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ORIGINAL PAPER

Hydrochemical evolution of the Reocín mine filling water (Spain)

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Abstract The Reocín mine is located in Cantabrian region, in northern Spain. Its exploitation ended in 2003 due to the exhaustion of its reserves. In November 2004, the controlled flooding of the openpit began. During this process, both the qualities of stored water and piezometric levels have been monitored to control the possible water detraction from the Saja-Besaya hydrographic system. This paper deals with the water chemistry analysis of the pit lake surface, as well as the different conditions of the area. Geological and hydrogeological contexts play an important role in the lake water chemistry. The lake water quality continues improving. The sulphate content and zinc concentrations are already below the permitted pouring limits. Three factors are distinguished: the washing

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of the mine shafts is completed; the water supply from the aquifer contributes to the dissolution of the salt content and the bedrock, and dolomite, which neutralizes acid waters and improves the water quality during the flooding process with a pH value of 8. Owing to these conditions, the stored water meets the necessary conditions for discharge and provides the opportunities to use it.

Keywords Water quality · Open pit lake · Hydrology · Geochemistry

Introduction

Mining lakes are formed when open-pit extraction ceases, and the created hole is flooded either by the inlet of groundwater flows, surface water flows or direct precipitation (Castro & Moore, 2000; Miller et al. 1996). These lakes change the landscape and may influence the natural flow of rivers and aquifers in the area. The physicochemical characteristics of these waters depend on several factors: the type of extracted ore, the type of fitting rock, the climate, the existing water flow, the morphology and depth of the gap, flood time, and the different water sources associated with this process (Delgado-Martín et al., 2013; Castro & Moore, 2000; Denimal et al. 2005; Castendyk & Webster-Brown, 2007; Werner et al. 2001a). Of these factors, the most important is the regional flow



systems and the geochemical rock properties. It is therefore necessary to determine both the water flow of the system and the hydrochemical reactions that take place, which will serve to determine the water quality and its possible subsequent uses (Werner et al., 2001b). One method for improving the physicochemical characteristics of the stored water consists in filling the gap quickly after mining ceases because most host rock oxidation occurs when the rock is above saturation level. As soon as the rock is below the water table, oxygen decreases and the oxidation rate is slower, thus improving water quality (Castro & Moore, 2000).

The mining industry proposes various strategies to reduce the environmental and social damage caused by mining. Most of these strategies consider the concept of sustainability, including the creation of sustainable livelihoods (employment, community development, and infrastructure), the optimization of resources, and the completion of mining operations (McCullough & Lund, 2006). The benefits that these types of trainings can bring into the area are mainly based on the type of use given to them (Gámez et al., 2019; Blanchette & Lund, 2016; Hinwood et al., 2012), whether recreation, biodiversity conservation, industrial use, or public water supply. Furthermore, Doupé and Lymbery (2005) established eight different end uses for mining lakes around the world: recreation and tourism, wildlife conservation, aquaculture, irrigation, water for livestock, drinking water, industrial water, and chemical extraction.

Aquaculture in well lakes is presented as a potential means of rehabilitating these environments, improving their appearance and productivity (Miller, 2008). The water used for this purpose must have suitable properties and, before selecting the biological species to be cultivated, basic studies must be conducted on water quality, availability, and sediment characteristics. Examples of this type of use are Paso de Piedra mine pit lake, Batán, Buenos Aires, Argentina (Hattingh, 2018; Mallo et al., 2010) and pit lakes in north-eastern Minnesota (Axler et al., 2004; Baeten et al., 2018). Stored water can be exploited as a strategic irrigation resource during drought periods as in North Pesteana, Romania (Izabela-Maria & Florea, 2018). Another viable use of stored water is human necessity due to seasonal increases in demand, as in Meirama Lake, La Coruña, Spain (Juncosa et al., 2016, 2018) or for animals

(Mhlongo & Amponsah-Dacosta, 2015). Mine water can also be used for heating and cooling, as in Springhill mine, New Scotland, Canada (Raymond and Therrien, 2014); Florence Mine, Cumbria, United Kingdom (Banks et al., 2019); Henderson molybdenum mine, Empire, Colorado, USA (Watzlaf & Ackman, 2006); Saturn coal mine, Czeladz, Poland (Malolepszy et al., 2005); and Barredo- Figaredo, Mieres, Asturias (Loredo et al., 2011).

The Reocín mine was a zinc exploitation located in the Cantabria province (Spain), where more than 7 million tons of zinc concentrates with more than a grade of 60.5% were extracted for 147 years until its closure due to the depletion of reserves (Fernández et al., 2008; Symons et al., 2009). The Reocín deposit was exploited through underground and openpit mining. The underground exploitation of the site began in 1909 while the openpit mining method reached its final design in 1976, alternating with underground mining in its early days (Fernández et al., 2008).

The flooding process of the mine began on November 1, 2004 and still continues, forming a mining lake with significant water resources for the region. Considering the openpit lake characteristics, this paper intends to analyse the chemical characteristics of stored water to observe how its quality evolved during the filling process, analysing the results obtained since they will stipulate the possible uses of the stored water.

Materials and methods

Study area

The Reocín mine is located in the Cantabria region, northern Spain (Fig. 1), on the western flank of the Santillana del Mar synclinal, specifically in the Reocín subsystem, bounded to the North and West by the Saja River and to the South and East, stratigraphically speaking, by a roof of Bedulian marly limestones with low permeability (Symons et al., 2009).

During the life of the mine, sphalerite, marcasite, and galena dismantled in the Gargasian dolomites around Torrelavega were extracted. These minerals are associated with the Aptian level (Gargasian-Clansayan) with a stretch of calcarenites and

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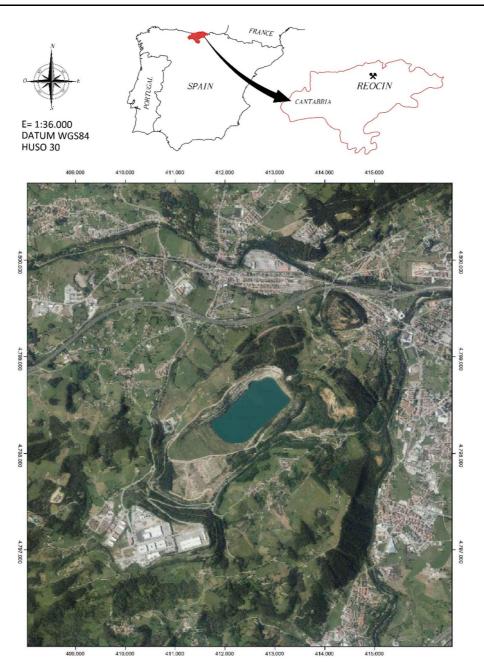


Fig. 1 The Reocín Mine location

limestones, dolomitized and irregularly shaped (Fernández et al., 2008).

Hydrogeology

The Reocín aquifer, where the mine is located, alternates with karstified limestones with high transmissivity and the low-karstified dolomites with low transmissivity (Fernández et al., 2008). Therefore, during the operation, it was considered that the water entered the mine from the west (watershed limestone zone), from both the Saja river and by recharging on the karstified outcrop. The aquifer is dolomitized, changing drastically into limestone on the edge of the mineralization on the west and gradually as it moves away to the east. Therefore, water flows into the mine from the west, to the river at the town of Golbardo. The refill is produced by the river itself and the outcrop, being the entrance to the lower east is due to the low transmissivity of the dolomites.

Therefore, given the existing lithology, the system is waterproof (Fernández et al., 1992), except for the upper area. Being an open basin, rainfall has a major influence on the mine hydrogeology, with a catchment basin of approximately 44 km². The average annual rainfall in the study area is 1100 mm. The lake that was created by mining activity occupies an area of 1.08 km² in the form of an ellipse, 1800 m long and 600 m wide, reaching a maximum depth of 280 m. Flooding occurs in successive filling stages, maintaining a certain level, from -272 m until reaching an equilibrium at + 60 m (Fernández et al., 2008).

The chemical quality of the filling water depends on several factors, including the type of fitting rock. Since the type of embedded rock is dolomite (magnesium calcium carbonate), which causes the neutralization of acid waters that originate when sulphide, oxygen, and water come into contact with the mine (Castro & Moore, 2000; Johnson & Hallberg, 2005; Kalin et al., 2006), correcting the pH to reach a value of 8.

Methodology

During the filling process, a thorough control of both piezometric levels is done using different piezometers located throughout the study area (Fig. 2) and the quality of the filling water. The process consists of different phases in which filling and other maintenance stages of the level occur: phase 1 (filling from level -272 m to level +13 m); phase 2 (filling from level + 13 m to level + 34 m) and phase 3 (at present), ending when the lake is completely filled. To control the process, the owner of the mine, placed 15 piezometers at the towns of Golbardo and Ganzo (Fig. 2). The study area was divided into three zones: the area where the mine is located; the northern area, where a number of sources and streams likely to be affected by flooding are located; and the area of the Saja and Besaya rivers. To study the condition of their water quality, two measuring stations were located in the Saja river, one upstream the Gargasian Albian outcrop (S1) and another downstream (S2). Two stations for discharge control are located in the Besaya river, upstream (BY1) and downstream (BY2). Floodwater is controlled at the following points: open pit (ECA), pumping (BOM), Pozo Santa Amelia (PSA), Rampa Jorge Valdés (RJV), Torres 1 (PT1), and Burco 1 (PB1). Surface water is controlled in Sameano stream (AS), La Teja Source (FTe), La turbera source (FTu), Saja river 1 (S1), Saja river 2 (S2), Besaya river 1 (BY1), and Besaya river 2 (BY2).

Geologically, the rock mass that separates the mine from the Saja River to the northwest is more likely to produce water leaks, since the carbonate formation in which the deposit is formed is in contact with the river. Samples were taken at all control points on the surface, with a periodicity of 4 measurements per month at the beginning of the flood, during 2004. Subsequently, the periodicity between 2005 and 2009 was reduced to two measurements per month, samples being taken once a month since 2010.

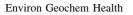
Water sampling and analytical procedures were conducted according to APHA (1998). The pH (both on site and at the laboratory), temperature, and specific conductivity were determined using electrometry. In addition, hydrochemical studies were done to determine the possible use for human consumption. Metal concentrations of zinc, iron, manganese, and lead were analysed at the laboratory using atomic adsorption spectrometry in flame, along with sulphate concentration using iron chromatography. Acceptable detection limits are 0.25 mg L⁻¹ for zinc, 0.05 mg L⁻¹ for iron and manganese, and 0.002 mg L⁻¹ for lead.

Results and discussion

The lake receives water from three different sources: the karst apparatus (the most important with almost 100 L/s, corresponding to 85% of the mine water), the diapiric (with a regular contribution all year round, corresponding to 1% of the mine water), and rain (corresponding to 14% of the mine water) (Fernández et al., 1992; Velasco et al., 2003).

Figure 3 shows the water level and precipitation data. The water level is currently at 45.69 because the pumps control the flooding. By observing the flooding process, the maximum level is expected to be at the 60th level, when balance is reached.

The chemistry of floodwater from open-cast mining is mainly caused by the dissolution of melanterite (FeSO₄·7H₂0) present in the underground exploitation that comes from the aerial alteration of the iron sulphates present in the deposit: pyrite, marcasite, and



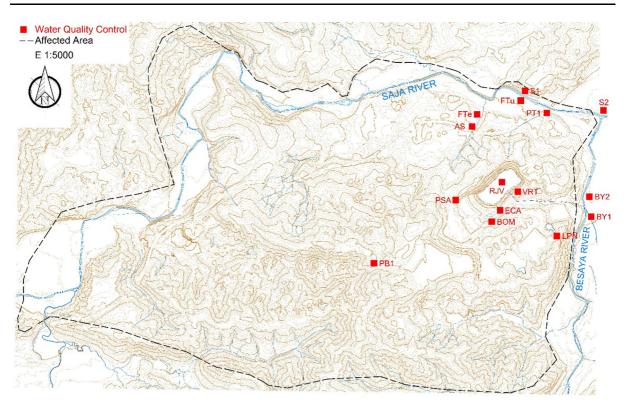


Fig. 2 Location of control stations

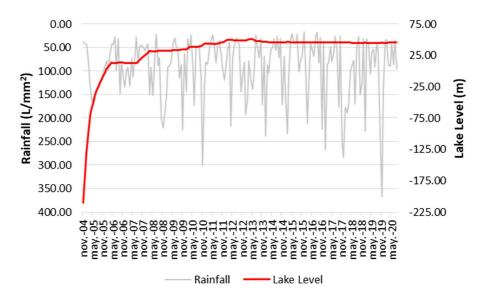


Fig. 3 Temporal evolution of water level

melnikovite. When melanterite dissolves, strong acidity is produced in the water, favouring iron, zinc, and manganese dissolution (Gammons et al., 2013; Pearce et al., 2016). Figures 4, 5 and 6 show the pH variation at different measurement points from the start of the flood in 2004 to 2020. From September 2005 to December 2006, the samples taken at the open-pit

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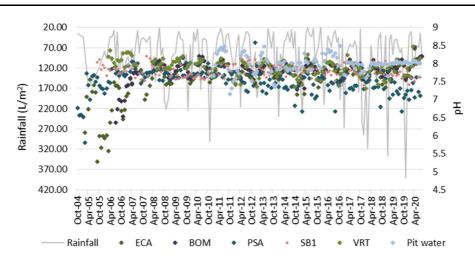


Fig. 4 Temporal pH evolution in the open pit mine zone

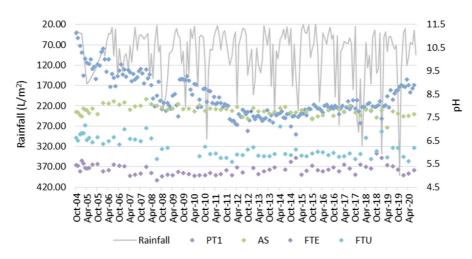


Fig. 5 Temporal pH evolution in superficial water

exploitation (ECA) show pH values between 5 and 6. The annual average pH in the area of the openpit exploitation in 2005, the first year after the start of the flood, was 6.56, indicating that at that time the waters were acid. As shown in Table 1, in the following years, the average values tended to rise and become basic due to the washing of the mining activity that took place in the first two years (Ordoñez et al., 2012). From that time on, the water quality began to improve, reaching values close to 8.

Since 2008, the evolution of the pH has shown a seasonal behaviour, with higher values in summer when rainfall is lower and lower values in heavy rainfall periods (Erdogan et al., 2020; Juncosa et al., 2019).

In the openpit area (Fig. 4), the pH value was acid (between 5 and 6.5) during the washing of the mining works, until April 2007. From this date on, the pH improved due to the type of fitter rock—dolomite (CaMg(CO3)₂)—since it reacts quickly in the presence of water with a pH lower than 7, causing both iron and different metals to precipitate and the pH to become basic (Castro & Moore, 2000; Elango & Kannan, 2007). As of 2007, pH values have been virtually constant at all measurement points, with values between 7 and 8.

In the north of the mine (Fig. 5), there are two areas with different pHs: the first one is the area where the sources of Turbera (FTu) and Teja (FTe) are located, with constant acid pH values throughout the flooding process, while the second one is the area of the

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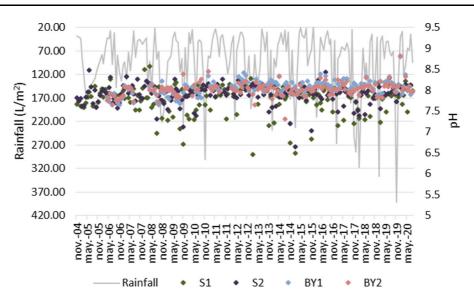


Fig. 6 Temporal pH evolution in the Saja and Besaya rivers

Table 1 pH variation with flooding time	Date	pH
	2006	6.67
	2007	7.49
	2008	7.74
	2009	7.74
	2010	7.80
	2011	7.84
	2012	7.63
	2013	7.83
	2014	7.68
	2015	7.68
	2016	7.80
	2017	7.61
	2018	7.66
	2019	7.78

Sameano (AS) and Torres1 (PT1) streams with a pH between 7.5 and 8.5. Due to the constant pH value over time, the flooding process does not strongly influence these areas. The same conclusion can be drawn from the north-east area (Fig. 6) of the mine where the pH variation remains almost constant during flooding.

Figure 7 shows the temporal conductivity evolution of the openpit water (ECA). The influence of precipitation is observed when assessing the highest conductivity values in the months when precipitation is scarce and the lowest in the rainiest months. Conductivity values tend to stabilize throughout flooding to an average of 2000 μ S/cm.

Water temperature variation is also reflected in conductivity variation since an increase in temperature facilitates ion transport, thus increasing conductivity, which results in higher values in the summer months (Akburak et al., 2020). Conductivity is also related to rainfall, with the highest values observed in drier periods and the lowest in periods of abundant rainfall. This trend, which has repeated over the years, is attributed to the washing of easily soluble mineral phases such as zinc (Juncosa et al., 2019). The conductivity data obtained relate the increased concentration of dissolved zinc ions to the increase in this parameter (Eyankware et al., 2018). The highest conductivity value appeared in January 2011 (3294 µSiemens/cm), corresponding to a dissolved solid value of 2305 mg/L. As zinc ions dissolved, conductivity decreased to about 2500 µSiemens/cm in December 2013.

The variation in dissolved iron concentration on the surface of the lake is shown in Figs. 8 and 9. The values observed in almost all measurement stations are very low and almost stable, without seasonal variations, except for two control points located at the Pozo Santa Amelia (PSA) and at the openpit exploitation (ECA). Samples taken at PSA reached high values in February (95.46 mg/L) and March (125.46 mg/L) 2005. At ECA, high values appeared from November 2005 (96.09 mg/L) to March 2006 (28.43 mg/L)

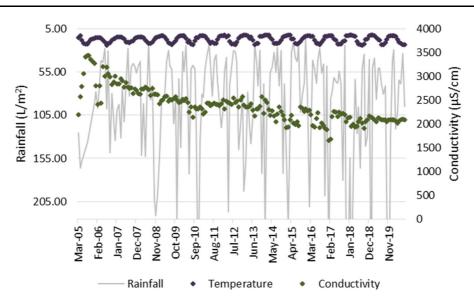


Fig. 7 Temporal evolution of conductivity and temperature

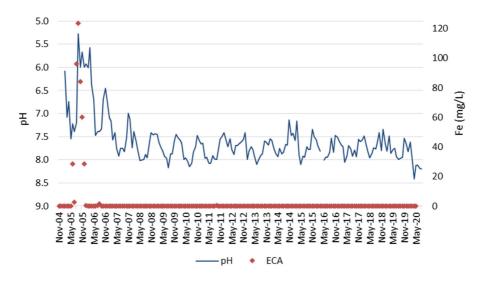


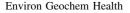
Fig. 8 Temporal evolution of dissolved iron at ECA

because, at the beginning of the flood, the pH values were acidic (between 5.28 and 5.58), favouring ferric hydroxide (Fe (OH)₃) precipitation and causing the lake waters to acquire a red tint according to Eq. $[Fe^{3+}] + 3H_2O = Fe(OH)_3 + 3[H^+]$ (Bachmann et al., 2001; Vandersluis et al., 1995).

As the flooding process progressed and due to the neutralizing effect of the dolomite, the pH values reached basic values, decreasing iron concentrations to values below 0.05 mg/L.

The 2D Table Curve software was used to establish the correlation between the pH and the iron contained in the floodwater (Salmon et al., 2017; Schultze et al., 2010; Shevenell, 2000). The exponential equation obtained relates both variables to a 0.86 correlation coefficient that sets the goodness of fit. Figure 10 shows the correlation established.

Figure 11 shows the temporal evolution of dissolved manganese. ECA behaviour decreases, with maximum concentration at the beginning of the flood (6.96 mg/L), corresponding to an acidic pH value (5.99). Subsequently, the values decreased during 2006 until reaching values below 1 mg/L in June



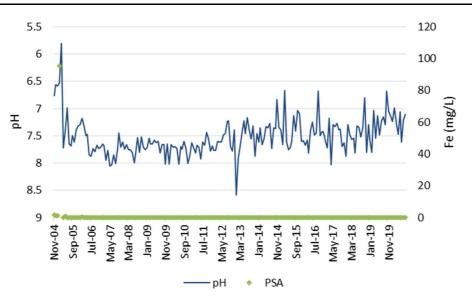


Fig. 9 Temporal evolution of dissolved iron at PSA

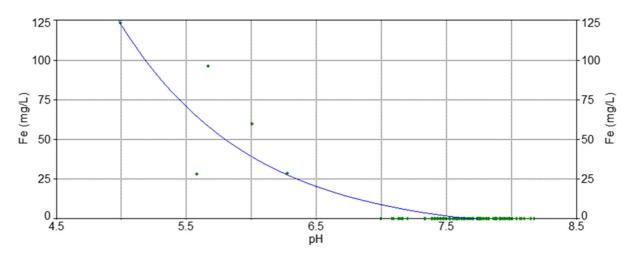


Fig. 10 pH-Fe correlation

2007, coinciding with an increase in the mine water pH.

At all the other measurement stations, manganese concentration has remained at low values mainly with concentrations below 0.05 mg/L.

Using the 2D Table Curve software, the correlation between pH and manganese was established (Denimal et al., 2005; Williams et al., 2019). The equation that relates both variables is exponential with a 0.93 goodness of fit, indicating the relationship between them. Figure 12 shows the correlation plot between pH and manganese. The temporal evolution of zinc concentration is shown in Fig. 13. Higher values appear at the beginning of the flood when mining work is washed out. The PSA values obtained were 37.67 mg/L in January 2005, 33.17 in February 2005, and 48.74 mg/L in March 2005, corresponding to 6.58, 6.52, and 5.81 pH values, respectively.

At ECA (Fig. 14), the highest concentrations occurred at the end of 2005 (21.47 mg/L in November and 18.37 in December), with pH values of 5.67 and 5.99.

Zinc concentration decreases as flooding progresses. So, the constant input of water from the

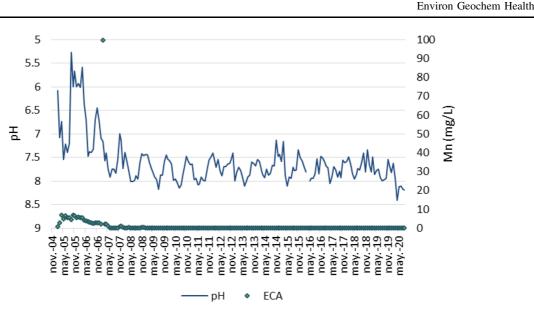


Fig. 11 Temporal evolution of dissolved manganese at ECA

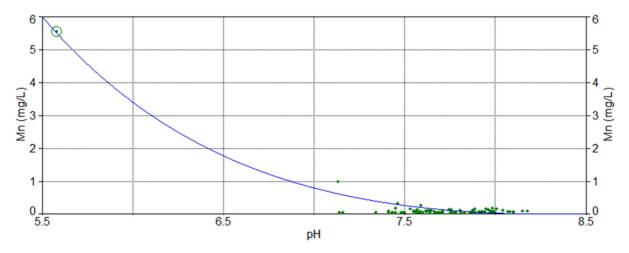


Fig. 12 pH-Mn correlation

aquifer causes salt content to dilute and the mining holes to be thoroughly washed, thus contributing to the improvement of the water quality (Islam et al., 2017; Villain et al., 2013). At all the other measurement stations, the concentration was low from the beginning of the flood, decreasing over time to reach values below 1 mg/L.

The water pH and the Zn concentration were correlated. The equation obtained is exponential with a 0.90 fit, indicating that the variation between both variables actually occurs, as shown in Figs. 13 and 14. Figure 15 shows the correlation between pH and Zn. Figures 16, 17 and 18 show the temporal evolution of sulphate. High values are found at both ECA and the samples taken from the filling water (BOM) at the beginning of the flood. At ECA, the maximum values appeared at the end of 2005: September (2804.60 mg/ L), October (2712.00 mg/L), November (2691.80 mg/ L), December (2588.20 mg/L), and January (2519.10 mg/L). These maximums can be associated with minimum pH values (5.28, 5.99, 5.67, 5.99, and 5.93, respectively) that occur at the time the gaps were created during mining were flooded, decreasing during the course of flooding to reach values below 1,500.00 mg/L from November 2014. Environ Geochem Health

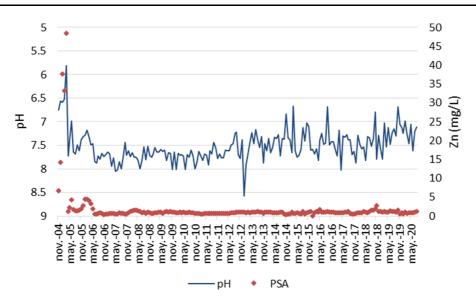


Fig. 13 Temporal evolution of dissolved zinc at PSA

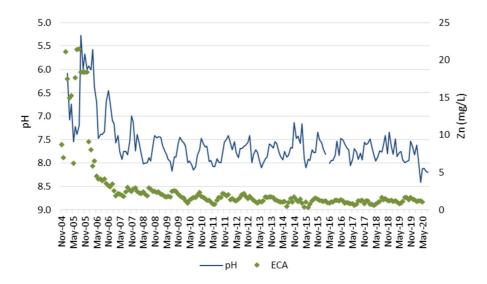


Fig. 14 Temporal evolution of dissolved zinc at ECA

Pumping began in July 2006, when the highest sulphate concentrations (2285.60 mg/L) and the lowest pH (6.36) were reached. With the flooding process, the values steadily decreased, reaching values below 1400.00 mg/L since February 2015.

Samples taken at the outlet of the treatment plant decanting raft (VRT) installed by the company at the beginning of the flood also show high concentrations (above 2000.00 mg/L), decreasing with the washing of the mining holes and the pH basicity, allowing the company to stop the plant operation, significantly improving the water quality.

The correlation between the pH and the dissolved sulphate was established to demonstrate their dependence (Luís et al., 2009; Sienkiewicz & Gasiorowski, 2016). Using the 2D table curve program, the fit equation and the degree of goodness of 0.66 were obtained. As shown in Fig. 19, the curve is greatly adjusted to the cloud of points obtained, reaffirming the correlation between the pH and the sulphates.

This study shows that the chemical characteristics of the water stored in the Reocín mine are influenced by geology and acid mine drainage (AMD) (Delgado et al., 2008). At the beginning of the flood, the water

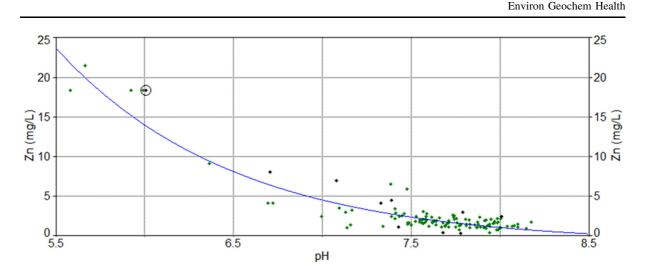


Fig. 15 pH-Zn correlation

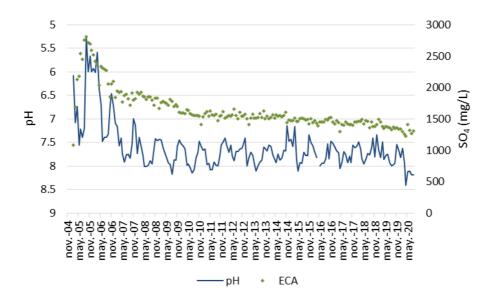


Fig. 16 Temporal evolution of dissolved sulphate at ECA

showed an acid pH (5.28) due to washing of the mining works, with high conductivity (3294.00 μ S/cm) and sulphate concentration (2804.06 mg/L). There was an increase in drainage water acidity and trace metal concentration when dissolution periods coincided with rainfall periods, which can be explained by leaching processes (Luís et al., 2009). Surface water analyses showed high metal concentration and low pH on the sites closest to the mining area (ECA, BOM, and PSA) at the beginning of the flooding. Meanwhile, on the sites further away from the mining area, the values remained low and constant throughout the process. Rock–water interaction also contributes to water chemistry improvement, as groundwater evolves chemically by interacting with minerals in the aquifer and the internal mixing between different groundwater along the flow paths (Belkhiri et al., 2012; Eang et al., 2018).

Conclusions

The flooding of the Reocín openpit mine began in November 2004 and has not yet been completed. The flooding is being controlled and does not present an



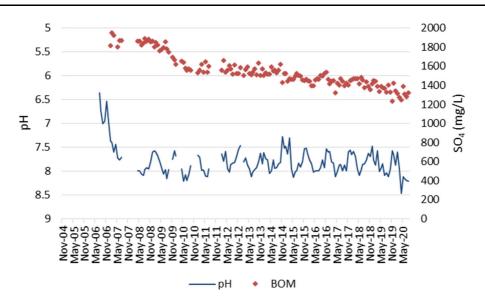


Fig. 17 Temporal evolution of dissolved sulphate at BOM

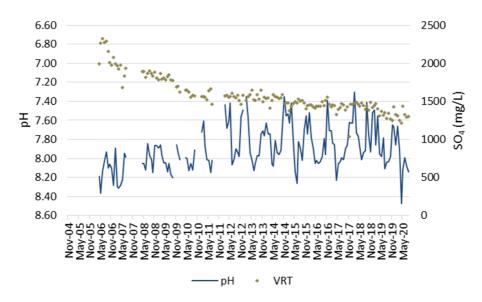


Fig. 18 Temporal evolution of dissolved sulphate at VRT

environmental risk as leaks have not been detected in any of the areas where the checkpoints are located.

The lake water continues to evolve, improving chemically, as indicated by the descending trend of each parameter. This, considering that during the flooding process improvement has continued, by decreasing the sulphate content and zinc concentration, which at the moment, is already below the permitted pouring limits.

This improvement in chemism is mainly due to three causes: the rapid washing of the mining holes (which occurred during the first years of the flood, 2005 and 2006); the constant water supply from the aquifer where the exploitation was located, which contributes to salt dilution; and the type of fitting rock (dolomite), which causes acid water neutralization, by reacting quickly in the presence of water with a pH less than 7, precipitating iron and other metals, thus improving the water quality.

The filling of the pit lake opens up a range of possibilities for using stored water, taking into account

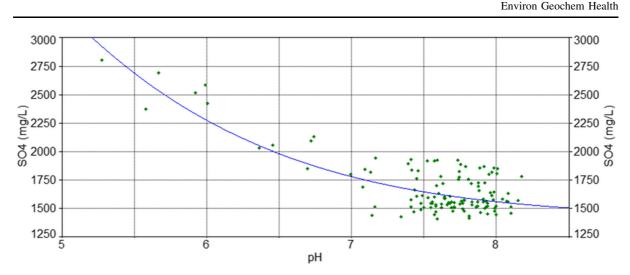


Fig. 19 Correlation between pH and dissolved sulphate

its quality and capacity, which makes it the second most important reservoir in the Cantabria province.

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Author contribution Noemí Barral: Conception/Research design/Acquisition of data/Analysis and interpretation of data/ Drafting the manuscript. Raúl Husillos: Conception/ Research design/Acquisition of data/Drafting the manuscript. Elena Castillo: Conception/ Research design/Acquisition of data/ Drafting the manuscript. Manuel Cánovas: Analysis and interpretation of data/Drafting the manuscript. Elizabeth Lam: Analysis and interpretation of data/Drafting the manuscript. All the authors approved the final version to be submitted.

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Declarations

Animal research Since this study did not involve animal research, no consents were required to participate and publish data belonging to animals. Therefore, the inclusion of these forms and other ethical issues related to the publication of this type of data do not apply to this study.

Consent to Participate Yes.

Consent to Publish All authors agreed to publish the manuscript respecting the current sequence of authors listed. Likewise, all authors agreed to designate Noemí Barral as the corresponding author.

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