Water **2015**, *7*, 2840-2850; doi:10.3390/w7062840

OPEN ACCESS

water

ISSN 2073-4441
www.mdpi.com/journal/water

Article

Hydraulic Behavior and Chemical Characterization of Lapilli as Material for Natural Filtering of Slurry

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Academic Editor: Miklas Scholz

Received: 13 April 2015 / Accepted: 5 June 2015 / Published: 15 June 2015

Abstract: Livestock effluents are a beneficial nutrient supply for crops, whereby their use is critical to ensure the sustainability of the farms global management. However, they can cause serious ecological problems if misused, polluting soils and groundwater. Combining “soft technology” and local materials is a low cost solution in terms of finance and energy. The REAGUA project (*REuso AGUA*, Water reuse in Spanish) analyzes the possibility of using “picon” (lapilli) as a material for the treatment of liquid manure from ruminants, for later use in subsurface drip irrigation system to produce forage and biofuels, in which the soil acts as a subsequent advanced treatment. A three-phase system, in which the effluent was poured with a vertical subsurface flow in an unsaturated medium, is designed. In order to determine the management conditions that optimize the filter, it was necessary to characterize the hydraulic behavior of lapilli and its ability to remove substances. Using three lapilli-filled columns, unsaturated flux, and a ruminant effluent, the reduction of chemical oxygen demand (COD), biochemical oxygen demand after 5 days (BOD_5) and ammonia, phosphorus and suspension solids (SS) obtained was over 80%, 90%, and 95% respectively, assumable values for irrigation.

Keywords: lapilli; slurry; advanced treatment; forage species

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1. Introduction

Bovine intensive farming in the Canaries represents 61% of total livestock and generates effluents that must be stored on farms and appropriately treated. These constitute a beneficial supply of nutrients for crops that are critical to ensure the sustainability of the management of farms. However, if used improperly, they can cause serious ecological problems. In this sense, agricultural intensification has changed the livestock effluent consideration from sub-product to a waste.

The treatment of slurry with local materials and technologies of low intensity results in low energy consumption and low financial costs. The REAGUA project analyzes the use of “lapilli” as a material for livestock wastewater treatment, in the same way as Baxter [1], who mentioned that volcanoes are a source of ore deposits and an important source of industrial material.

The goal is that the effluent produced is used in a drip irrigation system, for which the treatment should essentially be reduced in suspension solids (SS), COD, BOD_5 and nutrients, following the recommendations of Pescod and Ayars [2,3].

Studies of other authors [4], who used a treatment system in which one of its phases was filled with lapilli and which applied a horizontal subsurface flow, found a good reduction of organic matter (MO), the SS and pathogens, but also some risk of transmission of Salmonella. Therefore, improved health security that enables a subsurface drip irrigation (SDI) system is essential. In this regard, the system proposed here uses the soil as an advanced treatment medium so that the effluent is applied using the SDI to produce biofuels and forage.

Regarding the treatment conditions, in an interesting study, Zurita and White [5] compared the efficiency of various unconventional wastewater treatment systems, which were designed to improve the quality of irrigation water without producing a significant reduction nutrient input (these goals match those of the REAGUA project). These authors concluded that the most effective treatment is the subsurface one, by using a vertical two-phase flow (better than the horizontal flow), in which the reduction obtained was 85% ammonium (these authors associated it with an increase of nitrate) while *E. coli* reduction was 99.99%. The system designed by these authors coincides with the treatment used in this study, in which the effluent is introduced with a vertical subsurface flow in an unsaturated medium flowing through three phases (in series). The aim of this study is to obtain the water quality parameters after filtration, by reducing pollutants, to allow a sustainable livestock effluent irrigation using lapilli as a filter material and technologies of low intensity.

2. Materials and Methods

2.1. Behavior of Lapilli in Columns

A study was conducted in the laboratory with three columns of cylindrical glass 0.70 m and 0.048 m in diameter, filled with lapilli to a height of 0.55 m, sifted with a mesh of 0.002 m. The three columns were arranged in series and simulated a purification system with a vertical unsaturated flow.

2.1.1. Characterization of the Hydraulic Behavior of the Columns

By using distilled water, the following parameters were determined: bulk density (da), the ratio between the mass of dry soil (Ms) and total soil volume (Vt). These parameters were also determined and expressed as a percentage of the total volume of soil: real density (RD), the ratio of the dry soil mass (Ms), the volume occupied by the solid particles (Vs), the volume occupied by the pores, and porosity (ε).

For each column, the following parameters were measured: (i) the amount of water needed to reach saturation; (ii) the amount of free water, by measuring the amount of water lost by drainage over time; and (iii) the amount of water retained in the pores, which is the difference between the saturation and the volume lost by drainage.

The amount of volumetric content over time was calculated for each of the columns. These were the time intervals: 0, 0.5, 1, 2, 3, 4, 10, 20, 30 and 40 min.

2.1.2. Characterization of Lapilli as Filtration Medium

Using Non-Adsorbable or Biodegradable Substances

A tracer substance (Br at 50 $\text{mg}\cdot\text{L}^{-1}$) with low adsorption capacity was used. The solution was injected with a peristaltic pump at a flow rate of 1 $\text{mL}\cdot\text{min}^{-1}$. Once the column is in unsaturated conditions, and contains only retained (distilled) water, the Bromide injection starts. The concentration of the solution over time, once it has gone through each of the columns, was collected and analyzed by an ion chromatography. The sampled time intervals were the following: 0, 100, 120, 140, 180, 200, 240, 300 and 360 min (the latter being the time when all bromide was recovered).

Using Adsorbable Substances

A phosphate solution (as an example of adsorptive substance) at a concentration of 50 $\text{mg}\cdot\text{L}^{-1}$ (equivalent to 18.93 mg $\text{P}\cdot\text{L}^{-1}$) was used. As in the previous characterization, a peristaltic pump was used at a flow rate of 1 $\text{mL}\cdot\text{min}^{-1}$. The concentration of phosphate over time in each of the columns was measured by an optical emission spectrometry (ICP_OES).

Using a Livestock Effluent

In this test, the slurry was manually applied to each column at a rate of 10 mL every 10 min. Forty minutes were necessary to obtain a constant flow of 1 $mL\cdot min^{-1}$. Thus, the first sample (40 mL) was collected at 60 min. Half of the sample (20 mL) was used to fill the following column and the other 20 mL to be analyzed. The second and third columns started their wetting time in minutes 100 and 200, respectively, thus starting their sampling in minutes 160 and 260. The following sampling times were: 100, 140, 180, 220, 260, 300, 340, 380, 420, 460, 500, 540, 580, 600, 640, 700 and 740 for the corresponding columns.

2.2. Characterization of Slurry

Physic-chemical analysis of the samples collected in each of the columns was performed, determining pH, Electrical Conductivity (EC), nitrate, ammonium, COD, BOD_5, suspended solids and total phosphorus content, and major metal constituents (Na, K, Ca, mg, B, Cu).

2.2.1. pH and Electrical Conductivity (EC)

For the determination of these parameters, only two samples were taken: one before entering column 3 and the other one at the output of column 1.

2.2.2. COD and BOD_5

COD was estimated from the amount of total organic matter in each of the samples, by using a test kit (reference 0-29, 1500, Nanocolor CSB). The BOD_5 was obtained by measuring oxygen by selective electrode for 5 days. For this measure, 300 mL-gauged bottles -following the standardized BOD_5 method [6] were used.

The determination of these parameters is performed on four samples, the entry in column 3 (M1), the output from column 3 (M2), the output from column 2 (M3) and a final output from column 1 (M4). The percentage of COD reduction was calculated with the ratio between the value obtained after each of the columns and the initial value.

2.2.3. Suspended Solids (SS)

SS were measured from a sample taken before entering the column 3 and another one at the output from the column 1, following the standard methods [6].

2.2.4. N Determinations

One test kit (reference 985, 088, Nanocolor) was used to determine total N (N_T). By using segmented flow equipment, the ammonium present in the initial effluent (before entering column 3), in the output from the columns 3, 2 and 1, over time was determined.

Chromatograph concentration of nitrates and nitrites was measured, thus, determining organic and inorganic nitrogen (N_o and N_I, respectively).

2.2.5. Total Phosphorus

P concentrations present in the initial effluent, at the output from column 2 over time, and the concentration at the output from column 1, were analyzed by optical emission spectrometry (ICP_OES).

2.2.6. Other Nutrients

Determination of Na, K, Ca, Mg, B, and Cu was performed by using optical emission spectrometry (ICP_OES).

3. Results and Discussion

3.1. Hydraulic Behavior of the Columns

The results of the measurement of (i) the amount of water needed to reach saturation; (ii) the amount of free water; and (iii) the amount of water retained in the pores are presented in Table 1. Compared to other lapilli studies in the Canary Islands, the results of these quantities of water in this study are low, as some authors found values between 1.12 and 2.49 $g \cdot cm^{-3}$ with average values of 1.7 $g \cdot cm^{-3}$ [7]. The d_r values are also low, as these authors' values are between 2.45 and 3.70 $g \cdot cm^{-3}$, and lower than the density of basalt, which is 3.01 $g \cdot cm^{-3}$, [8], demonstrating the vacuole nature of pyroclastic. As for porosity, the samples show intermediate values, being those of the aforementioned study, between 18% and 57%. The micro porosity defined by Miller Donahue [9], considers micro pores as those able to retain water after drainage, among 0.1 and 80 µm. The micro porosity of the studied lapilli is lower than those obtained in the Canary Islands (12.33% to 35.02%), although a different methodology to determine them was used in this study.

Table 1. Weight, height, column volume (V_c), bulk density (d_a), real density (d_r), volume of solid (V_s), porosity (ε) and amount of water saturation (A_s), free water (A_l), and micro porosity (M).

Column	Weight (kg)	Height (m)	V_c (m^3)	d_a ($kg \cdot m^{-3}$)	V_s (m^3)	d_r ($kg \cdot m^{-3}$)	ε %	A_s (m^{-3})	A_l (m^{-3})	M %
1	1.117	0.55	0.89×10^{-3}	1260	0.488×10^{-3}	2290	45	0.397×10^{-3}	0.091×10^{-3}	8.56
2	1.130	0.55	0.89×10^{-3}	1280	0.460×10^{-3}	2460	48	0.425×10^{-3}	0.096×10^{-3}	4.24
3	1.126	0.55	0.89×10^{-3}	1270	0.466×10^{-3}	2420	47	0.419×10^{-3}	0.090×10^{-3}	9.79

Figure 1 presents the volumetric water content over time for the three columns, showing a rapid drainage. As can be seen, in the first 30 s, drainage between 25% and 45% of the water occurs. After 4 min, only 11 to 20% of water remains, and after 40 min, water retained is only between 5.5% and 6.0%. Therefore, there is a significant preferential flow, thus columns filter better in unsaturated conditions. When pumping a water flow of 1 $mL \cdot min^{-1}$, 100 min are required to reach the balance condition (collecting 1 $mL \cdot min^{-1}$).

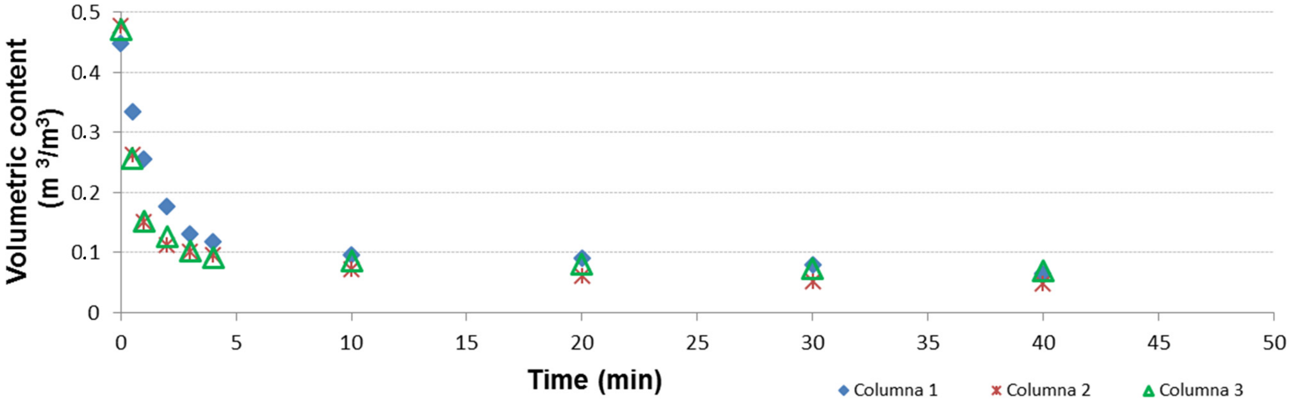
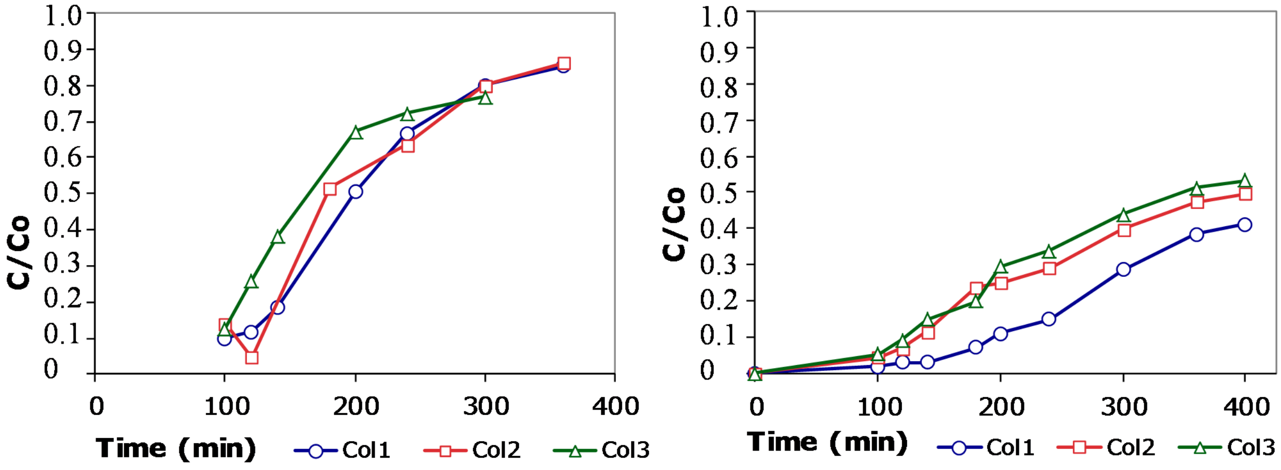


Figure 1. Volumetric water content versus time in each of the columns.

3.1.1. Characterization of the Behavior of Columns with Non-Absorbable or Biodegradable Substances (Use of Bromide)

In Figure 2a, Br concentration over time, after going through the column, was presented. It is observed that, after 100 min, Br is starting to be collected, and its recovery is very rapid, thereafter yielding almost 90% of the amount initially provided after four hours. These results agree with other studies, which state that Br is an ion that has minimal reaction behavior with the environment, thus it is used as a tracer in soil studies [10].



(**a**) (**b**)

Figure 2. Bromide tracer concentration (**a**) and phosphate (**b**) *versus* time of sampling.

3.1.2. Characterization Debugger Potential of Absorbable Substances (Use of Phosphate)

Figure 2b, presents P concentration *versus* time, showing that 60% of P in the column is recovered. These results agree with those obtained by other authors [11]. Comparing Figure 2a,b, column 1 is the one with the maximum adsorption, as it presents less preferential flow.

3.2. Characterization of Slurry

3.2.1. pH and EC

In Table 2, the values of pH and EC of the effluent were presented before entering column 3 and the output of column 1. The reduction in this study contrasts with the observed EC increase obtained by Quintero [11]. In their study, when lapilli was wetted with distilled water, there was an EC increase in the second and third day after being moistied.

Table 2. Values of pH and EC of the effluent before entering column 3 (pHe and ECe) and output column 1 (pHs and ECs) at 25 °C.

pHe	ECe (μs·cm^{-1})	pHs	ECs (μs·cm^{-1})
7.58	4740	7.10	863
7.61	4706	7.09	864
7.60	4730	7.01	860
Average	Average	Average	Average
7.60	4740	7.07	862.33

3.2.2. COD, BOD_5, and SS

In Table 3, the results of COD analysis were presented. The reduction percentage of the total MO is 78%. The initial value of BOD_5 in the effluent is 380 mg·L^{-1} O_2. Regarding SS, a reduction percentage of 92.48% from the effluent is obtained, because 10.53 mg·L^{-1} enters while 0.79 mg·L^{-1} comes out. The next step of this project will be to determine the useful life of "lapilli" and the best way to reclaim it once used.

Table 3. Effluent COD values along the system.

Sample	COD mg·L^{-1}
M1 (before entry to the column 3)	526
M2 (at the exit from column 3)	425
M3 (at the exit from column 2)	320
M4 (at the exit from column 1)	115

3.2.3. Nitrate

Table 4 shows N quantity of the effluent (in its different forms), added at the entry and recovered at the output of the system. Additionally, N_O and N_I are expressed as percentage of this N_T (1 and 2). Percentages of different forms of N_I are also calculated. As seen in the table, 90% of N_T is inorganic nitrogen (N_I) (all in ammoniac form) and only 10% is organic nitrogen (N_O). After going through the columns, adsorption and transformation of organic and ammonia forms in nitrate (N) occur. As a result of this, removal or transformation rates obtained with lapilli unsaturated filtering are the following: 97% of the ammonium, 79% of N_I, 99% of N_O and, consequently, 81% of the N_T added by the effluent is removed.

Table 4. Values of total nitrogen (N_T), total organic nitrogen (N_O), ammonium (N_{NH4^+}), nitrites (N_{NO2^-}), nitrates (N_{NO3^-}), and total inorganic nitrogen (N_I) at the input and output of the three columns and the removal thereof.

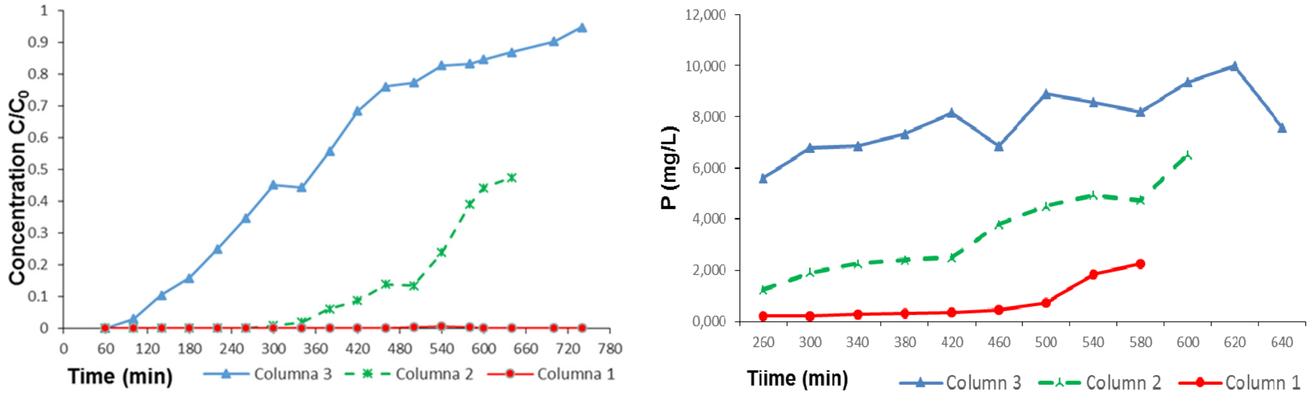
Source	Entry		Output		Removal
Parameter	**g**	**%**	**g**	**%**	**g Output/g Entry (%)**
Vol (L)	0.84		0.78		
N_T (g)	161.28	100	31.15	100	80.68
N_O (g)	15.60	9.7 (1)	0.15	0	99.04
N_{NH4^+}	145.68		3.89	12.5 (3)	97.33
N_{NO2^-}	--		3.09	10.0 (4)	
N_{NO3^-}	--		24.03	77.0 (5)	
N_I (g)	145.68	90.3 (2)	31.00	99.5 (6)	78.72

(1) N_0 (%) = (N_O/N_T) (3) N_{NH4^+} (%) = (N_{NH4^+}/N_I) (5) N_{O3^-} (%) = N_{NO3}/N_I)

(2) N_I (%) = N_I/N_T Entry (4) N_{O2^-} (%) = N_{NO2}/N_I) (6) N_I (%) = N_I/N_T) Output

3.2.4. Ammonium

In Figure 3a, ammonium concentrations over time in each of the columns were shown. Reduction yields of 10%, 50%, and 100% are obtained after filtering through columns 3, 2, and 1, respectively. This reduction is consistent with that obtained by Zurita and White [4], who mention a reduction of 85% (these authors associate this reduction with increased nitrate) by using a vertical subsurface flow in only two stages. Therefore, these results show that, in order to obtain a good reduction, at least two columns are required. The decrease in ammonium is consistent with the high adsorption capacity of this substance [12] and its nitrification capability. Nitrate was also determined, both in the effluent and at different time intervals, giving values below the limit of quantification, which suggests that, originally, all mineral N was in the form of ammonium. However, after five hours, some nitrification may have occurred.



(a) (b)

Figure 3. Values of ammonium concentration (**a**) and phosphorus (**b**) over time at the three columns.

3.2.5. Total Phosphorus

In Figure 3b, Ptotal concentrations (mg·L^{-1}) over time after filtration through each column are represented. The initial effluent concentration was 33 mg·L^{-1}, so the reduction of P is significant (33%) since it goes through the first column. After filtering through the second column, about an order of magnitude in reduction is obtained. The total observed reduction is enough to obtain less than 2 mg·L^{-1}. This result is consistent with the presence of organic P (which would be retained with the particles in suspension). In conventional sand irrigation filters, and by using treated water, only a 15% reduction of total P was obtained [13]; while Hernandez Moreno [7], working with artificial effluent, but with lapilli, obtained a significant reduction of phosphate, because organic compounds adhere to the interchangeability of lapilli. Therefore, the material chosen for the processing system is very suitable.

3.2.6. Metals

In Table 5, the filtration results for other nutrients from the effluent are presented. It is observed that, in general, after going through column 3, and after sufficient time has elapsed, a slight decrease in concentration is produced. However, after going through columns 2 and 1, a significant reduction occurs.

Table 5. Other nutrients concentration (mg·L^{-1}) from the effluent over time, are presented.

Column	K	Na	Ca	Mg	B
Initial effluent	329.4	448.4	169.2	75.9	1.02
C3-100	45.7	176.7	108.7	51.4	0.62
C3-300	250.5	413.4	164.6	78.7	0.95
C3-680	331.9	480.1	152.3	79.4	1.08
C2-100	5.9	36.8	19.4	12.0	0.20
C2-300	27.6	174.8	107.4	55.4	0.40
C2-460	101.5	298.8	157.8	70.8	0.55
C1-100	28.3	69.2	26.5	12.8	0.22
C1-300	23.3	62.4	23.8	12.1	0.19
C1-420	14.4	39.9	15.6	8.1	0.12

4. Conclusions

The treatment of ruminant slurry for later use in subsurface drip irrigation of forage species and biofuels, by using local materials (in our case, lapilli) and low intensity technologies (vertical filtration by using subsurface flow under unsaturated conditions, circulating three phases in series) has been shown as a feasible treatment due to its simplicity and low financial and energy cost.

The hydraulic behavior of lapilli and its ability to remove substances were characterized, by using three columns filled with lapilli and a real ruminant effluent at a discontinuous flow of 10 mL·min^{-1}. Reductions obtained for different parameters were the following: COD and N 80%, BOD5, 90%, while, for ammonium, SS and P exceeded 95%. These values can be accepted to be injected into a subsurface irrigation system, in which the soil acts as an advanced treatment. Our experiments show that it is feasible to reuse an effluent with a low intensity of treatment if subsequent irrigation management conditions let the soil act as an advanced treatment.

Acknowledgments

This work was supported by Ministry of Economy and Finance. Subprogram Basic Research Projects (CGL2012-39520-C03-03) and Research Canary Agency, of the Gobierno de Canarias (SolSubC200801000012). We also appreciate the cooperation of the Phytopathological Laboratory and Agri-Food and Agricultural Experimental Farm of Cabildo de Gran Canaria.

Author Contributions

Juan Ramón Fernández Vera, Jose Manuel Hernández Moreno, Vanessa Mendoza-Grimón and Maria del Pino Palacios-Diaz had the original idea for the study and, with all co-authors carried out the design. V. Mendoza-Grimón and MP Palacios-Diaz were responsible for follow-up of study participants. Nereida Falcón Cardona, Mendoza-Grimón, Idaira Hernández Brito and Juan Ramón Fernández Vera were responsable for carried out the analyses, while Jose Manuel Hernández Moreno, Sebastian O. Pérez Báez, Axel Ritter and María del Pino Palacios Díaz were responsible for data cleaning and analysis. Nereida Falcón Cardona, Mendoza-Grimón and Palacios-Diaz drafted the manuscript, which was revised by all authors. All authors read and approved the final manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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