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CHEMICAL AND AGRONOMIC CHARACTERIZATION OF SEWAGE WATER FROM THE CASTELLANO DEPOSIT, PINAR DEL RÍO

Caracterización química y agronómica de las aguas residuales del yacimiento Castellano, Pinar del Río

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ABSTRACT. The reuse of sewage water in agriculture results in a future need in many countries, since this sector employs almost 70 % of available freshwater, and there have been reports on critical levels of water deficit in many regions around the world. Cuba is not exempt from these problems, and its national politics towards water promotes the implementation of this practice. Therefore, in the current paper, an attempt has been carried out to characterize and to assess the quality of mining sewage water and its use in agriculture. To achieve successfully this aim, five punctual samples were collected from the creek "Biajaca", where sewage waters coming from Minas Castellano, in Minas de Matahambre, Pinar de Río are discharged. These five punctual samples to form one composed sample were combined and so the pH and electrical conductivity were measured. After filtering and keeping the proper conservation, the following elements Na, K, Ca, Mg, B, Al, Bi, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, Tl, Zn, As, Be, Sb, Se, Mo, V, Ti, S, P, Rb were determined through digestion in microwave and analysis by ICP-OES. On the other hand, the ions F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-} were determined by ion-exchange liquid chromatography. The results showed that it is a type of water characteristic of the study area, with an acid pH and salinity, permeability and toxicity indicators suitable for its usage in agriculture, as long as its acidity is pre-neutralized.

Key words: water quality, permeability, water reuse, salinity, toxicity

RESUMEN. La reutilización de las aguas residuales en la agricultura es una necesidad futura en muchos países, ya que este sector emplea casi el 70 % del agua dulce disponible y se han reportado niveles de déficit hídrico críticos en muchas regiones del mundo. Cuba no está exenta de estos problemas y su política nacional del agua promueve la aplicación de esta práctica. Por esta razón, en el presente trabajo se caracterizó y evaluó la calidad de las aguas residuales mineras para su uso en la agricultura. Se colectaron cinco muestras puntuales en el arroyo "Biajaca" donde descargan aguas residuales provenientes de las Minas Castellano, en Minas de Matahambre, Pinar de Río. Las cinco muestras puntuales se combinaron formando una muestra compuesta y se midió el pH y conductividad eléctrica. Tras el filtrado y la adecuada conservación, se determinaron los elementos Na, K, Ca, Mg, B, Al, Bi, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, Tl, Zn, As, Be, Sb, Se, Mo, V, Ti, S, P, Rb, mediante digestión con microondas y análisis por ICP-OES. Además, los iones F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-} se determinaron por Cromatografía Líquida de Intercambio Iónico. Los resultados mostraron que es un tipo de agua característica de la zona de estudio, con un pH ácido, con indicadores de salinidad, permeabilidad y toxicidad adecuados para su uso en la agricultura, siempre que se pre-neutralice su acidez.

Palabras clave: calidad del agua, permeabilidad, reutilización de aguas, salinidad, toxicidad

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INTRODUCTION

Humanity is growing at an accelerated rate and for its development it requires superficial and underground waters, which are increasingly scarce and of lower quality (1). Critical levels of permanent or seasonal water deficit have been reached and it is accepted as a fact that in a large part of the planet there is a water crisis (2,3). It is expected that by 2025, 80 % of the Earth’s population will live under conditions of high scarcity of water resources (4).

Agriculture is in fact the main user of fresh water in the world (5) and represents approximately 70 % of drinking water (6). Due to this, different competent administrations have considered the reuse of reclaimed wastewater as one more component of the water cycle and the legal regime for the reuse of treated water has been published (Royal Decree 1620/2007, BOE 294 of 7 December) and the United Nations World Report on the Development of Water Resources 2017 (7).

The treated wastewater, applied with adequate irrigation systems, should be the water of the future for the irrigated land under irrigation, optimizing the resources in those conditions that allow it (8). In some cases, the treated wastewater is of better quality than the water in the area, presenting a total adequacy for its use in irrigation (9). Plants need at least 14 elements for adequate nutrition and they usually obtain them from soil, fertilization and irrigation water. But the increasing anthropogenic activity and the consequent increase in the concentration of heavy metals have modified the chemical composition of the water and often limit its usefulness for irrigation (10). Hence, the reuse of wastewater requires the adoption of measures to protect public health. For this purpose, there are international standards that allow the safe use of treated wastewater (11,12).

In Cuba, the loss of water quality is also a growing problem. Some investigations have observed eutrophication of lakes and increased metal content in aquifer areas, as a consequence of high exploitation and, in surface waters, due to dumping of industrial and livestock waste insufficiently treated (13).

Severe drought problems have also occurred in recent periods that can significantly affect harvests in the coming years (14). Therefore, due to the convenience of giving a productive use to wastewater, the National Water Policy in Cuba adopts within its 22 principles to promote the reuse of these waters and introduce efficient techniques in agricultural irrigation (15).

The fundamental activity in “Minas de Matahambre”, Pinar del Río, is mining, but a small non-negligible part of the land is destined for agricultural use (16), which is why it is the objective of this work to characterize and evaluate the quality of the sewage water from mining activities in this area, for use in agriculture.

MATERIALS AND METHODS

Sampling and preparation of mining wastewater samples

Five point samples were collected at different sites of the “Biajaca” stream, where they discharge wastewater from the Castilian Mines, Pinar del Río, located at 22º37’27.50 “North Latitude and 83º59’34.96” West Longitude, at a height of 22 m a.s.l Sampling was carried out in April 2016, dry period with relatively low flow. Samples were collected every 30 m in five sites of the stream channel (Figure 1), following the sampling and preservation procedures established in the ISO standard for the design of sampling programs (17).

Figure 1. Sampling sites in the Biajaca stream, Minas de Matahambre, Pinar del Río

50 L were taken in the five sampled sites and combined to form a composite sample of 250 L. This volume of sample was collected in order to evaluate, in subsequent studies, the effects of residual water in tomato hydroponics. Next, pH and electrical conductivity were measured in triplicate and 1 L of the sample was conserved for subsequent analysis of alkalinity, in accordance with standard NMX-AA-036-SCFI-2001 (18). After homogenizing the composite sample, it was filtered using a filtration system with five-position ramps and vacuum pump and 0.45 µm cellulose nitrate filters. Three subsamples with a volume of 10 mL each were separated from the filtered sample, kept in plastic containers with HNO_3 (pH=2) and refrigerated until further chemical analysis.

Characterization of mining wastewater

0.7 g of each liquid sample was weighed in a 25 mL digestion tube and 4 mL of concentrated nitric acid and 1 mL of 33% hydrogen peroxide were added. The digestions were performed in a Milestone Ultraclave microwave heating up to 220 °C for 20 minutes and then rooted in 10 mL.

The total elements Na, K, Ca, Mg, B, Al, Bi, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, Tl, Zn, As, Be, Sb, Se, Mo , V, Ti, S, P and Rb were determined by analysis by atomic emission spectrometry with induction-coupled plasma (ICP-OES). The determinations were made in an atomic emission spectrometer model THERMO ICAP 6 500DUO with capacity to perform measurements in axial and radial, the instrumental conditions were similar in both modes: flow rate (nebulizer-auxiliary gas)=0.55-0, 5 L min^{-1}, power RF (W) = 1 150 W, speed of introduction of the sample=30 rpm, Number of repetitions = 2.

The solutions for the calibration were prepared by dilution with ultrapure water; from the certified multielemental mother-plasma Plasma CAL supplied by the company SCP Science. The F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-} ions were determined by Liquid Ion Exchange Chromatography (Metrohm) (19). The quality of the chemical analysis was evaluated by calculating the difference between anions and cations:

$$e = \frac{\sum_{i=1}^{n} cations \; - \sum_{i=1}^{n} anions}{\sum_{i=1}^{n} cations \; + \sum_{i=1}^{n} anions} * 100 = 4{,}58\ \%$$

RESULTS AND DISCUSSION

The “Biajaca” stream sampling site is located in the upper member of the San Cayetano formation, close to the drainage area of the Castellano deposit. It is formed by pelitic, carbonaceous schists, frequently calcareous, with intercalations and lenses of dolomitized limestones, sandstones and siltstones. The parameters that characterize the general composition of the composite water sample representative of the stream under study are shown in the Table 1.

Figure 2 shows the hydrogeochemical pattern of the water under study according to Fagundo *et al*. (2012) (1). According to the chemical composition of the same and considering that its acidity has been neutralized, it is classified as sulphated bicarbonated type calcium chloride magnesium sodium (SO_4=HCO_3> Cl-Na = Ca> Mg) (20), with a mineralization of 0, 21 g L^{-1} and a total hardness of 83.54 mg L^{-1} $CaCO_3$.

The results correspond to the studies carried out in this area by Ponce *et al*. (1997), which identified three horizons that feed mainly on deeper aquifers. Among them is the *Upper Jurassic aquifer Horizon*, located towards the south-east of Castellano. The chemical composition of these waters in their majority, are sodium sulphates and their values of mineralization and total hardness are close to those obtained in the present study (20).

The chemical composition of the water under study is acquired, in the case of sulphates, by the arrival of surface waters close to the mining area that have high concentrations of sulfuric acid as a result of the oxidation and hydrolysis processes of sulfide minerals (FeS2). Other major ions, such as sodium and chloride, showed a ratio in meq L^{-1} of Na/Cl equal to 1.38; higher than the threshold (0.862) established by Schlesinger *et al*. (2000) (21). This result indicates that the water under study retains the ionic ratio (Na/Cl) characteristic of seawater.

On the other hand, it is attributed that the majority elements Ca, Mg and alkalinity owe their contents in the water studied to the interaction of the same with the rocks of the San Cayetano formation.

In most of the elements, the most common oxidation states and simple ions prevail (Na^+, Ca^{2+}, Mg^{2+}, K^+, Fe^{2+}, Al^{3+}, Mn^{2+}, Sr^{2+}, Zn^{2+}, T^{l+}, Cu^{2+}, Ni^{2+}, Rb^+, Cl^-). While for N, S and B the ions and complex species predominate, NO3-, SO42- and H3BO3, respectively. At the pH of 4.36, the H_2CO_3 present in 98 % is the predominant species in the balance of carbonates; however, at neutral pH, bicarbonate ions (HCO_3^-) predominate. Figure 3 shows the distributions of the species of the elements: S, Mg, Ca, Al. The rest of the elements are almost entirely in a single species, obtained by hydrogeochemical modeling using the PHREEQC program with the base of MINTEQ data (22).

The evaluation of the quality of water collected for agricultural use was carried out according to the standards established by FAO 1985 on the basis of the following categories: salinity, alkalinity, toxicity and miscellaneous (11).

The combined water sample has a high content of hydronium ions, with a pH of 4.36; so it is not suitable for agricultural use according to FAO. The decrease in pH is due, among other factors, to the oxidation-dissolution processes that sulphides undergo when interacting with air and water, which leads to the formation of sulfuric acid. These processes also cause a significant enrichment in sulfates (23).

Similar pH values were obtained in environmental studies of the areas affected by the mining activity of the Santa Lucía deposit (16,24,25).

These pH values show the negative impact that the acid drainage of the mines causes on the superficial waters near the exploitation area.

Table 1. Parameters that characterize the chemical composition of the water coming from the Arroyo "Biajaca", Pinar del Río and reference values established by FAO and other environmental standards

Parameters	Measured value	Standard error	Reference (FAO, 1976)	Resolution 0631. Colombia	Rule Ecuador
pH	4,4	0,2	6,5-8,4	6-9	6,5-9
CE (µS cm^{-1})	270	15	200-750	-	-
Na (meq L^{-1})	1,02	0,02	0-40	-	-
Ca (meq L^{-1})	1,02	0,02	0-20	-	-
Mg (meq L^{-1})	0,65	0,08	0-5	-	-
K (meq L^{-1})	0,10	0,02	0-2	-	-
Fe (mg L^{-1})	0,41	0,07	5	2	0,3
Al (mg L^{-1})	0,28	0,00	5	-	0,1
Mn (mg L^{-1})	0,17	0,01	0,2	-	0,1
Sr (mg L^{-1})	0,07	0,02	-	-	-
Zn (mg L^{-1})	0,05	0,01	2	3	0,18
B (mg L^{-1})	0,03	0,01	0,75	-	-
Tl (mg L^{-1})	0,03	0,00	-	-	-
Cu (mg L^{-1})	0,01	0,00	0,2	1	0,02
Ni (mg L^{-1})	0,01	0,00	0,2	0,5	0,025
Rb (mg L^{-1})	0,01	0,00		-	-
Alkalinity (meq L^{-1})	1,03	0,03	1,5	-	-
Cl^- (meq L^{-1})	0,73	0,06	4	7,1	-
NO_3^- (mg L^{-1})	14	2	<30 for crops not sensitive to N	-	-
SO_4^{2-} (mg L^{-1})	51	3	-	250	-
Br^- (mg L^{-1})	0,10	0,00	-	-	-
R.A.Sadj	1,46	-	6	-	-

Electrical Conductivity (CE), Adjusted Sodium Adsorption Ratio (R.A.S adj.)

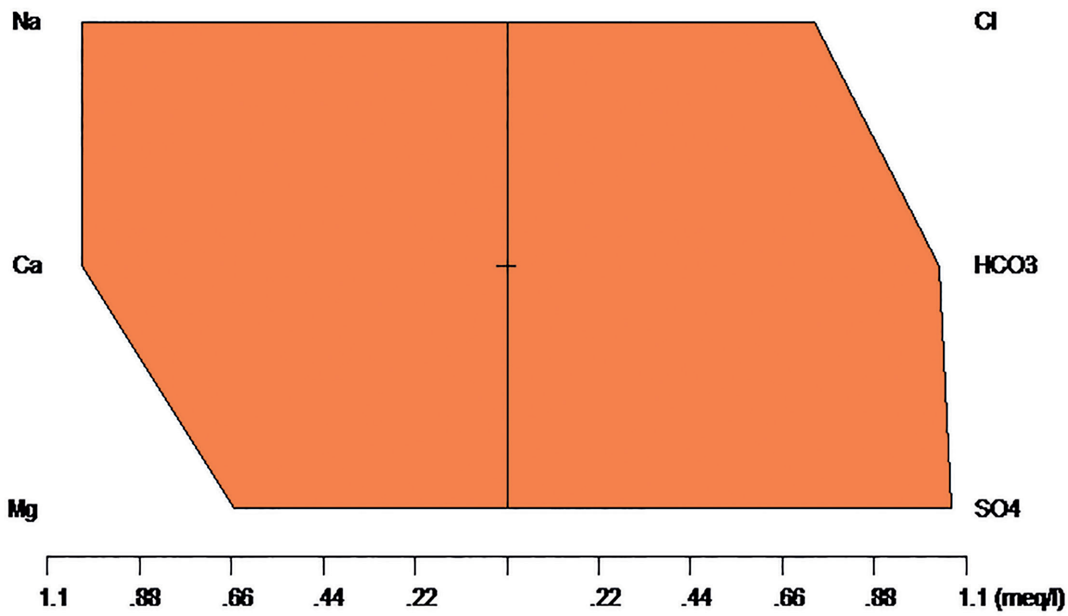

Figure 2. Hydrogeochemical pattern of the water collected in the Biajaca stream, Minas de Matahambre, Pinar del Río

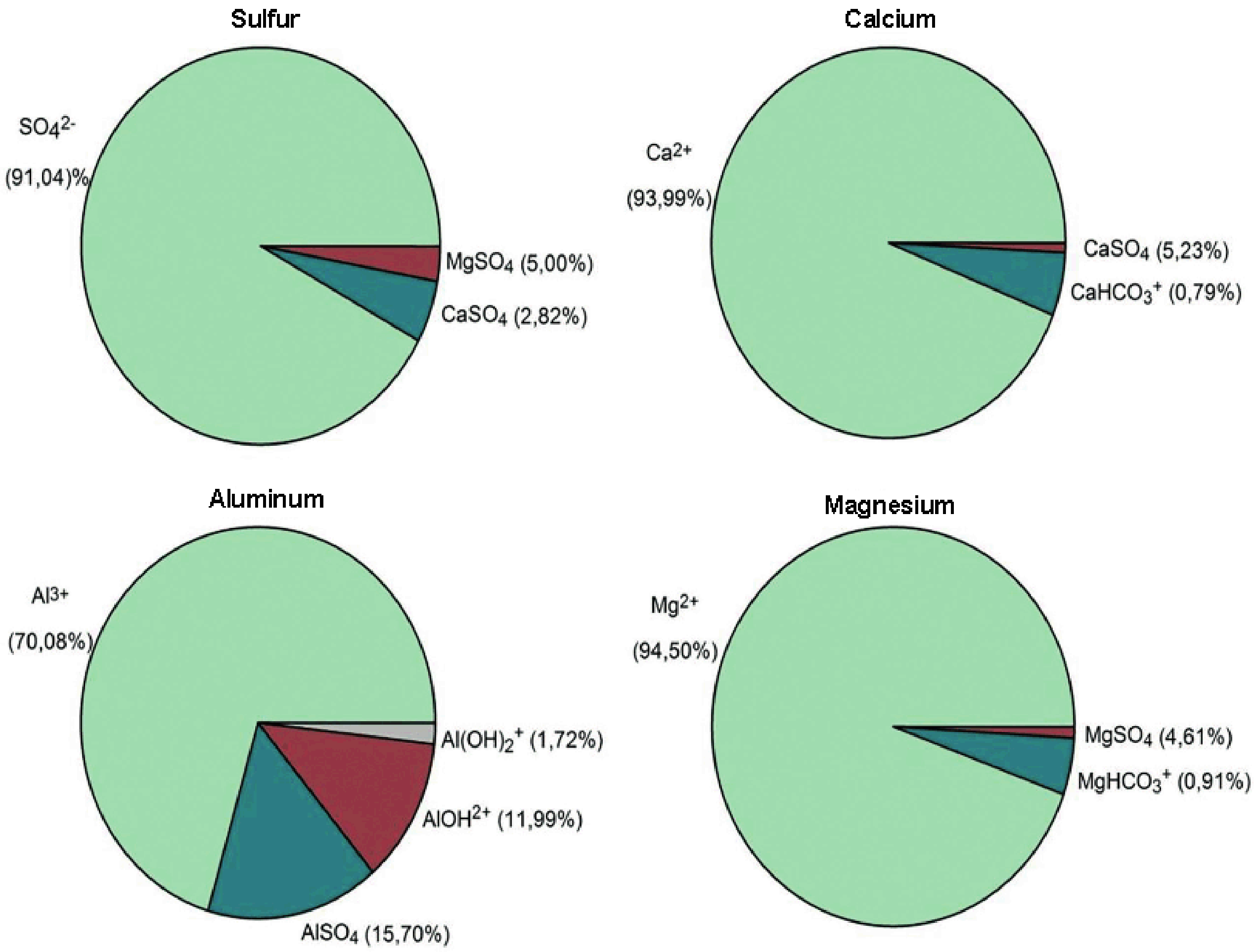

Figure 3. Distributions of the species Sulfur, Calcium, Aluminum and Magnesium in the water collected in the Biajaca stream

The electrical conductivity (C.E) in the water sample was 269.7 µS cm^{-1}, lower than the upper limit established by the FAO (750 µS cm^{-1}), so it is not a problem of soil salinization. However, salinization below 200 µS cm^{-1} represents a problem of water permeability in the soil. The electrical conductivity measured in the sample is very close to this value; therefore, it is water with possible difficulty of infiltration to the root zone.

The Adjusted Sodium Adsorption Ratio (R.A.S adj) was 1.46, less than the value established by the FAO. This indicator is related to the permeability of water in the soil and shows that the measured sodium content does not represent a risk of sodification. Therefore, the wastewater studied does not present limitations for its agricultural use.

The problems of infiltration of water into the soil are due, among other factors, to the combination of the effects associated with sodicity and water salinity. The simultaneous evaluation of the indicators R.A.S and CE in the FAO diagram (1985) showed that the collected sample shows a slight or moderate reduction in the infiltration rate in the soil. For these reasons its use is recommended without neglecting the possible corrosive effects

The concentration of all the elements analyzed (Table) is lower than the permissible limits established by FAO for its use in agriculture. Its absorption does not cause damage to crops and many of them (N, K, Ca, Mg, Fe, Mn, Zn, B, Cu and S) favor the nutrition of plants. The elements not reported in the table presented concentrations below the limit of detection in each case.

Different results were obtained by Milián *et al*. (2012) (16) and Cañete *et al*. (2011) (23), which found high concentrations of Al, As, Cd, Co, Cr, Cu, Fe Mn, Pb, Zn and SO_4^{2-} in surface streams from the Santa Lucia Mine (16,23). The concentrations of the toxic elements in the study water are all lower than those found by different authors (16). These differences are attributed to the fact that the sampling sites are not the same, the Biajaca stream is closer to the Castellano deposit than to the Santa Lucía one.

However, the distance between both deposits is only 2 km (26) with connected rivers that give the possibility of extending the contamination between the mining deposits. If so, another hypothesis regarding these differences found from 2012 to 2016, could be given by the possible dilution with uncontaminated waters, which constitutes an important environmental achievement. However, these criteria are not conclusive since the authors do not report the data related to the flow and time of the year of sampling.

When comparing the results with other international standards, it was observed that the concentration of all the elements is lower than the maximum permissible values in the discharges of mining residuals to surface water bodies (27). However, the levels of Al and Fe, despite being lower than those established in Resolution 0631, exceed the maximum limits allowed (0.1 and 0.3 mg L^{-1} respectively) for the preservation of flora and fauna in warm fresh waters established in the Ecuadorian Standard of environmental quality and discharge of effluents (28).

The total concentration of salts was 5.91 meq L^{-1} and the ratio of the percentage of sodium to the total content of cations present in the water was 35.7 %. The arrangement of this type of water in the Greene diagram (FAO) allowed concluding that good quality water is available for irrigation, provided that its acidity is neutralized before use.

CONCLUSIONS

- The chemical composition of the water of the Biajaca stream in Minas de Matahambre, Pinar del Río, reflects the characteristics of the formation to which it belongs and also the negative impact it receives from nearby mining activities. Its high acidity is the product of the acid drainages of the mines and represents the main limitation for direct use in agriculture.
- The salinity, sodicity and toxicity indicators comply with the requirements established by the FAO. The combination of C.E. and R.A.S results in that the collected water presents slight infiltration problems in the soil. Such problems can be reduced by adding plaster to water or soil, or by mixing two sources of water. Although, generally these problems are not necessary to correct them, unless the crop demands a large quantity of water, therefore, the previous neutralization of the pH, converts the collected water into a good quality innocuous resource for its use as irrigation water.

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