Effects of pumice mining on soil quality

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Abstract

México is the world’s fourth most important maize producer; hence, there is a need to maintain soil quality for a sustainable production in the upcoming years. Pumice mining, a superficial operation, modifies large areas in Central Mexico. The main aim was to assess the present state of agricultural soils differing in elapsed-time since pumice mining (0–15 years), in a representative area of the Calimaya region in the State of Mexico. The study sites in 0, 1, 4, 10 and 15 year-old reclaimed soils were compared with adjacent undisturbed site. Our results indicate that soil organic carbon, total nitrogen, microbial biomass carbon and microbial quotients were greatly impacted by disturbance. A general trend of recovery towards the undisturbed condition with reclamation age was found after disturbance. Recovery of soil total nitrogen was faster than soil organic carbon. Principal components analysis was applied. The first three components together explain 71.72% of the total variability. First factor reveals strong associations between total nitrogen, microbial biomass carbon and pH. The second factor reveals high loading of urease and catalase. The obtained results revealed that the most appropriate indicators to diagnose the quality of the soils were: total nitrogen, microbial biomass carbon and soil organic carbon.

1 Introduction

Land degradation refers to processes induced by human activities that cause the decrease of biological productivity or biodiversity, as well as the current capacity and/or potential to sustain human life (Oldeman, 1998). Land degradation and desertification affects many regions of the world (Cerdà et al., 1999; Bai et al., 2013; Izzo et al., 2013; Wang et al., 2013; Yan et al., 2015) and there is a need to restore those areas affected by land degradation processes (Guénon et al., 2013; Özcan et al., 2013; Kröpfl et al., 2013; Li et al., 2013; Tejada et al., 2014; Zucca et al., 2015). There are examples over studies in opencast coal mines, magnesite mines and limestone mines (Haigh
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Surface or opencast mines, are a form of operation designed to extract near-surface volcanic minerals whose deposits are horizontal, shallow and relatively thick of considerable lateral extension that uses the method of mechanical mining (or dry) opencast mining (Hartman, 1987); this type of mining degrades the soils 2–11 times more than underground mining (Li, 2006).

The open pit mining has great economic importance in Mexico therefore taking the volume of production of non-metallic minerals as an indicator, the State of Mexico is the largest producer of non-metallic minerals i.e. mainly extracted sand and gravel. Similarly have most damaged area by surface mining (Jiménez et al., 2006). In agricultural area of the Municipality of Calimaya highlands in state of Mexico, is carried out opencast mining. After extractive operations in the pumice areas, agricultural use is continued soil although productivity declines while the landscape and soil characteristics are substantially altered. These facts make evident the need of suitable correction management actions to accelerate the succession process and return of the degraded area to an environmentally acceptable and productive condition (Rogowski and Weinrich, 1987). Pumice is a volcanic rock with trapped gas bubbles formed during volcanic eruptions (Whitham and Sparks, 1986); is made up of Si, Al, K, Na, Fe oxides, with a small percentage of Ca, Mg, Mn, Ti oxides (Liguori et al., 1984).

The main causes of land degradation during mining operation are (1) removal of vegetation cover and topsoil, (2) excavation and dumping of overburden, (3) changes in the landscape (Mukhopadhyay et al., 2013), (4) disruption of surface and subsurface hydrologic regimes, (5) transformation of fertile cultivated land into wasteland and in some cases (6) serious environmental pollution and ecological degradation, which can lead to the loss of biodiversity (Keskin and Makineci, 2009). Soil in mined sites is replaced by overburden, which differs substantially from developed soils (Huggett,
with adverse properties such as severe depletion of organic matter, erosion risk, toxicity, and nutrient deficiency, which commonly reduce productivity in post mining landscapes (Sourkova et al., 2005).

In a post-mining landscape, it is necessary, the regeneration of the uppermost soil layer, organ-mineral horizon, and soil biota, which transform organic matter (Frouz et al., 2001). The accumulation of organic matter (OM) is critical because this results in positive changes in physical and chemical soil properties, such as water holding and sorption capacities, nutrient content and availability, soil bulk density, buffering capacity, increases microbial biomass and extractable carbon, microbial community structure and biodiversity. Moreover, OM is an energy source for the soil microorganisms, which drives decomposition and mineralization of plant residues, thereby releasing nutrients (Sourkova et al., 2005; Laudicina et al., 2015).

Soil quality included soil physical, chemical and biological properties, as well as soil processes and their interactions. The selection of some properties for to asses soil quality is an effective way. Some authors using independent indicators, others preferred their combinations into models or expressions in which various properties are involved, these expressions are called indices (Zornoza et al., 2015). The establishment of multiparametric indices has been used as an adequate tool for integrating greater information of soil quality. It provide a more holistic measurement of soil quality (Brevik et al., 2015; Zhang et al., 2015). Several studies have generated indices from a data set using physical, chemical and biological indicators. Organic carbon, microbial biomass and enzymes activity have been widely used to assess impact of land use change and reclaimed soils (Chodak and Niklinska, 2010).

Measurement of enzyme activities is widely used to examine nutrient cycling processes in soil (Nannipieri et al., 1990; Tabatabai and Dick, 2002). Moreover, enzyme activities can provide indications of quantitative changes in SOM and are usually related to the presence of viable microorganisms and their oxidative activities (Gianfreda et al., 2005), they could be sensitive indicators of the effect of land degradation on soil microbial activity. Measurements of soil hydrolases provides an early indication of

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changes in soil fertility, since they are related to the mineralization of important elements such as nitrogen, phosphorus and carbon and may provide some insight, into the metabolic capacity of the soil (Shaw and Burns, 2006; García-Orenes et al., 2010). Urease plays an important role in soil nitrogen cycle because it can hydrolyze urea an important fertilizer in agricultural systems to ammoniacal nitrogen (Sinsabaugh et al., 2000; Caldwell, 2005). Catalase has a great effect on changing soil redox, chemical properties of soil solution, and accelerating transformation of organic matter (Wang, 2012). The metabolic quotient estimates the activity and efficiency of decomposition (or C use) by soil microbes (Anderson and Domsch, 1990) and has resulted a suitable indicator to provide evidence of soil perturbation (Zornoza et al., 2015).

Little information exists about how mining affects soil in cropland regions of the world and especially those of the central highlands of Mexico. The present work aimed to assess the changes produced in the agricultural soils differing in elapsed-time since pumice mining (0–15 years). With the information from this study we examine valuable indicators of surface mine reclamation progress in open cast mine.

2 Materials and methods

2.1 Study site

This study was conducted in Calimaya, state of Mexico (Central Mexico; 19°1’25” N; 99°4’02” W), where the mean annual temperature is 14 °C and the mean annual rainfall is 800 mm (INEGI, 2009). The dominant climate is subhumid with summer rains. Dominant soils are Ochric Andosols (GEM-ICEGEM, 2012). The main type of land use in the region is cropland based on maize. Cultivation techniques consist in monoculture, removal of crop residues, and use of N fertilizer, herbicides and pesticides. The land use change has caused a decrease in cultivated land from 7508 to 5350 ha between 2010 and 2011 (GEM, 2011–2012). Following standard practice on surface mining sites, the
topsoil was stripped and stockpiled until mining operations were completed; stored soil was then spread on top of overburden.

2.2 Sampling and processing

February 2011, five mine sites, differing in elapsed-time since reclamation (0–15 years) of opencast pumice mine spoils in the Central of Mexico, were chosen on the basis of similarity of aspect as well as proximity to one another and an adjacent undisturbed site, for comparison, located about 2800–2950 m a.s.l. The selected sites were newly mined (S), a year (S₁), four years (S₄), ten years (S₁₀), fifteen years (S₁₅) and old mined and undisturbed soil (S). These sites had been continuously cultivated since reclamation of mine spoils, surface soil samples (0–15 cm depth) were taken in February (during the dry season), June (onset of the rainy season), and March 2012 (during the dry season), stored at 4°C for biochemical analyses. Afterwards, they were passed through a 2 mm mesh sieve.

The following parameters were analyzed: gravimetric moisture content (GMC) was measured gravimetrically, water holding capacity (WHC) according to Forster (1995), soil bulk density as described by Domínguez and Aguilera (1987). The soil pH and electrical conductivity were determined in soil/water (1 : 2.5 w/v) suspension with a pH meter and a conductivity meter, respectively (Thomas, 1996). Content of soil organic carbon (SOC) was determined by the Walkley-Black method (Nelson and Sommers, 1996) and TN with Kjeldahl digestion (Bremner, 1996).

MBC of soil samples was estimated by chloroform fumigation and extraction method (Vance et al., 1987). Basal respiration (BR) was estimated by quantifying the carbon dioxide (CO₂) released by microbial respiration in 33 days of incubation at 25°C adjusted to 40% water holding capacity (WHC). For this purpose, 25 g soil was filled into flasks, together with small flasks containing 10 mL of 0.2 mol L⁻¹ NaOH, to capture the released CO₂, and hermetically sealed. CO₂ was determined by titration with 0.2 mol L⁻¹ HCl, after precipitation of the barium carbonate resulting from the addition
of BaCl₂ to the NaOH solution, using phenolphthalein diluted in 100 mL ethanol (60 %, v/v) as indicator (Alef and Nannipieri, 1995).

Catalase activity was measured by titrating the residual H₂O₂ added to soil and not degraded by catalase with KMnO₄ (Johnson and Temple, 1964), acid phosphatase activity was measured by spectrophotometry (400 nm) of p-nitrophenol released from 1.0 g soil after a 60 min incubation at 37 ℃ with a 0.025 mol L⁻¹ p-nitrophenyl phosphate substrate, in 4 mL of 0.17 mol L⁻¹ MUB (universal buffer), at pH 5 (Tabatabai and Bremmer, 1969), urease activity was determined as the amount of NH₄⁺ released from 5.0 g soil after a 120 min incubation with a substrate of 0.2 mol L⁻¹ urea at 37 ℃, 4.5 mL of THAM (Tris buffer) (Alef and Nannipieri, 1995). The metabolic quotient (qCO₂) was calculated as the ratio of basal respiration to MBC (Anderson and Domsch, 1990).

With the aim of identifying relationships between the properties evaluated a correlation analysis was performed. Then Principal Components Analysis (PCA) was applied using data of soil physical and chemical properties as well as enzyme activities to determine the degree of association of the variables analyzed. The number of components was determined by the eigenvalue-one criterion. Subsequently, a hierarchical cluster analysis (Ward’s method) was performed using the software STATGRAPHICS 5.1.

3 Results

3.1 Soil physic-chemical properties

Gravimetric moisture content ranged from 1.37 % (0 year old) to 2.81 % (15 year old) and were correlated with the water holding capacities (r = 0.74; p < 0.01). TN and SOC increased at a slower rate. The SOC level was found higher in no mine soil than mined soil.
3.2 Microbial properties

MBC values shown in Table 2, the highest value of MBC was found in the undisturbed soil, which coincides with the highest value of organic matter with respect to the mined soils. After extraction of pumice MBC content, $S_0$ decreases $>50\%$ with respect to $S$. Table 2 shows the values for enzymatic activity and soil microbial properties (MBC, respiratory activity and $q\text{CO}_2$). The highest values were obtained in undisturbed soil, the rest of soils show similar values. This result indicated that the land degradation is associated to a strong decrease in the SOC content. The respiration rate was higher in undisturbed soils and lower in mined soil.

Enzyme activities increased with increasing age of mined: undisturbed soil showed the highest enzyme activity values, while the mined soil the lowest (Table 2). Soil phosphatase activity, which is a good indicator of the potential mineralization of P-containing organic compounds in the soil (Dick and Tabatabai, 1993), was lower in mined soil. In this study, urease activity in undisturbed soils was particularly high, and although the value of the activity was similar to mined soils, and the maximum value of activity in the mined soils ($13\text{ mmol NH}_4\text{ g}^{-1}\text{ h}^{-1}$) was almost 81 undisturbed soils ($16\text{ mmol NH}_4\text{ g}^{-1}\text{ h}^{-1}$).

The $q\text{CO}_2$ values were higher in mined soils compared to the undisturbed soils (Table 2), meaning that the microorganisms were more efficient in conserving C resources in the no mined sites as compared to the mined sites.

3.3 Correlations

Correlation matrix revealed that a large number of soil variables were significantly correlated with each other (Table 4). For a few physical variables, negative correlation was observed between soil pH and MBC and TN. Closer correlations were found between chemical and biological variables. The MBC was positively correlated with $\text{CO}_2$, WHC, and TN while significantly positive correlations among catalase, $\text{CO}_2$ and urease activity were found. Additionally, both SOC and WHC were positively correlated with TN.
and \(q\text{CO}_2\) negatively correlated with catalase, MBC, WHC and moisture. \(\text{CO}_2\) was positively related with the urease activity, MBC, and negatively related with EC, pH and \(q\text{CO}_2\). MBC was significantly correlated with water holding capacity, \(\text{CO}_2\) and TN. SOC contents were significantly correlated with TN (Table 3).

Principal components analysis (PCA) was performed using all the data for soil characteristics to explore the differences between soils the main results are shown in Table 3. The weighted loading values in each component were used to select the indicators, with ten percent of the highest weighted loading as a threshold for selection (Govaerts et al., 2006). The first component accounted for 33.27 with TN followed by MBC and pH. Contribution from the second component to the total variance was 23.97 was found with catalase and urease. PC3 accounted for 14.74 According to the PCA results pH and TN were the most important characteristics that affected MBC.

### 3.4 Cluster analysis

To determine group associations and to assess the affinity among different variable, Cluster analysis is often used (Hussain et al., 2008). The dendrogram obtained using Euclidean distance as similarity criterion (Fig. 1) revealed a clear separation into two major groups. The first cluster comprises S, S\(_{15}\) and S\(_{10}\), the second one S\(_0\), S\(_1\), and S\(_4\). The first cluster includes the soil with more time mining and undisturbed soil suggesting a higher similitude with S\(_{15}\). The second cluster corresponds to soils with less extraction time of being more similar soils S\(_1\) and S\(_4\).

In the present study, Dendogram Analysis (DA) was applied to differentiate soils with different age of mined as well as to determine whether the difference was actually significant. Three major soil clusters were evident. Canonical scores indicate that S\(_{15}\) reclamation site is more similar to the S\(_{10}\) site that of the others mined sites (Fig. 2). DA although clearly able to discriminate between disturbed and undisturbed ecosystems, indicated a trend towards the undisturbed condition with reclamation age. The graph showed two major groups, the first consisting of the three most disturbed soils (S\(_0\), S\(_1\) and S\(_4\)) and the second group corresponds from the two least disturbed (S\(_{15}\) and S\(_{10}\)).
4 Discussion

Mining operations resulted in a decrease in gravimetric moisture content and water holding capacity; increased pH, CE and bulk density; decreased SOC and TN in recently returned topsoil (0 year old) compared with undisturbed site (Table 1). The loss of SOC and TN observed in soils post-pumice mining is typical of major ecosystem disturbance (Kimble et al., 2001). The depletion of organic C produces a decline in soil quality associated with the reduction in available water capacity, nutrient concentration and soil structure (Schwenke et al., 2000).

Our results also indicate that disturbance was highly detrimental to SOC. Reclaimed SOC averaged only 45 undisturbed site. In addition, direct photodegradation by solar radiation may contribute to the loss of soil C (Austin and Vivanco, 2006) as well as the mixing of surface soil with deeper soil layers (Ward, 2000). The quantity of organic matter (C and N) and rates of microbial C mineralization to CO$_2$ (respiration) were recovered with age in mining soil. Shrestha and Lal (2011), aimed at quantifying the effects of mining and reclamation processes on physical and chemical properties of reclaimed soils for three dominant soil series in Ohio. Mining and reclamation activities decreased soil organic carbon (SOC) declined by 52 to 83 % of undisturbed sites and nitrogen (N) pools declined by 42 to 75 % of undisturbed sites.

The recovery of MBC in these soils was faster than organic C. This may indicate that the proportion of bio-available C in 15 year-old soils has become similar to that of undisturbed soils but may also be the result of an increased availability of TN (evidenced by lower C/N ratios in older soils) and inorganic P (as a result of fertilization). All measured components of the microbial biomass remain greatly reduced from the undisturbed condition. Similarly, mined soil MBC was estimated to average only 76 % of amounts found for undisturbed soils. Insam et al. (1991) and Rodríguez et al. (2005), also suggested that TN availability has little effect on MBC, although their results showed relationship between MBC and TN of soil.
The increase in soil basal respiration in mined soil can be explained by an increase in the contents of SOC and nutrients, which would enhance microbial activity (Emmerling et al., 2000), and biomass cycling, thus leading to an increase in basal respiration (Fernandes et al., 2005).

To estimate the activity and efficiency of decomposition (or C use) by the soil microbesthe $q\text{CO}_2$ was calculated (Anderson and Domsch, 1990). That establishes, as the MBC becomes more efficient in using the resources available, less C is lost as $\text{CO}_2$ through respiration (García-Orenes et al., 2010). Soil microbial quotients in several ecosystems have been found to increase immediately post-disturbance and subsequently decline with age (Insam and Domsch, 1988; Schipper et al., 2001; Graham and Haynes, 2004). This pattern of microbial quotient has been interpreted as indicative of a decrease in C bio-availability in the soil organic matter over time. The microbial quotient of 0 year-old was higher than other mined soils and increased with age. The respiration rate per unit of microbial biomass (respiratory quotient) is a variable that can be interpreted more easily (Fernandes et al., 2005). The lower $q\text{CO}_2$ values at S site may suggest that is a more stable ecosystem with high levels of biological activity. The $q\text{CO}_2$ is a valid indicator of the efficiency of energy use by microbes (Yan et al., 2003). High $q\text{CO}_2$ values suggest that the soil microbes are competing intensely for the small amount of C available.

The principal components analysis indicated that TN, MBC followed similar accumulating trend and, after 15 years of restoration point to successful recovery of the reference state conditions.

5 Conclusions

After several cycles of the microbial biomass and enzyme activities were recovered apparently as a result of the continuous additions of organic matter. Indicators of quality of soil evaluated MBC and NT, stand in the multivariate analysis, these biochemical indicators provide information to diagnose of quality of soil that have remove pumice.
Data from this study as decreasing OC, NT and MBC in mined soils, suggest that removal of pumice has an adverse impact on the quality of maize acreage; this situation needs to be analyzed in order to consider measures for sustainable land management. Selected indicators are a minimum number of easy and repeatable measure variables which represents local conditions.

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Table 1. Chemical and physical properties of soil samples with different years of extraction of pumice.

| Sample | GMC (g 100 g⁻¹) | WHC (soil: H₂O) | pH 1:2.5 (dS m⁻¹) | EC (g cm⁻³) | BD (mg kg⁻¹) | P (mg kg⁻¹) | TN (g 100 g⁻¹) | SOC (mg kg⁻¹) | C/N |
|--------|-----------------|-----------------|-------------------|-------------|-------------|-------------|---------------|--------------|-----|
| S₀     | Feb 2.33 ± 0.11  | 31 ± 0.57       | 5.5 ± 0.31        | 0.13 ± 0.00 | 1.18 ± 0.01 | 16.23 ± 0.19 | 0.08 ± 0.03  | 1.30 ± 0.12  | 18 ± 5.30 |
|        | Jun 2.27 ± 0.11  | 29 ± 0.58       | 5.9 ± 0.16        | 0.17 ± 0.00 | 1.17 ± 0.02 | 16.23 ± 0.14 | 0.11 ± 0.00  | 0.72 ± 0.11  | 7 ± 1.00  |
|        | Mar 1.37 ± 0.09  | 30 ± 0.58       | 6 ± 0.08          | 0.16 ± 0.01 | 1.12 ± 0.01 | 16.91 ± 0.14 | 0.10 ± 0.01  | 1.20 ± 0.05  | 12 ± 2.12 |
| S₁     | Feb 2.48 ± 0.13  | 33 ± 2.08       | 5.1 ± 0.30        | 0.08 ± 0.00 | 1.21 ± 0.01 | 16.23 ± 0.03 | 0.11 ± 0.00  | 1.23 ± 0.11  | 11 ± 1.05 |
|        | Jun 2.40 ± 0.01  | 30 ± 0.58       | 6.2 ± 0.16        | 0.35 ± 0.01 | 1.15 ± 0.01 | 17.80 ± 0.01 | 0.11 ± 0.00  | 0.84 ± 0.11  | 8 ± 1.05  |
|        | Mar 1.60 ± 0.17  | 30 ± 0.00       | 6.2 ± 0.01        | 0.17 ± 0.01 | 1.12 ± 0.01 | 17.02 ± 0.26 | 0.11 ± 0.00  | 1.30 ± 0.05  | 12 ± 0.42 |
| S₄     | Feb 2.59 ± 0.03  | 35 ± 0.00       | 5.2 ± 0.04        | 0.07 ± 0.00 | 1.21 ± 0.00 | 16.72 ± 0.20 | 0.17 ± 0.00  | 1.50 ± 0.11  | 9 ± 0.65  |
|        | Jun 2.63 ± 0.05  | 30 ± 0.00       | 6.2 ± 0.56        | 0.30 ± 0.02 | 1.2 ± 0.04  | 17.80 ± 0.07 | 0.11 ± 0.00  | 0.72 ± 0.11  | 7 ± 1.00  |
|        | Mar 1.67 ± 0.02  | 30 ± 0.00       | 6.3 ± 0.04        | 0.21 ± 0.00 | 1.2 ± 0.02  | 17.57 ± 0.42 | 0.12 ± 0.02  | 1.25 ± 0.08  | 11 ± 1.00 |
| S₁₀    | Feb 1.68 ± 0.02  | 30 ± 0.00       | 4.9 ± 0.14        | 0.10 ± 0.00 | 1.31 ± 0.00 | 21.95 ± 0.64 | 0.19 ± 0.03  | 1.56 ± 0.20  | 8 ± 0.66  |
|        | Jun 1.71 ± 0.04  | 27 ± 0.00       | 5.4 ± 0.20        | 0.45 ± 0.11 | 1.20 ± 0.03 | 20.71 ± 0.07 | 0.17 ± 0.00  | 1.60 ± 0.20  | 9 ± 1.15  |
|        | Mar 1.289 ± 0.25 | 27 ± 0.00       | 5.5 ± 0.03        | 0.27 ± 0.00 | 1.24 ± 0.02 | 23.93 ± 0.49 | 0.17 ± 0.00  | 1.50 ± 0.12  | 9 ± 0.72  |
| S₁₅    | Feb 2.14 ± 0.02  | 34 ± 0.58       | 5.1 ± 0.09        | 0.18 ± 0.01 | 1.23 ± 0.04 | 18.89 ± 0.19 | 0.17 ± 0.00  | 1.63 ± 0.23  | 10 ± 1.32 |
|        | Jun 2.81 ± 0.06  | 31 ± 0.58       | 5.4 ± 0.10        | 0.47 ± 0.01 | 1.10 ± 0.01 | 20.43 ± 0.13 | 0.11 ± 0.00  | 0.78 ± 0.00  | 7 ± 0.00  |
|        | Mar 1.80 ± 0.37  | 29 ± 0.58       | 6.0 ± 0.05        | 0.19 ± 0.00 | 1.10 ± 0.00 | 19.07 ± 0.13 | 0.11 ± 0.00  | 1.09 ± 0.08  | 12 ± 0.73 |
| S      | Feb 3.16 ± 0.07  | 40 ± 3.78       | 4.7 ± 0.18        | 0.10 ± 0.00 | 1.11 ± 0.01 | 16.77 ± 0.19 | 0.32 ± 0.03  | 2.28 ± 0.11  | 7 ± 1.00  |
|        | Jun 3.11 ± 0.02  | 34 ± 0.58       | 5.6 ± 0.14        | 0.13 ± 0.00 | 1.02 ± 0.02 | 15.74 ± 0.05 | 0.23 ± 0.02  | 2.67 ± 0.11  | 12 ± 0.75 |
|        | Mar 2.15 ± 0.10  | 33 ± 0.58       | 5.4 ± 0.11        | 0.24 ± 0.00 | 1.02 ± 0.03 | 16.68 ± 0.12 | 0.19 ± 0.00  | 2.73 ± 0.00  | 14 ± 0.00 |

GMC: Gravimetric moisture content, WHC: water holding capacity, EC: electrical conductivity, BD: bulk density; P: available phosphorus, TN: total nitrogen, SOC: soil organic carbon, S₀: recently mined, S₁: one year old mined, S₄: 4 years old mined, S₁₀: 10 years old mined, S₁₅: 15 years old mined, S: undisturbed soils. Mean ± SD.
Table 2. Enzymatic activity and soil microbial properties of soil samples with different years of extraction of pumice.

| Sample | Feb    | Jun    | Mar    |
|--------|--------|--------|--------|
| S₀     | 5.33 ± 1.15 | 6.67 ± 0.58 | 11.33 ± 1.16 |
| S₁     | 7.00 ± 1.00 | 10.67 ± 0.58 | 12.67 ± 1.16 |
| S₄     | 9.67 ± 0.58 | 6.33 ± 1.10 | 13.00 ± 0.00 |
| S₁₀    | 10.33 ± 0.58 | 4.00 ± 1.00 | 10.67 ± 0.58 |
| S₁₅    | 11.67 ± 1.52 | 10.33 ± 1.53 | 14.00 ± 0.58 |
| S      | 11.00 ± 1.00 | 8.34 ± 2.10 | 16.00 ± 1.73 |

| Sample | (µmoles N-NH₄⁺ g⁻¹ h⁻¹) | Specific activity | (µmoles PNP g⁻¹ h⁻¹) | Specific activity | (µmoles H₂O₂ consumed g⁻¹ h⁻¹) | Specific activity | MBC (mg C kg⁻¹ h⁻¹) | MBC (mg C kg⁻¹ s⁻¹) | CO₂ (µmoles L⁻¹ h⁻¹) | qCO₂ (µmoles L⁻¹ h⁻¹) |
|--------|--------------------------|-------------------|-----------------------|-------------------|-----------------------------|-------------------|-------------------|------------------|------------------|------------------|
| S₀     | 2.70 ± 0.26              | 4.25              | 0.11 ± 0.06           | 2.11              | 246 ± 28                    | 1847 ± 49         | 2.6 ± 0.34        |                  |                  |                  |
| S₁     | 5.22 ± 0.42              | 7.63              | 0.12 ± 0.01           | 3.04              | 134 ± 25                    | 837 ± 39          | 3.1 ± 1.25        |                  |                  |                  |
| S₄     | 2.60 ± 0.04              | 5.90              | 0.09 ± 0.02           | 2.13              | 284 ± 98                    | 1849 ± 22         | 2.2 ± 0.15        |                  |                  |                  |
| S₁₀    | 2.01 ± 0.13              | 1.02              | 0.14 ± 0.02           | 2.43              | 150 ± 19                    | 861 ± 00          | 2.0 ± 0.23        |                  |                  |                  |
| S₁₅    | 1.40 ± 0.09              | 9.83              | 0.29 ± 0.05           | 1.10              | 166 ± 20                    | 1804 ± 16         | 3.8 ± 0.40        |                  |                  |                  |
| S      | 2.76 ± 0.42              | 9.41              | 0.16 ± 0.03           | 3.95              | 150 ± 19                    | 862 ± 00          | 2.0 ± 0.23        |                  |                  |                  |

MBC: microbial biomass C, CO₂: respiratory activity, qCO₂: metabolic quotient, S₀: recently mined, S₁: one year old mined, S₄: 4 years old mined, S₁₀: 10 old years mined, S₁₅: 15 years old mined, S: undisturbed soils. Mean ± SD.
Table 3. Results of principal component analysis.

| Variable         | Component | Component | Component | Component | Component |
|------------------|-----------|-----------|-----------|-----------|-----------|
|                  | 1         | 2         | 3         | 4         | 5         |
| GCM              | 0.371     | -0.513    | -0.464    | 0.451     | -0.337    |
| WHC              | 0.634     | -0.037    | -0.655    | 0.271     | -0.132    |
| pH               | -0.864    | 0.151     | 0.081     | 0.303     | -0.040    |
| EC               | -0.258    | -0.204    | 0.777     | 0.259     | -0.307    |
| Bulk density     | -0.019    | -0.125    | 0.055     | -0.957    | -0.130    |
| MBC              | 0.803     | 0.181     | -0.483    | -0.243    | 0.016     |
| TN               | 0.880     | 0.127     | -0.042    | 0.184     | -0.193    |
| SOC              | 0.762     | 0.193     | -0.043    | 0.386     | 0.353     |
| C/N              | -0.035    | 0.050     | -0.158    | 0.150     | 0.942     |
| Urease           | 0.096     | 0.891     | -0.043    | 0.235     | -0.064    |
| Phosphatase      | 0.523     | -0.694    | 0.240     | 0.173     | -0.018    |
| Catalase         | 0.090     | 0.938     | 0.228     | 0.083     | 0.072     |
| P                | 0.147     | 0.143     | 0.785     | -0.481    | -0.221    |
| CO$_2$           | 0.392     | 0.707     | -0.225    | -0.269    | 0.397     |
| qCO$_2$          | -0.512    | 0.418     | 0.517     | 0.018     | 0.358     |

Bold values indicates the higher weight variables are considered highly weighted within 10% of variation of the absolute values of the highest factor loading in each PC. GCM: gravimetric moisture content, WHC: water holding capacity, EC: electrical conductivity, MBC: microbial biomass C, TN: total nitrogen, SOC: soil organic carbon, P: available phosphorous, CO$_2$: activity respiratory, qCO$_2$: the metabolic quotient.
Table 4. Correlation matrix between the different parameters determined.

| Parameter  | EC  | Catalase | MBC  | SOC   | CO₂  | WHC  | GMC  | TN   | pH   | qCO₂ | Urease |
|------------|-----|----------|------|-------|------|------|------|------|------|------|--------|
| EC         | 1   |          |      |       |      |      |      |      |      |      |        |
| Catalase   | NS  | 1        |      |       |      |      |      |      |      |      |        |
| MBC        | −0.67 | NS  | 1    |       |      |      |      |      |      |      |        |
| SOC        | NS  | NS       | 0.53 | 1     |      |      |      |      |      |      |        |
| CO₂        | −0.58 | 0.67 | 0.64 | NS  | 1    |      |      |      |      |      |        |
| WHC        | NS  | NS       | 0.75 | NS   | NS  | 1    |      |      |      |      |        |
| GMC        | NS  | NS       | NS   | NS   | NS  | 0.74 | 1    |      |      |      |        |
| TN         | NS  | NS       | 0.68 | 0.78 | NS  | 0.64 | NS  | 1    |      |      |        |
| pH         | NS  | NS       | −0.76 | NS | NS  | NS  | NS  | −0.64 | 1    |      |        |
| qCO₂       | NS  | −0.77    | −0.61 | NS | NS  | −0.66 | −0.77 | NS  | NS  | 1    |        |
| Urease     | NS  | 0.84     | NS   | NS   | 0.60 | NS   | NS   | NS  | NS  | NS  | 1      |

EC: electrical conductivity, CO₂: activity respiratory, WHC: water holding capacity, GMC: gravimetric moisture content, TN: total nitrogen, qCO₂: metabolic quotient, NS: not significant, p < 0.01.
**Figure 1.** Dendogram obtained (Ward method) for cluster analysis of different age mined and undisturbed soil. S$_0$: recently mined, S$_1$: one year old mined, S$_4$: 4 years old mined, S$_{10}$: 10 old years mined, S$_{15}$: 15 years old mined, S: undisturbed soils.
Figure 2. Plot of canonical scores calculated by discriminant analysis for the different age of mined soil. $S_0$: recently mined, $S_1$: one year old mined, $S_4$: 4 years old mined, $S_{10}$: 10 old years mined, $S_{15}$: 15 years old mined, $S$: undisturbed soils.