that there were significant differences in the correlation coefficients for the chalk and non-chalk sites at a level of $p < 0.001$ showing that the differences in the relationships of these variables at chalk and non-chalk sites is not likely to be due to chance.

Table 8 near here

Comparison of the results of different methods to determine particle size

Figure 3 shows the textural class of the topsoil determined by different methods for the four landscape units at Yattendon and for soil data at sites not on chalk parent material. All the diagrams show that determinations of psd based on sedimentation generally have less clay and more silt than those based on laser methods; the 8 µm threshold has been used for the latter. The divergence between the plotted points for the two methods is very small for sites where the soil was not developed on chalk (Figure 3e), and greatest for the plateau and south-facing slope at Yattendon (Figure 3b and c); the latter had the most inorganic carbon (Table 2) and the soil was the lightest in colour (Figure 2). For the north and south-facing slope and valley (Figure 3 a, c and d) hand-texturing indicated similar proportions of clay and silt to the sedimentation method, but less sand. The plateau area had large organic matter contents as reflected in the dark colour of the soil (Figure 2).

Hand-texturing was less reliable at determining the clay- and silt-sized fractions of this soil and the sand fraction was markedly over-estimated.

Figure 3 shows the differences in soil textural class based on the pipette and laser methods where as Figure 4 shows the relationship between the values determined by these methods for each particle size fraction. When Chalk and non-chalk samples are included together for regression (Figures 4 a, c and e) the relationship is strongest for sand and weakest for clay. This might be expected given that many of the problems with both methods of particle size determination are associated with the clay fraction and the appropriate sizing of non-spherical phyllosilicates. When separate regression lines are fitted for the non-chalk soils and samples with > 4% $CaCO_3$, it is evident that the slope of the lines for sand are similar (Figure 4b), but the lines for samples with > 4% $CaCO_3$ are steeper than for the non-chalk soils for silt and clay (Figures 4 d and f). This difference in steepness is most marked for clay (Figure 4 f). This difference in slope suggests that the 8 µm cutoff might not be appropriate for chalk soils as the relationship between laser and sedimentation methods is different.

Figures 3 and 4 near here

Scanning electron microscope (SEM) analysis

The sample from field 215 at Yattendon (Figure 2) was analysed by SEM by two independent observers. The first observed that the sand-sized fraction comprised mostly porous chalk particles, a small quantity of angular quartz and some rounded quartz grains.

Chalk particles and fragments, diagenetically smoothed quartz crystals and rounded aggregates of calcite particles of <10 µm were present. Some weathered feldspars could also be seen in this size range. At ≤5 µm crystalline calcite rhombs were clearly visible. At <1 µm, the material was largely calcite in the form of rounded grains with identifiable faceted crystal faces. At <1 µm there were fewer fragments of clay minerals than one would expect; illite was clearly visible and kaolinite might be present. Coccoliths are visible throughout the size ranges and some fragmented coccoliths were probably included in the <1 µm aggregates of calcite material (*Pers. Comm.*, Peter Bull, Department of Geography, University of Oxford).

Based on SEI results, the second observer noted that a substantial proportion of the pure chalk fragments in the soil comprise coccoliths; the outer walls of minute planktonic algae (coccolithophores). Coccoliths are formed of calcium carbonate plates, each less than 3µm, that are aggregated to form composite particles, up to 10 µm in diameter, that typically have ring-like structures. Coccolith rings and degraded component calcite plates comprise most of sub-sample A (silt and sand-sized fractions, Figure 5). Most of the clay-sized fraction in the sub-sample is dominated by crystalline calcite <2 µm in size. Coarser crystals of calcite (some of which could be part of the primary, bioclastic sediment) are dispersed within a clay mineral matrix (Figures 5 and 6).

Figures 5 and 6 near here

Sub-samples A and B are quite different; the latter comprises disaggregated mineral grains and possibly organic debris. The mineral grain size varies between 1 and 40 µm, and most of the calcite grain edges are rounded and etched. The mineral grains are often interlocked and the pores are frequently blocked by clay-sized particles. Secondary electron images suggest that coccoliths and foraminifera bioclasts are degraded to smaller, irregular calcite particles.

The SEM results for both sub-samples suggest that there is some dissolution of chalk in the soil and that secondary porosity could be developed within the component chalk clasts. These pores are more than 10–30 µm, larger than those in the background chalk matrix because of dissolution and leaching of calcite grains. The morphology of the leached voids resembles the original mineral or clast morphology (Figure 7). The matrix of both sub-samples is composed of calcite particles (up to 2 µm) with rounded edges and overgrowth zones. The proportion of clay minerals is relatively small, however, authigenic tubular alumino-silicate fibres up to 5 µm in length and about 0.25 µm in diameter are observeable. Some of the clay minerals form a “mesh” of tubes and cylinders that bind mineral particles together which contributes to the blocking of pores. Other clay minerals, such as illite and kaolinite might also be present, although they cannot be clearly defined from the existing observations.

Figure 7 near here

The NSI and G-BASE surveys

Figure 8 shows the ternary diagrams for four soil parent materials; Chalk, Blown Sand, Mudstone and Tidal Flats using psd information determined by sedimentation for the NSI and by laser for the G-BASE survey. Two diagrams are given for each parent material to show the range of soil textures for the 2 and 8 μm thresholds when psd is determined by the laser method. Ideally, comparisons of the two methods should be made with soil samples from the same locations, however, such data are not currently available. Nevertheless, there are two reasons to suggest that a comparison based on samples from independent sites would not affect the results adversely. First, the samples from each survey cover a broad geographic area with a significant proportion of lithomorphic chalk soil (Figure 1) and similar range of soil types. Second, the ternary diagrams (Figures 8a and 8e) and histograms (not shown) show that the samples encompass a broad range of soil psd and have similar distributiosn, and there is also a wide range of values for the sand-sized fractions, the fraction that is identified most reliably by both methods (i.e. NSI sedimentation, 18–95 % sand; G-BASE laser, 10–85% sand). We feel justified, therefore, in drawing some general conclusions from a comparison of these two datasets. Here we are not interested in the textural class that the chalk soil falls into given the different boundaries, but the range of values of sand, silt and clay that are identified for two surveys that cover the same broad geographic region.

Figure 8 near here

Table 9 gives the mean and range of percentage calcium (expressed as weight percent oxide) in samples from the G-BASE survey over four parent materials. For the chalk soil, the concentration of calcium will be dominated by the quantity of carbonate (calcite) in each sample. For soil developed on the other three parent materials, Ca reflects the quantities of all Ca bearing minerals, including calcite, phyllosilcates and feldspar. The greater Ca concentrations in the chalk soil (Table 9) indicate it contains markedly larger amounts of calcite than soil samples from the three other parent materials.

Table 9 near here

Figure 8 a–d shows that when a 2 μm threshold is used for the laser results, the range of particle size fractions for the NSI and G-BASE data is not comparable. The particle size fractions determined by laser from the G-BASE survey appear to have less clay and more silt than those determined by sedimentation from the NSI survey. The 8 μm threshold essentially redistributes part of the silt fraction to the clay fraction. Figure 8 f–h shows that when the 8 μm threshold is used for soil developed on non-calcareous parent materials, the psd identified by sedimentation and laser methods in the two surveys overlaps. The 8 μm threshold was suggested by Konert and Vanenberghe (1997) for general use, but when used for soil developed on chalk (Figure 8e) samples measured by laser from the G-BASE survey appear to have more clay and less silt than those from the NSI survey, which used sedimentation. This is the same pattern as was observed for the Yattendon data. The ternary diagrams for chalk soils (Figure 8 a and e), suggest that the 8 μm threshold over-corrects for chalk samples because the laser results change from under-estimating clay content when the 2 μm threshold is used, to over-estimating it with the 8 μm threshold.

While Figure 8 is concerned with differences in the texture of the soil on different parent materials using the 2 and 8 µm cut-offs, Figure 9 shows the relationships between laser and sedimentation methods for each parent material and particle size fraction. As the NSI and GBase surveys used different sampling points, but the broad geographical areas and histograms for each size fraction for each of the surveys were similar in range and mean etc., correlations were calculated between summary statistics (minimum, maximum, mean, median, lower quartile and upper quartile) for each survey on a given parent material. The only exception to this procedure was for the G-BASE survey for Blown sand where following examination of the histogram, two extreme outliers with very low sand contents were removed before calculation of the summary statistics.

The most important feature in Figure 9 is the consistency in the slope of the regression lines for clay (Figure 9c) for parent materials other than chalk (0.38-0.47x) and which correspond to similar gradients identified in previous studies. Eschel et al. (2004) found a gradient of 0.35x, Konert and Vandenberghe (1997), 0.36x, Beuselinck et al. (1998), 0.35x and Buurman et al. (2001), 0.29x and 0.39x for Marine and Loess sediments, respectively. In contrast to these gradients, that for chalk soil is 0.71x approximately double that reported by others, and here, for non-chalk soils. Also, the gradient for the line for chalk soils (8 µm) in Figure 4 for the Yattendon site was slightly more than double that of the line for non-chalk sites. These differences in regression line slopes for chalk and on-chalk soils indicate how differences in the mineralogy can significantly influence the particle size determined by both methods.

The results confirm that the 8 µm cut-off is suitable for non-chalk soils. The differences in psd determined by the laser and sedimentation methods when the 8 μm threshold is used for laser results (Figures 3 and 8) and the differences in the slopes of the regression lines for chalk and non-chalk soils for clay (Figure 9c) can be accounted for by differences in particle density and shape for chalk soils. Mineralogically, calcite is denser than quartz (Klein and Hurlbut, 1993). However, fine (a few microns in diameter) calcite particles in chalk soil will aggregate to form chalk fragments (Sumbler, 1996), which are porous and less dense than quartz particles of the same size. Therefore, they take longer to fall a given distance through water as a smaller particle would. This means that the proportion of clay- and silt-sized particles can be over-estimated by the sedimentation analysis of a chalk soil. It seems that the average particle density of 2600 kg m^{-3} is not appropriate for particle size determination of chalky soil.

The over-estimation of the clay-sized particles with the 8 μm threshold and the steeper slopes of the regression lines for chalk soil can also be attributed to differences in particle shape. When using laser methods, this threshold essentially adjusts for the fact that much of the clay-sized fraction identified by sedimentation is dominated by platy, phyllosilicate clay minerals. If these minerals are oriented so that the laser beam intercepts them along their long-axis they would appear larger than their average diameter suggests. Pabst *et al*. (2000) found that for samples rich in clay minerals, modification of Stokes' law to deal with the plate-like shape of such minerals meant that sedimentation results were in good agreement with laser methods. Lerman *et al*. (1974), Lu *et al*. (2000) and Pabst *et al.* (2001) have also looked at Stokes' theory of settling velocities for particles of different shape. This suggests that it is the sedimentation methods that need to be adjusted. The issue of particle shape does not need to be compensated for with chalk soil where the clay-sized fraction is composed largely of tiny calcite crystals.

Although adjusting thresholds between the size fractions to gain agreement between laser and sedimentation methods is basically unsound, it provides an interim practical solution for comparison with the large amount of particle size analysis that has been done in the past using sedimentation methods. The 2 μm threshold (or some threshold less than 8 μm) would correspond with the proportions of sand-, silt- and clay-sized particles if particle shape was taken into account when psd is determined by sedimentation. Typically, particle shape is not accounted for with sedimentation methods, but Scott-Jackson and Walkington (2005) suggested a boundary of between 6.5 and 7.5 μm for direct correlation between sedimentation and laser methods for a clay with flints soil developed over chalk. For this parent material, chalk contributes to less of the mineral matter than for a lithomorphic chalk soil. Therefore a threshold less than this should be suitable for lithomorphic chalk soil. Figure 9 shows ternary diagrams of the psd of the top-soil determined by laser and sedimentation methods for the NSI and G-BASE soil samples developed on chalk with a 4 µm cut-off for the clay:silt boundary for laser methods, cut-offs of 5 and 6 µm were also investigated but are not shown. This diagram shows that there is considerable overlap between the sedimentation (NSI survey) and laser methods (G-BASE survey) when a 4 µm cut-off for the clay/silt boundary is used for laser methods. Clearly an appropriate threshold for the clay/silt boundary for laser methods will depend on soil carbonate content and its particle size.

Figure 9 near here

Discussion

Our results have considerable implications for determining the psd of soils developed on chalk which contain a significant proportion of lithogenic calcium carbonate. For example, in England chalk is the parent material for soil over 8825 km^2, about 7 % of the soilscape. Based on land cover data (Fuller et al., 1994), about half of the area of lithomorphic chalk soil is used for arable agriculture, which is some 8.5% of the total arable area in England. In addition, there are seven NSAs underlain by chalk parent material in England (Figure 1; MAgiC, 2008a; British Geological Survey, 2006). Based on the map of soil parent material (British Geological Survey, 2006), we have determined that about 70% of the area of lithomorphic chalk soil (6178 km^2) is classified as an NVZ (MAgiC, 2008b). There is clearly scope for improving recommendations for fertilizer application rates based on an improved understanding of the psd of chalk-soil and water-holding properties to reduce the leaching of nitrate to water.

Given the problems of laser and sedimentation methods for determining psd on chalk, hand-texturing should be used initially to give an indication of soil texture that can be compared with the results from sedimentation and laser methods. Soil surveyors have noted that soil developed on chalk often feels more silty than the analytical results indicate, but the psd is usually based on sedimentation methods which can over-estimate the clay sized fraction when phyllosilicates are present.

Typical agricultural soil often has clay-sized fractions dominated by chemically reactive, moisture holding phyllosilicate minerals and sand-sized fractions dominated by chemically inert quartz grains that do not readily hold moisture. The SEM analysis showed that moisture-holding, porous chalk fragments comprise much of the sand-sized fraction in chalk soil and that fine calcite crystals, which do not readily hold water, constitute a significant proportion of the clay-sized fraction. Correlation analysis showed that these differences in mineralogy had implications for the behaviour of the soil and that relationships were different for chalk and non-chalk soil. Relations between soil texture and important soil properties in chalk soil were the reverse of those commonly observed in soil dominated by silicate minerals, and the strength of these relationships increased with the proportion of inorganic carbon in the soil. For chalk soil with significant quantities of calcite in the clay-size fraction, the 8 μm threshold over-corrects the partition between clay and silt because the clay-sized fraction is not dominated by phyllosilicates with their non-spherical geometry. Sedimentation methods can slightly over-estimate the clay content when using an average particle density for calculations, but this effect appears to be smaller than phyllosilicate mineral shape.

Conclusions

From this study, we suggest an interim solution to the problems associated with analysis of psd in chalk soil would be to use a clay/silt boundary of 4 μm for the psd determined by laser to correspond with the results from sedimentation. This is not an optimal solution; these two methods are measuring slightly different things soshifting boundaries between the size fractions does not solve the problem. However, based on our observations in this study, it does provide a means of overcoming this difficulty in the short-term, until a more theoretically sound solution is found.

Land managers need to know how agrochemicals will move through the soil which is strongly related to fundamental soil properties, including psd and mineralogy. This is a complex problem for soil developed on chalk because there are major differences in the physical and chemical behaviour of the minerals in the clay- and sand-sized fractions in comparison to most other soil types, as highlighted by the correlation and SEM analysis in this study. It is difficult to predict soil properties such as water-holding capacity because of the differences in composition of the sand and clay fractions. Further investigation of the mineralogy and behaviour of the particle size fractions of chalk soil is required so that fertilizer and pesticide recommendations can be adjusted accordingly. This is important because 70 % of the Chalk outcrop in England is classified as an NVZ.

On a more theoretical note, the relative merits of the Fraunhofer model and Mie theory need to be assessed for laser psd of chalk soils given the porosity of the sand-sized particles. There is clearly a requirement for these two models to be combined; the greater accuracy of the former for small size fractions with the benefits of the latter for the larger size fractions. Further research is also needed to determine an appropriate particle density figure for sedimentation analysis of chalk soil. An adaptation of standard sedimentation methods might also be needed to account for differences in shape and mineralogy of the size fractions. This would be a more theoretically sound way of proceeding than the application of an arbitrary boundary between clay and silt for laser methods because it would vary with the proportion of carbonate in the soil. However, it would probably result in various soil types being attributed with different behaviour than is traditionally associated with them because most notions of soil behaviour are extrapolated from standard sedimentation analysis.

When measuring the psd of chalk soil, practitioners need to consider the impact their chosen method will have on their results. An alternative procedure would be to measure psd before and after the removal of calcite from their samples using acid. This would estimate the proportion of calcite in each size fraction and this could be used to infer the behaviour of agrochemicals in such soil. We suggest that samples should not be shaken or treated with ultrasound to avoid destroying the fragile, porous chalk fragments that make up much of the sand-sized fraction. With further research, agro-chemical manufacturers should provide separate application guidelines for chalk soil, given its unique properties which are related to its composition. It is beyond the scope of this paper to redefine the textural classes based on sedimentation. However, to apply and compare laser methods properly to sedimentation methods, such a major change in standard methodology may be needed. This is particularly so given the savings in time and money with the use of laser methods and the importance to practitioners who need to know the psd of many sample for precise management.

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Figure and Table Captions

Figure 1. The location of lithomorphic Cretaceous Chalk soils in England, the Yattendon site and NSI and GBase samples collected from different parent materials. Nitrate Sensitive Areas shown with numbered notations are: (1) Kilham, (2) Springwells, (3) North Newbald, (4) North Lincolnshire Wolds, (5) Sedgeford, (6) Slip End, (7) Ogborne St George. NSI textures © Cranfield University (National Soil Resources Institute), 2008.

Figure 2. Aerial photograph of the Yattendon site showing sampling point locations

Figure 3. Ternary diagrams showing the particle size distribution of the top-soil determined by different methods for the (a) north-facing slope, (b) plateau, (c) south-facing slope and (d) valley at the Yattendon site and for (e) other sites not on chalk.

Figure 4. Regression plots showing the relationship between laser and pipette methods for the Yattendon and some non-chalk sites: (a-b) sand, (c-d) silt and (e-f) clay fractions. Separate regression lines for non-chalk soils, and chalk soils with > 4% inorganic carbon are shown in b, d and f.

Figure 5. High magnification SEM microphotograph of coccoliths outer ring (subsample A)

Figure 6. High magnification SEM microphotograph showing rhombic and prismatic crystals of calcite (subsample A). These represent component plates of coccoliths, and possible coccolith spines derived from the primary chalk sediment

Figure 7. High magnification SEM microphotograph of pores in subsample B.

Figure 8. Ternary diagrams showing the particle size distribution of the top-soil determined by laser and sedimentation methods for the NSI and GBase soil samples developed on: (a, e) chalk, (b, f) blown sand (c, g) Mudstone, (d, h) Tidal flats using 2 µm and 8 µm cutoffs, respectively for laser methods. NSI textures © Cranfield University (National Soil Resources Institute), 2008.

Figure 9. Regression plots showing the relationship between laser and pipette methods for the different parent materials in the NSI and GBase surveys for the (a) sand, (b) silt and (c) clay fractions.

Figure 10. Ternary diagrams showing the particle size distribution of the top-soil determined by laser and sedimentation methods for the NSI and GBase soil samples developed on chalk with a 4 µm cut-off for the laser methods. NSI textures © Cranfield University (National Soil Resources Institute), 2008.

Table 1. Field and Laboratory Methods

Table 2. Inorganic carbon as a proportion of total carbon in each of the landscape units at the Yattendon site

Table 3. Correlation Coefficients between soil properties and raw clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for all areas at the Yattendon site

Table 4. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for all areas at the Yattendon site

Table 5. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 214

Table 6. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 215

Table 7. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 217

Table 8. Summary of correlations of key variables with CLR clay and sand for Chalk sites and non-chalk sites

Table 9. Proportion of carbonates in samples from NSI samples on different parent materials

Table 1. Field and Laboratory Methods

Soil property	Method
Depth (cm)	Auger and tape measure
Loss on ignition (LOI) (%)	500ºC, 10g air dry < 2 mm
Moisture Correction Factor (MCF) (%)	100ºC, 10g air dry < 2 mm
Munsell value	Air dry < 2 mm
Stoniness (%)	Standard charts
Texture – Sand, Silt, Clay (%)	Finger-texturing, pipette method, laser granulometry
Volumetric water content (VWC) (%)	Delta-T Theta probe calibrated for soil type

Table 2. Inorganic carbon as a proportion of total carbon in each of the landscape units at the Yattendon site

Location	Inorganic carbon as a proportion of total carbon (%)
North slope	36
Plateau	42
South slope	63
Valley	48

Table 3. Correlation Coefficients between soil properties and raw clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for all areas at the Yattendon site

	clay	depth	LOI	MCF	sand	silt	stones	value	VWC
clay	1.000								
depth	-0.373	1.000							
LOI	**-0.541**	0.000	1.000						
MCF	**-0.501**	0.076	0.833	1.000					
sand	-0.807	0.405	**0.254**	**0.339**	1.000				
silt	-0.658	0.113	0.589	0.413	0.087	1.000			
stones	-0.169	-0.139	0.097	-0.003	-0.069	0.374	1.000		
value	0.669	-0.286	-0.599	-0.669	-0.627	-0.330	0.024	1.000	
VWC	**-0.351**	0.239	0.283	0.430	**0.415**	0.064	-0.180	-0.428	1.000

Table 4. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for all areas at the Yattendon site

	CLR-clay	depth	LOI	MCF	CLR-sand	CLR-silt	stones	value	VWC
CLR-clay	1.000								
depth	-0.387	1.000							
LOI	**-0.487**	0.000	1.000						
MCF	**-0.472**	0.076	0.833	1.000					
CLR-sand	-0.908	0.401	**0.299**	**0.370**	1.000				
CLR-silt	-0.100	-0.079	0.390	0.186	-0.326	1.000			
stones	-0.130	-0.139	0.097	-0.003	-0.035	0.378	1.000		
value	0.670	-0.286	-0.599	-0.669	-0.631	-0.014	0.024	1.000	
VWC	**-0.371**	0.239	0.283	0.430	**0.413**	-0.144	-0.180	-0.428	1.000

Table 5. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 214

	CLR-clay	depth	LOI	MCF	CLR-sand	CLR-silt	stones	value	VWC
CLR-clay	1.000								
depth	-0.045	1.000							
LOI	**-0.166**	-0.321	1.000						
MCF	**0.084**	0.023	0.334	1.000					
CLR-sand	-0.815	-0.026	**0.086**	**-0.171**	1.000				
CLR-silt	0.277	0.092	0.037	0.194	-0.783	1.000			
stones	-0.031	-0.382	0.179	-0.205	0.057	-0.061	1.000		
value	0.044	-0.294	-0.097	-0.361	0.008	-0.061	0.436	1.000	
VWC	**-0.027**	0.332	0.033	0.316	**0.003**	0.023	-0.493	-0.454	1.000

Table 6. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 215

	CLR-clay	depth	LOI	MCF	CLR-sand	CLR-silt	stones	value	VWC
CLR-clay	1.000								
depth	-0.429	1.000							
LOI	**-0.664**	0.287	1.000						
MCF	**-0.635**	0.312	0.561	1.000					
CLR-sand	-0.917	0.308	**0.466**	**0.556**	1.000				
CLR-silt	0.215	0.117	0.206	-0.075	-0.587	1.000			
stones	-0.155	-0.019	0.178	0.016	0.050	0.192	1.000		
value	0.579	-0.321	-0.432	-0.457	-0.445	-0.085	-0.139	1.000	
VWC	**-0.350**	0.176	0.218	0.544	**0.320**	-0.074	-0.052	-0.175	1.000

Table 7. Correlation Coefficients between soil properties and centred log-ratio transform of clay, silt and sand percentages determined by laser methods with an 8 µm cut-off for Yattendon field 217

	CLR-clay	depth	LOI	MCF	CLR-sand	CLR-silt	stones	value	VWC
CLR-clay	1.000								
depth	-0.160	1.000							
LOI	**-0.500**	0.171	1.000						
MCF	**-0.591**	0.152	0.662	1.000					
CLR-sand	-0.881	0.049	**0.293**	**0.376**	1.000				
CLR-silt	-0.291	0.209	0.471	0.542	-0.144	1.000			
stones	-0.478	-0.081	0.316	0.463	0.402	0.309	1.000		
value	0.736	-0.137	-0.520	-0.762	-0.510	-0.576	-0.499	1.000	
VWC	**-0.200**	0.131	0.097	0.262	**0.193**	0.075	0.158	-0.307	1.000

Table 8. Summary of correlations of key variables with CLR clay and sand for Chalk sites and non-chalk sites

Site	Parent Material	Variables	Correlation	Variables	Correlation
Yall	Chalk	CLR clay and LOI	-0.487	CLR sand and LOI	0.299
Y214	Chalk	CLR clay and LOI	-0.166	CLR sand and LOI	0.086
Y215	Chalk	CLR clay and LOI	-0.664	CLR sand and LOI	0.466
Y217	Chalk	CLR clay and LOI	-0.500	CLR sand and LOI	0.293
Yall	Chalk	CLR clay and MCF	-0.472	CLR sand and MCF	0.370
Y214	Chalk	CLR clay and MCF	0.084	CLR sand and MCF	-0.170
Y215	Chalk	CLR clay and MCF	-0.635	CLR sand and MCF	0.556
Y217	Chalk	CLR clay and MCF	-0.591	CLR sand and MCF	0.376
Yall	Chalk	CLR clay and VWC	-0.371	CLR sand and VWC	0.413
Y214	Chalk	CLR clay and VWC	-0.027	CLR sand and VWC	0.003
Y215	Chalk	CLR clay and VWC	-0.350	CLR sand and VWC	0.320
Y217	Chalk	CLR clay and VWC	-0.200	CLR sand and VWC	0.193
CM	Lower Greensand	CLR clay and LOI	0.833	CLR sand and LOI	-0.860
FF	Lower Greensand	CLR clay and LOI	0.502	CLR sand and LOI	-0.598
UW	Oxford clay	CLR clay and LOI	0.782	CLR sand and LOI	-0.453
Wall	plateau gravel	CLR clay and LOI	0.374	CLR sand and LOI	-0.371
CM	Lower Greensand	CLR clay and MCF	0.413	CLR sand and MCF	-0.434
FF	Lower Greensand	CLR clay and MCF	0.483	CLR sand and MCF	-0.494
UW	Oxford clay	CLR clay and MCF	0.839	CLR sand and MCF	-0.669
Wall	plateau gravel	CLR clay and MCF	0.709	CLR sand and MCF	-0.700
CM	Lower Greensand	CLR clay and VWC	0.792	CLR sand and VWC	-0.816
FF	Lower Greensand	CLR clay and VWC	0.103	CLR sand and VWC	-0.396
UW	Oxford clay	CLR clay and VWC	0.459	CLR sand and VWC	-0.443
Wall	plateau gravel	CLR clay and VWC	0.593	CLR sand and VWC	-0.632

Table 9. Proportion of carbonates in samples from NSI samples on different parent materials

Location	Carbonates of NSI samples (%)	
	Range	Mean
Chalk sites	15.8-52.7	27.03
Blown sand sites	0.7-4.2	1.95
Mudstone sites	1.1-7.7	2.62
Tidal Flats sites	0.9-8.1	2.77

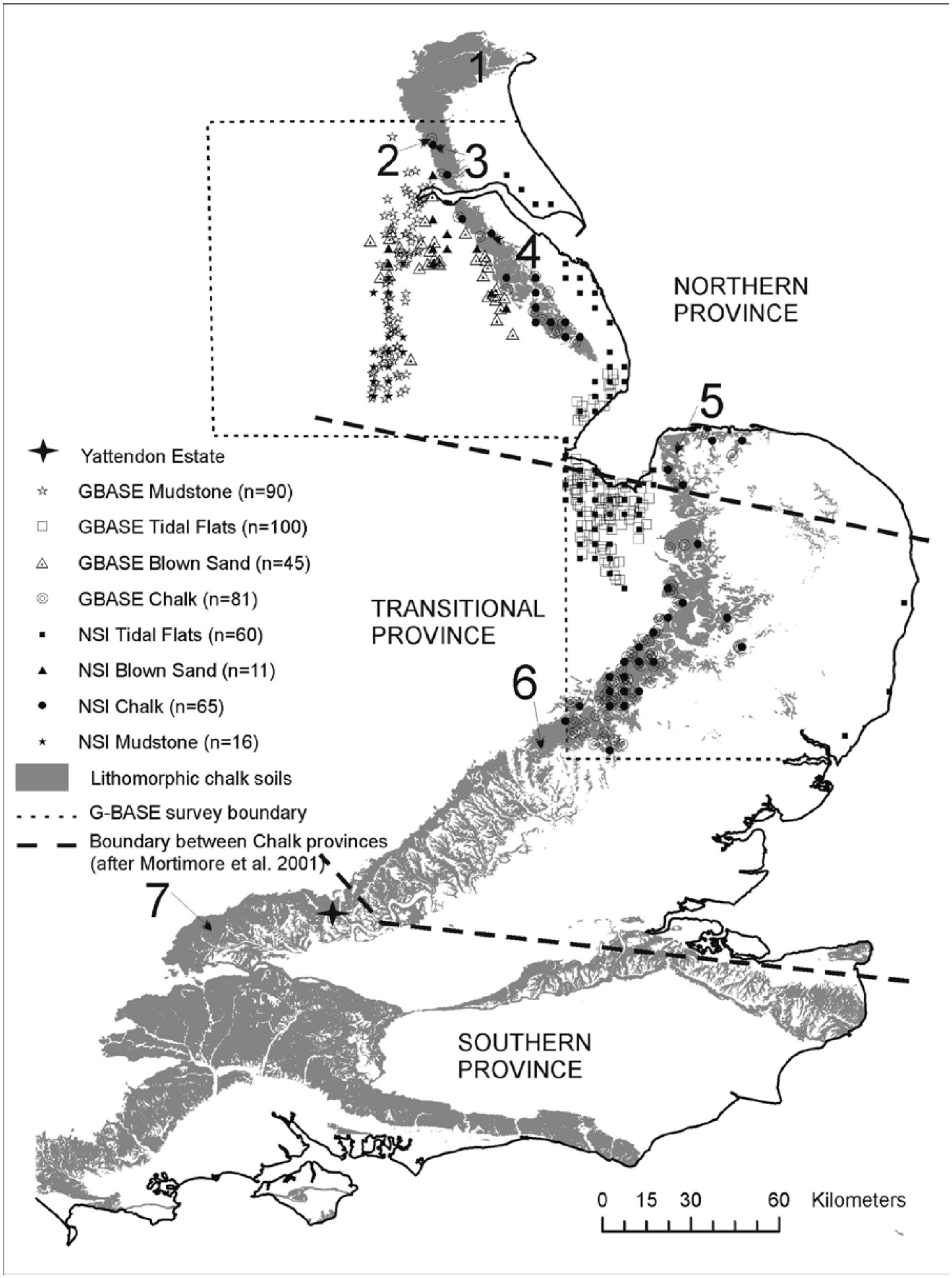


Figure 1. The location of lithomorphic Cretaceous Chalk soils in England, the Yattendon site and NSI and GBase samples collected from different parent materials. Nitrate Sensitive Areas shown with numbered notations are: (1) Kilham, (2) Springwells,

(3) North Newbald, (4) North Lincolnshire Wolds, (5) Sedgeford, (6) Slip End, (7) Ogborne St George. NSItextures © Cranfield University (National Soil Resources Institute), 2008

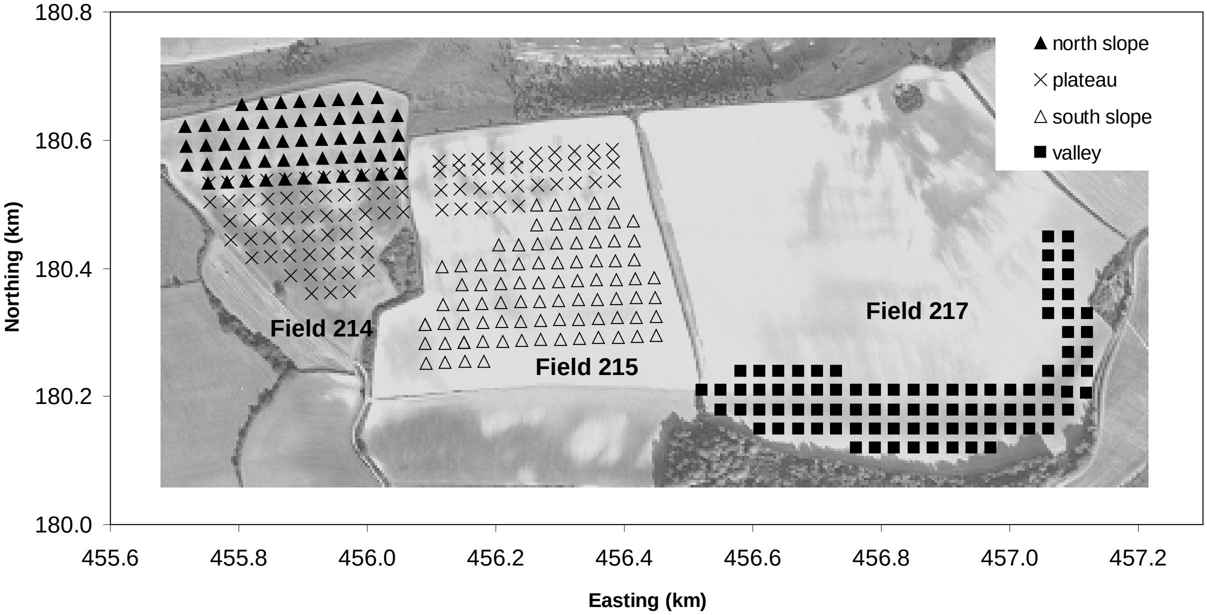


Figure 2. Aerial photograph of the Yattendon site showing sampling point locations

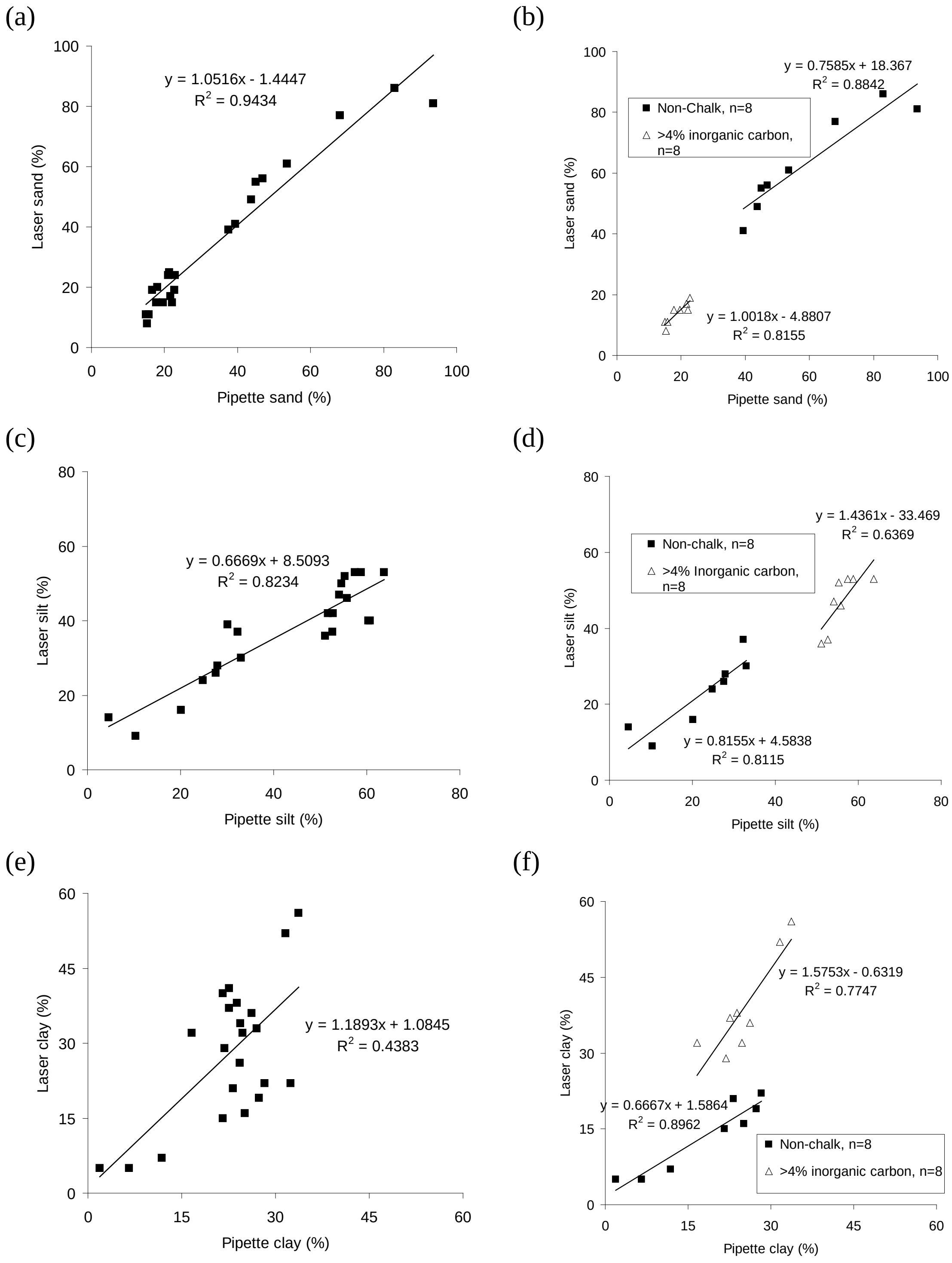


Figure 4. Regression plots showing the relationship between laser and pipette methods for the Yattendon and some non-chalk sites: (a-b) sand, (c-d) silt and (e-f) clay fractions. Separate regression lines for non-chalk soils, and chalk soils with > 4% inorganic carbon are shown in b, d and f.

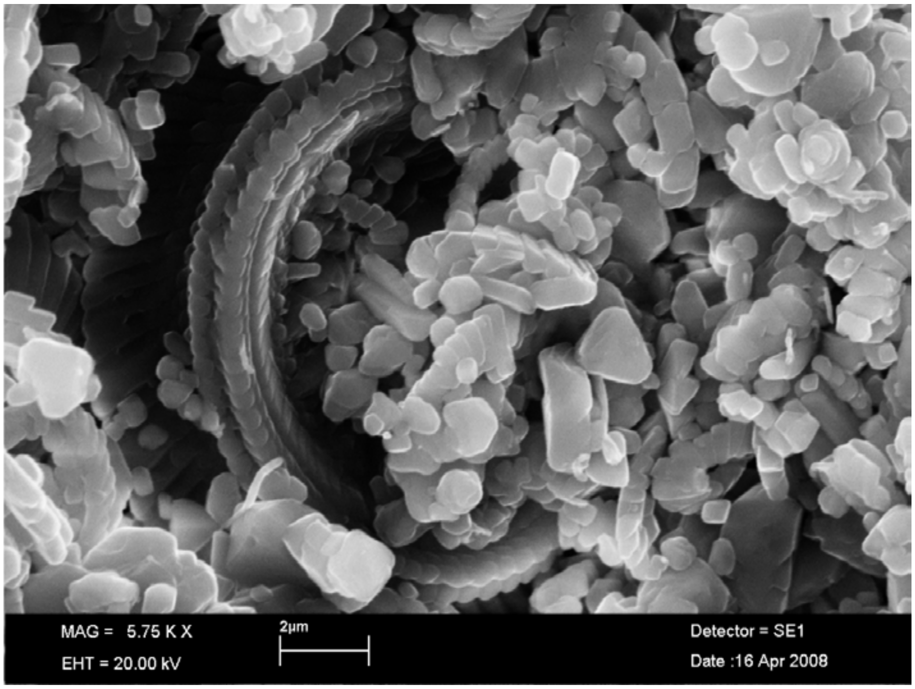


Figure 5. High magnification SEM microphotograph of coccoliths outer ring (subsample A)

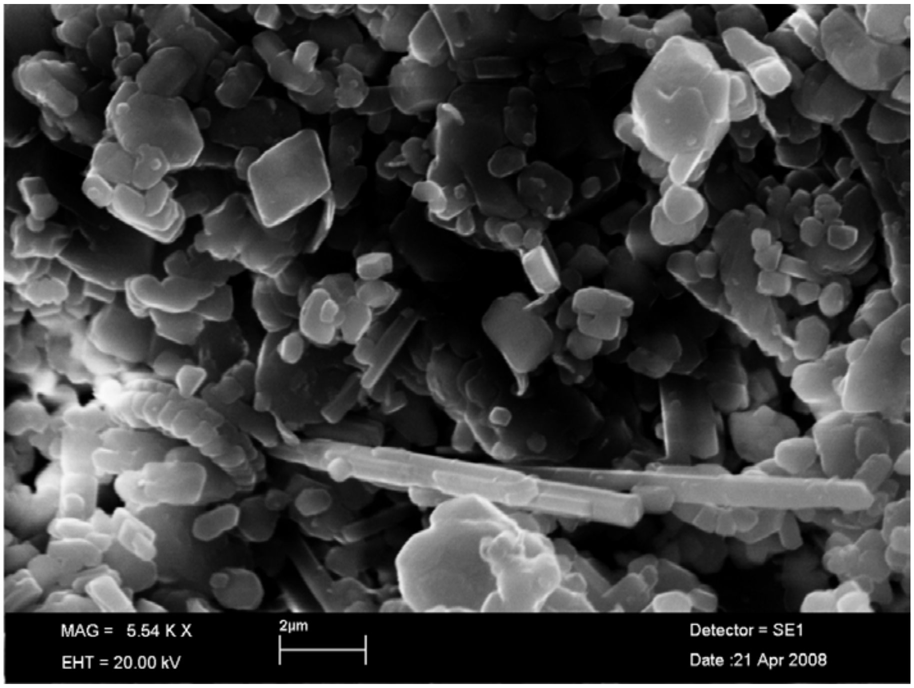


Figure 6. High magnification SEM microphotograph showing rhombic and prismatic crystals of calcite (subsample A). These represent component plates of coccoliths, and possible coccolith spines derived from the primary chalk sediment

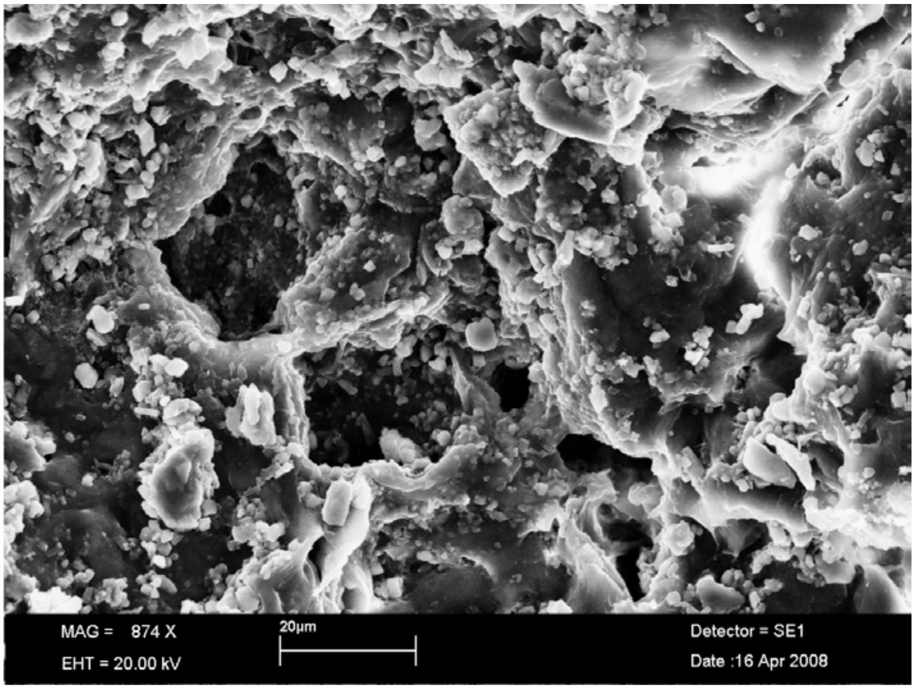


Figure 7. High magnification SEM microphotograph of pores in subsample B.

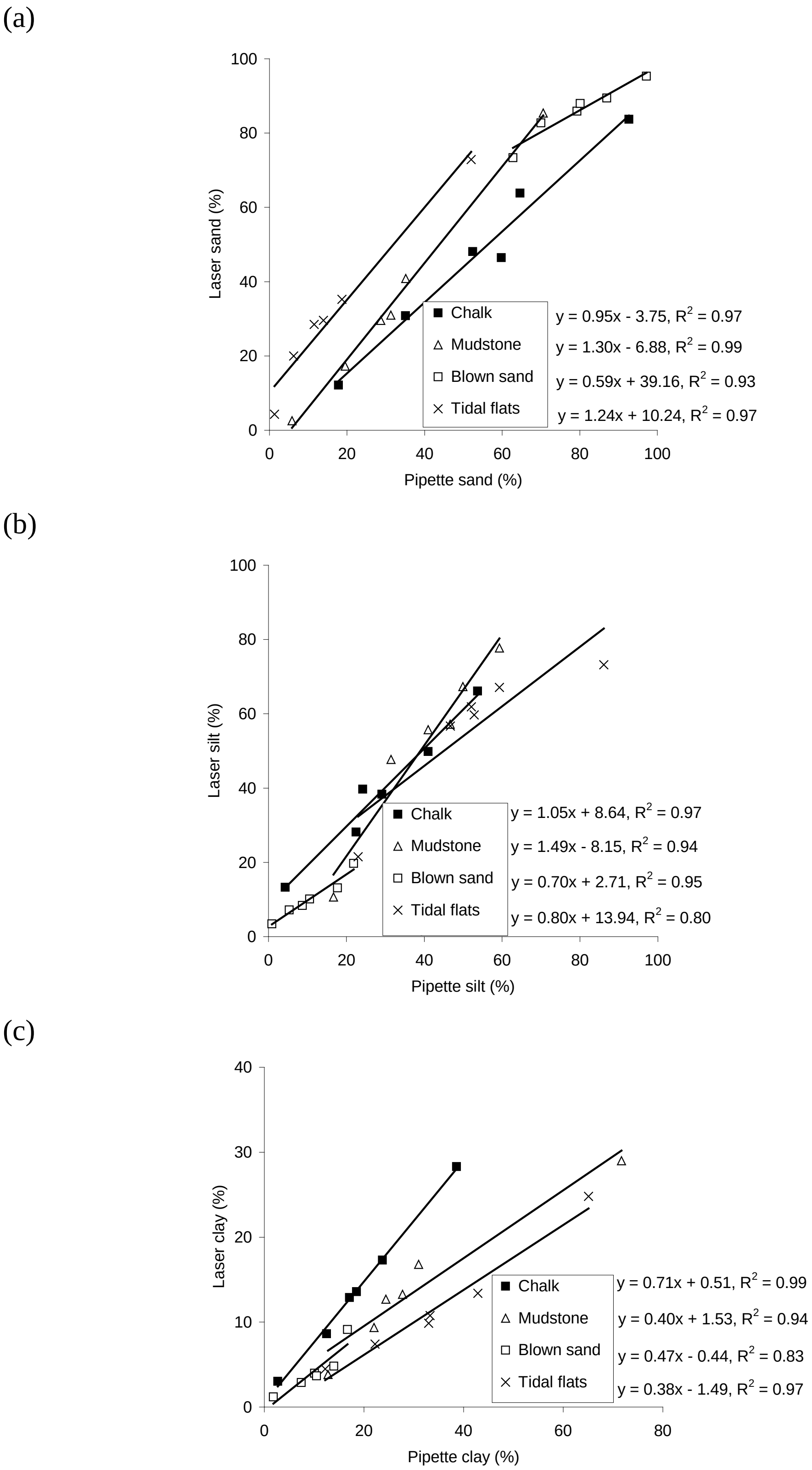
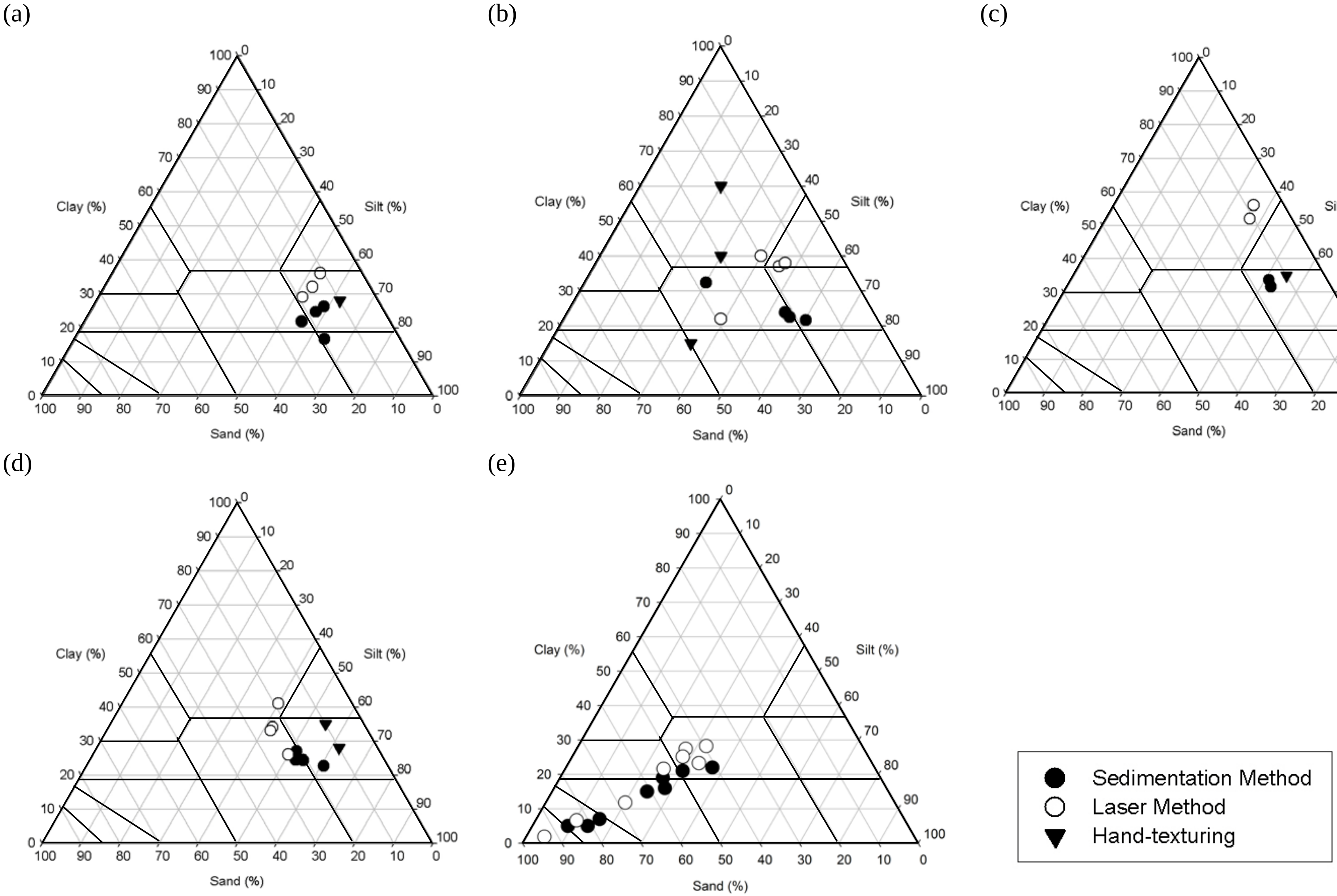


Figure 9. Regression plots showing the relationship between laser and pipette methods for the different parent materials in the NSI and GBase surveys for the (a) sand, (b) silt and (c) clay fractions.


(a)
(b)
(c)
(d)
(e)
Clay (%)
Silt (%)
Sand (%)
0
10
20
30
40
50
60
70
80
90
100
● Sedimentation Method
○ Laser Method
▼ Hand-texturing

* 2 µm cut-off used for sedimentation methods and 8 µm cutoff used for laser methods

Figure 3. Ternary diagrams showing the particle size distribution of the top-soil determined by different methods for the (a) north-facing slope, (b) plateau, (c) south-facing slope and (d) valley at the Yattendon site and for (e) other sites not on chalk.

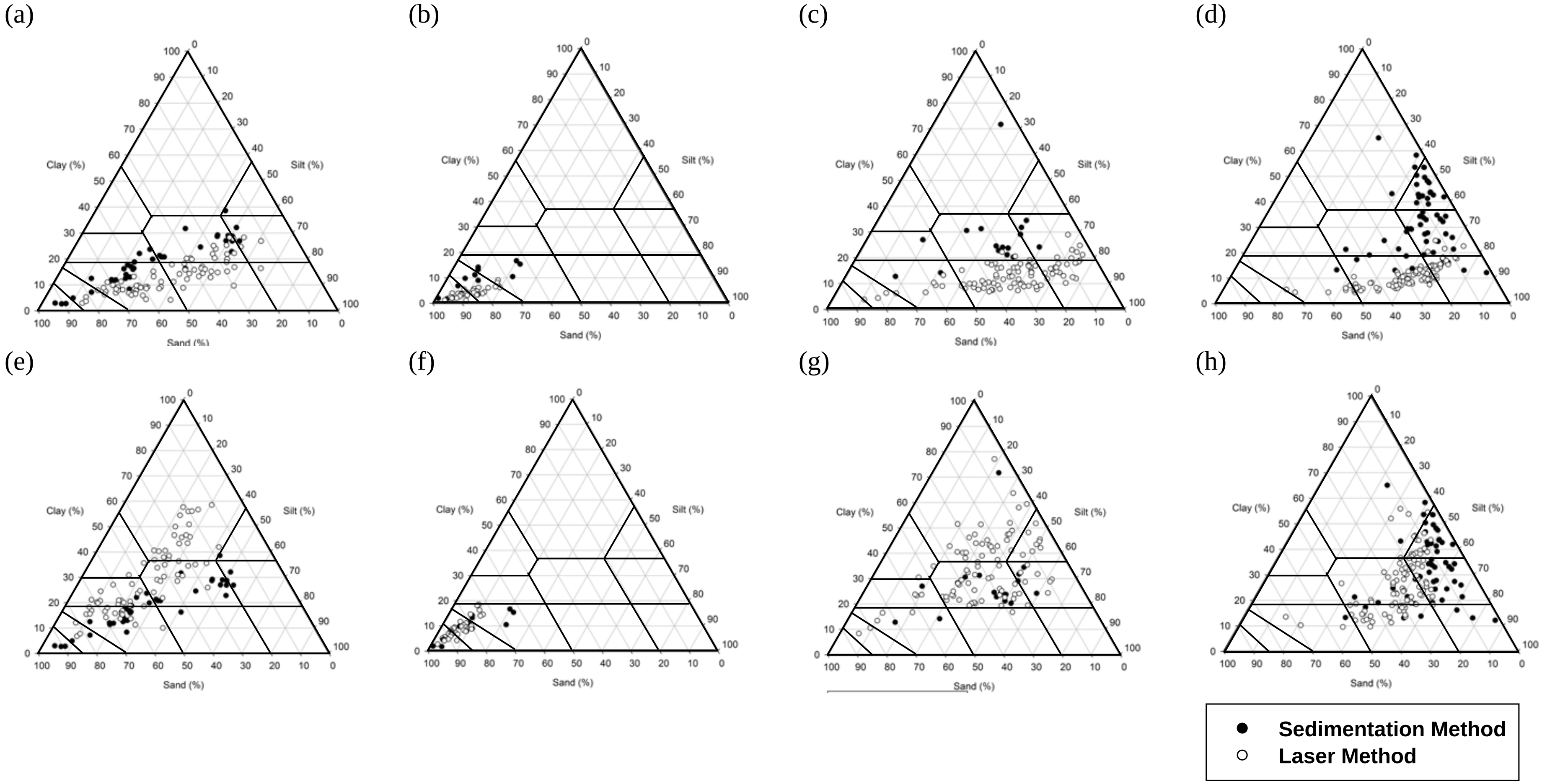


Figure 8. Ternary diagrams showing the particle size distribution of the top-soil determined by laser and sedimentation methods for the NSI and GBase soil samples developed on: (a, e) chalk, (b, f) blown sand (c, g) Mudstone, (d, h) Tidal flats using 2 μm and 8 μm cutoffs, respectively for laser methods. NSItextures © Cranfield University (National Soil Resources Institute), 2008

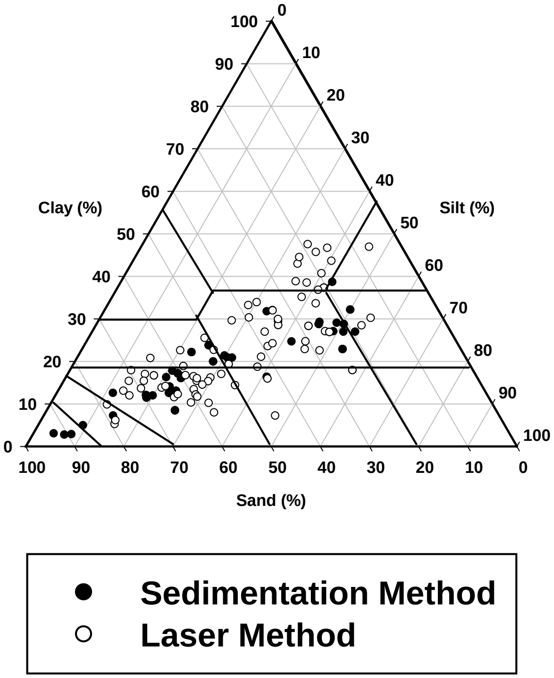


Figure 10. Ternary diagrams showing the particle size distribution of the top-soil determined by laser and sedimentation methods for the NSI and GBase soil samples developed on chalk with a 4 µm cut-off for the laser methods. NSItextures © Cranfield University (National Soil Resources Institute), 2008