

Wetting of agricultural soil measured by a simplified capillary rise technique

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Summary

We describe the use of a capillary rise method to measure the wettability of 10 samples of agricultural soil from Rothamsted long-term experimental sites. The samples have very similar clay contents, but organic carbon (C) contents range from 11.5 to 31.2 g kg⁻¹. Their wetting rates were interpreted by an improved method of data analysis, consistent with the Washburn equation, and showed an increase in the effective contact angle between the water meniscus and the soil with increasing C content. This corresponds to a decrease in wettability with increasing C content, and accords with other results reported in the literature. By contrast with water, we found that capillary rise for *n*-hexane into soil did not depend on the soil's bulk density or C content. *A priori* calculations of the expected wetting rates from fluid properties and an effective hydraulic radius estimated by other methods gave magnitudes and trends that agreed with our experimental data. The results show that estimates of effective hydraulic radius can provide a useful approximation for characterizing soil wetting, but that further modelling should be carried out.

Introduction

Water repellence of soil cannot be measured directly. Instead it must be inferred from an indirect method, commonly the sorption of water or other liquids or both into the soil matrix. A rapid assessment of the water repellence can be made from the time that it takes for a drop of water to sorb into the soil matrix. This method is imprecise but useful in the field (Letey *et al.*, 2000). Variants of it use mixtures of water and ethanol, with a corresponding range in surface tensions, to provide more quantitative data. More sophisticated approaches involve the use of mini-tension-infiltrometers, and compare the tension-regulated infiltration of water and water-ethanol mixtures (Hallett *et al.*, 2003, 2004).

Liquid repellence can be expressed in terms of the contact angle that the liquid would make with a hypothetical non-porous flat surface of the soil. A contact angle of zero occurs if the liquid wets perfectly, and a contact angle of 90° corresponds to no wetting of the surface. If the contact angle is greater than 90°, as for mercury intruding into soil, the fluid has to be forced in (Gomendy *et al.*, 1999). The capillary rise method (Ellerbrock *et al.*, 2005; Goebel *et al.*, 2005) is a convenient way to measure contact angle on small samples of soil in the laboratory. It is useful because it gives an indication of the macroscopic response of a soil to a wetting liquid. However, the fluid is wicked up into the porous matrix of the soil, so the wetting

rate and hence the inferred contact angles depend on both the surface properties of the soil and its pore structure. This effect needs to be taken into account when one interprets contact angles in the context of soil treatments. Recently, Goebel *et al.* (2004) explored the effect of soil pre-treatment, and they showed that contact angles measured on homogenized soil samples were somewhat larger than those measured with soil aggregates. They found that the soil's hydration state could influence estimated contact angles.

In this paper we describe the use of the capillary rise method to measure the wettability of soils from the long-term experiments at Rothamsted Research in the southeast of England. The soils we used are similar in their clay content but differed by a factor of nearly three in their organic carbon contents as a result of different management. The method that we propose requires a laboratory balance that can be logged several times per second, together with a specially constructed wetting chamber. We also present an improved mathematical analysis of the results. The results are compared with those from other agricultural soils.

Materials and methods

Theory of the capillary rise method for measuring contact angle

When wetting liquids enter a porous structure, capillary forces at the interface between the fluid, pore walls and displaced fluid

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(usually air or vacuum) cause the fluid to enter the pore network. The fluid must accelerate from rest against its inertia. Individual wetting events are described approximately by the Bosanquet equation (Bosanquet, 1923), which equates the inertial and viscous forces to the force due to applied pressure and the capillary force, respectively:

$$\frac{d}{dt} \left(\pi r^2 \rho x \frac{dx}{dt} \right) + 8 \pi \eta x \frac{dx}{dt} = P_e \pi r^2 + 2 \pi r \gamma \cos \theta. \quad (1)$$

Here x is the distance travelled by the liquid front in time t into a cylinder of radius r , ρ is the liquid density, η its dynamic viscosity, γ is the interfacial tension, θ is the contact angle of the fluid meniscus with the wall of the tube and P_e is the external pressure applied at the entrance of the capillary tube.

Wetting according to the Bosanquet equation causes advancing wetting tracks, often in features of small volume, which occur at very short time intervals. These tracks of wetting fluid penetrate the porous matrix well ahead of the line of the average wetting front (Schoelkopf *et al.*, 2000). At longer times, the wetting process integrates (Bodurtha *et al.*, 2005) to a behaviour described by Washburn (1921) and Lucas (1918). They obtained a time-dependency for the uptake of a wetting fluid into a cylinder by assuming that the flow was laminar and could therefore be described by both the Poiseuille and Laplace equations. The Lucas–Washburn equation is

$$x^2 = \frac{r_h \gamma t \cos \theta}{2 \eta}, \quad (2)$$

where r_h is the effective hydraulic radius and η is the dynamic viscosity. The equation assumes there is no applied pressure and there is no effect due to a hydrostatic head. Many permeation experiments show at least superficial agreement with Equation (2), with an uptake distance x approximately proportional to \sqrt{t} . The equation therefore continues to be used, despite the fact that when wetting in porous networks, r_h , θ and γ have no precise physical basis. Despite the crudity of the implicit approximations, the effective hydraulic radius can be used to provide a simple means of characterizing soil porous networks, as shown later.

For a sample of porosity ε in a sample tube of inner radius R and cross-sectional area πR^2 , the weight of water w taken up is

$$w = \varepsilon \rho \pi R^2 x, \quad (3)$$

provided the experiment is done at such short times that the hydrostatic head on the liquid is negligible compared with the capillary forces. Combining Equations (2) and (3) gives

$$w^2 = \left(\frac{\varepsilon^2 \pi^2 R^4 r_h}{2} \right) \left(\frac{\rho^2 \gamma \cos \theta}{\eta} \right) t = c K t, \quad (4)$$

where c and K are the lumped terms in the respective parentheses; c is a geometric parameter (Siebold *et al.*, 1997), whereas K depends on the characteristics of the fluid and the way in

which the fluid interacts within the soil. Taking the logarithm of the square root of Equation (4) leads to

$$\ln w = \beta \ln(c K t), \quad (5)$$

where $\beta = 0.5$.

In practice, there is a wetting jump at the moment of contact between the sample and the water, which causes a rapidly dying weight oscillation δw (Schoelkopf *et al.*, 2000). This causes an oscillation in the observed weight w_{obs} at the observed time t_{obs} , and masks the exact start time t_0 of the wetting process. The working equation becomes

$$\ln \{ w_{\text{obs}}(t_{\text{obs}} - t_0) - w(t_0) + \delta w(t_{\text{obs}} - t_0) \} = \beta \ln \{ c K (t_{\text{obs}} - t_0) \}. \quad (6)$$

Equation (6) contains two unknowns, t_0 and δw , and therefore cannot be solved. However, because the two unknowns are linked experimentally, we can find a solution by stepping through values of t_0 near the likely observed start of the wetting process, until $\beta \Rightarrow 0.5$. If, however, δw is large when t is close to, or equal to t_0 , no solution can be found; this was the case in two measurements of capillary rise reported here. Apart from those cases, it was possible to find solutions to Equation (6) for which $0.43 < \beta < 0.57$.

The solution of Equation (6) gives a series of weight and time readings for the solved value of β , so the product cK can be found for any soil n . We now compare values of $c_n K_n$ for the same sample and the two different liquids, water and n -hexane. For the same sample, the values of c will be the same as they only depend on the sample, not the liquid. So, from Equation (4) it follows that

$$\frac{c_n \cdot K_{\text{water},n}}{c_n \cdot K_{\text{hexane},n}} = \frac{K_{\text{water},n}}{K_{\text{hexane},n}} = \frac{\rho_{\text{water}}^2 \gamma_{\text{water}} \cos \theta_{\text{water},n} \eta_{\text{hexane}}}{\rho_{\text{hexane}}^2 \gamma_{\text{hexane}} \cos \theta_{\text{hexane},n} \eta_{\text{water}}}. \quad (7)$$

On the right-hand side of the equation, if we assume that n -hexane is fully wetting, $\theta_{\text{hexane}} = 0$, so the only unknown is $\theta_{\text{water},n}$, which can therefore be found.

In comparison with existing methods to make capillary rise measurements, Equation (6) is a significant advancement because it provides the means to precisely identify the start of wicking that conforms to the Lucas–Washburn equation.

Experimental technique

The rate of liquid uptake was measured on a digital microbalance, namely a Mettler Toledo XP504 balance with a precision of 0.1 mg linked to a computer. Measurements were made with Mettler's BalanceLink software, which captured 34 measurements per second via an RS232C interface. The measurements tended to be constant for groups of three or four successive readings, so in practice the reading changed every 0.1 s. We checked the sensing and equilibration rate of the balance by

gently dropping a 10-mg standard weight on to the balance. The reading changed from zero to 11.3 mg in 0.095 s, thus registering the deceleration force on the dropped weight, and then diminished to 10.2 mg, 0.28 s after the last zero weight reading before the drop. For the current dynamic wetting experiments, we corrected any constant time lag in measurement by taring the time, so we estimated any weight error to be less than 0.2 mg and any time error to be less than 0.1 s.

The soils were contained in stainless steel tubes of length 45 mm and internal diameter 21.7 ± 0.2 mm. The base of each tube was covered with gauze, which was clamped to the tube with a plastic ring clip (Figure 1). We packed the soils into the sample tube by repeated additions of volumes of 1.0 ± 0.1 cm³ of soil. We compacted each new volume by dropping a specially constructed 562-g plunger five times from the top of the sample tube. The soils had previously been air-dried and homogenized. In the context of the findings of Goebel *et al.* (2004) described above, the use of air-dry soil seemed a reasonable standard soil preparation to adopt.

Each soil tube was mounted into a wetting apparatus with a design based on that of Schoelkopf *et al.* (2000). The top of each tube had a lid attached by side screws, with a fitting that allowed it to be clamped to a slider on vertical rods, visible in Figure 1. The slider was in turn connected to a vertical screw thread that screwed through the top of the wetting chamber. When this screw was manually rotated, the sample descended towards the liquid without itself rotating. The screw thread was pitched so that one full rotation lowered the sample by 1 mm.

The wetting chamber comprised a metal box with glass front and no base, which fitted on the base of the balance without touching the balance pan. It is shown in Figure 1 with its front removed for clarity. The wetting liquid was contained in a glass dish, which stood on the balance pan. At the rear of the chamber

were three troughs, which were filled with the wetting liquid to ensure a saturated vapour and minimize evaporation from the glass dish. The wetting chamber was in turn mounted in the balance chamber. Inside the balance chamber was the bulb of a high-precision mercury thermometer, which was checked to ensure that the temperature of the experiments was $20.0 \pm 0.2^\circ\text{C}$.

We began the experiment by manually lowering the sample on to the liquid until the balance indicated a wetting jump had occurred, and then quickly giving one extra turn to lower the sample by a further 1 mm. Figure 1 shows the apparatus at the moment of the wetting jump, which has caused a ripple visible for about 0.1 s, estimated with a video camera, as the liquid jumps up to the sample. The displacement of the fluid by the descending sample, the wetting jump and ensuing ripple together result in the weight oscillations δw in Equation (6).

Description of the soils

The soil used in this experiment was collected from the two ley-arable rotation experiments at Rothamsted Research, some 40 km north of London ($51^\circ48'03''\text{N}$, $0^\circ21'10''\text{W}$). One of the original objects of these field experiments was to investigate the effect of various cropping and rotation practices on soil organic carbon. The experiments were established on these sites in 1949 on land that had a long history of permanent pasture (Highfield) or long-continued arable cropping (Fosters) (Johnston, 1972). Since the start both sites have had the same management, with cultivations, drilling and harvesting done on the same or succeeding days.

Approximately 1 kg of soil was collected from the upper 10-cm horizon of each plot and returned to the laboratory and air-dried until required. The samples were passed through a 5-mm aperture sieve, and larger pieces of organic matter such as straw

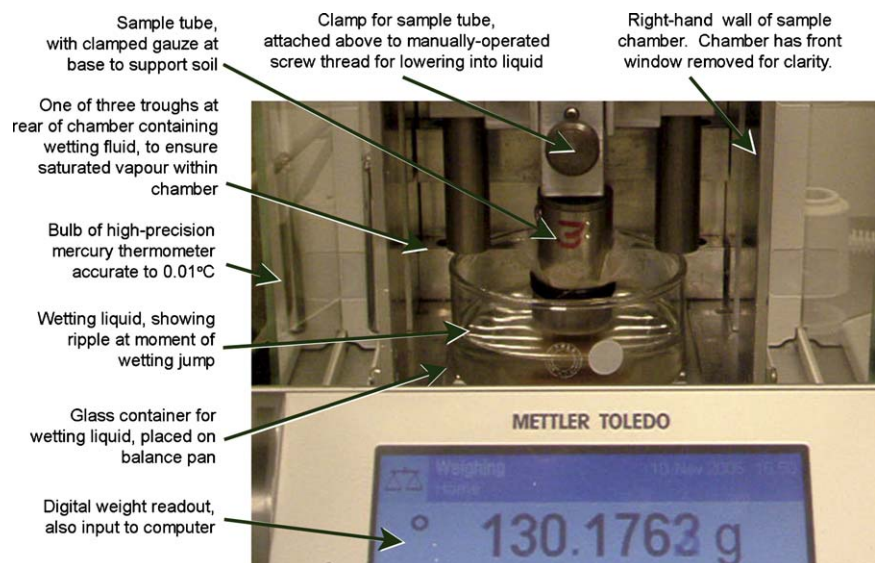


Figure 1 Sample tube, wetting chamber, and balance assembly.

and roots were discarded. A sub-sample from each treatment was crumbled by hand, and the particle-size distribution and organic carbon (British Standard Institution, 1975) was determined on it (Table 1). The C data reported in Table 1 were determined from soil collected from the upper 10 cm, whereas those reported by Johnston (1972) were collected from the upper 23 cm, so the two sets of data differ somewhat.

Results

Figure 2 shows the bulk densities of the repacked, replicated samples used for capillary rise experiments. The differences in bulk density between treatments used in the *n*-hexane and water capillary rise measurements were small and not statistically significant. Increased organic carbon (C) content (from 10 to 30 g kg⁻¹) resulted in a decrease in bulk density (from 1.55 to 1.15 g cm⁻³) of the packed soil samples.

Figure 3 shows two example results, namely slowest wetting with water, and a typical wetting result with *n*-hexane similar to the fastest wetting with water. It can be seen that there was a weight loss of the liquid in the container as it was absorbed by the soil. The saturation of the vapour inside the sample chamber ensured that none of this apparent weight loss was due to evaporation, a feature confirmed for each measurement by a recorded weight stable to ± 0.1 mg before each experiment. The solution of Equation (6) corresponds to a taring of time and weight, and a straightening of the wetting curves between the linear-gradient start and end points when plotted against \sqrt{t} , as in Figure 3. Apart from the taring, the results are as captured from the balance and show the initial weight oscillations. We chose the gradient start and end points to select the portion of the curve between the initial oscillations and the final tail-off as the sample approached full saturation or the wetting front reached a height at which drag due to its hydrostatic head became significant.

The solution of Equation (6) gives the product of the lumped constants, c and K , for water and *n*-hexane, which are plotted as a function of the C content in Figure 4. The lines fitted to these data were obtained from grouped regression. The accumulated analysis of variance from this regression showed that the source of the soil (i.e. Highfield or Fosters) was not significant ($F = 1.23$ with 1 and 39 d.f. and $P = 0.274$) beyond its effect on C content.

The cK values for *n*-hexane do not show any trend with increasing C content. By contrast, the cK estimates for water depend strongly on C content, and they are widely scattered at small C contents. Those soils with the greatest C content have the smallest values of cK , which correspond to the slowest rates of water sorption. The values of $\cos\theta$ for water were calculated with Equation (7) from the mean cK data for water and *n*-hexane of each plot, on the assumption that $\theta = 0$ for *n*-hexane. Figure 5 shows them plotted against C content. The line fitted in Figure 5 explains 55% of the variance in $\cos\theta$. When contact angle θ was regressed against C content, 47% of the variance was explained.

Discussion

Water repellence and soil organic carbon

Estimated values of $\cos\theta$ decreased as C content increased (Figure 5). These results accord with other reports in the literature described below. They are derived from Equation (7) and are based on ratios of the cK values shown in Figure 4. However, there is no evidence within the results that soil management (i.e. arable or grass) *per se* affects contact angle. Despite a reduction in density, the cK values for *n*-hexane do not alter with increasing C content. It is tempting to associate this lack of change with a lack of alteration of pore structure as C increases. However, in our study it is probable that cK values for *n*-hexane do not change with changing C content,

Table 1 Details for the soils used in this study

Treatment	Plot	Sand (0.60–2.0 mm)	Silt (20–60 μm)	Clay (< 2 μm)	Organic carbon (C)
		/g kg ⁻¹			
Highfield					
Bare fallow	PF	178	525	297	11.5
Arable	9/10	189	504	306	14.5
Ley–arable (arable)	11/12	190	496	314	16.0
Ley–arable (grass)	15/16	188	490	322	16.3
Re-seeded grass	13/14	185	503	312	24.3
Permanent grass	23/24	179	487	333	31.2
Fosters					
Arable	03/04	268	472	259	12.8
Ley–arable (arable)	01/02	277	463	260	13.5
Ley–arable (grass)	17/18	298	472	229	13.3
Re-seeded grass	07/08	264	423	314	17.7

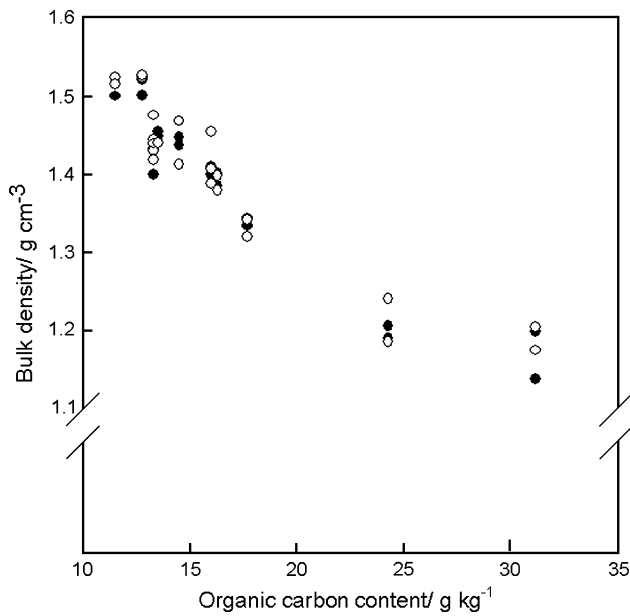


Figure 2 Bulk density of soil used in capillary rise experiments with water (○) and *n*-hexane (●).

because both the porosity and surface area increase with increasing C, and together these changes cause the effective hydraulic radius to remain approximately constant with respect to C. Hallett *et al.* (2001) observed that ethanol sorptivity provides an estimate of fluid transport properties of soil and that soil structure needs to be inferred from this. Our results illustrate the danger in making inferences about soil structure solely from the wicking of non-polar liquids such as *n*-hexane.

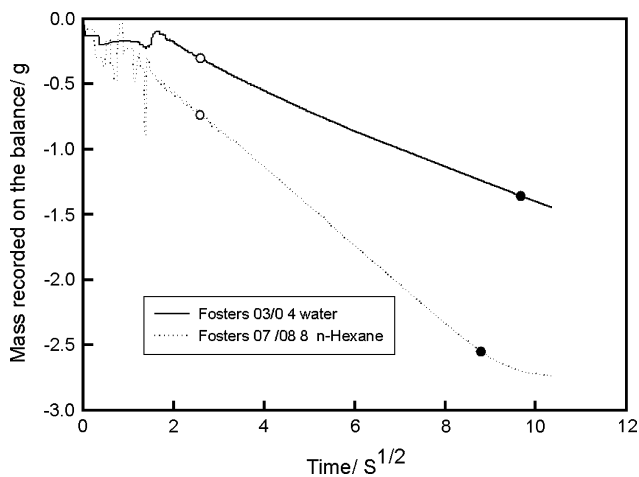


Figure 3 Examples of the weight recorded from the balance during the liquid sorption experiments plotted against the square root of time. The open symbols correspond to the start of the linear relationship between fluid wicking and the square root of time determined from the solution to Equation (6) for $\beta \Rightarrow 0.5$. The closed symbols show the end point of the linear relationship determined from inspection.

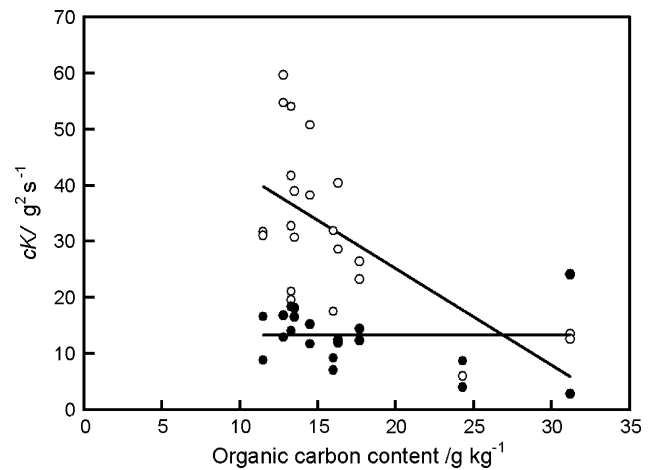


Figure 4 Values of cK plotted against soil organic carbon content for *n*-hexane (●) and water (○). The lines show the regressions of cK on C content.

Calculation of geometric and fluid parameters

In the calculation of contact angle, the unknown terms in Equation (4) are eliminated in Equation (7). However, it is instructive to investigate how well estimates of the various terms in Equation (4) can be used to replicate the experimental data in Figure 4. The soil porosity ε as a function of C content can be estimated from the soil's bulk density (Figure 2). The contact angle θ is assumed to be zero for *n*-hexane (fully wetting), and for water it is assumed to be as shown in Figure 5. Although

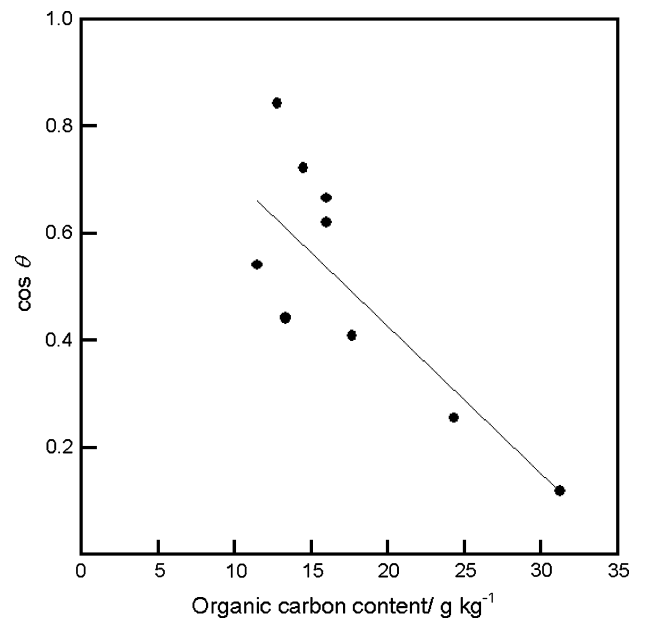


Figure 5 Values of $\cos\theta$ calculated with Equation (7) and the data in Figure 4, plotted as a function of C content. The line is the regression on $\cos\theta$ on C content. It explains 55% of the variance in $\cos\theta$.

the effective hydraulic radius r_h is ill defined, Dunstan & White (1986) proposed the following:

$$r_h = \frac{2(1 - \phi)}{\phi \rho_s A}, \quad (8)$$

where ϕ is the volume fraction of solids, ρ_s is the particle density and A is the surface area of the soil. Here the surface area, A , was estimated from the relationship between water content at a matric potential of -1.5 MPa and surface area (Petersen *et al.*, 1996). The estimates of effective hydraulic radius r_h for the soil we used are shown in Figure 6 and from these data we assume a typical and constant value for r_h of $8 \mu\text{m}$. In Figure 7 we have plotted estimates of cK against C content using Equation (4). For each C content we used measured porosities ε (calculated from the data in Figure 2), an effective hydraulic radius r_h of $8 \mu\text{m}$, the values of $\cos\theta$ for water in Figure 5 and $\cos\theta = 1$ for *n*-hexane, as well as the known fluid properties (ρ , η and γ). We note that for *n*-hexane, r_h is the only variable in Equation (4) that is not known with any certainty. So the agreement in Figure 7 between the measured and calculated values of the product cK supports the validity of Equation (8) for estimation of effective hydraulic radius of soil.

Comparison with other published data

Figure 8 shows the contact angles we measured for water where they are compared with contact angles in other studies of agricultural soils. Although the data are scattered, one can see the contact angle increases ($\cos\theta$ decreases) for all soil types with increasing C content, and that the present data follow this general trend. The different relationships between C and contact angle might

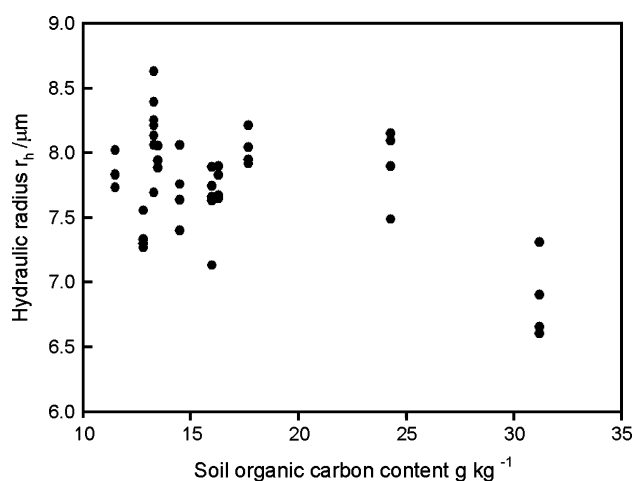


Figure 6 Effective hydraulic radius estimated from Equation (8) plotted as a function of organic carbon. We used the data in Figure 2 to apply the Dunstan & White (1986) equation for effective hydraulic radius. The surface areas of our soils were estimated from the data of Petersen *et al.* (1996) for the relationship between surface area and the water content at a matric potential of -1.5 MPa.

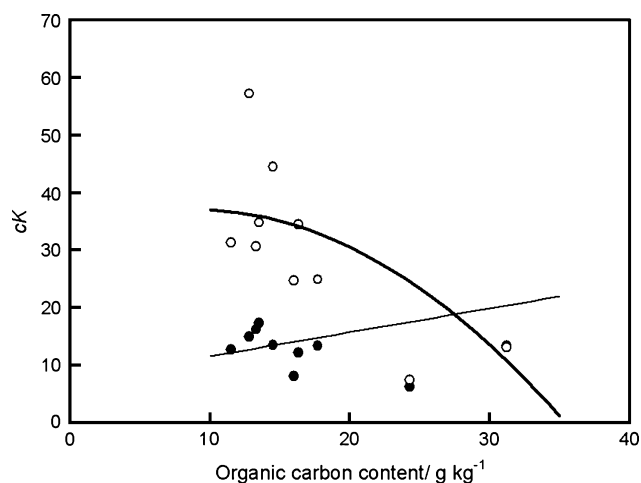


Figure 7 Predictions (shown as lines) of the product cK calculated with Equation (4) for each C content, for water (\circ) and *n*-hexane (\bullet) using our best estimates of the effective hydraulic radius (Figure 6). We assumed that r_h had a constant value of $8 \mu\text{m}$ and porosity was expressed as a function of C content using the data in Figure 2. Thus the product cK can be expressed as function of C content.

be due to different amounts and types of hydrophilic and hydrophobic functional groups within the soils for a given C content (Ellerbrock *et al.*, 2005). The extent to which soil structure can also affect wetting, and even cause super-hydrophobicity (McHale *et al.*, 2005), remains to be determined.

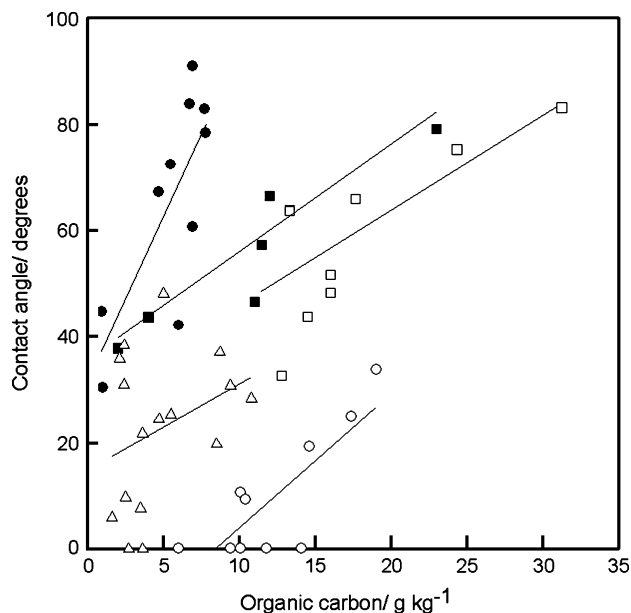


Figure 8 Contact angle data from the literature for agricultural soils. The data sets are Adenstedt from Woche *et al.* (2005) (\circ), Lietzen from Woche *et al.* (2005) (\bullet), this work (\square), Goebel *et al.* (2005) (\blacksquare), Rotthalmuenster from Woche *et al.* (2005) (\triangle). The lines show linear regressions fitted to these data.

Conclusions

A capillary rise method has been used to study samples of agricultural soil, by a fast laboratory balance and wetting chamber. We present an improved method of data analysis to identify when capillary rise data accord with the Washburn equation. For a set of soil samples with similar clay content, wettability (or contact angle) was related to organic carbon content. We found that *n*-hexane is wicked into soil at similar rates irrespective of bulk density and is thus a poor indicator of differences in soil structure. Calculations of the expected wetting rates, incorporating an effective hydraulic radius estimated from the equation of Dunstan & White (1986), gave satisfactory agreement with the experimental data.

Acknowledgements

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