

# Time dependence of soil water hysteresis in a minesoil reclaimed by sewage sludge amendments

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## ABSTRACT

One of most important problems in soil thermodynamics is related to soil water hysteresis, which arises mainly from differences in the processes of emptying and filling individual pores during wetting and drying. In a minesoil coming from excavations, amended with six different types of sewage sludge (composted or thermally dried), it was observed that the critical point ( $\psi_c, w_c$ ) that separates the region dominated by complete pore filling/emptying from that dominated by partial pore filling/emptying could be found using the linear relationship between suction and time. Differences in temporal variation rates of soil gravimetric water content, before and after this critical point were more evident during the soil drying process. The selected minesoil required a mean of 1.6 min to change their water content from dryness to wilting point ( $\Delta t_{total}$ ) under the wetting process, but under the drying process around 130 min was required for a similar water content change at the same temperature, verifying soil hysteretic conditions. It was observed, mainly at short term, that composted sludge increased soil water holding time ( $\Delta t_{total}$ ) under both the drying and wetting processes. Changes in the temporal dependency of hysteresis could be explained partially by the relationship with soil organic carbon.

## INTRODUCTION

The drying and wetting of soils are two processes which occur continuously in the field. The dependence of soil water content ( $\theta$ ) on its energy state or suction ( $\psi$ ) differs under both processes and this hysterical behavior arise from differences in the processes of filling or emptying soil pores [1] during wetting and drying. Soil water hysteresis is caused by several factors such as the incomplete connectivity of pore spaces, shrink–swell phenomena, thermal gradients, the presence of entrapped air, the “ink bottle” capillary effect, and contact angle hysteresis, all of which are closely related to soil structure. In minesoil reclamation processes, sewage sludge is currently used as organic amendment in order to improve many soil properties such as fertility, soil water retention and aggregate stability [2, 3]. Organic matter makes the aggregates more resistant by increasing their internal cohesion, binding soil particles by organic polymers or physical enmeshment by roots or fungi [4], or decreasing their watability [5]. Under these processes water is retained in the soil structure by capillary forces at high saturations (low suction regime) [6], while at low saturations (high suction regime) the forces of molecular attraction are predominant [7]. However, the time dependency of the wetting and drying processes is rarely compared. The main objective of this study is to assess the effects of two kinds of sewage sludge (composted and thermally dried) on the time involved in the wetting and drying process of a minesoil. We apply a segmented model to quantify temporal changes in soil water content. The model can thus identify the critical point where soil water retention becomes dominated by complete pore filling/emptying (low suction regime) rather than dominated by partial pore filling/emptying (high suction regime). The relationship between water holding time with soil organic carbon, measured by the wet oxidation method, was analyzed.

## PROPOSED MODEL

The critical gravimetric water content ( $w_c$ ) was found using the relationship between gravimetric water content ( $w$ ) and time ( $t$ ), in both low and high suction regimes:

$$w = a_1 t + b_1 \quad t_e \leq t \leq t_c \quad (1)$$

$$w = a_2 t + b_2 \quad t_c \leq t \leq t_\infty \quad (2)$$

where  $a_1$  and  $a_2$  are constants,  $t_c$  is the time when air/water entry suction occurred and  $t_c$  is the time when low suction regime changes to high suction regime. The sign of  $a_1$  and  $a_2$  depends on whether soil was under a wetting or drying process. The interception of both straight lines (Equations 1 and 2), corresponding to the transition point where low suction regime changes to high suction regime, is determined by a change of slope (Figure 1). The  $t_c$  and  $w_c$  values were calculated and the  $\psi_c$  value was determined from the water retention curve (WRC) using the  $w_c$  value. The composite fractal model of Ojeda et al. (2006) was used to fit WRC, as this model covers both the low and high suction regimes (Figure 2), where  $A_1$  and  $A_2$  (Figure 2) are constants with different physical meanings, depending on which suction regime dominates the wetting or drying cycle, while  $D_1$  and  $D_2$  are pore-solid and surface fractal dimensions, respectively. The parameter  $A_1$  is related to changes in soil structure stability, while  $A_2$  is related to changes in soil aggregate surface. It suggested that  $a_1$  and  $a_2$  parameters could be related to corresponding soil properties due to their similar soil water retention domain.

## MATERIALS AND METHODS

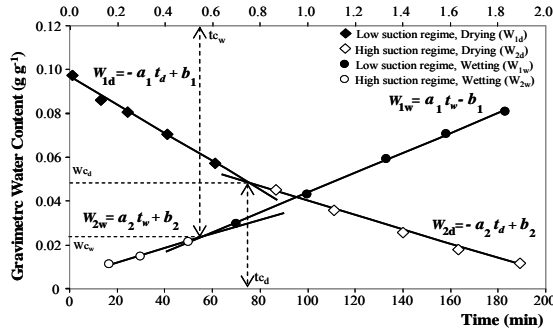
The soil samples were obtained from a limestone quarry (Begues municipality, Barcelona, Spain) that produces a waste soil (minesoil) resulting from excavation processes. It has characteristics of a red Mediterranean soil, with a loamy texture, being low in organic matter (organic C =  $0.47 \pm 0.09\%$ ), but rich in lime ( $\text{CaCO}_3 = 39.3 \pm 8.0\%$ ) and coarse fragments ( $62.0 \pm 6.6\%$ ). Minesoil was amended with six types of treated sludge produced in various municipal waste-water treatment plants from medium-sized towns of Catalonia (NE Spain). All sludges were digested and partially dewatered (20% dry matter) before being subjected to either a composting or a thermal drying process. Dewatered sludge from Manresa was composted with crushed pinewood bark, while dewatered sludge from Blanes and Vilaseca was composted with pinewood splinters. All sludges were applied to soil as granules, except thermally dried sludge from Sabadell that was added as a pellet (1 cm in diameter). Table 1 shows the origin, composition and treatments applied to selected sludges.

A nominal dose of about  $35 \text{ Mg ha}^{-1}$  (dry weight) of each sludge was mixed with the minesoil and distributed in 28 lysimeters of 150 L with a surface area of  $0.3 \text{ m}^2$ . There were 4 replicates per treatment. The experiment was maintained in the quarry for 13 months. Samples were taken after one month (sampling one: S1) and twelve months (sampling two: S2) after the start of the experiment. Soil samples (0 – 20 cm depth) of each lysimeter were air-dried at room temperature, passed through a 2 mm sieve, and stored at  $4^\circ\text{C}$  in the dark before analysis. Table 2 gives information about treatments and the main physicochemical properties of the amended soils at S1.

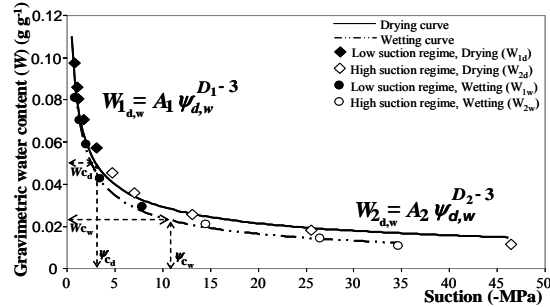
The main wetting and drying branches of the water retention curve were measured in the laboratory using a WP4 Dew Point PotentiaMeter (Decagon Devices, Inc. Pullman, WA). For the wetting curves, subsamples of approximately 1.2 g of air-dry soil from each plot sample were placed in sample cups (40 mm diameter  $\times$  10 mm high) resulting in a monolayer of aggregates that completely covered the bottom of the sample cups as recommended by the manufacturer. Each sub-sample was vapor wetted for a different length of time using an ultrasonic humidifier. Wetting up times of 5, 10, 20, 40, 60, 80, 100, 120, 140, 160 and 180 seconds were chosen to produce a series of approximately equally spaced suction values within the operating range of the WP4 (from -0.5 to -50 MPa). After wetting up, the samples were allowed to equilibrate for 1 h. The suction was then measured with the WP4 instrument. At the end of each measurement, the samples were oven dried at  $40^\circ\text{C}$  for 24 h, and values of  $w$  (gravimetric water content) were calculated. Paired measurements of  $w$  and  $\psi$  (suction) were obtained in this way for each wetting branch.

For the drying curves approximately 1.2 g of air dry soil from each plot sample, was placed in a sample cup (both the sample and sample cup were previously weighed) and saturated by capillarity for 10 minutes using one band of filter paper in contact with a free water table approximately 10 mm above the soil surface. A balance and the WP4 Dew Point PotentiaMeter were then used to record  $w$  and  $\psi$  periodically over time, as the sample dried by evaporation [8]. Approximately 8-12 paired measurements of  $w$  and  $\psi$  were obtained in this way for each drying branch.

The time required for the soil wetting and drying processes was measured using a chronometer and the relationships between gravimetric water content and time were analyzed. The wetting or drying time that elapsed between wilting point ( $\psi_{1.5}$ ) and suction at 25 MPa ( $\psi_{25}$ ), called  $\Delta t_{\text{total}}$ , was considered as a parameter.



**Figure 1.** Temporal variation of gravimetric water content and time dependence of critical gravimetric water content, where  $w_c$  is the point that separates the region dominated by complete pore filling/emptying from that dominated by partial pore filling/emptying ( $t_c$ : critical time; subscript d: drying, w: wetting; Lower X-axis: Drying state; Upper X-axis: Wetting state).



**Figure 2.** Example of the segmented fractal model used to fit the main drying and wetting branches of the water retention curve applied to a sample from the experimental minesoil ( $\psi_c$ : critical matrix potential,  $W_c$ : critical gravimetric water content; subscript d: drying process, w: wetting process).

**Table 1.** Treatments, composition and identification code of the sewage sludge types used as organic amendment.

Origin <sup>†</sup> (WWTP)	Type of digestion	Code*	Organic Matter (%)	Stability Degree <sup>‡</sup> (%)	N (%)	P (%)	pH	EC (dS m <sup>-1</sup> ) <sup>¶</sup>
Blanes	Anaerobic	C <sub>BL</sub>	56.6	29.0	3.2	7.0	6.5	7.6
Manresa	Anaerobic	C <sub>MR</sub>	55.5	40.6	2.3	4.3	7.1	3.9
Vilaseca	Aerobic	C <sub>VL</sub>	58.3	35.8	3.0	5.8	6.9	8.5
Besós	Physico-chemical	T <sub>BS</sub>	72.3	8.6	2.2	4.0	6.1	1.4
Mataró	Anaerobic	T <sub>MT</sub>	74.0	40.4	3.5	3.3	6.2	5.8
Sabadell	Anaerobic	T <sub>SB</sub>	62.2	39.5	3.9	5.8	7.3	0.9

<sup>†</sup> WWTP: identification of municipal waste water treatment plant. \* Composted sludge from Blanes (C<sub>BL</sub>), Manresa (C<sub>MR</sub>) or Vilaseca (C<sub>VL</sub>), and thermally-dried sludge from Besós (T<sub>BS</sub>), Mataró (T<sub>MT</sub>) and Sabadell (T<sub>SB</sub>). <sup>‡</sup> Stability degree: percentage of organic matter resistant to acid hydrolysis. <sup>¶</sup> EC: Electrical conductivity (1:5 water extract).

**Table 2.** Mean values of the physicochemical properties of <2 mm fraction of the minesoil amended with different types of sewage sludge.

Type of sludge	Treatment <sup>†</sup>	Sand %	Silt %	Clay %	pH (H <sub>2</sub> O)	E.C. (dS m <sup>-1</sup> ) 1:5	SOM* %	N %	P-Olsen mg kg <sup>-1</sup>
Composted	C <sub>BL</sub>	44,2	35,2	20,6	8,0	1,1	1,8	0,15	51
	C <sub>MR</sub>	45,7	34,3	20,0	8,1	0,9	1,6	0,10	46
	C <sub>VL</sub>	38,3	36,2	25,5	8,0	1,0	2,4	0,22	101
Thermally dried	T <sub>BS</sub>	46,2	34,0	19,8	8,2	0,5	1,9	0,12	35
	T <sub>MT</sub>	38,6	41,3	17,4	8,1	0,5	2,9	0,16	49
	T <sub>SB</sub>	51,0	31,6	20,1	8,3	0,7	1,2	0,08	10
Control	O <sub>MS</sub>	42,5	35,0	22,5	8,5	0,5	0,8	0,06	6

<sup>†</sup> See Table 1 to identify sewage sludge treatment. \*SOM: organic matter. O<sub>MS</sub>: minesoil without sludge.

The straight lines ( $w$  vs.  $t$ ) for wetting and drying states at low and high suction regimes were fitted separately using regression analysis, calculated with Statview© [9] (SAS Institute, 1998) statistical software, and estimates of  $a_1$  and  $a_2$  parameters (Equations 1 and 2) were produced. Hypothetical intercepts with the Y-axis ( $b_1$  and  $b_2$ ) were not analyzed. The main wetting and drying branches also were fitted separately using segmented non-linear regression analysis, taking ( $\psi_c$ ,  $w_c$ ) values as break point, using the PROC NLIN (Newton method) in the SAS/STAT® statistical software program [10], and soil water contents for wilting point ( $\psi_{1,5}$ ) and suction at 25 MPa ( $\psi_{25}$ ) was estimated from them. The effects of the different kinds of sludge applied on the soil water retention parameters were

measured by a three-way variance analysis (sludge type – state – sampling), using PROC GLM from the statistical software program SAS/STAT© [10]. When significant differences were found, the t-test (Least Significant Difference) was used to make paired comparisons between the different treatments.

## RESULTS AND DISCUSSION

The measured values of  $w$  and  $\psi$  for amended minesoil under drying and wetting processes ranged from 0.01 to 0.15 g g<sup>-1</sup> and from -0.5 to -50 MPa, respectively, where increases in  $\psi$  always resulted in decreases in  $w$ , and vice versa. The time required to wet or dry a minesoil sample varied from 0.08 to 4 min for wetting process and up to 210.7 min for drying process. Measured values of  $a_1$  and  $a_2$ , corresponding to the rate of temporal variation of soil gravimetric water content, showed no significant differences between treatments (Table 3). Globally,  $a_1$  and  $a_2$  values were higher at sampling one (S1) than at sampling two (S2) and were higher for wetting process than for drying process. Interaction between state and sampling time (Table 3) for  $a_1$  parameter is related to two main causes: (a) under wetting process the uptake rate of soil gravimetric water content ( $a_{1w}$ ) in S1 was higher than in S2 and, (b) under drying process the loss rate of soil gravimetric water content ( $a_{1d}$ ) in S1 was higher than in S2. During wetting and drying processes, water is present in soil at different places e.g. totally filling pore soil, at the asperities of pore walls, in pendular rings between grains, in bridges between grains separated by small gaps, in pore throats between larger pores, or in structures formed by a combination of these water morphologies [11, 12, 13]. Under wetting/drying processes, all of them are communicated by film water which increases/decreases their thickness depending on the increase/decrease of soil water content. The slope change is possibly related to a critical point ( $\psi_c$ ,  $w_c$ ) where the water film that links the different water morphologies starts to disappear (drying process), leaving isolated wet areas, or starts to appear (wetting process), linking isolated wet areas by a film which increases their thickness.

Differences in  $a_1$  and  $a_2$  values, observed between dry and wet states, are referred to different soil water movement processes with different time scales ( $a_{1d}:a_{1w} = 44:1$ ;  $a_{2d}:a_{2w} = 71:1$ ). The rate of temporal variation in soil gravimetric water content during the wetting process corresponds to a positive slope, whilst for the drying process this corresponds to a negative slope (Figure 1). Comparing  $a_1$  and  $a_2$  parameters in each treatment, sampling and state by t-test (Table 4), it was observed that  $a_1 > a_2$  under a drying process in all sludge treatments at both samplings, whilst under a wetting process,  $a_1 > a_2$  was only observed for treatments with composted sludge at S1 and in two treatments (minesoil control and minesoil treated with composted sludge from Vilaseca) at S2.

However, when analyzing the  $\Delta t_{total}$  parameter (water holding time), the influence of composted or thermally dried sludges was observed between treatments, samplings and states, with significant interactions between all of them (Table 3). This means that differences of  $\Delta t_{total}$  between treatments must be analyzed at each sampling time and drying/wetting process, as presented in Figure 2. During the drying process, all composted sludge treatments increased the  $\Delta t_{total}$  time of minesoil at S1, while at S2 thermally dried sludge from Sabadell reduced  $\Delta t_{total}$  time of minesoil. During the wetting process, only composted sludges from Blanes and Vilaseca increasing the  $\Delta t_{total}$  time of minesoil at S1, while at S2 thermally dried sludges from Besos and Sabadell reduced the  $\Delta t_{total}$  time of minesoil. The comparatively low  $\Delta t_{total}$  time of thermally-dried sludge from Sabadell ( $T_{SB}$ ) at S1 can be explained by its particle size (pellets of ca. 1cm diameter), which presented a smaller decomposition than the other composted and thermally-dried sludges that were supplied as granules. However, at S2, reductions in water holding time ( $\Delta t_{total}$ ) under the wetting and drying processes, as observed in thermally dried sludge treatments from Besós and Sabadell, could be explained by improvements in soil wettability or drainage, according to Ojeda et al. [14], which observed that thermally-dried sludge increases soil infiltration.

The water retention of minesoil calculated from WRC (Figure 1) was significantly higher under the drying process than under the wetting process (unpublished data). The minesoil required a mean of 1.6 minutes to change their soil water content from dryness to wilting point under wetting process, while under drying process changes from wilting point to dryness required a mean of 130 minutes. This means that soil hysteresis depends not only on differences of soil water retention between drying and wetting curves. Soil hysteresis also involved differences in the time required to absorb or loose soil water.

**Table 3.** Summary of three-way variance analysis of sludge treatment (composted or thermally dried), sampling time (S1 and S2) and hysteresis (wetting-drying processes) for soil water retention parameters measured on the minesoil.

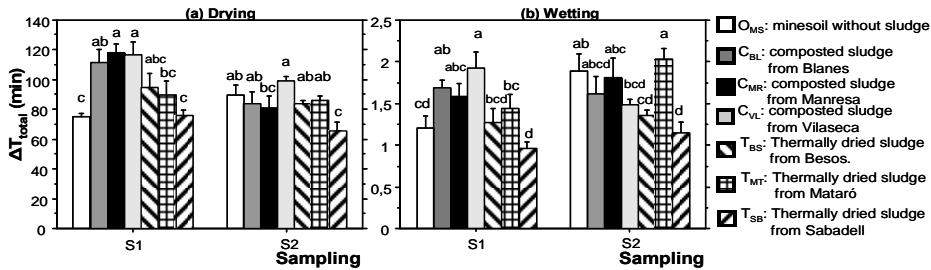
Parameters <sup>†</sup>	F Value						
	T	S	H	T*S	S*H	T*H	T*S*H
a <sub>1</sub>	NS	27.9***	912.4***	NS	28.7***	NS	NS
a <sub>2</sub>	NS	14.1**	505.6***	NS	NS	NS	NS
Δt <sub>total</sub>	6.6***	13.8**	2386.8***	3.0*	14.1**	6.4***	2.9*
w <sub>wp</sub>	50.4***	101.9***	28.9***	7.1***	NS	NS	NS
w <sub>c</sub>	8.4***	12.2**	17.5***	2.7*	NS	NS	NS
w <sub>25</sub>	62.1***	8.7*	156.8***	5.0**	NS	NS	NS

<sup>†</sup> All values are significant at p < 0.0001 (\*\*\*), 0.001 (\*\*) and 0.05 (\*), except for those denoted by NS. T = treatment, S = sampling time, H = hysteresis: wetting-drying processes.

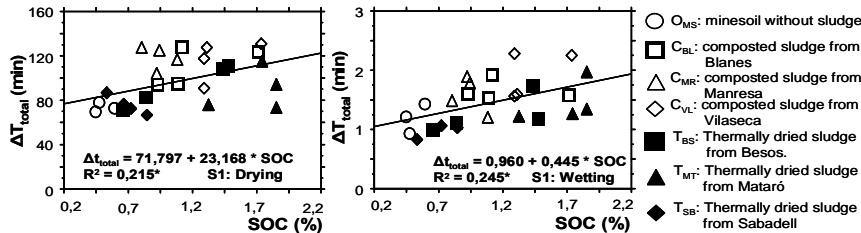
**Table 4.** Paired t-test analysis between a<sub>1</sub> and a<sub>2</sub> parameters of a minesoil amended with different types of sewage sludge.

Type of sludge	Treatment	t-value			
		Sampling one (S1)		Sampling two (S2)	
		Drying	Wetting	Drying	Wetting
Composted	C <sub>BL</sub>	-9.1*	5.0*	-3.8*	NS
	C <sub>MR</sub>	-33.8***	4.9*	-6.1*	NS
	C <sub>VL</sub>	-6.9*	3.3*	-12.4*	4.3*
Thermally dried	T <sub>BS</sub>	-8.6*	NS	-11.1**	NS
	T <sub>MT</sub>	-5.5*	NS	-17.0**	NS
	T <sub>SB</sub>	-6.6*	NS	-6.6*	NS
Control	O <sub>MS</sub>	-10.1*	NS	-5.2*	4.0*

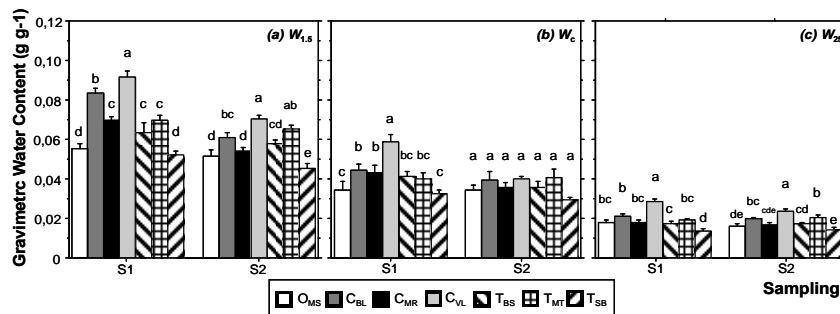
All values are significant at p < 0.0001 (\*\*\*), 0.001 (\*\*) and 0.05 (\*), except for those denoted by NS.



**Figure 3.** Mean values of time elapsed between wilting point ( $\psi = -1.5$  MPa) and dryness ( $\psi = -25$  MPa) called  $\Delta t_{total}$  under (a) drying or (b) wetting process of a minesoil amended with different types of sewage sludge, measured at sampling time one (S1) and two (S2). Different letters indicate significant differences for each sampling ( $p < 0.05$ ).



**Figure 4.** Relationships between soil organic carbon (SOC) with water holding time ( $\Delta t_{total}$ ), as the time required to change gravimetric water content from wilting point (-1.5 MPa of suction) to dryness (-25 MPa of suction) and vice versa, at sampling one (S1).



**Figure 5.** Mean values of gravimetric water contents at wilting point ( $W_{1.5}$ ), critical point of suction regime change ( $W_c$ ), and dryness ( $W_{25} = \text{GWC at suction at } -25 \text{ MPa}$ ) of a minesoil without sludge ( $O_{MS}$ ) and treated with composted sludge from Blanes ( $C_{BL}$ ), Manresa ( $C_{MR}$ ) or Vilaseca ( $C_{VL}$ ), or thermally-dried sludge from Besos ( $T_{BS}$ ), Mataró ( $T_{MT}$ ) and Sabadell ( $T_{SB}$ ), measured at sampling time one (S1) and two (S2). Different letters indicate significant differences for each sampling ( $p < 0.05$ ).

Under the wetting process, the observed increases in water holding time ( $\Delta t_{\text{total}}$ ) time could be a consequence of a reduction in minesoil wettability, as occurred at S1 for composted sludge amendment, or to increases in soil organic carbon contents [2] (Figure 3) and persistent hydrophobic substances from the applied sewage sludge [15]. Under the drying process, a positive relationship between  $\Delta t_{\text{total}}$  and SOC was also observed (Figure 4). Possibly, increases in water holding time were related to modifications of soil porosity produced by sludge amendments in the short term, as observed by Sort and Alcañiz [16] in other sludge treated soils where pore irregularity highly limited soil water drainage.

Finally, mean values of gravimetric water content retained at wilting point, at critical points where suction regime changed, and at dryness (soil suction = -25 MPa) were higher in sludge treatments, mainly at wilting point in S1 (Figure 5). Since water holding time ( $\Delta t_{\text{total}}$ ) was affected by sludge amendments, but no changes in the loss or uptake rates of water ( $a_1$  and  $a_2$  parameters) were observed, the influence of composted and thermally dried sludges on water retention of this minesoil can be mainly explained by changes in the adsorption-desorption properties of pore wall surface, rather than changes in soil structure or aggregate stability.

## CONCLUSIONS

The time dependence of wetting and drying processes was observed in a minesoil amended with composted and thermally dried sludges. Soil water hysteresis could be identified by differences in soil water retention and by the elapsed time for wetting and drying.

The critical point ( $\psi_c$ ,  $w_c$ ), where low suction regime change to high suction regime, was determined by timing the soil wetting and drying processes. Water holding time ( $\Delta t_{\text{total}}$ ), defined as the time to retain water in soil, was affected by sludge amendments for both the wetting and drying cycles.

Composted sludges decreased the wettability and water loss of the minesoil in the short term (one month after sludge amendments). When considering longer time frames (one year), wettability was higher in the minesoil treated with thermally dried sludge from Besós and Sabadell, while losses were only higher in the latter.

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