

Long-term field pH manipulation influence on microbial activity, water repellency and physical properties of soil

Anastasia Fountouli^{1,2*}, Graeme I. Paton¹, Christine A. Watson², Robin L. Walker², Annette Raffan¹, Paul D. Hallett¹

¹ Institute of Biological and Environmental Sciences, University of Aberdeen, AB24 3UU, Scotland.

² Crop & Soils Systems, SRUC Aberdeen Campus, Craibstone Estate, Aberdeen AB21 9YA, Scotland.

* Corresponding author. Tel.: +33 6971727371. E-mail: ana.fountouli@gmail.com

Abstract: Studies across multiple soils find increasing pH decreases water repellency. In this study, water repellency and a range of other soil physical properties of bulk soils, aggregates and intact specimens were measured on a long-term pH field experiment on a single sandy loam soil under a ley-arable crop rotation, with soil pH adjustments occurring annually by adding FeSO_4 or CaCO_3 , to lower or raise the pH, respectively. Crop impacts were investigated by comparing 3rd year grass-white clover to spring oats, at the beginning (May) and end (September) of the growing season to allow soil structure comparisons. As in previous research, increased CO_2 microbial respiration ($p < 0.05$) was found with increasing pH along the gradient, but in this study, we found only the aggregate and soil bulk density affected by soil pH. Soil-water contact angles differed between crops ($p < 0.05$), as well as the repellency index of soil aggregates, however, there was no soil pH effect. Overall, differences in data were found to be a result of the various crops in the rotation rather than by soil pH, indicating only minor impacts on soil physical characteristics after > 55 years of chemical additions to amend soil pH.

Keywords: Soil physical behaviour; Water repellency; Soil pH; Long-term experiments; Aggregates.

INTRODUCTION

Soil pH is commonly manipulated in agriculture to improve chemical conditions for crop productivity, and this intervention is known to have major impacts on a range of soil processes (Chen et al., 2022). Whilst the impacts of soil pH on soil chemical and biological processes have been widely investigated (Msimbira and Smith, 2020; Rousk et al., 2009), dating back to early work on soil fertility (Pettinger, 1935), the impact on soil physical behaviour has been less widely researched (Diehl et al., 2010; Neina, 2019). Field studies examining the influence of soil pH on physical processes reported that it influences soil surface properties, soil texture, aggregate stability and soil water repellency (Bonanomi et al., 2024). However, these studies relied on natural pH differences driven by a combination of climate, soil mineralogy and vegetation, so it is difficult to disentangle whether it is soil pH or a confounding factor that caused these differences.

Soil water repellency (SWR) has substantial effects on crop production, soil use and land management, with SWR severity usually associated with climate and soil texture. Sándor et al. (2021) reported that climatic conditions can override or inhibit the impact of vegetation on SWR despite variations in soil pH. The same researchers also reported that finer soil textures have a lower risk of exhibiting SWR, even though this contradicts Gao et al. (2018) who demonstrated that as soil particle size increased, the soil's ability to repel water reduced.

The effect of pH on SWR is complex and varies among different climates, possibly due to temperature, soil mineralogy and soil water content (Bonanomi et al., 2024; Gao et al., 2018; Sándor et al., 2021). Decreased soil pH is generally associated with increased SWR (Lebron et al., 2012). In Spain, researchers reported that SWR was dominant in acid soils and that it increased as pH reduced (Mataix-Solera et al., 2007). With

additions of substances that shift soil pH, improved soil wettability has been observed (Fielding et al., 2022; Woudt, 1959).

Soil pH potentially controls SWR through changes in the variable soil surface charge of hydrophobic organic coatings. A study by Diehl et al. (2010) applied soil pH changes through the gas phase in controlled laboratory conditions, so there was minimal shift in the soil moisture content, resulting in a more accurate response of SWR to pH manipulations. The study reported that in some soils there was a decrease in repellency with decreasing pH, possibly due to the abundance of proton accepting surface sites with a significant amount of positive surface charge. Furthermore, there was no decrease in repellency with decreasing pH in soils with insufficient proton accepting surface sites to develop significant positive surface charge in the soil. Whilst Diehl et al. (2010) and other laboratory-based studies have shifted pH under controlled experimental conditions, it is not known whether similar results would be observed in the field where plants, microbes, and fluctuations in temperature and moisture would have an impact. Additionally, where pH impacts on SWR have been measured in the field, it has been in contrasting soils with natural pH differences (Mataix-Solera et al., 2007), but to date SWR has not been measured following long-term field pH manipulation of a single soil.

Under different pH in the same soil, plant growth and microbiology could further affect SWR through different hydrophobic compounds that coat soil particles (Hallett, 2007; Lebron et al., 2012). The origin of these coatings comes from various backgrounds, potentially including organic compounds from fungal hyphae (Rillig, 2005), humic acids (Spaccini et al., 2002), microbial biomass and decomposed organic matter (Doerr et al., 2000). Water repellency is also associated with microbial community composition (Seaton et al., 2019) and plant root exudates (Hallett et al., 2003), which again differ depending upon soil pH (Robles-Aguilar et al. 2019).

The water repellency of soil is of high importance for the future of agriculture and sustainability (DeBano, 2000; Seaton et al., 2019). Whilst high levels of water repellency can restrict plant growth, reduce water availability and contribute to soil erosion (Diehl et al., 2010), small levels of water repellency may stabilise soil physical structure against slaking (Hallett, 2007) and even lead to a lower water competition between deep-rooted and shallow-rooted plants (Zeppenfeld et al., 2017). The position in the soil profile where water repellent substances are accumulated plays a major role in water flow as it causes unsaturated preferential water flow rather than piston flow and a direct water flow between aggregates (Ellerbrock and Gerke, 2004; Rye and Smettem, 2017). Complementary to this observation, Goebel et al. (2004) reported that there was sorption of hydrophobic materials on the surface of small aggregates (< 1 mm) and congested hydrophobic soil organic matter in bigger aggregates (2–4 mm). These studies demonstrate the need for measurements at the aggregate scale when assessing SWR to understand the preferential flow of water and irregular moisture patterns in the soil.

A soil pH field experiment, that had been established for over 55 years, was used in this study to explore the impacts on SWR and underlying drivers such as microbial activity and the soil pore structure. It had pH plots ranging from pH 4.5 to pH 7.5. The main objective of this study was to compare the effect of soil pH and various crops in a rotation on soil physical and microbiological properties. The crop was either pH-sensitive spring oats, thereafter, called oats, or pH-tolerant grass. Whereas the oats were planted into tilled soils, the grass was 3 years old, so the soil was less recently disturbed. A secondary objective was to explore how the form of the soil sample affected water repellency measurements by comparing soil aggregates (Rücknagel et al., 2007), intact cores, and sieved soil. The study tests the hypothesis that decreasing soil pH will increase the water repellency of an agricultural soil, exacerbated by changes to soil physical structure and biological activity, with impact on soil physical properties and a large effect on water repellency. It is expected that any soil physical properties will be negatively affected at the 4.5 pH sub-plot (as opposed to the pH 6 and 7.5 sub-plots) due to lack of Ca^{2+} concentrations causing renewed flocculation of clay colloids that bond soil particles together and improve soil structure. The uniqueness of this long-term pH field experiment allows us to gather data regarding the adaptation of soil physical structure after decades of pH amendments, with the hope of understanding potential knock-on impacts to field pH

manipulation beyond crop nutrient availability and yield response.

MATERIAL AND METHODS

Study Site

Soil was sampled from the Woodlands long-term pH field experiment at the Craibstone Estate, SRUC, Aberdeen (57° 11' N, 2° 12' W), Scotland, UK. It started as a single plot pH gradient in 1949 and it was followed by an eight-year crop rotation x pH gradient in 1961 for the demonstration of the pH influence on crop growth (NOSCA, 1960). There does not appear to be any other long-term artificially maintained pH experiment of such a smooth gradient reported in the literature. This makes the Woodlands long-term pH field experiment an extremely valuable resource to examine the effect of soil pH on soil physical properties, plant and root performance and the activity of microorganisms in soil.

The site consisted of an eight-year rotation that included winter wheat, potatoes, spring barley, swedes, spring oats and a three-year grass-clover ley as seen in Figure 1. Every crop was grown annually and as the rotation sequence advanced, the location of each crop inside the experiment shifted to the right. Annual soil tests were performed, usually in February (before ploughing takes place) and the pH gradient was maintained by additions of FeSO_4 and CaCO_3 to lower or raise the pH, respectively. The pH gradient went from 4.5 to 7.5 with a 0.5-unit increment.

The soil in this area is a freely draining Humic Entic Podzol (Baxter, 2007) with sandy loam texture consisting of 68% sand, 25% silt and 7% clay, organic matter of approximately 5 g 100 g⁻¹ and a natural pH value of 5.5 (Schmidt et al., 2004). The climate class of the region is Cfb, according to the Köppen-Geiger climate classification (Kottek et al., 2006).

Yield variations in response to the pH treatment have been reported through the years (per comms. Robin Walker, SRUC). Each plot within the rotation was divided into seven smaller plots (5 m wide × 6 m long, = 30 m²), thereafter called pH sub-plots. The experimental design was not replicated, so each crop is represented only once in the field although all individual treatments (crop x pH) are present in every year. The value of the long-term pH manipulation at this site outweighs this limitation in experimental design, and the plots have been used extensively for high impact research (Bartram et al., 2014; Herold et al., 2012; Nicol et al., 2008).

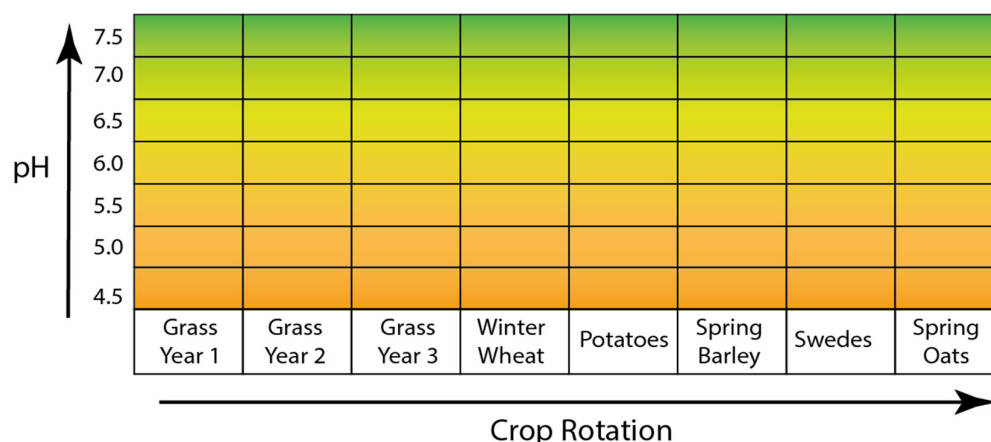


Fig. 1. Plan of the Woodlands long-term pH field experiment rotation in 2013.

Yield variations in response to the pH treatment have been reported through the years (per comms. Robin Walker, SRUC). In 2014, oats had the highest yields at pH 5.5 (6.84 t ha⁻¹ dry matter), but it dropped by 15% at pH 4.5 and by 95% at pH 7.5, demonstrating pH sensitivity. Grass-clover had the highest yields at pH 6 (7.96 t ha⁻¹ dry matter), but smaller drops of <15% at pH 4.5 or pH 7.5. For our study, samples were collected from the extreme (pH 4.5 and 7.5) and the middle (pH 6) sub-plots under either: (i) 3rd year grass-clover that is a mixture of perennial ryegrass (*Lolium perenne* L. var. mixture Fornax, Elgon and Cooper) and white clover (*Trifolium repens* L. var. mixture Alice and Grasslands Demand), or (ii) spring oats (*Avena sativa* L. var. Firth) that followed swedes (*Brassica napus* var. napobrassica). For simplicity, these crop treatments are referred to as grass and oats from herein. There were two different sampling periods, the first samples were taken before summer (May 2014), a few weeks after the oats were sown and the soil structure of this plot had, therefore, recently been disturbed by tillage. The second sampling was carried out at the end of summer (September 2014), after the oats were harvested and, therefore, the assumption was that the soil structure would be more stable after the crop season. Wheelways from the harvester and other machinery were avoided.

In both sampling periods, the grass plot was undisturbed and had been in place for 3 years, thereby contrasting the oats plot in terms of disturbance by tillage. In each pH sub-plot, soil from the top 5 cm of mineral soil was collected from 9 randomly selected locations. To assess potential artefacts imposed by the sampling method, and to measure structural impacts on soil physical properties, three different samples were collected; intact samples with cores, aggregates, and loose soil that was subsequently passed through a 2 mm sieve. The intact soil cores were 60 mm diameter x 50 mm height and taken using an auger to minimise horizontal movement and damage to the pore structure. Soil samples were transported to the laboratory (14–18 °C) and were stored at 4 °C, with all microbial analyses performed within two weeks. The following soil and site characteristics were determined for each sample and used in the subsequent statistical analyses: pH_(water), gravimetric soil moisture content, bulk density, and aggregate density to understand density on different soil size levels, aggregate sorptivity, soil water contact angles, and a range of biological measurements as described below. Details of the soil and site characteristics, the methods used to determine these characteristics, and each of the samples included in this study are described later. The pH, water content and carbon (C) of the different pH plots are shown in Table 1. Soil C was measured with a Carlo Erba Instruments (Wigan, UK), NA 2500 Elemental

Micro Analysis, with soil treated with 0.1 M HCl to remove inorganic C.

Soil pH

Soil pH was determined on air-dried loose soil samples in water at a soil: deionised water ratio of 1:2.5 w/w according to Rousk et al. (2009). The pH was determined with a combined glass/reference (ORION Star 211) pH meter electrode. For the calibration of the instrument commercial pH buffers (pH = 4.0 and 7.0, Fischer Scientific, UK) were used and the calibration was checked once every 15 samples.

Soil microbial biomass

Soil microbial biomass C (SMBC) was determined on loose soil samples using the fumigation-extraction method (Brookes et al. 1985). Soil samples (5 g at 55% Water Holding Capacity) were fumigated with ethanol-free CHCl₃ for 24 h at 18 °C ambient temperature. Fumigated and non-fumigated soils were extracted with 0.5 M K₂SO₄ for 30 minutes (1:5 soil: extractant ratio w/v) on an orbital shaker. Microbial C in filtered extracts (Whatman No. 1) was determined by an aqueous analysis system, LABTOC 5000 analyser equipped with its own autosampler (PPM Ltd, Kent). The microbial C flush was obtained as the difference between extractable C from the fumigated and non-fumigated samples and converted to SMBC using a k_{EC} factor of 0.45 (modified from Sparling and West, 1990).

Soil microbial respiration

Soil respiration was determined by an incubation method according to Paton et al. (2006). Field moist loose soil samples (1 g) were placed in sealed plastic vacuettes of 9 ml capacity. The vacuettes were incubated for 24 hours at ambient temperature (18 °C) and the headspace analysed for CO₂ concentrations using a gas chromatograph (Chrompak 9001, Middleberg, The Netherlands) with an attached methanizer and flame ionisation detector (FID). The gas chromatograph was calibrated with certified gas standards (Linde Gases, Aberdeen). A sample of the headspace was removed from the vial using a syringe and injected into the GC flow injection loop (250 µL) system with a nitrogen carrier gas (20 ml min⁻¹) onto an 80/100 mesh Poropak Q column (2 m × 1 / 8" OD × 2 mm). The oven temperature remained constant at 100 °C and the FID temperature was set at 250 °C. Respiration values were expressed as mg CO₂ g soil⁻¹ day⁻¹ following subtraction of a blank vial containing atmospheric CO₂ only, incubated for the same time.

Table 1. Target and actual pH_(water), soil water content (SWC) for May and September, and soil organic carbon (C) results of the target treatments. Grass refers to 3rd year grass-white clover and oats refers to undersown spring oats.

Crop	Target pH	Actual pH	SWC- May (g g ⁻¹)	SWC- September (g g ⁻¹)	C (g 100 g ⁻¹)
grass	4.5	3.94 ± 0.04 ^a	0.38 ± 0.001 ^a	0.37 ± 0.01 ^a	6.08 ^a
grass	6	5.19 ± 0.08 ^b	0.36 ± 0.001 ^a	0.39 ± 0.01 ^a	6.08 ^a
grass	7.5	6.75 ± 0.03 ^c	0.36 ± 0.001 ^a	0.40 ± 0.02 ^a	5.83 ^b
oats	4.5	3.94 ± 0.08 ^a	0.40 ± 0.001 ^a	0.40 ± 0.02 ^a	5.58 ^a
oats	6	5.15 ± 0.05 ^b	0.42 ± 0.001 ^a	0.37 ± 0.01 ^a	5.78 ^a
oats	7.5	6.68 ± 0.01 ^c	0.44 ± 0.002 ^a	0.39 ± 0.02 ^a	6.14 ^b

Values are presented as mean (n = 9) ± standard error (SE). Statistical significance ($p \leq 0.05$) between the different pH sub-plots and for each soil separately is shown with superscript letters. Pearson correlation showed a relationship between pH and CEC for grass ($p = 0.007$) and for spring oats ($p = 0.001$).

Soil water repellency

All water repellency measurements were conducted on soil that had been oven-dried (40 °C for 48 hours), following the approach of Hallett and Young (1999). Soil was cooled in a desiccator to minimise humidity changes.

Sieved soil - Soil-water contact angles by capillary rise

For this measurement, 5 g of sieved soil was placed in a powder wetting setup attached to a One Attension Force Tensiometer (Sigma 700/701, Stockholm, Sweden). The powder wetting setup consisted of a metal tube with a porous base lined with filter paper and a spring plunger that packs the specimen. The base of the set up was slowly immersed into a beaker filled with liquid to 1 mm depth. The infiltration rate (slope) was calculated via the Lucas–Washburn equation based on the capillarity of the liquid. The measurement was conducted for both water and ethanol so that the contact angle could be determined. Ethanol has a much lower surface energy than water and so it behaves in water repellent soil in a similar way to that in which water would behave in the same soil if the soil were wettable. Since SWR affects infiltration of water but not ethanol in the soil, comparisons of their infiltration rates are used to characterize SWR. Using the capillary rise data, the sorptivities of ethanol (S_e) and water (S_w) were calculated using Philip's equation.

Aggregates and Soil Cores- Sorptivity

Aggregate sorptivity was measured with a miniature infiltrometer device, with water or ethanol used as the wetting liquid so that water repellency could be determined (Hallett and Young, 1999). Soil aggregates of no larger than 2 cm diameter collected from the surface (0–3 cm) of the pH sub-plots were air-dried. The liquid infiltrated into each aggregate from a 2 mm diameter infiltrometer tip that was set at 5 mm above the liquid reservoir.

Water repellency index

The water repellency index (R) of both soil cores and the aggregates was determined from S_w and S_e by:

$$R = 1.95(S_e/S_w). \quad (1)$$

The constant 1.95 accounts for differences in viscosity and surface tension between water and ethanol. A value of $R = 1$ indicates that a soil has no water repellency. Whereas S_w is affected by soil pore structure and water repellency, S_e is affected only by pore structure which allows for determination of the intrinsic sorptivity (Hallett and Young, 1999). Thus, R is the reduction in water sorptivity caused by hydrophobic coatings covering soil pore walls.

Bulk and aggregate density

Bulk density was determined from the dry mass and volume of the soil cores. Oven-dried (40 °C for 48 hours) aggregates of ≤ 2 cm size (0.29 g being the smallest aggregate measured and 0.56 g the biggest) were used to measure aggregate density according to the Archimedes principal (Pansu and Gautheyrou, 2006). Thread was attached around each aggregate and the weight of the aggregate plus thread was noted. Paraffin wax beans were melted in a beaker at 57 °C temperature and each

aggregate was quickly dipped into the wax and removed immediately. The procedure was repeated several times until the aggregate was entirely sealed and covered with wax. A beaker containing water was placed on a balance and the weight was noted. Each waxed aggregate was carefully introduced to the beaker, avoiding contact with the side of the beaker, and the weight difference was recorded. The density of wax coating was 0.9 g cm⁻³ and the aggregate volume was calculated by subtracting the volume of wax coating from the total volume. The final density was calculated as mass divided by volume of soil.

Statistical analysis

All data were found to be normally distributed between the different pH sub-plots. Results were analysed using ANOVA with SPSS 22 statistical software. For all measurements, unless stated otherwise, there were 9 independent replicates used from each pH sub-plot. For each sampling period and crop, results were calculated separately between the different pH sub-plots. For multiple comparisons between means of the pH sub-plots for each sampling period and crop separately, the Fisher's protected least significant difference (LSD) intervals were used. Pearson's correlation was also used. p -values are not mentioned if not significant.

RESULTS

Summary statistics of all measurements are provided in Table 2.

Soil microbial respiration and biomass

The basal CO₂ respiration (Figure 2) was affected by the soil pH treatments ($p < 0.05$), although on closer examination May grass and May oats at pH 7.5 were the most different. The highest values were observed in soil samples collected in May at the pH 7.5 sub-plot. Soils from under grass respired 3.52 mg CO₂ g⁻¹ day⁻¹, which was double in comparison to the 1.64 CO₂ g⁻¹ day⁻¹ respiration for soils under oats at the same pH. These data indicate that the microbial communities of the soils were more active in neutral pH conditions than the acidic pHs, regardless of the crop in the field. Soils from the pH 4.5 and 6 sub-plots had similar CO₂ respiration for both crops and between sampling periods, apart from oats in September for pH 6, where there was a significant decrease ($p < 0.05$). This was reflected in the sampling period effect for both crops ($p < 0.05$). Furthermore, at the pH 7.5 sub-plot, microbial respiration was greater for samples taken in May than in September, particularly under grass. There was a sampling period effect also for both crops ($p < 0.05$).

Soil microbial biomass C (Figure 3), on the other hand, was not affected by pH, but was affected by crop and sampling period ($p < 0.05$). Apart from soil sampled at the pH 4.5 sub-plot, there was less of an impact on SMBC between sampling periods for the perennial grass in its third year of growth than for annual oats. These results indicate that SMBC increased in the presence of vegetation in the 3rd year grass treatment. In the time between planting and harvesting of the oats, SMBC had decreased markedly.

pH and crop effect on the soil physical properties

The average field soil water content at sample collection varied ($p < 0.05$) between crops (0.36 g g⁻¹ for grass and 0.42 g g⁻¹ for oats in May, and 0.38 g g⁻¹ for grass and 0.39 g g⁻¹ for oats in September) but did not vary in between pH sub-plots or

sampling periods, so no effect due to soil pH manipulations or the different sampling period was observed (Table 1). Dry bulk density of soil cores (Figure 4) varied along the pH sub-plots ($p < 0.05$) and with time ($p < 0.05$). The aggregate density (Figure 5) also varied along the pH sub-plots ($p < 0.05$) and with time, but also between sampling periods (Table 2).

Contact angle measurements in soil samples collected in May for both crops between the different pH sub-plots, had higher average values than those of soil samples collected in September

(Figure 6). As the contact angle measurements for soils were $< 90^\circ$, the soils between the different pH sub-plots, for both crops and sampling periods were sub-critically water repellent. There were no significant differences in the contact angle values between the pH sub-plots, however, differences were found among the crops ($p < 0.05$). The most hydrophilic soil was sampled from the pH 6 grass sub-plot in September (52°) and the most hydrophobic soil was sampled from the pH 7.5 oat sub-plots in May (77°). For soil aggregates, water sorptivity varied

Table 2. Statistical analysis of pH $\times$ crop $\times$ sampling period interactions. Values reported are the F-values from the ANOVA and asterisk indicating the level of significance. NS not significant at the $p = 0.05$ level and * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$.

Property	pH	crop	sampling period	pH $\times$ crop	pH $\times$ sampling period	pH $\times$ crop $\times$ sampling period
CO ₂ soil respiration	11.58***	NS	8.16**	NS	5.11**	3.45*
Microbial biomass C	NS	7.68**	NS	NS	NS	NS
Dry bulk density	3.94*	NS	29.07***	NS	NS	4.04*
Aggregate density	257.44***	441.63***	29.20***	941.61***	227.48***	26.58***
Soil contact angle	NS	6.45*	NS	NS	NS	NS
Aggregate water sorptivity	NS	NS	NS	NS	NS	3.42*
Aggregate ethanol sorptivity	NS	151.64***	NS	3.44*	NS	3.50*
Aggregate repellency index	NS	28.31***	NS	NS	NS	NS
Soil repellency index	NS	NS	10.90**	NS	NS	NS
Soil water content	NS	11.55**	NS	NS	NS	4.02*

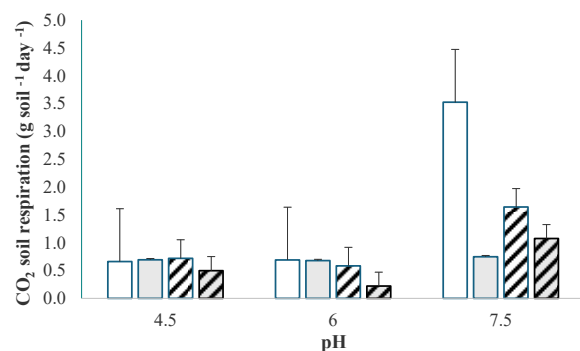


Fig. 2. CO₂ soil respiration as affected by field pH treatment for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). Statistical significance ($p \leq 0.05$) between the different pH sub-plots for each soil separately is shown in Table 2. Grass refers to 3rd year grass-white clover and oats refers to undersown spring oats.

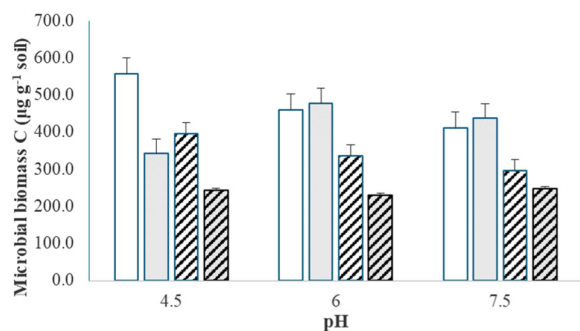


Fig. 3. Microbial biomass C as affected by field pH treatment for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

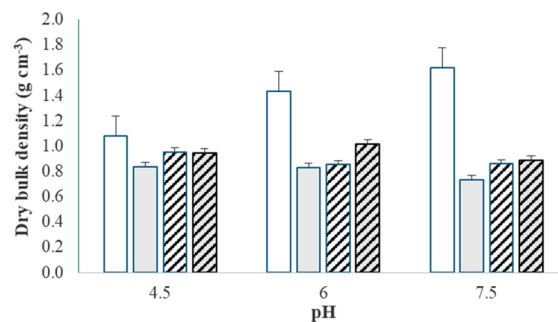


Fig. 4. Dry bulk density at different soil pH for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). Statistical significance ($p \leq 0.05$) between the different pH sub-plots for each soil separately is shown in Table 2. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

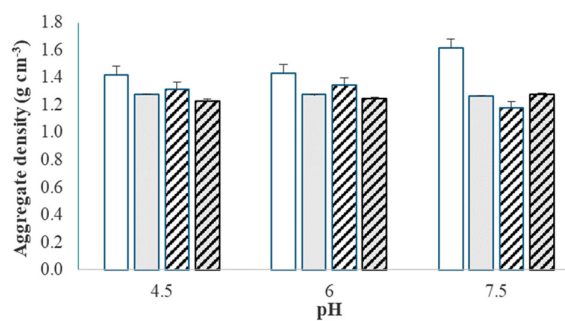


Fig. 5. Aggregate density measurements at different pH for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). Statistical significance ($p \leq 0.05$) between the different pH sub-plots for each soil separately is shown in Table 2. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

between different crops ($p < 0.05$) but not between different pH treatments (Figure 7). Aggregate water sorptivity of soil under oats during May was less at the pH 4.5 sub-plot and increased at the pH 6 and 7.5 sub-plots. However, following harvest in September, an opposite trend was observed for soil sampled from the same crop plot with the greatest aggregate water sorptivity values reported at the pH 4.5 sub-plot. At the pH 6 sub-plot, values were similar for soil sampled from both crop plots and sampling periods. The aggregate ethanol sorptivity (Figure 8) was almost two times greater for soil under grass compared to oats, but it was not affected by soil pH or the sampling season. The largest values were observed for the pH 6 grass sub-plot in May and September, 5.24 $\text{mm s}^{-1/2}$ and 6.59 $\text{mm s}^{-1/2}$ respectively. The smallest values were observed at pH 6 sub-plot for oats in May (2.02 $\text{mm s}^{-1/2}$) and at pH 4.5 for oats in September (2.05 $\text{mm s}^{-1/2}$).

Soil cores had a greater R than aggregates (Figure 9, 10), particularly from soil under the grass plot, which had a greater R than aggregates collected in the oat plot. Soil pH had no effect on the aggregate and soil core R for samples collected from the different pH sub-plots, but there was a seasonal effect ($p < 0.05$).

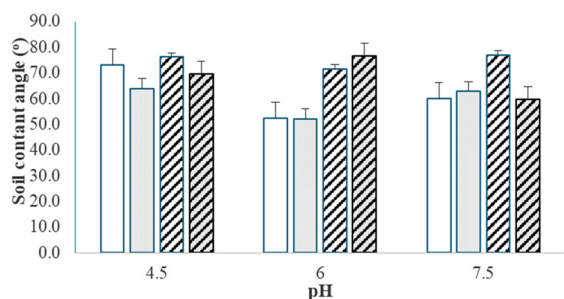


Fig. 6. Soil contact angles plotted against the pH gradient oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

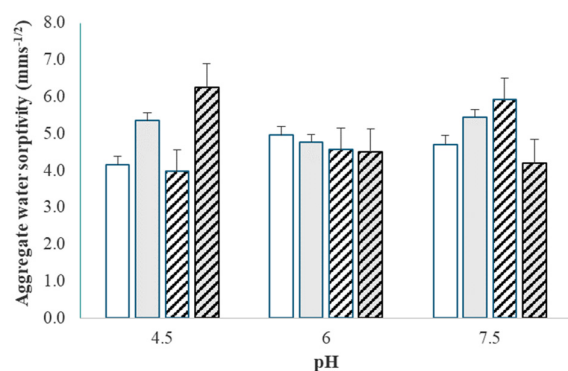


Fig. 7. Aggregate water sorptivity plotted against the pH gradient for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

DISCUSSION

We observed minimal difference in soil physical properties in a controlled ley-arable rotation field experiment that had

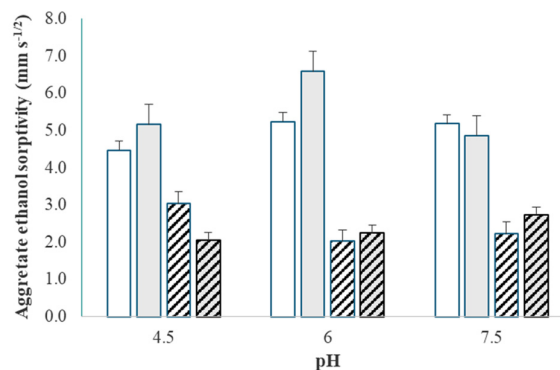


Fig. 8. Aggregate ethanol sorptivity plotted against the pH gradient for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

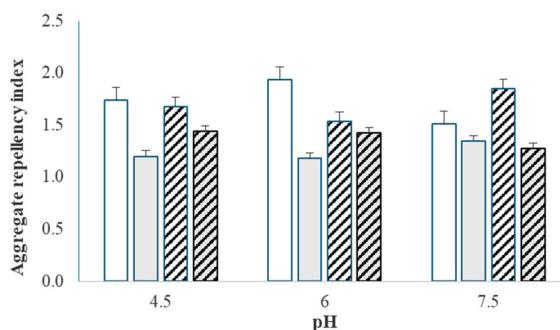


Fig. 9. Aggregate repellency index (R) plotted against the pH gradient for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

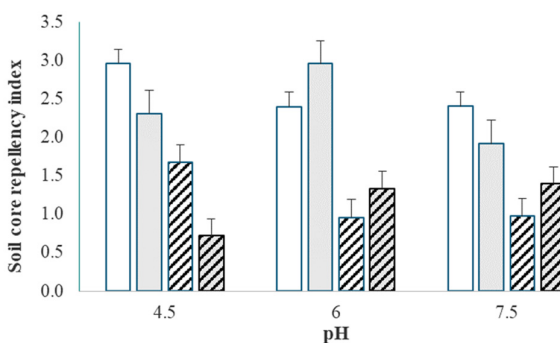


Fig. 10. Soil core repellency index (R) plotted against the pH gradient for oats (no fill) and grass (diagonal fill) sampled just after tillage in May (white) and after harvest in September (grey). Values are mean ($n = 9$) $\pm$ standard error (SE). No significant differences were found between the different pH sub-plots. Grass refers to 3rd year grass-white clover and oats refers to undersown oats.

received pH amendments for over 55 years. Soil pH affected only soil respiration and the soil density. Any other differences found were a result of the different crops in the rotation (oats and 3rd year grass) rather than the pH amendments, demonstrating the importance of plants to soil processes.

In addition, we found that different sampling periods influenced the physical properties of the soil, possibly due to soil structure disturbance in May in the oats plot followed by physical restructuring of the soil by the developing crop roots up until the time of harvest in September. We assumed that spatial variability would have minimal influence on the results since there was minimal variation in soil texture and the entire field was underlain by an indurated soil layer which lies 35–40 cm below the surface.

Soil microbial activity and soil pH

It appears that liming improved the soil's microbial activity, as basal CO₂ respiration was particularly influenced by the pH amendments, especially in soil sampled from the pH 7.5 sub-plot that received liming to increase its pH to this level. This finding is in line with those of Fielding et al. (2022), who demonstrated that adding lime to the soil raises its basal respiration.

SMBC was influenced by the different crops. This influence could be related to differences in C input by preceding and growing crops in the rotation, and their response to soil pH. Swedes preceding the spring oat crops sampled, had the greatest yields at pH 7 of 9.86 t ha⁻¹ dry matter (DM). Under grass, pH 6 had the greatest dry matter production at 7.96 t ha⁻¹ DM. In the spring oat plots, the greatest grain yield was observed to be from the pH 5.5 sub-plot and found to be 6.84 t/ha DM, with the highest oat straw yield being 4.33 t ha⁻¹ DM from the pH 6.5 sub-plot (pers. comm. Walker 2014).

Plant residue incorporation in the field increases the levels of the primary forms of organic C and nutrients used by the microbial biomass, by increasing the amount of organic matter available to soil microorganisms (Kemmitt et al., 2006). We suggest that soils at the pH 7.5 sub-plots exhibited increased basal respiration during May due to the perennial vegetation covering the grass plot, and the high residual fertility caused by the incorporation of swede residues in the spring oat plots during ploughing. From the yield data, it was clear that for the oats plot, the greatest plant residue input from the previous cultivation was around the pH 7 sub-plot, which would explain an increase in the soil respiration for oats in May. These findings not only confirm previous studies suggesting that microbial activity levels are linked to vegetation abundance (Hernández-Cáceres et al., 2022), but also suggest that incorporating residue of preceding crops in a rotation, just before sowing, acts as an additional driver of microbial activity stimulation and, thus, nutrient cycling enhancement.

Microbial communities have been shown to differ with pH at the Craibstone pH experiment that we studied (Herold et al., 2012). Rousk et al. (2009) examined soils from the Hoosfield acid strip (Rothamsted Research, Harpenden, UK) and reported a one-third decrease in the basal respiration, in the pH interval of 8.3 to 4.5 due to fungal and bacterial functional redundancy. They found a five-fold increase in fungal growth between these pH plots, while bacterial growth decreased more than five-fold. Similarly, when bacterial and fungal responses to N fertilisation were examined at the 150-year-old 'Park Grass' experiment (Rothamsted Research, Harpenden, UK), Rousk et al. (2011) found that the ratio of fungal to bacterial growth increased up to 30-fold between pH 8.3 to pH 4.5, from about 0.15 to about 5. Previous studies using soil from the pH plots we examined found the fungi to bacteria ratio to increase with decreasing pH (Nicol et al., 2008). Our data between pH 7.5 and pH 4.5 indicate a one-fourth reduction in the basal respiration in grass soil samples collected in May and 50% reduction in the basal respiration in oats soil samples collected at the same time, supporting these studies.

Impact of soil pH shifts on soil and aggregate density

With yield variations across the pH gradient and soil structure disturbance in the oats plot in May followed by crop harvest at the end of summer, we expected to see major differences in the soil densities of both crops. However, even though soil pH affected both the bulk and the aggregate density, we did not notice any major fluctuations in either of them.

We found the aggregate density to be significantly affected by soil pH, crop and sampling period and to be generally higher than the density on all pH sub-plots with samples from grass collected in May displaying the highest values on both bulk soil and on aggregate level.

When comparing the sampling periods, both bulk and aggregate density dropped in soil sampled from grass, however, values did not change in the oats soil. Soil bulk density is higher under pasture (Abindaw et al., 2023) but in our study, difference in densities in the grass plot could be attributed to changes in soil organic carbon and microbial activity as when soil water content is high enough and temperatures rise, earthworm activity, abundance, and biomass tend to increase affecting soil structure and aggregation (Singh et al., 2020). Gong et al. (2019) also reported that "earthworms increase soil macro-aggregation, thereby changing the composition of bacterial and fungal communities" as they demonstrated that earthworms alter the microbial community in soil through increasing the soil aggregation and the formation of macro-aggregates (>2 mm). The grass plot remained undisturbed, unlike the oats plot, so this could be the reason for displaying a slightly higher soil bulk and aggregate density. We expected soil bulk density to increase in the oat plot in September as we assumed the crop would improve the soil's structure but this was not the case. This could be attributed to the sandy loam texture of the soil.

Impact of soil pH shifts on water repellency

Biology and physics are intertwined processes in soils (Hallett et al., 2013), as are pH influences on chemical processes that could alter organic matter/mineral complexes, inter-particle bonding and other drivers of soil structure formation. Lime addition to soil increases the available negative surface charges, allowing ions to be attracted to these sites by electrostatic attraction or adsorption forming chemical bonds (Ameyu, 2019). Alkaline cations that are potentially attracted to these sites include Ca, Mg and K that increase crop production, microbial activity and, thus, soil structure.

Despite many investigations reporting a large influence of pH on SWR (DeBano, 2000; Doerr et al., 2000), our research found no difference between the different pH sub-plots, demonstrating that the years of pH manipulations in the soil, surprisingly, had no impact on the SWR of the soil studied. Pearson's correlation between the soil contact angles data and the soil *R* showed a strong positive relationship ($r = 0.647$, $p < 0.001$) at the 0.01 level, as soils with high contact angles would be expected to have a high soil water *R*. The subcritical water repellency observed would likely have larger scale impacts to water flow and retention. Water in the soil would first saturate the big pores and then the small pores, potentially due to hydrophobic coatings covering the soil aggregates that lead water to fill the big pore spaces in the soil profile. Smaller pores would saturate after the soil reached a certain moisture level. In our study, aggregates had a greater *R* especially in the grass plots than the oats when compared to the soil *R*. Reduction of SWR has been observed in soils following soil structure disturbance as the abrasion of particles can remove hydrophobic coatings from soil surfaces

and increase the hydrophilic soil surface area (Buczko et al. 2006). Soil was sieved prior to the contact angle measurement, which could explain the lower R compared to the intact aggregate soil samples. Additionally, R of grass aggregates was greater than oat aggregates, likely because the grass plot remained undisturbed by cultivation for three years. On the sieved soil, abrasion may have decreased the differences observed between crops.

It is difficult to compare our results to existing field experiments that have measured natural pH impacts on water repellency as these studies compared a range of soils of different origin. From laboratory manipulation of soil pH, Diehl (2010) showed a large influence of pH on water repellency, so we had anticipated similar impacts would be observed in the field. In fact, soil pH driven physical effects were postulated to be exacerbated under field conditions due to plant growth and shifts in microbial community structure that would favour fungal importance at lower pH (Rousk et al., 2009; Young et al., 2012), and thus enhance soil aggregate stability through the fungal network. Fungal domination in acid soil conditions is attributed to either Al toxicity or lack of substrates due to weak plant growth (Aguilera et al., 2015). Given that fungi are major drivers in the development of SWR (Young et al., 2012), it was surprising that we found no difference. A possible explanation for the lack of pH impact on water repellency and more specifically increase in repellency at the 4.5 pH sub-plots would be, as suggested by Diehl et al. (2010), that the soil had an insufficient number of proton accepting surface sites to develop significant positive surface charge in the soil that would induce repellency.

Influence of plants on the soil properties

With previous crop data and other studies finding oats to be very responsive to pH change (Holland et al., 2019), it was expected that pH would affect the physical properties of soil under this plot. Decreased biomass production due to pH shifts could decrease residues incorporated back into soil and the action of plant roots in restructuring soils. Moreover, greater biological inputs, such as root biomass, would be expected at the pH 5.5–6.5 sub-plots when growing conditions are more favourable for oats. Oat yield was suppressed at the pH 7.5 sub-plot, while grass/clover was relatively insensitive to pH with minimal yield variation between the different pH sub-plots (Craibstone data). Grass is a relatively pH-insensitive crop and the cultivation of it seems to reset any physical impacts that would be expected by the pH shifts. In the pH 4.5 sub-plot under oats, greater water repellency was expected due to the lower amounts of crop residue and likely more fungi (Rousk et al., 2009).

In the presence of a rotation including a grass/clover ley the soil is less vulnerable to human practices that affect soil quality, such as years of pH amendments (Willoughby et al., 2023). It could be that the 3 years of grass/clover cultivation in the rotation dominates biological impacts on soil physical behaviour, with the yield insensitivity of this crop to pH masking the pH effects we expected to observe. The impact of these 3 years when the soil is uncultivated last through the remainder of the crop rotation and remove any pH impact on the physical properties of the soil that would otherwise be observed. This finding is of major importance for the future of agriculture as it demonstrates the importance of crop rotation for soil quality improvement and maintenance even after decades of chemical manipulations of this nature. Moreover, the maritime temperate climate of the region has rainfall far exceeding evaporation, so leaching could

diminish the development of water repellency (Bonanomi et al., 2024). Further research in drier climates, across a wider range of soil types of setup with controlled pH gradients and with different crop rotations is required to study pH effects on SWR with greater confidence. Unfortunately, few long-term pH experimental sites are available in the world to make this possible.

CONCLUSIONS

The long-term amendment of soil pH in the field was not found to affect many soil physical properties, including water repellency. This was not expected given findings from other researchers who have demonstrated large pH driven impacts to water repellency in controlled laboratory experiments and across different soils with varying pH. This experiment had an eight-year crop rotation that included three years of perennial grass/clover mixture, which is less yield sensitive to pH changes compared to oats. It is likely that this grass/clover crop had such a large impact on soil physical behaviour, that pH driven changes were masked for other crops grown later in the rotation.

Further studies on the impact of pH on crop roots in this field experiment are needed, as differences in root traits are expected to affect soil physical properties. There is also a need for research on pH impacts on soil physical properties in an arable system without a ley in the rotation, to determine if the presence of pH tolerant grass over-ride expected pH impacts on soil physical behaviour.

Acknowledgements. We thank Michael McGibbon and David Galloway for technical assistance in the laboratory. The research was made possible by the existence the SRUC Woodlands Field pH experimental field plots that were funded by the Scottish Government RESAS and maintained by SRUC technical staff (Michael Coutts and Derek Simpson). The PhD studentship was funded jointly by SRUC, the Roland Sutton Trust and the University of Aberdeen, Scotland, UK.

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Received 19 March 2024

Accepted 12 June 2024