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RESEARCH ARTICLE

Effect of liquid humus and calcium sulphate on soil aggregation

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Abstract

An *Ultic Haploxeralf* soil collected from ridges built into the slopes of the Coastal Mountain Range of Central Chile was used in applications of humic and fulvic acids (HFA) extracted from sludge from sewage treatment plants, in combination with gypsum ($CaSO_4$). A total of 12 treatments were applied by combining four doses of HFA (0, 20, 30 and 40 t ha^{-1}) with three doses of gypsum (0, 1.2 and 6.0 t ha^{-1}). The effect of these treatments was assessed using three indicators of the degree of soil aggregation: macroporosity (MA), bulk density (Da) and microinfiltration (MI). The experiment was set up in a laboratory using a completely randomised design (CRD). Factorial variance analysis was also performed using two factors: gypsum in three doses and HFA in four doses. For all three indicators, MA, Db and MI, it can be seen that there is interaction between the HFA treatments and the gypsum treatments in the studied soil. The macroporosity increased with HFA treatments with 20 or 30 t ha^{-1} (9%) and with gypsum of 1.2 t ha^{-1} plus 20 t ha^{-1} HFA (11%). It can also be seen that the gypsum treatments have no effect on Db when HFA is not applied. Microinfiltration is the variable that shows the greatest effects from the treatments applied. The results also clearly show that high amounts of HFA (40 t ha^{-1}) and of gypsum (6.0 t ha^{-1}) cancel out the differential effects and cause negative effects on the three indicators for the studied soils. The use of liquid humus and its combination with calcium sulphate (20/1.2 t ha^{-1}) improves soil aggregation.

Keywords: Erosion, humic acid, gypsum, contour farming

Abbreviations: HFA (Humic and Fulvic Acids), MA (soil macroporosity),Db (soil bulk density),MI (soil microinfiltration rate of water)

1. Introduction

The use of ridges on sloped hillsides is a farming technique that offers agricultural advantages but which eventually leads to the degradation of the soil. In terms of its advantages, the technique improves edaphoclimatic limitations such as soil depth and drainage, and decreases the risk of frosts (Gardiazabal *et al*., 2004; Gardiazabal, 2007; Lemus *et al*., 2005). Its disadvantages with regard to the soil include degradation, mixture of soil horizons and total or partial elimination of vegetal coverage, which increases the possibility of erosion due to rainfall (Kay, 2000; Plante *et al*., 2005; Sagredo, 2005; Youlton *et al*., 2010). This means that the technique provides important economic advantages, while at the same time leading to negative processes for the environment.

One alternative to mitigate the effects of erosion is by improving soil structure. This can be achieved by using organic and inorganic aggregates. This technology modifies the physical properties of the soil, such as porosity, structural stability, infiltration and others which contribute to increasing water absorption in the soil, thus decreasing surface runoff and avoiding the erosion of soil particles (Cucunubá-Melo *et al*., 2011; Tejada and Gonzalez, 2008; Xiao-Gang *et al*., 2007; Zhang *et al*., 2007; Pagliai *et al*., 2004). Nevertheless, the use of soil aggregates on steep slopes involves high costs and presents practical difficulties. One way to resolve this difficulty would be the use of liquid products that can be applied via the irrigation system.

Liquid humus is a product that provides the same benefits as solid organic aggregates and which can be distributed through the irrigation system (Fataftah *et al.*, 2001; Stevensons, 1994). The humus is mainly composed of humic and fulvic acids (HFA), which represent between 60 and 80% of the organic matter in the soil (Porta *et al*., 2003). No difference has been found in the stability of the aggregates after application of humus formed from sludge, in comparison with the commercial humus made from leonardite (Merino, 2007). This researcher also identified an optimum dose of 21 t ha^{-1} HFA. However, the effect of higher doses or the effect of combining liquid humus with some other inorganic aggregate in order to improve soil aggregation has not been studied.

In addition, the application of calcium as an inorganic aggregate has important effects on soil aggregation. The flocculating power of Ca^{+2} generates bridges between the clays and the particles of organic matter. However, different sources of calcium, such as carbonates, sulphates, oxides can lead to different results (Alfaro and Bernier, 2008). Rivera (2007) performed different aggregate essays to prove that calcium sulphate at a dose of 1.2 t ha^{-1} increases the stability of aggregates in two types of soil. It should be noted that applying calcium through irrigation implies the need to use formulations of micronized calcium carbonate (Oster, 1982; Sawyer, 1982). The effects on the soil make it an efficient aggregate that is able to reduce susceptibility to soil erosion (Bronick and Lal, 2005).

Humic substances (HFA) together with calcium ions produce a synergic effect on soil aggregation. The calcium cations possess a preferential affinity for the carboxyl group in the HFA substances, which are essential for establishing organo-mineral bridges that are able to unite the clays (Macedo, 2002). Therefore, they are both principle elements of soil aggregation (Bronick and Lal, 2005; Majzik and Tombácz, 2007; Pedra *et al*., 2007).

In accordance with the above, it is believed that the use of liquid humus (HFA) applied via drip/micro sprinkler irrigation combined in some proportion with calcium sulphate can improve soil aggregation. The objective of this research, therefore, is to evaluate the effect of different doses of HFA and its interaction with two doses of calcium sulphate on some physical properties of the soil that are indicative of its degree of aggregation.

2. Materials and Methods

2.1. Soil sampling and preparation

In order to perform the experiment under controlled laboratory conditions, soil samples were collected from ridges built into the slopes of the Coastal Mountain Range in Central Chile. The soil is an example of *Ultic Haploxeralf,* derived from granitic rock with a depth of between 50 and 100 cm and slopes with gradients between 20 and 50%. The samples were gathered at the Quintil farm owned by the *Pontificia Universidad Católica de Valparaíso* (PUCV) located in the district of Quillota, Central Chile (32º53´ S; 71º12´ W). The area has a temperate climate with a prolonged dry season and precipitation concentrated in the winter months, with average annual rainfall of 454 mm (CIREN, 1997; Santibañez and Uribe, 1990).

The samples were taken from the erodible soil surface, which is the surface stratum (0 – 20 cm in depth) (Pedra *et al*., 2007). In the laboratory the soil was dried in air and then sieved (< 2 mm). It was spread out in order to perform the physical and chemical characterisation in accordance with conventional methods of the PUCV Soils Laboratory (Table 1). Finally, 200 g of soil were deposited in 500 ml containers before applying the treatments.

Table 1. Physical and chemical soil characterisation with interpretation class.

COMPONENT	SIMBOL	VALUE	UNIT	CLASS
Sand	s	52.00	%	Sandy loam
Silt	St	28.00	%	
Clay	C	20.00	%	
Bulk density	B_d	1.29	$g\ cm^{-3}$	Medium
Water holding capacity	W_{HC}	34.60	ml	Medium
pH		7.65		Optimal
Electric conductivity	EC	0.40	$dS\ m^{-1}$	Not salty
Sodium	Na^{+1}	0.56	$mmol\ L^{-1}$	Low
Calcium	Ca^{+2}	1.23	$mmol\ L^{-1}$	Low
Magnesium	Mg^{+2}	1.79	$mmol\ L^{-1}$	Medium
Carbonate	$CO_3^{=}$	< 0.001	$mmol\ L^{-1}$	Low
Bicarbonate	HCO_3^{-}	1.40	$mmol\ L^{-1}$	Low

2.2. Extraction of HFA

The extract of humic and fulvic acids (HFA) was prepared from sludge from sewage treatment plants, using a method of alkaline extraction (Sanderson, 1994). Quantification of fats, proteins, carbohydrates and HFA in the extract was performed using the following procedure. The extract is degreased and its fat content then quantified through liquid-liquid extraction (Skoog, 2001), the protein level is then determined (Peterson, G. L., 1977), followed by carbohydrates (Dubois *et al,* 1956) and COD (AOAC, 2000). The degreased extract is then ultra-filtered with a 3000 Da membrane (Liu and Fang, 2002), whereupon the proteins, carbohydrates and COD of the filtrate are measured. The HFA correspond to the organic matter that is retained by the ultrafiltration, discounting the proteins and carbohydrates. The theoretical conversion factors used are 1.11 g O_2/g proteins; 1.06 g O_2/g carbohydrates; and 1.70 g O_2/g HFA (Table 2).

2.3. Treatments

A total of 12 treatments were applied by combining four doses of HFA (0, 20, 30 and 40 t ha^{-1}) with three doses of calcium sulphate ($CaSO_4$) (0, 1.2 and 6.0 t ha^{-1}). The control treatment (T_0) corresponds to the absence of $CaSO_4$ and HFA.

Table 2. Secondary sludge composition

Compound	Total solid (TS) mg g^{-1}	%
Humic substances	89.0 ± 8.2	68.4
Proteins	16.3 ± 1.4	12.5
Carbohydrates	5.2 ± 0.7	4.0
Fat	18.1 ± 2.6	13.9
Other	1.6 ± 0.4	1.2

2.4. Determining the application dose

2.4.1. Humic and fulvic acids

In order to establish the equivalent doses for the laboratory tests, the weight of one hectare was estimated as 2.4 x 10^3 t ha^{-1}, considering a depth of 20 cm and a bulk density of 1 g cm^{-3} (Table 3). As the total volume of the solution applied exceeded the water retention capacity of the soil, it was necessary to divide the applications into 11 weekly cycles until the required volume was reached (Table 4). For both the control treatment (HFA = 0; $CaSO_4$ = 0) and the treatments of 20 and 30 t ha^{-1} HFA, distilled water was applied until the maximum solution volume applied in the treatment of 40 t ha^{-1} HFA was reached. The experiment was extended for one more cycle, completing a total of 12 weeks.

Table 3.Equivalent doses of HFA for experimental containers.

HFA t·ha^{-1}	Volume(*) cm^3	Weight(**) g
0	-	-
20	210	1.7
30	315	2.5
40	420	3.3

(*) Total volume applied per container of HFA solution of 8 gL^{-1}
(**) Equivalent weight of HFA applied per container

Table 4. Volume of HFA applied per treatment and per cycle. The total volume after all cycles is also shown along with its equivalence in doses in t ha^{-1}.

	Units	HFA, t ha^{-1}		
		20	30	40
Cycle 0	ml	70	70	70
Cycle 1-10	ml	14	25	35
Total	ml	210	315	420

2.4.2. Calcium sulphate

The amount of calcium applied was calculated for the required dose in accordance with the equivalency between the weight of the arable layer of one hectare and the amount of soil in the container. As with the case of the HFA, the amount per cycle was calculated. Therefore, when applying a dose of 6.0 t ha^{-1} of $CaSO_4$, 0.5 g of Ca^{+2} is required, which is equivalent to 2.15 g $CaSO_4$ •$2H_20$ per container. The calcium was applied in the same cycles along with the HFA. The calcium sulphate was therefore crushed and stirred to dilute it in the distilled water (Table 5).

Table 5. Dose of $CaSO_4$ in 200 g of soil

Calcium sulphate kg ha^{-1}	Ca/200 g suelo g	$CaSO_4(2H_20)$ g
0	-	-
1200	0.1	0.43
6000	0.5	2.15

2.5. Soil aggregation indicators

2.5.1. Macroporosity, MA

Macroporosity corresponds to the volume of pores considered as big. The term big is fairly ambiguous, macropores are those > 15 or in some cases > 100 µm in diameter (Bouma *et al*., 1977; Kadžienž *et al*., 2011) or those described as big as holes made by worms, roots and cracks. This study considers macroporosity as the volume of water extracted from the saturated soil under a pressure of 33.4 kPa (Arriagada *et al*., 1999).

2.5.2. Microinfiltration, MI

A mini-disk infiltrometer (MDI) (Decagon Devices, Inc.) was used to measure this indicator. The MDI measures the volume of water (ml) that passes from the infiltrometer to the soil in a set time. The MDI is placed on the surface of the soil, the suction exerted by the soil on the porous disk breaks the surface tension of the water and the water begins to seep out of the infiltrometer into the soil (Robichaud *et al*., 2008). In this study, infiltration was measured over 5 minutes at intervals of 1 min.

2.5.3. Bulk density, Da

Bulk density was measured using the cylinder method. Metal cylinders 1.15 cm in length and 2.10 cm in diameter were used to collect soil samples. The soil was then weighed and dried at 105 °C for 48 h. The bulk density is the ratio between the dry weight of the soil and the volume of the cylinder (MAPA, 1986).

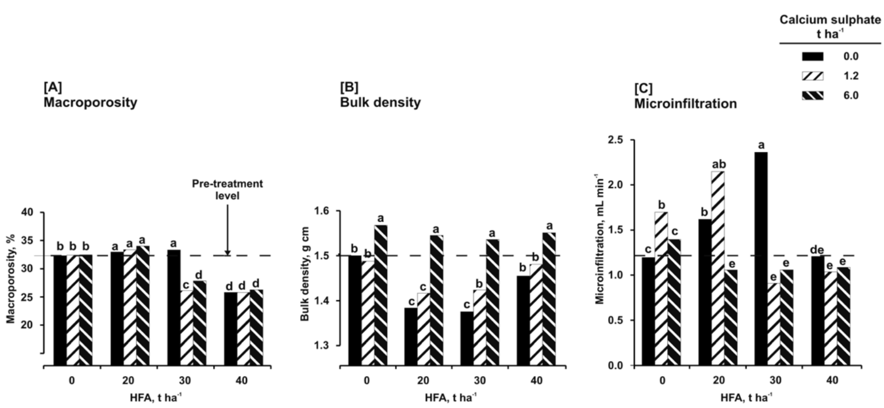
2.6. Experimental design and data analysis

The experiment was set up in the laboratory in a total of 48 containers distributed in a completely randomised design (CRD) with 12 treatments and 4 repetitions. The data were analysed in a 4 x 3 factorial system.

Factorial variance analysis was performed using two factors: $CaSO_4$ in three doses and HFA in four doses. In order to identify differences between the treatments, the multiple-comparison Tukey test was applied ($p \leq 0.05$). Statistics program Minitab, version 16, was used to perform the calculations.

3. Results

It can be seen both for MA and Db and MI that there is interaction between the HFA treatments and the gypsum treatments. In general, the results indicate that the mean doses of HFA and/or gypsum produce favourable effects on the soil structure indicators; on the other hand, higher doses of both compounds produce an unfavourable effect on the indicators (Figure 1).



For each soil variable (A, B, C) similar letters indicate that no significant difference was found (Tukey, p < 0.05)

Figure 1. Effect of the four doses of humic acids (HFA) and the three doses of gypsum on the soil aggregation indicators: A. Macroporosity; B. Bulk density; C. Microinfiltration

3.1. Macroporosity (MA)

The effects of gypsum and HFA on the macroporosity of the soil differ depending on the combination of the elements and their respective doses, and can be grouped into three response categories. MA increases an average of 10% when 20 t ha^{-1} HFA is applied with 1.2 t ha^{-1} gypsum, or with 30 t ha^{-1} HFA without gypsum. The gypsum dose of 6 t ha^{-1} produces a detrimental effect on the soil, irrespective of the HFA dose. The same detrimental effect is obtained with 30 t ha^{-1} HFA plus 1.2 t ha^{-1} gypsum, or 40 t ha^{-1} HFA without gypsum (Figure 1A).

3.2. Bulk density (Da)

It can be seen that there is no effect from the gypsum treatment on bulk density when no HFA is applied. With HFA treatments of 20 or 30 t ha^{-1}, without gypsum, a reduction (10%) can be seen, and with the HFA treatment of 40 t ha^{-1} there is no difference from the control sample. In addition, with gypsum at 1.2 t ha^{-1} and HFA of 20 t ha^{-1}, bulk density falls 10% and there is no difference with HFA at 30 t ha^{-1}. Finally, all HFA treatments with 6.0 t ha^{-1} of gypsum increase bulk density by an average of 5% (Figure 1B).

3.3. Microinfiltration (MI)

Microinfiltration is the variable with most sensitivity to the treatments used. Without adding gypsum, MI increases 60% when 20 t ha^{-1} HFA is applied, with regard to the control sample without HFA. This value increases to 81% when HFA is applied at a dose of 30 t ha^{-1}. When 20 t ha^{-1} HFA plus 1.2 t ha^{-1} of gypsum is applied, an increase of 49% is obtained, which is similar to the increase seen with 20 t ha^{-1} HFA without gypsum. Finally, the opposite effect is seen with doses above 30 t ha^{-1} HFA together with gypsum. The application of 30 or 40 t ha^{-1} HFA reduced MI by 27% with 1.2 t ha^{-1} of gypsum and by an average of 25% with 6.0 t ha^{-1} of gypsum for all HFA treatments (Figure 1C).

4. Discussion

The behaviour of the three indicators shows that the variables MA, Db and MI respond favourably or unfavourably to the treatments applied. While MA reaches its maximum with treatments of 20 t ha^{-1} HFA, 30 t ha^{-1} HFA and 20 t ha^{-1} HFA plus 1.2 t ha^{-1} gypsum, Db reaches a minimum with the same treatments and MI reaches its maximum with 30 t ha^{-1} HFA.

Unfavourable effects for the soil are seen for all three indicators for the treatments of 40 t ha^{-1} HFA plus 1.2 t ha^{-1} gypsum and the combination of 20, 30 and 40 t ha^{-1} HFA with 6.0 t ha^{-1} gypsum. The favourable effect caused by treatments with 20 and 30 t ha^{-1} HFA is similar to what was found by Merino (2007) with liquid humus and is more effective for increasing the stability of the aggregates and reducing bulk density than the application of biosolids in doses of 3 or 5% (w/w) (Salazar *et al*., 2012; Medina and Azcón, 2010; García-Orenes *et al*., 2005).This confirms the increase in aggregation and structural stability of the soil which can in turn decrease the possibility of erosion (Bounani, 2002; Bronick and Lal, 2005; Graetz, 1997; Pagliai *et al*., 2004; Salgado, 2001).

Furthermore, the results obtained clearly show that high amounts both of HFA (40 t ha^{-1}) and of gypsum (6.0 t ha^{-1}) cause negative effects in the three soil indicators included in this study. This fact coincides with results from the application of humus in a solid state (38 to 80 t ha^{-1}) which reduces macroporosity and increases bulk density (Amer, 2012; Macedo, 2002). Studies reveal that the application of high doses of gypsum (5 to 5.7 t ha^{-1}) reduces infiltration and seedling emergence due to the formation of a surface seal (Borselli *et al*., 1996a; Borselli *et al*., 1996b; Roth *et al*., 1991).

5. Conclusions

The use of liquid humus and its combination with gypsum improves soil aggregation. The hypothesis of this study is achieved by combining 20 t ha^{-1} HFA with a low dose of gypsum, corresponding to 1.2 t ha^{-1}. Therefore, the present research represents a contribution to minimising the dose of HFA and gypsum (20/1.2 t ha^{-1}), maximising the beneficial effects on indicators that are closely related to reduce soil erosion. In addition, the application of these aggregations in liquid form constitutes a significant contribution as it allows distribution over space and time through mechanised irrigation systems. It is important to mention that for field testing, liquid or soluble formulation of calcium must be used, such as micronized calcium carbonate.

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