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Gypsum amendment effects on micromorphology and aggregation in no-till Mollisols and Alfisols from western Ohio, USA

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ABSTRACT

Synthetic gypsum, a by-product of electricity generation, is used as a soil amendment to overcome water ponding, improve soil and water quality, improve field conditions to support farm equipment, and reduce the variability of crop yield in no-till fields by improving hydrology. Gypsum is a source of soluble calcium (Ca) that improves physical properties of the soil by promoting clay aggregation, thereby increasing water infiltration rates and movement through the soil profile. Undisturbed soil samples from Brookston and Celina soils in Ohio, USA were collected to a depth of 75 cm in agricultural fields treated with gypsum for 0, 4, and 12 years to determine changes in chemical and physical properties. Gypsum applications increased exchangeable Ca and Ca: Mg ratios, and promoted clay flocculation and improved soil structure. Mean weight diameter of aggregates increased with gypsum treatment at most depths in both soils. Micromorphological analysis showed variations in porosity (ϕ), pore size distribution, pore shape and aggregate size related to gypsum treatment, soil, and soil depth. There were no consistent responses to years of gypsum application. Gypsum treated soils had higher porosity than untreated soils in all depths <75 cm and a higher percentage of micropores and mesopores compared to the control. Also, gypsum treated soils had larger aggregates than the control for all soil depths examined. Aggregates <100 μm predominated in the Brookston control soils, and <200 μm aggregates dominated the Celina control soils. However, there was no prevailing aggregate size for gypsum treated soils. In conclusion, our study found positive effects of gypsum on most properties measured; although, not consistently related to years of gypsum applications to both soils.

1. Introduction

Ohio is located on the eastern edge of the American corn belt and benefits from highly fertile soils. However, approximately 55% of Ohio's agricultural land needs drainage intervention to improve infiltration and reduce soil erosion, reduce water logging in the plant root-zone, modify unfavorable field conditions for farm equipment in the spring and fall, and to reduce year to year crop yield variability caused by inconsistent infiltration and water movement (Ohio State University Extension, 1995; Tirado-Corbalá, 2010). Drainage issues, especially extended periods of profile saturation and surface ponding after snowmelt and/or excessive seasonal rainfall, may be particularly challenging to the adoption of no-tillage (NT) systems by Ohio farmers (Rusinamhodzi et al., 2011; Tirado-Corbalá et al., 2013). Artificial drainage (e.G. *tile* drainage) in poorly drained NT fields is often inadequate to prevent ponding after heavy rains (Tirado-Corbalá et al., 2013). Readily available flue gas desulfurization gypsum (FGDG) has been used as a soil amendment by some Ohio farmers, facilitating successful implementation of NT practices. High-purity FGDG is a coal combustion by-product which is readily available in Ohio due to electricity generation from many coal-fired power plants (Tirado-Corbalá, 2010). Coal continues to be an important fuel globally, and the combustion process that generates electricity often requires the removal of SO_2 from flue gases to meet clean air regulations. The materials produced during the scrubbing process are called FGD by-products and are initially mostly composed of $CaSO_30.5\ H_2O$ (calcium sulfite hemihydrate) and any unreacted sorbent (Chen et al., 2001; Laperche and Bigham, 2002). When forced air oxidation procedures are applied to $CaSO_30.5\ H_2O$, high quality FGDG ($CaSO_42H_2O$) is produced (Chen et al., 2005) and is deemed to be a worthy source of Ca and S for soils (Dontsova et al., 2005; Dick et al., 2006; USEPA, 2008).

Abbreviations: FGDG, Flue gas desulfurization gypsum; CT, Control treatment; ST, Short-term gypsum treatment; LT, Long-term gypsum treatment; D_b, Bulk density; MWD, Mean weight diameter; WSA, Water stable aggregates; PSD, Pore size distribution.

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Since new coal-fired power plants must meet stricter emissions regulations and as old units are retrofitted with modern scrubbing technologies, the availability of FGDG for agricultural markets is expected to be maintained. FGDG's potential to improve soil quality and agricultural productivity, when applied to many agricultural soils, has created renewed interest in the merits of gypsum as a soil amendment (Chen et al., 2001; Clark, 2002; Dick et al., 2006). Gypsum is a good source of soluble Ca and can improve soil physical properties by promoting aggregation, especially in high clay soils (Shainberg et al., 1989; Chan and Heenan, 1998). Surface-applied gypsum increases the electrolyte concentration of infiltrating water and compresses the electric double layer around clay particles. It also provides Ca^{2+} to the soil exchange complex where it has selectivity over more dispersive Mg^{2+} and Na^{+} ions (Shainberg et al., 1989; Dontsova and Norton, 2001).

Many studies have been conducted on the effect of gypsum on surface soil structure, crusting (Zhang and Miller, 1996), infiltration (Miller, 1987; Flanagan et al., 1997) and erosion (Norton and Dontsova, 1998); however, relatively few studies have analyzed the structural and hydrologic effects of gypsum in the subsurface horizons of non-sodic soils. The use of gypsum to improve soil water relationships on some poorly drained soils in Ohio has been documented (Tirado-Corbalá et al., 2013; Bigham, 2012 and Dick et al., 2006). Tirado-Corbalá et al. (2013) reported approximately 60% more cumulative drainage for gypsum treated soils (i.e. 12 yrs. FGDG-Brookston and 4 yrs. FGDG-Brookston) compared with untreated soils in a greenhouse water balance study. This greater drainage response was attributed to calcium redistribution to lower depths.

Few studies have examined the effects of gypsum application on soil structure at the soil aggregate scale.The aim of this study was, therefore, to determine the effects of long-term applications of FGDG gypsum (0, 4 and 12 yr) on soil structure (size and spatial arrangement of primary particles and voids) in the surface and subsurface horizons of two non-sodic soils with contrasting drainage (i.e., a moderately well drained and a poorly drained soil).

2. Materials and methods

2.1. Study site and undisturbed sample collection

Soil samples for soil characterization (section 2.2), and large undisturbed cores for a greenhouse study (Tirado-Corbalá et al., 2017) and for micromorphological analysis (section 2.3), were collected during March and April 2008, from no-till agricultural fields in Preble County, OH, USA (39° 45′17” N, 84° 40′28” W). The soils sampled were Celina silt loam (fine, mixed, active, mesic Aquic Hapludalfs, < 6% slope) and Brookston loam (fine-loamy, mixed, superactive, mesic Typic Argiaquolls, 1% slope) (Fig 1). These are two common soil series found in the glaciated till-plain landscape of central and western Ohio. The fields had been under no-tillage corn (*Zea mays* L.) and soybean (*Glycine* max L.) rotation since 1998. Twenty-four undisturbed soil columns (30.5 cm diameter x 75 cm long) were collected for the greenhouse study, described in Tirado-Corbalá et al. (2013 and 2017). Four columns were collected from each of three treatment areas that included: 1) a control treatment (Control) with no gypsum application; 2) a short-term treatment (ST) consisting of annual applications of gypsum at a rate of 1680 kg/ha over the previous 4 yr (i.e. since 2004) for a total of 6720 kg/ha; and 3) a long-term treatment (LT), consisting of annual applications of gypsum at a rate of 1680 kg/ha for the previous 12 yr for a total of 20,200 kg/ha. For the LT treatment, the first six gypsum applications were waste drywall gypsum (WDG). The subsequent six applications of gypsum on the LT fields and all four applications on the ST fields were made using FGDG. All gypsum applications for the ST and LT treatments were applied each year during the month of December. Both WDG and FGDG were applied to the fields using a double spinner lime spreader (Tirado-Corbalá et al., 2017). The FGDG was obtained from the William H. Zimmer Power Station near Moscow, OH. The FGDG was of high purity with <3% water insoluble residues and trace element content. The content of Ca, Mg and S were 19.8, 0.02 and 16.2%, respectively. The content of P and B were 16.7 and 26.7 mg kg^{-1}, respectively. Dontsova et al. (2005) reported that WDG contained 21.9, 0.22 and 18.9% of Ca, Mg and S, respectively, and 51.6 and 7.3 mg kg^{-1} of P and B, respectively.

2.2. Sampling for soil characterization

Prior to the collection of undisturbed soil columns, a series of sampling steps were enacted to provide soil for chemical and physical characterization. Duplicate soil samples from each field were taken at four depths (0–20, 20–40, 40–60, and 60–75 cm) using a 3-in. (7.5 cm) diameter auger; two samples from each depth from separate auger borings were composited. The moist soil was air-dried and sieved through a 2-mm sieve prior to physical and chemical characterization.

Sub-samples (50 g) were collected in triplicate per soil/treatment/depth to perform water aggregate stability (WSA) measurements by

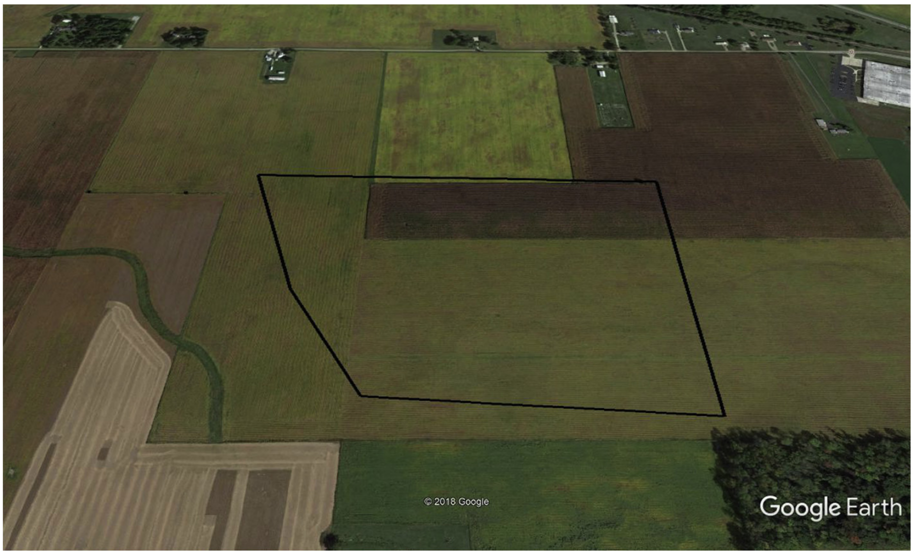

Fig. 1. Delimited area from no-till agricultural fields (39° 45′17” n, 84° 40′28” w) in preble county, ohio, usa; from where was collected the undisturbed soil columns of celina silt loam (aquic hapludalfs) and brookston loam (typic argiaquolls).

wet sieving (Kemper and Rosenau, 1986). The soil was spread evenly on top of a nest of sieves with openings of 4.0, 2.0, 1.0, 0.5, and 0.25 mm. The sieves were placed in a wet sieving apparatus with a stroke length of 3.7 cm at 30 oscillations per minute, based on the design of Kemper and Rosenau (1986), and sieved in a water column for 30 min. The aggregates retained on each sieve were washed into pre-weighed, 100-ml volumetric beakers, oven-dried at 105 °C for 24 h, and weighed. The mean weight diameter (MWD) of aggregates collected from each experimental treatment was calculated as $\text{MWD} = \Sigma X_i \text{WSA}_{i,}$ where $_i$ was the $_i$th size fraction (> 4 mm, 2–4 -mm, 1–2 -mm, 0.5–1-mm, and 0.25–0.5 mm aggregate size fractions), and X was the mean diameter of each size fraction, based on the mean inter-sieve size. Total WSA (TWSA) is equal to the sum of all aggregates size fractions. Material passing through the sieve with the smallest openings (i.e. 0.25 mm) was not counted under TWSA, is considered non-stable material. This non-stable fraction (NSF) was determined by calculation, i.e. by subtracting the total weight of stable aggregates from the weight of the initial sample.

Triplicate soil samples were collected from each treated field area and used to measure total C via combustion method. Soil pH was measured in a 1:1 soil/distilled water suspension and soil electrical conductivity was determined in a 1:5 soil-water mixture (Thomas, 1996). Exchangeable Ca^{2+} and Mg^{2+} were extracted using 1 M NH_4OAc (Warncke and Brown, 1998), and a flocculation/dispersion (F/D) study was performed to determine the behavior of soil clays under the influence of different gypsum treatments. For this study, three, 10-g samples of <2 mm soil/treatment/depth were placed in 750-ml sedimentation bottles. No chemical dispersing agent was used and no organic matter was removed. The 750-ml bottles were filled with 500 ml of deionized water and each sample was stirred for exactly 1 min with an electric mixer and placed in a sedimentation tank to equilibrate. After 24 h,10 ml aliquots were extracted at 3.5 cm depth to measure light transmittance at 420 nm wavelength using a Spectra Max Plus 384 Spectrophotometer (Dontsova and Norton, 2001).

Additional sub-samples were collected in triplicate from the field to calculate water retention curves (WRC) (Dane and Hopmans, 2002). These samples were taken using a 92-cm split soil sampling tube, which was driven by a truck-mounted Giddings Probe. The soil sampler fits twelve (7.62 × 7.62 cm) rings. Data from four depth intervals (0–20, 20–40, 40–60 and 60–75 cm) were collected for this study. Bulk density (Bd), total porosity (Φ) and pore size distribution (PSD) percentages were calculated using results from the water retention curves. Total porosity (Φ) was computed as [1 – (Bd/ Pd)], where Bd was the bulk density and Pd was the soil particle density (assumed to be 2.65 g/cm^3). The PSD data was divided into three pore size ranges (<0.2, 0.2–2 and > 2 μm).

2.3. Soil collection and impregnation for micromorphological study

The soil columns were vertically bisected using a Milwaukee reciprocating saw (blade- 12 in. length and 5/8 threads per inch) after completing an alfalfa lysimeter study previously described by Tirado-Corbalá et al. (2013 and 2017). Undisturbed soil samples from each soil/treatment/depth were collected from the vertical and horizontal faces of the half-columns by using circular tins (6 cm × 2.5 cm).

In this study, we included only results from thin sections generated from vertical faces of the columns. The soil samples were oven-dried at 105 °C for 48 h., saturated with 3 M Scotchcast™ electrical liquid epoxy resin, and placed inside a vacuum desiccator to remove air from the pores and facilitate the entrance of resin into the pore space. The resin-impregnated samples were cured overnight in an oven at 110 °C, and oriented cuts were performed using a diamond trim saw until the soil sample could fit on a 50 × 75 mm glass slide. Further impregnation of the undisturbed soil samples, grinding in oil, embedding, and dying of the thin sections was performed at National Petrographic Service, Inc. in Houston, Texas.

2.4. Thin section image acquisition

The evaluation of thin sections (TS) was performed in a semi-quantitative way using NIS-Elements BR 3.0 image analysis software with image stitching capabilities. The dimensions of the TS were 35 × 60 mm × 30 μm thick. The TS were used to analyze total porosity (Φ), pore size distribution (PSD) and aggregate sizes (AS). For PSD, data were grouped into three size ranges: macropores, > 1000 μm; mesopores, 100–1000 μm; and micropores, 50–100 μm. The aggregate dimensions were grouped into four size ranges: 0–100, 100–200, 200–300, and > 300 μm and shape into three categories: cracks, irregular and circular.

The TS from each soil/treatment/depth were examined under a Nikon 55i microscope with an LED light source and a polarizer/analyzer with a trinocular head at several levels of resolution. For TS image acquisition, the Nikon 55i microscope with manual stage was connected to a Nikon Digital Sight DS-U2 camera operated in conjunction with the NIS-Elements BR 3.0 image analysis software. The images were collected using a low power (2×) objective under plain light. To be able to acquire and analyze a sufficiently representative area of the thin sections, at high resolution, several smaller sections of image were digitally stitched to obtain a larger image of the actual thin section. The camera settings were set to "best quality image" (i.e. 2560 × 1920 pixels; exposure, ME 60 ms at +1.0 EV; and gain, 1.20 x) with 15% image overlap.

2.5. Image segmentation

The PSD and AS were quantified by directly measuring the number of pixels that conformed to each feature in the binary image. The actual image (i.e. the original captured after stitching) was transformed to binary colors with the NIS-Elements Br image program. After capturing the image of the whole TS, the red and green channels were removed to reduce the size of the data file and to speed up the image analysis using only the blue channel. The blue channel image was transformed to black and white where the black area represented the aggregates (soil matrix) and the white areas the pores (Fig. 2). Both were quantified by counting the pixels that conformed to each feature. Measurements were done using a single point threshold tool where one pixel equals 2.42 μm. A mathematical morphology basic option was used to smooth rough edges and matrix settings under the binary image option, and to reduce any noise, variability or irregularities resulting from thin section polishing. Objects with an area smaller than 20 pixels were eliminated corresponding with pores equal to or smaller than 50 μm.

For PSD, macropores, mesopores and micropores corresponded to areas with a size of >413, 41.3–413 and<41.3 pixels, respectively. Since the soil aggregates did not have a well-defined geometry, we used the parameter area (A), divided by the perimeter (P) to represent the size of the aggregates. Pore shape (sphericity) was derived from the following equation: $\text{Sphericity} = 4\pi A/P^2$ where values ranged from 0 to 1 and a value of 1 is a perfect circle. Sphericity results were divided into three classes: (cracks, 0–0.30; irregular-shaped pores, 0.31–0.50; and circular-shaped pores, 0.51–1.0) according to Mooney et al. (2000). The variables were quantified by direct measurement of the number of pixels that conformed to each feature in the binary image following the procedures detailed in Lebrón et al. (1999) and described using standard methods (Stoops, 2003; Soil Survey Staff, 2014).

2.6. Statistical analysis

For statistical purposes, the duration of gypsum application (Control, ST and LT) was randomly assigned to the field areas and variability was assumed to be primarily due to soil type and gypsum rate. The analysis of variance (ANOVA) was performed by soil. Also, the ANOVA and Tukey's test ($p < 0.05$) for mean comparisons were performed using

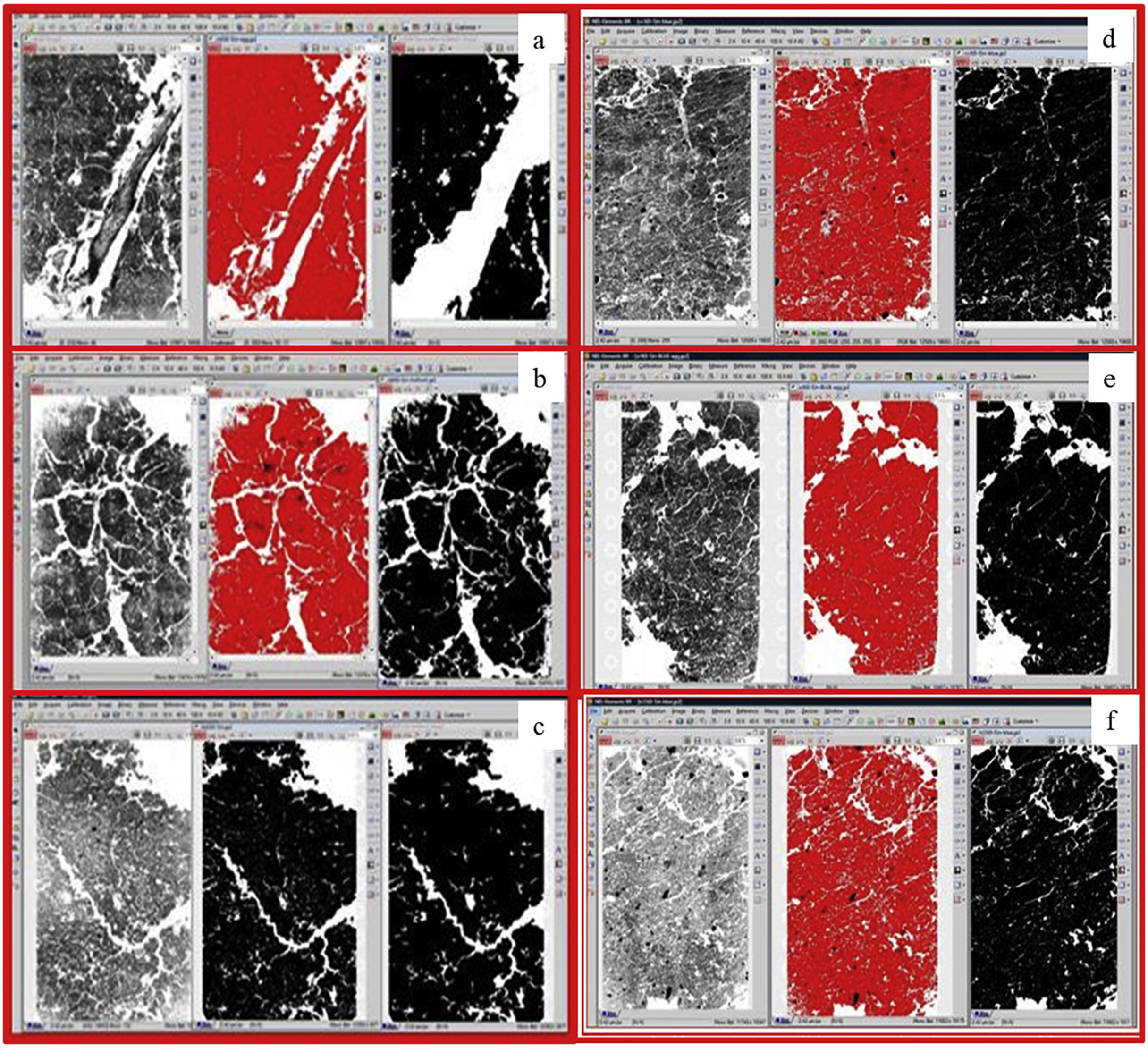

Fig. 2. Example of images from Brookston (left blocks) and Celina (right blocks) soils from the three treatments: a,d) untreated, CT; b,e) short-term gypsm treatment, ST and c,f) long-term, LT gypsum treatment as viewed in thin sections of samples from 0 to 20 cm depth. Pictures on the left side of each row in a block represent the original image under a blue channel, the central image in each row in a block is an image created after applying thresholds and transformations to red (or black on c image) and white, and the right image of each row in a block is after applying a smooth binary layer. In the right images, black areas represent aggregates (i.e. the soil matrix) whereas white areas represent pore space. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the Statistical Analysis System JMP Version 9.0 (SAS Institute, Cary, NC, USA). The treatments were compared for Ca: Mg ratio; clay flocculation/turbidity; bulk density (Bd), total porosity (ϕ), mean weight diameter (MWD), and pore size distribution (PSD) determined from the water retention curves (WRC); and total porosity (ϕ), pore size distribution (PSD), pore shape (PS) and aggregate size (AS) information from the soil micromorphological study.

3. Results and discussion

3.1. Soil direct measurements

3.1.1. Soil chemical and physical properties

The effects of gypsum on several chemical and physical properties obtained from undisturbed soil samples (Table 1) indicated that gypsum-treated soils had more exchangeable Ca and less exchangeable Mg than the untreated soils (Tirado-Corbalá et al., 2013). Ratios of exchangeable Ca to Mg generally increased in the gypsum-treated soils compared to the controls. The highest Ca: Mg ratios were found in the first 0–40 cm depth, and the LT treatments yielded higher Ca: Mg ratios than the ST treatments within this depth. At 60–75 cm depth in the Celina soil, higher Ca: Mg ratios were found in both gypsums treated soils when compared to the control.

Dontsova and Norton (2001) reported a positive effect of increased exchangeable Ca: Mg ratios on the structural stability and surface infiltration of four typical Midwestern soils. Based on the Ca: Mg ratios obtained in this study, we would expect soil clays from the gypsum treatments to exhibit more flocculation (i.e. less dispersion) than those in the controls. Optical density measurements from suspended clays from the Brookston and Celina LT-gypsum treatments allowed higher light transmittance than the control treatment clays at all depths (Table 1). However, results from the ST-samples were less consistent. Curtin et al. (1994) and Emerson and Chi (1977) have attributed the differences in dispersion of suspended clays as being due to the hydrated radii properties of Ca and Mg. Calcium has a smaller hydrated radius than Mg and can maintain higher electrostatic forces at clay surfaces. In contrast, Mg at low ionic strengths promotes clay dispersion, enhances slaking, and increases the susceptibility of soil aggregates to raindrop impact. We attribute the improved physical properties described in this paper (increased MWD of soil aggregates, etc.) as being

Table 1
Selected properties obtained from undisturbed Brookston and Celina soils under different gypsum application rates and soil depth intervals.

Soil	Treatment	Depth	Ca[a]	Mg[a]	Ca:Mg[a]	TC[b]	F/D[a]
		-cm-	mg kg^{-1}		Ratio	%	
Brookston	Control[b]	0–20	2367 b[c]	464 b	5.1 b	1.90	77.7 b
	ST		2663 a	605 a	4.4 b	2.05	81.9 b
	LT		2605 a	296 c	8.8 a	1.77	94.2 a
	Control	20–40	3033 c	831 a	3.7 b	1.36 b	56.5 b
	ST		3763 a	741 b	5.1 a	2.06 a	65.8 a
	LT		3106 b	506 c	6.1 a	1.49 b	70.3 a
	Control	40–60	2559 c	831 a	3.1 b	1.20 a	44.9 c
	ST		3393 a	804 b	4.2 a	0.67 b	56.5 b
	LT		3175 b	709 c	4.5 a	1.32 a	83.9 a
	Control	60–75	2279	765	3.0	0.71 b	56.1 b
	ST		2852	719	4.0	0.35 c	44.5 c
	LT		2854	732	3.9	1.18 a	74.7 a
Celina	Control	0–20	1139	225 b	5.1 b	1.22	61.4 b
	ST		1755	492 a	3.6 b	1.13	50.6 c
	LT		1518	187 b	8.1 a	1.30	80.7 a
	Control	20–40	1201 c	428	2.8 c	0.44 c	70.0 a
	ST		2037 a	532	3.8 b	3.19 a	42.3 b
	LT		1585 b	251	6.3 a	0.69 b	75.2 a
	Control	40–60	1884	778 a	2.4 c	0.34 c	35.4 c
	ST		3071	305 b	10.0 a	4.41 a	42.2 b
	LT		2097	338 b	6.2 b	2.27 b	52.5 a
	Control	60–75	1688 b	419 a	4.0 b	3.06 b	33.4 b
	ST		3189 a	177 b	18.0 a	4.60 a	36.2 b
	LT		3214 a	161 b	20.0 a	4.78 a	48.9 a

[a] Ca = exchangeable Ca, Mg = exchangeable Mg, Ca: Mg = calcium and magnesium ratio, TC = total carbon, and F/D = Flocculation/dispersion study determined with light transmittance (420 nm) data.

[b] Control = no gypsum application, ST = 4 years' annual gypsum application (6720 kg/ha total) and LT = 12 years' annual gypsum application (20,200 kg/ha total).

[c] Means followed by the same letter or no letters in a column for each soil depth are not significantly different ($P < 0.05$, Tukey HSD).

directly related to the increased ratios of exchangeable Ca to Mg achieved by long-term surface applications of gypsum.

The effect of the gypsum treatment at depth is attributed to the fact that the LT- gypsum application to the soil surface resulted in greater amount of dissolved calcium and sulfate that moved downward into the Brookston and Celina profiles (Tirado-Corbalá et al., 2013). This movement does not change soil texture (i.e. clay, silt and sand content), pH, EC, and CEC which remained constant among treatments for each soil type (Table 2). However, the exchangeable Ca and Mg concentrations changed with years of gypsum application (Tirado-Corbalá et al., 2013 and Tirado-Corbalá et al., 2017). Similar results were found by Lebrón et al. (2002) who reported soil texture remained constant when sodic soils were reclaimed with gypsum; only with changes on soil chemical properties with time. Toma et al. (1999) assessed the effect of 16 yrs. application of gypsum on soil chemical properties. They found Ca moved quickly from the top soil during the first two years after application, and then continued to translocate to and be absorbed in the subsoil for even longer periods of time. Agassi et al. (1998) found prominent dissolution-precipitation reactions after a severe heavy rain, probably due to dissolution of gypsum and formation of carbonate products. In our study, significant total C (TC) differences were found at deeper depth than 20 cm in both soils among treatments (Table 1). However, this increase in TC occurred primarily in Celina soils due to changes in inorganic C concentrations with the longer periods of gypsum application (Tirado-Corbalá et al., 2013 and Tirado-Corbalá et al., 2017). Higher calcite (calcium carbonate) content as percentage (%) of TC was observed in LT-gypsum (8.1 ± 1.1) than ST-gypsum (3.9 ± 0.3) and control (1.3 ± 0.2) (Tirado-Corbalá et al., 2013 and Tirado-Corbalá et al., 2017). Also, in our study (Tirado-Corbalá, 2010), we found visible calcite deposition in LT-gypsum pores at 60–75 cm soil depth that it was not observed under the other two treatements. As Wang and Anderson (1998), we attributed this finding after applying high electrolyte water with gypsum to a calcareous system, where Ca concentration increases in solution, above calcium equilibrium, dissolving Ca, Mg and carbonates under saturated conditions and precipitating some of the dissolution products as secondary carbonates under unsaturated conditions.

3.1.2. Other physical soil properties

Soil cohesion and aggregate strength usually increase with decreased water content due to an increased number of particle contact points and capillary forces (Horn et al., 1994; van Breemen, 1998). Horn et al. (1994) reported that soil drainage depended on the effectiveness of swelling and shrinking pressures and, in general, better drainage was observed in soils amended with either a 100% lime-saturated solution or a 100% gypsum-saturated solution. Amezketa (1999) emphasizes that Ca from gypsum acts as an inorganic stabilizing agent which prevents dispersion of aggregates. The percentage of WSA and MWD are often used as quantitative indicators of soil aggregation (Márquez et al., 2004).

In this study, the Brookston and Celina soils exhibited no consistent response to gypsum applications for TWSA and NSF (Table 3) within the depths sampled. Differences between treatments for WSA by aggregate size were not clearly linked to treatment effects or to soil depth (Table 3). In Brookston soils, positive effects of long-term application of gypsum are observed for the larger WSA (> 4 mm) in the 0–20 and 60–75 cm soil depths compared with untreated soils. In contrast, the Celina soils had greater amounts of the larger WSA (> 2 mm) at 60–75 cm soil depth under the ST-gypsum treatment versus the other two treatments. Among soil types, treatments and soil depths, MWD varied from 0.8 to 6.5 mm. WSA from the gypsum-treated soils clearly possessed greater MWDs than the control soils at virtually all depths (Table 4). Walia and Dick (2018) reported around 1.2 times higher MWD of aggregates from Hoytville clay loam and Wooster silt loam soil treated with gypsum (26.9 Mg ha^{-1}) plus glucose (4.5 Mg ha^{-1}) versus control (untreated) soil. However, these authors did not find differences between gypsum rates only (8.9 Mg ha^{-1} and 26.9 Mg ha^{-1})

Table 2
Textural class, particle size distribution, pH and CEC of Brookston and Celina soils [a].

Soil	Depth	Texture class	Clay	Sand	Silt	pH	EC[b]	CEC[b]
	cm		%				dS m^{-1}	cmol^{+}/kg
Brookston	0–20	Loam	24.8 (0.9)	28.0 (2.0)	46.7 (1.7)	5.7 (0.5)	0.89 (0.2)	23.7 (7.2)
	20–40	Clay loam	35.0 (3.1)	22.6 (4.3)	42.3 (1.9)	5.9 (0.3)	1.06 (0.3)	24.9 (7.8)
	40–60	Clay loam	37.3 (1.5)	24.5 (4.2)	38.2 (3.4)	6.3 (0.2)	1.03 (0.3)	22.1 (5.9)
	60–75	Clay loam	31.9 (0.5)	32.3 (4.7)	35.8 (4.6)	6.6 (0.2)	0.86 (0.2)	18.5 (1.9)
Celina	0–20	Loam	16.0 (4.4)	27.9 (5.4)	56.0 (9.7)	6.5 (0.3)	0.85 (0.4)	11.6 (5.2)
	20–40	Clay loam	36.4 (4.1)	9.9 (3.3)	53.7 (1.8)	6.6 (0.4)	0.81 (0.3)	11.6 (3.5)
	40–60	Clay loam	43.6 (2.1)	19.8 (3.5)	36.6 (2.2)	7.6 (0.5)	0.86 (0.2)	15.2 (3.4)
	60–75	Clay loam	32.0 (6.1)	31.3 (6.5)	36.7 (7.7)	8.0 (0.4)	0.61 (0.2)	14.5 (7.1)

[a] Each entry is the mean of measurements across all three sites (i.e., the control or no gypsum application, the ST site represented by 4 years' annual gypsum application and the LT site represented by 12 years' annual gypsum application). Number in parentheses indicates difference between maximum and minimum values from the three treatments.

[b] EC = electrical conductivity and CEC = cation exchange capacity.

Table 3
Percentage of water stable aggregates and total water stable aggregates of Brookston and Celina soils under different gypsum application rates and soil depth intervals.

Soil	Treatment[a]	Depth	WSA retained on sieve sizes shown below [c]					TWSA[c]	NSF[c]
			> 4	4–2	2–1	1–0.5	0.5–0.25		
		cm	mm					%	
Brookston	Control	0–20	77.7 b	6.4 a[b]	5.8	4.8	1.0 b	95.7	4.3
	ST		91.5 a	1.9 b	2.2	2.2	2.1a	99.9	0.1
	LT		88.6 a	1.9 b	3.9	1.6	2.7 a	98.7	1.3
	Control	20–40	42.7	17.8	20.3	11.8 a	4.2	96.8	3.2
	ST		79.5	8.4	5.4	3.7 b	1.2	98.2	1.8
	LT		49.7	19.9	12.8	9.6 a	5.0	97.0	3.0
	Control	40–60	46.1	7.7	18.1	15.7	7.0	94.6	5.4
	ST		90.4	3.4	1.3	3.0	0.3	98.1	1.9
	LT		79.2	6.1	5.6	3.8	1.5	96.2	3.8
	Control	60–75	11.5 c	11.3	22.0	24.8 a	14.4	84.0	16.0
	ST		40.0 b	23.0	20.0	5.0 b	3.6	91.6	8.4
	LT		66.2 a	9.0	6.0	5.0 b	2.8	89.0	11.0
Celina	Control	0–20	50.2	5.4	9.8	10.1	8.3	85.8	14.2
	ST		76.3	3.9	3.3	4.6	4.5	92.5	7.5
	LT		74.1	4.7	4.1	4.6	4.1	91.6	8.4
	Control	20–40	10.7	9.7	2.8 b	8.0	11.9	43.1	56.9
	ST		45.3	23.0	11.7 a	10.0	3.2	93.0	7.0
	LT		39.7	22.0	14.3 a	18.6	4.9	99.5	0.5
	Control	40–60	21.4	6.2 b	14.8	16.6	18.4	77.4	22.6
	ST		11.0	3.9 b	15.5	17.3	11.3	59.0	41.0
	LT		17.5	15.0 a	13.4	13.8	11.0	29.3	70.7
	Control	60–75	11.6 b	9.9 a	10.9 b	15.2 a	14.3	61.9	38.1
	ST		18.1 a	19.9 a	5.2 c	7.2 b	13.2	63.6	36.4
	LT		7.1 c	6.1 b	61.6 a	3.7 c	6.9	85.4	15.6

[a] Control = no gypsum application, ST = 4 years' annual application (6720 kg/ha total) and LT = 12 years' annual gypsum application (20,200 kg/ha total).
[b] Means followed by the same letter or no letters in a column for each soil depth are not significantly different ($P < 0.05$, Tukey HSD).
[c] WSA = water stable aggregates (> 4, 4–2, 2–1, 1–0.5 and 0.5–0.25 mm), TWSA = total water stable aggregates (%) and NSF = non stable fraction (%).

Table 4
Mean weigh diameter, bulk density, total porosity and pore size distribution of Brookston and Celina soils under different gypsum application rates and soil depth intervals.

Soil	Treatment	Depth	MWD[bc]	BD[b]	Φ[b]	PSD[b]		
						<0.2 µm	0.2-2 µm	> 2 µm
		cm	mm	g cm^3	%	%		
Brookston	Control[a]	0–20	5.5	1.18	55	20.0	10.0	12.0
	ST		6.1	1.21	54	19.0	10.0	11.0
	LT		5.8	1.29	51	17.0	6.0	13.0
	Control	20–40	3.8	1.29	51	18.0	15.0	11.0
	ST		5.5	1.30	51	9.0	6.0	11.0
	LT		4.5	1.31	51	9.0	10.0	18.0
	Control	40–60	3.6	1.42	46	16.0	15.0	13.0
	ST		6.1	1.32	50	5.0	8.0	13.0
	LT		5.6	1.33	50	9.0	8.0	19.0
	Control	60–75	4.7 b	1.52 a	43 b	19.0 a	6.0 b	11.0 b
	ST		5.5 a	1.37 b	48 a	6.0 b	8.0b	20.0a
	LT		5.3 a	1.37 b	48 a	9.0 b	20.0 a	19.0 a
Celina	Control	0–20	1.5	1.20	55	9.0	12.0	8.0
	ST		1.4	1.30	52	17.0	9.0	6.0
	LT		1.3	1.30	52	16.0	11.0	8.0
	Control	20–40	2.3 b	1.30	52	15.0	7.0	8.0
	ST		4.5 a	1.40	48	19.0	7.0	6.0
	LT		5.0 a	1.30	49	15.0	7.0	8.0
	Control	40–60	0.8 c	1.50	44	11.0	4.0	7.0
	ST		2.3 b	1.50	43	16.0	7.0	7.0
	LT		3.2 a	1.40	49	16.0	11.0	9.0
	Control	60–75	1.5	1.60	40	13.0	8.0 b	8.0 b
	ST		2.1	1.60	40	13.0	19.0 a	13.0 a
	LT		3.6	1.40	48	15.0	8.0 b	8.0 b

[a] Control = no gypsum application, ST = Four years' annual application (6720 kg/ha total) and LT =12 years' annual gypsum application (20,200 kg/ha total).
[b] MWD = Mean weight diameter, BD = bulk density, Φ = total porosity, and PSD = pore size distribution (determined from samples collected for water retention curves).
[c] Means followed by the same letter or no letters in a column for each soil depth are not significantly different ($P < 0.05$, Tukey HSD).

and the control on the Hoytville clay loam and Wooster silt loam topsoils. The Hoytville and Wooster soils generally have higher clay contents and have been managed under no-till for longer periods of time.

Bulk density and total porosity (ϕ) differences ($P < 0.05$) were only found for the 60–75 cm interval in the Brookston soils (Table 4). Higher Bd and lower ϕ were identified in the control soils compared with both Brookston gypsum-treated soils. Even though there was no statistical difference ($P > 0.05$) between treatments for the other depth intervals, LT- gypsum treated soils had 10% higher Bd compared with control soils in the first 20 cm (Table 4). Samples from the first 40 cm of both Celina gypsum rate treatments had higher Bd and lower ϕ compared with the control. However, for the 40–75 cm soil depth, the LT gypsum treated soils had lower Bd compared with controls and the ST gypsum treated soils (Table 4). Walia and Dick (2018) found that gypsum treatment had no effect on Bd and ϕ in any Hoytville clay loam and Wooster silt loam layer. However, Narra (2010) reported a positive effect on structural development when gypsum was added to clayey sediments. In addition, he found higher Bd values on clayey sediments treated with gypsum and lime in comparison to untreated soils. Greater Bd values were attributed to more clay aggregation in the sand-clay mixtures, as a result of packing where clay coated sand particles.

Statistical differences between treatments ($P > 0.05$) in Brookston soils for the 60–75 cm depth range were found for pore size distribution (PSD) calculated from the water retention curve data (Table 4). The controls had ~ 65% more <0.2 µm pores compared with both gypsum-treated soils. The LT- gypsum treated soils had ~ 70% more 0.02–2 µm pores than the other treatments. Both Brookston gypsum treated soils had ~ 45% more >2 µm pores than the untreated, control soil. Even though there was no statistical difference between treatments for the remaining intervals, the control treatment soil had >60% more <0.2 µm and 0.2–2 µm pores compared with gypsum treated soils. The LT-gypsum soils had more >2 µm pores than the other two treatments (Table 4).

In Celina soils, statistical difference was found in PSD for <0.2 µm pores in the 0–20 cm interval and for 0.02–2 and > 2 µm pores in the 60–75 cm depth interval (Table 4). Both Celina gypsum treated soils had around 45% more <0.2 μm pores than untreated soils in the first 20 cm of soil. However, ST-gypsum had 58 and 38% more 0.02–2 and > 2 μm pores than the other soils, respectively (Table 4). Gypsum additions tended to induce larger (> 2 μm) pores in Brookston, unlike in the Celina soil. The higher clay content of Brookston soils may have influenced the formation of larger aggregates and inter-aggregate pores more strongly than for Celina.

3.2. Soil indirect measurements and-micromorphological analysis

The aggregate sizes (AS), total ϕ and PSD of samples varied with soil treatment, depth and natural drainage. Lebrón et al. (2002) emphasized that pore size, geometry and connectivity are affected by aggregate arrangement. Changes in chemical composition (i.e. concentration of exchangeable Ca, Mg and Na) directly affect aggregate size and stability and, consequently, the hydraulic properties of the soil (Lebrón et al., 1999–Lebrón et al., 2002). Several researchers emphasize that image analysis has become a useful technique to measure AS, total ϕ and PSD (Mermut, 2009; Shen et al., 2015). To better understand how chemical composition affects PSD and pore shape, it was necessary to analyze AS from images. Aggregate size is considered an indicator of aggregate stability, similar to traditional aggregate stability tests. For these measurements, aggregates were divided into four sizes (0–100, 100–200, 200–300 and > 300 μm) (Table 5).

Statistical differences were found for both soil types between treatments in 0–20 cm depth for the four aggregate sizes. For Brookston soil, ST-gypsum treated soils had about 30% more 0–100 μm aggregates compared with the other treatments. However, LT-gypsum treated soils had 35% more 200–300 μm aggregates versus the other treatments (Table 5). For the other AS's at 0–20 cm soil depth, untreated soil had higher AS than gypsum-treated soil. For the Celina soil, differences in percentages of aggregates were found for both gypsum-treated soils versus the control soil for the 0–100 and > 300 μm AS's (Table 5). Both Celina gypsum-treated soils had 20% more 0–100 μm aggregates compared with the control. Control soils had 50% more 100–200 μm aggregates than the LT-gypsum treated soils. At depths of 20–75 cm no statistical differences were encountered between treatments in either soil, and no specific trend was found for each soil depth and aggregate sizes (Table 5).

For ϕ, statistical differences ($P < 0.05$) were found for the 0–20, 40–60 and 60–75 cm soil depth intervals in the Brookston soil. In the first 20 cm, higher ϕ was found for both Brookston soils treated with gypsums versus the control soil (Table 5). The LT- gypsum treated soils had 54% and ST- gypsum treated soils had 45% more ϕ, respectively, compared with the control soil (Table 5). For the 40–60 cm depth interval, both rates of gypsum in the Brookston soil had 50–60% more ϕ than the control soil. Finally, both rates of gypsum in Brookston soils also had higher ϕ than the untreated soil for the 60–75 cm soil depth interval (Table 5).

For the Celina silt loam, statistically greater ϕ was obtained at 0–20 cm depth for both gypsum rate treatments versus the control soil. Both gypsum treatments yielded around 32% more total ϕ in Celina soils (Table 5). At 20–40 cm depth, the soil from the LT-gypsum treatment had around 37% more ϕ than the other treatments. At 40–60 cm depth, both gypsum treatments had around 50% more ϕ than the control (Table 5).

For the Brookston soil, statistical differences ($P < .05$) in microporosity (50–100 μm), were encountered for all depth intervals (Table 5). At 0–20 cm, higher microporosity was found under both gypsum treatments compared to the control. The LT and ST gypsum treated soils had 59 and 50% more microporosity, respectively than the control soil. At the 40–60 and 60–75 cm intervals, both Brookston gypsum treated soils had around 50% more micropores than the untreated soil (Table 5). Statistical differences ($P < 0.05$) in mesoporosity (100–1000 μm), were also found for the 0–20 and 60–75 cm depth intervals in the Brookston soil (Table 5). About 50% greater mesoporosity was observed under both gypsum treatments compared with the control soil. For macroporosity (> 1000 μm), statistical differences were encountered for the 0–20 cm and 60–75 cm intervals where the Brookston control soils had higher macroporosity than the gypsum treated soils (Table 5).

Table 5
Aggregate size, total porosity and pore size distribution size percent of soil samples measured by using NIS-Elements Br image analysis software.[a]

Soil	Treatment	Depth	Aggregate size (μm)				Pore size distribution (μm)			Total ϕ
			0-100[b]	100–200	200-300	>300	50–100	100–1000	>1000	
		cm	%				%			
Brookston	Control	0–20	57 b[c]	14 a	8 b	21 b	25.8 b	4.8 b	1.4 a	32.0 b
	ST		71 a	9 b	6 b	14 b	51.2 a	6.8 a	1.2 b	59.2 a
	LT		55 b	5 b	29 a	11 b	63.0 a	5.9 a	1.2 b	70.1 a
	Control	20–40	83	10	7	0	34.7	6.1	1.2	42.0
	ST		17	55	7	21	57.9	6.8	1.1	65.7
	LT		64	12	7	17	59.8	5.1	1.3	66.2
	Control	40–60	55	27	8	10	12.2 b	1.8	0.8	14.8 b
	ST		0	56	0	44	25.8a	4.4	1.5	31.7 a
	LT		52	35	6	7	34.2 a	9.3	1.2	44.7 a
	Control	60–75	37	24	4	35	15.7 b	3.3 b	1.6 a	20.5 b
	ST		8	30	0	62	42.8 a	6.6 a	1.2 b	50.6 a
	LT		0	27	0	73	56.3 a	7.5 a	1.2 b	64.6 a
Celina	Control	0–20	49 b	47 a	1.6 a	2.4	34.9 b	4.9 b	1.4 a	41.2 b
	ST		61 a	25 b	0 b	14	53.6 a	6.4 a	1.2 b	61.2 a
	LT		67 a	0 c	0 b	33	59.1 a	7.3 a	1.2 b	67.6 a
	Control	20–40	95	5	0	0	40.0 b	5.6	1.3	46.9 b
	ST		83	5	0	12	44.4 b	6.2	1.2	51.8 b
	LT		63	3	8	16	68.6 a	5.9	1.2	75.7 a
	Control	40–60	32	18	15	35	26.9 b	2.7	0.7	30.4 b
	ST		27	33	35	20	53.4 a	6.4	1.2	61.0 a
	LT		18	45	15	7	52.6 a	7.0	1.2	60.9 a
	Control	60–75	14	15	0	71	30.2	3.2	0.7	34.1
	ST		42	0	5	43	37.4	6.4	1.2	48.7
	LT		21	60	7	12	46.4	10.2	1.2	54.0

[a] Control = no gypsum application, ST = 4 years' annual application (6720 kg/ha total) and LT = 12 years' annual gypsum application (20,200 kg/ha total).
[b] ϕ = total porosity due to micropores (50–100 μm), mesopores (100–1000 μm) and macropores (>1000 μm).
[c] Means followed by the same letter or no letters in a column for each soil depth are not significantly different ($P < 0.05$, Tukey HSD).

Table 6
Sphericity (pore shape) percentage measured by using NIS-Elements Br image analysis software.

Soil	Treatment	Depth	Mesoporosity			Macroporosity		
			Crack	Irregular	Circular	Crack	Irregular	Circular
		cm	%					
Brookston	Control[a]	0–20	0	4.1	95.9	14.9 b[b]	20.6	65
	ST		0	4.3	95.7	4.8 c	18.9	76.3
	LT		0	2.1	97.9	37.2 a	6.0	56.8
	Control	20–40	0	8.5	91.5	0	20.3	79.7
	ST		0	5.0	95.0	0	19.3	80.7
	LT		0	1.7	98.3	86.5	13.5	0
	Control	40–60	0	1.9	98.1	20.9	10.9	68.2
	ST		0	1.9	98.1	12.4	16.5	71.1
	LT		0	2.8	97.2	13.4	9.89	76.7
	Control	60–75	0	2.8	97.2	9.2	15.3	75.5 a
	ST		0	1.1	98.9	18.1	15.1	66.8 b
	LT		0	1.8	98.2	8.7	11.1	80.2 a
Celina	Control	0–20	0	8.5 a	91.5	2.0	21.0	59.0
	ST		0	5.0 b	95.0	14.0	19.0	67.0
	LT		0	1.7 c	98.3	10.0	13.0	77.0
	Control	20–40	0	4.2 a	95.8	15.6	22.1 b	62.3 a
	ST		0	1.2 b	98.8	10.0	90 a	0 b
	LT		0	5.3 a	94.7	12.8	87.8 a	0 b
	Control	40–60	0	5.3	94.7	12.8 b	17.8 b	69.4 a
	ST		0	4.0	96.0	35.5 a	1.7 c	63.3 a
	LT		0	7.8	92.2	0 c	83.8 a	62 b
	Control	60–75	10 b	11.5 b	88.5 b	11.6	26.8	61.6
	ST		0 c	3.1 c	96.9 a	10.9	14.6	74.5
	LT		29.7a	49 a	21.3 c	3.8	14.5	81.7

[a] Control = no gypsum application, ST = 4 years' annual application (6720 kg/ha total) and LT = 12 years' annual gypsum application (20,200 kg/ha total).
[b] Means followed by the same letter or no letters in a column for each soil depth are not significantly different (P < 0.05, Tukey HSD).

For the Celina PSD, statistical differences were found for microporosity (50–100 μm) throughout the top 60 cm. The LT- gypsum soils had more micropores compared with the control soils at all sampling depths within this range (Table 5). The ST-gypsum soils also had more micropores than the control soils in the 0–20 and 40–60 cm soil depth intervals. For mesoporosity (100–1000 μm), both gypsum treatments yielded more mesopores at 0–20 cm than in the CT soil. The LT and ST-gypsum treated soils had 33 and 24% more mesopores than the CT soil, respectively (Table 5). No statistical differences were encountered for the remaining depths (Table 5). Higher macroporosity (>1000 μm) was found in the untreated soils (~14%) in the top 40 cm.

The importance of pore shape to the hydraulic properties of a soil have been shown by several authors (Phillips et al., 2003; Tuller et al., 1999). In these studies, pore shape was found to be a good descriptor of the soil system and was a more realistic indicator of soil behavior than other parameters. Pore shape-sphericity was calculated according to Mooney et al. (2000) where macropores and mesopores were divided into three classes: cracks (0–0.30), irregular-shaped pores (0.31–0.50) and circular-shaped pores (0.51–1.0). In the Brookston soil, mesopores had either irregular or circular shapes (~ 90%) at all depths and, cracks were not present (Table 6). For Brookston soil, circular macropores were also the most predominant but variability was much greater than with the mesopores, and both cracks and irregular shaped pores comprised a greater proportion of the macroporosity. Statistical difference ($P < .05$) was encountered for the 0–20 cm interval, with the LT-gypsum treated soils having around 37.2% of all macropores classified as a crack (Table 6). This compared with 7.8 and 3.1% for the ST-gypsum treated soil and control, respectively (Table 6).

The mesopores in the Celina silt loam also had irregular or circular shapes (~ 90%) at most depths (Table 6). Cracks were observed only in the 60–75 cm samples. A statistical difference was observed among the three treatments at the 60–75 cm soil depth for circular shape. The LT-gypsum treated soil had less circular mesopores compared with the other two treatments. The LT- gypsum treated soils had 30% cracks, 49% irregular and 21% circular pore shapes for mesopores for the 60–75 cm soil layer. Generally, the untreated Celina soils had more irregular pores in the first 40 cm compared with gypsum-treated soils (Table 6). For crack shape, statistical difference was found in the 60–75 cm soil interval. The LT- gypsum treatment had almost three times more crack pores than the other two treatments (Table 6). These differences in pore shape may indicate that gypsum additions are promoting increased flocculation of clay and re-aggregation with successive wetting and drying cycles.

4. Conclusion

Increased gypsum applications to non-sodic soils in Ohio resulted in increased exchangeable Ca: Mg ratios and clay flocculation. Also, higher percentages of WSA were found in gypsum-treated soils at most depths. A micromorphology study revealed total ϕ, PSD, AS and pore shape (sphericity) of undisturbed soil samples varied with treatment and soil. Gypsum-treated soils had higher total ϕ to depths of 75 cm compared to control soils. For both soils, gypsum treatments had a positive effect on the percentage of total ϕ characterized as micropores and mesopores, and a higher percentage of pores in the macropore range was found in the control (untreated) soils. The AS varied with respect to treatment and soil depth. Gypsum-treated soils had larger aggregates than the control soil throughout the investigated depths. Aggregates <100 μm and < 200 μm predominated in the Brookston control soil and Celina control soil, respectively. There was no predominant AS for gypsum-treated soils. Pore size distribution was related to AS which was affected by soil chemistry. In our study, we found positive effects of gypsum on most physical properties measured, but the effects were not always related to the number of years of gypsum application to the Brookston or Celina soils.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found in the online version, at doi: https://doi.org/10.1016/j.geodrs.2019.e00217. These data include the Google map of the most important areas described in this article.

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