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Physical characterization of an extensive volcanic rock in México: “red tezontle” from Cerro de la Cruz, in Tlahuelilpan, Hidalgo

Caracterización física de una roca volcánica abundante en México: ”tezontle rojo” proveniente del Cerro de la Cruz, en Tlahuelilpan, Hidalgo

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ABSTRACT

Tezontle is an extrusive volcanic rock, coming in black or red in color. Despite its many uses and applications, there are few studies concerning the attributes of this rock. Therefore, this work to the physical characterization of red tezontle samples, taken from Tlahuelilpan, Hidalgo, México. After grinding and sieving, the following properties were obtained: humidity (0.37% to 0.48%), weighed moisture-retention capacity (7.50% – 43.56%) and volumetric moisture-retention capacity (5% – 25%), which justifies its hydroponic and gardening uses; particle density (2.37 g cm^{-3} - 2.83 g cm^{-3}), dry bulk density (0.87 g cm^{-3} - 1.33 g cm^{-3}), total pore space (52.83% - 63.90 %), specific surface area (5.558 m^2 g^{-1} and 9.66 m^2 g^{-1}) and cation exchange capacity (4.04 Cmol(+) kg^{-1}), whose low values contradict the high efficiency reported for cation removal. The preliminary analysis of the X-ray diffraction pattern shows intense peaks of quartz and suggests the presence of plagioclases (anorthite and albite).

RESUMEN

El tezontle es una roca volcánica extrusiva y de coloración negra o rojiza. A pesar de sus múltiples usos y aplicaciones, existen pocos estudios respecto a sus atributos. Por ello, el objetivo de este trabajo consiste en la caracterización de muestras de tezontle rojo provenientes de Tlahuelilpan, Hidalgo, México. Después de molido y tamizado se determinaron las propiedades: (0.37% - 0.48%), grado de hidratación (7.50% – 43.56%), hinchamiento (5% – 25%), que justifican su aplicación en hidroponía y jardinería; densidad real (2.37 g cm^{-3} - 2.83 g cm^{-3}), densidad aparente (0.87 g cm^{-3} - 1.33 g cm^{-3}), porosidad total (52.83% - 63.90%), área superficial (5.558 m^2 g^{-1} y 9.66 m^2 g^{-1}) y capacidad de intercambio catiónico (4.04 Cmol(+) Kg^{-1}), cuyos bajos valores contradicen la alta eficiencia reportada para remoción de cationes. El análisis preliminar del patrón de difracción de rayos X muestra picos intensos de cuarzo y presencia de plagioclasas (anortita y albita).

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Tezontle; roca volcánica; caracterización física.

Keywords:
Tezontle; volcanic rock; physical characterization.

INTRODUCCIÓN

Physical properties of volcanic materials are subject of interest from physics and geologists, due to the relation with physical processes and thermal compressions during the solidification process (Pola, Crosta, Fusi, Barberini & Norini, 2012).

Tezontle is one of the materials most widely used as a substrate in the production of vegetables and ornamental plants in México, mainly due to its low cost and availability (Meric, Tuzel, Tuzel & Oztekin, 2011). Different proportions of soil and tezontle were evaluated in greenhouse tomato production efficiency (Ojodeagua-Arredondo, Castellanos-Ramos, Muñoz-Ramos, Alcantar-González, Tijerina-Chávez, Vargas-Tapia & Enríquez-Reyes, 2008). Pineda-Pineda, Castillo-González, Morales-Cárdenas, Colinas-León,

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Valdéz-Aguilar & Avitia-García (2008) recommended tezontle as substrate to improve the production and quality of poinsettia (*Supjibi Red*). This volcanic rock is also widely used in construction and decoration.

Tezontle is an igneous rock, reported as pyroclastic scoria (Rodriguez & Castillo, 2001) and is formed by the solidification of mafic magma from volcanic eruptions towards the surface (Hubp, 2002). When magma cools on the exterior of the crust, extrusive or igneous rocks are formed, as it is the case of tezontle (Marsh & Coleman, 2009; Cornelis & Cornelius 1997).

The Cerro de la Cruz is an isolated tezontle hill, located in the Mezquital Valley, on the Trans Mexican Volcanic Belt (Gómez-Tuena, Orozco-Esquivel & Ferrari, 2007; Silva-Mora, 1997). This hill was created during the Miocene Epoch, near the Xithí and La Joya volcanoes. Basaltic and dacitic rocks are observed around the Cerro de la Cruz (Mooser, Montiel & Zúñiga, 1996; Silva-Mora, 1997).

Silva (1997) first reviewed the geochemical composition of more than forty igneous rock samples in the Mezquital Valley area, including calderas and volcanoes. This study reported various compositions, from acidic to alkaline rocks. Their structures differ significantly in SiO_2, Al2O_3 and Fe_2O_3 content.

There is only one report about the physical characterization of a Mexican red tezontle, which in this case is from seven different mines in Celaya, Guanajuato. This work analyzes the effect of particle size (<0.125 mm to 12.60 mm fractions) on the physical properties: particle densities vary from 2.71 g cm^{-3} and 2.17 g cm^{-3}; dry bulk densities, from 1.16 g cm^{-3} to 0.61 g cm^{-3}; total space, from 57.4% to 77.1% and volumetric moisture-retention capacity from 50.2% to 12.9% (Vargas-Tapia, Castellanos-Ramos, Muñoz-Ramos, Sánchez-García, Tijerina-Chávez, López-Romero, Martínez-Sánchez & Ojodeagua-Arredondo, 2008). There is an interesting result in this report: regardless of the actual place where the samples were taken, no significant differences between the properties of the same particle size are observed. The main influence is due to the granulometry, which allows for significant differences.

Tezontle has been studied as a removal material for wastewater treatment. It can be successfully used as a filter bed, together with aerobic processes in municipal wastewater management, which reduced total suspended solids by 80% and chemical oxygen demand (COD) by up to 50% (González-Matínez, Millán-Salazar & González-Barceló, 2010).

Metal ions (Cd^{2+}, Co^{2+}, Cu^{2+}, Hg^{2+}, Mn^{2+}, Ni^{2}, Pb^{2+} and Zn^{2+}) are adsorbed by red and black tezontle in high removal percentages (Ortiz-Polo, Richards-Uribe, Otazo-Sánchez, Prieto-García, Hernández-Ávila & Acevedo-Sandoval, 2007). Also, UO_2^{2+} ion adsorption was also studied considering the surface charge of the tezontle at different pH values (López-Muñoz, Durán-Blanco, Iturbe-García, Olguín-Gutiérrez, 2010).

Considering the potential application of tezontle in environmental technologies, the aim of this work was to characterize the physical properties of red tezontle from another Mexican site, Cerro de la Cruz, in Tlahuelilpan, Hidalgo. We considered it very important for any process to analyze the variations of the physical properties of the tezontle as a material due to the particle size of the samples. This study includes the Specific Surface Area (SSA) and the Cation Exchange Capacity (CEC) characteristics. A preliminary report of X rays diffraction pattern is included. It would be interesting to compare these results with to the previous ones, reported in the state Guanajuato.

MATERIALS AND METHODS

A random sampling was conducted in Cerro de la Cruz, located in the municipality of Tlahuelilpan, Hidalgo, México: 117°13'33'' W and 20°08'57'' N, at 2 060 m altitude. The tezontle (N=14 samples, aprox. 5 kg [11 lbs] each) was collected at different sites (zig zag) of the Cerro de la Cruz hill at a height 1.5m (4.9 ft). The collected material (72.5 kg [159.8 lbs]) was mixed together, crushed in a Glen Creston 19 jaw crusher and then grinded using a Sturtevant roller mill equipped with two smooth rollers each 12.5 cm (4.9 in) in width and 20 cm (7.9 in) in diameter. The representative sample was sieved, washed with deionized water, and air-dried for further analysis.

The physical properties of tezontle were measured three times. An analysis of variance was used to determine the significance of the differences between the figures obtained for each fraction, MINITAB 14 was utilized.

Humidity (*Hum*%) and weighed moisture-retention capacity (*W* %)

A fast and uniform heating MB45 thermobalance with a full digital calibration halogen lamp was used to determine the humidity of the fractions obtained from the natural material. The *W* % was calculated using equation 1 (Nuñez, 2000; Nuñez, 2006; Llorca & Bautista 2006; UNE-EN 13040:2012, 2012).

$$W\% = \left(\frac{W_w - W_o}{W_o}\right) \cdot 100 \ . \tag{1}$$

Where: W_o is the dry weight of the material and W_w is the wet weight after allowing it to reach maximumweighed moisture-retention capacity (over 1h). These masses were weighed on an analytical balance. W_w was obtained after air-drying to remove excess water.

Volumetric moisture-retention capacity (W_s%)

Volumes were determined before and after adding water into a burette. The volume was measured every 5 min after manual agitation. The W_s% is calculated using equation 2.

$$W_s\% = \left(\frac{V_s - V_D}{V_D}\right) \cdot 100 \ . \tag{2}$$

Where: V_s is wet volume and V_D is dry volume.

Particle density (ρ)

Determined for each fraction with a 50 ml pycnometer (SIMAX) at 20 °C using equation 3 (NOM-021-SEMARNAT-2000, 2000). The powder (approximately 5 g [0.18 oz]) is placed inside the pycnometer, and weighed. The pycnometer is then filled with a fluid of known density, in which the powder is not soluble. The volume of the powder is determined by the difference between the volume as shown by the pycnometer, and the volume of liquid added (i.e. the volume of air displaced).

$$\rho = \frac{S}{S + A - (S + a)} = \frac{S}{A - a} = \frac{S}{V_s} \ . \tag{3}$$

Where: ***S*** is the sample weight, ***A*** is the water weight of the total pycnometer volume and ***a*** is the water weight of the pycnometer volume not occupied by the sample. Finally, the sample volume $\boldsymbol{V_s}$ is in the denominator.

Dry bulk density (ρA)

The graduated cylinder method was used. It consists of filling it with samples up to the line and then recording the weights. Equation 4 was used.

$$\rho A = \frac{\overline{X}}{V} \ . \tag{4}$$

Where: X is the average of the weights of the sample and V is the volume of the test-tube.

Total pore space (P %)

Determined by equation 5 (Leiton-Soubannier,1985; Soriano & Pons, 2001; Porta, López-Acevedo & Poch, 2008).

$$P\% = 100 - \left(\frac{\rho_A}{\rho}\right) \cdot 100 \ . \tag{5}$$

Where: ρA is the particle density, ρ is the dry bulk density.

Specific surface area (SSA)

The BET method was used with Micrometrics ASAP 2050. Each sample (0.5 g [0.18 oz]) is degassed for 2 h and then nitrogen adsorption isotherm at 77 °K is recorded.

Cation exchange capacity (*CEC*)

Expressed in Cmol(+) kg^{-1}, it is calculated using equation 10 with 1N solution of ammonium acetate, pH 7.0.

$$CEC = 200\ (V)\ (N) \ . \tag{6}$$

Where: V is the HCl solution volume (ml) and N is the HCl concentration (mol L^{-1}).

Phase analysis by X-ray diffraction

A D8 ADVANCE in Bragg-Brentano configuration was used connected to a copper anode. The components were identified using the Joint Committee on Powder Diffraction Standards, International Centre for Diffraction Data database (JCPDS-ICDD).

RESULTS AND DISCUSSION

Percent Humidity (*Hum*), percent volumetric moisture-retention capacity (W_s) and percent weighed moisture-retention capacity (W)

Table 1 presents the physical characterization of red tezontle fractions with different mean particle diameters: >2000 µm, 1425 µm, 725 µm, 512.5 µm and < 425 µm. The humidity (% *Hum*) of the natural material is very low in all fractions, from 0.37% to 0.48%. This feature is typical of tezontle since it is a non-clay volcanic mineral rock with very low content of organic matter (Hernández-Jiménez, Ascanio-García, Morales-Díaz, Bojórquez-Serrano, García-Calderon & García-Paredes, 2006) compared to zeolites, which are characterized by the ability to retain and release water without modifying their structure (Chica-Toro, Lodoño-Benítez & Álvarez-Herrerra, 2006).

W_s value is higher in fractions of smaller particles, since the grinding process enables the exposure of occluded pores while increasing the specific surface of the material. Therefore, interaction with water is favored. This result agrees with the swelling kinetics (W_s) for different size particles, as seen in figure 1. The smaller particles achieve equilibrium in less than 1 min and the plateau corresponds to the smaller ones W_s (5%). Figure 1 shows that the larger diameter particles (> 2000 μm to 512.5 μm) fractions W_s% achieve equilibrium in 15 min-20 min.

Table 1.
Physical characterization of red tezontle from Cerro de la Cruz.

Ø (μm)	*Hum* (%)	*Ws* (%)	*W* (%)	ρ (g cm^{-3})	ρA (g cm^{-3})	*P* (%)
>2000	0.41	25.0±0.3a	7.5±0.2a	2.37±0.04a (2.26)	0.87±0.02a (0.64)	63±1a (76.3)
1425	0.43	25.0±0.2a	17±1b	2.68±0.04b (2.44)	1.088±0.005b (0.67)	59.4±0.2b (75.2)
725	0.37	20.00±0.04b	31.8±0.7c	2.74±0.04c (2.56)	1.042±0.005c (0.75)	62.0±0.2a (72.4)
512.5	0.46	20.0±0.2b	43.6±0.6d	2.80±0.04c (2.60)	1.0±0.1d (0.80)	64.2±0.6a (70.7)
<425	0.48	5.00±0.00c	30.5±0.8c	2.83±0.04c (2.70)	1.3±0.2d (1.03)	52.8±0.7c (62.13)

Column results with the same letter have no significant differences. (Turkey, $p \leq 0.05$). **Ø** = mean particle diameter, ρ = particle density, ρA = dry bulk density ***P*** = Total pore space; ***Hum*** = Humidity; ***W*** = weighed moisture-retention capacity and W_s= volumetric moisture-retention capacity.
Source: Authors own elaboration.

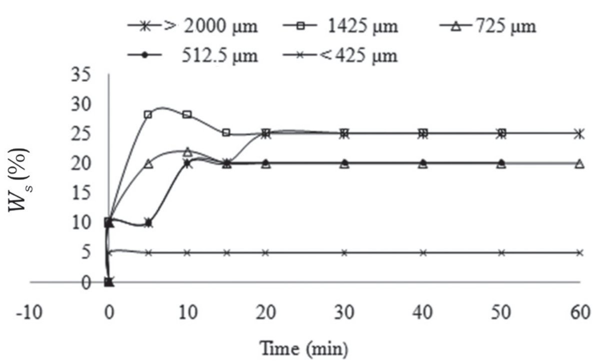

Figure 1. Swelling kinetics of different mean-particle-diameter fractions of red tezontle.
Source: Authors own elaboration.

Fractions of 725 μm and 512.5 μm show similar behavior as the graph lines superimpose. It is generally observed that as the average diameter decreases, W_s drops. As W_s values are based on volume measurements, lower graining fractions show greater compactation with the addition of water and the powder stacks, therefore, volumes shrink (Oelkers & Schott 1995).

Likewise, *W* is related to the weighed water content of the studied fractions. The smaller mean diameter particles show higher *W* values (table 1). It is due to cavities beign exposed as a consequence of the milling process and the greater accessibility of water to the internal pores. Note that the fraction with less graining (< 425 μm), also presents the higher humidity percentage on the starting material.

Particle density (ρ) and dry bulk density (ρA)

The experimental data for ρ and ρA of red tezontle fractions are presented in table 1. ρA values are higher than those determined for ρ, as expected. They are within the range 0.87 g cm^{-3} - 1.33 g cm^{-3} range. According to standards (NOM-021-SEMARNAT-2000, 2000), values are representative of volcanic minerals.

Both increase as the mean-particle diameter decreases, due to the greater compaction that occurs with smaller particles. The same tendency is reported in tezontle samples from the state Guanajuato, but the values obtained in the present work are higher (Vargas-Tapia *et al.,* 2008) for the same particle sizes. The differences between the values are statistical significant ($\rho \leq 0.05$). The difference is might be due to the solidification rate of the pyroclastic material in state of Hidalgo, where gas outflow might be facilitated.

Total pore space (*P*)

P was calculated based on ρA and ρ values, using equation (5) and values are shown in table 1. The calculated percentage includes two types of porosity: one related to the space between grains and the other promoted for the grinding process fractures, which allows a greater pore space due to the closed pores that are exposed to the surface.

P values vary of >2000 μm to 512.5 μm fractions lie in a range of 59.4% to 64.2% with slow or no significant differences. The <425 fraction shows the lowest pore space (52.8%). This fraction behaves differently than the others.

The previous work from state of Guanajuato reported a total pore space of 67.4% for the 1000 μm

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particle size fraction and 70% for the 2000 μm particle size fractions (Vargas-Tapia, *et al.,* 2008). These values are higher than those obtained in this work and are in accordance with the lower ρA and ρ values reported.

This suggests that the volcanic rocks from both sites are not geologically related. Other volcanic materials, such as lavas, present lower % P, due to physical compression during the solidification processes (Pola, *et al.,* 2012).

Tezontle is reported as pyroclastic scoria (Rodríguez & Castillo, 2001) and that is why, %P values are higher than those from lavas.

Specific surface area (SSA)

BET experiments were performed for two particle size fractions: 425 μm and 297 μm. The results are shown in table 2. By way of an illustration, figure 2 shows the N_2 adsorption-desorption isotherm (BET) for the 425 μm size fraction. There is a total pore fill up followed by the desorption process. The graphic also shows the presence of mesoporosity in the material.

Table 2.
BET results of 425 μm and 297 μm fractions of red tezontle from Cerro de la Cruz.

Fraction particle size (μm)	SSA ($m^2\ g^{-1}$)	Pore Volume (cm^3g-1)	Pore diameter (nm)
425	5.6	0.016	11.8
297	9.7	0.019	12.4

Source: Authors own elaboration.

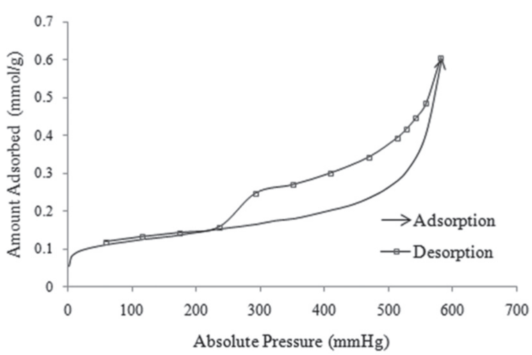

Figure 2. N_2 adsorption-desorption isotherm of tezontle for average 425 μm size fraction.
Source: Authors own elaboration.

Both fractions present low SSA, pore volume and diameter.These small values are in agreement with the low P values (table 1), that are typical of igneous rocks.

Diameter cavities correspond to the classification of mesopores (Everett, 1972), although, considering the low SSA, they might be scarce on the surface.

Despite comprising internal and external areas of the particle, the SSA obtained is smaller than those reported for natural zeolites (17.8m² g-1-25.4m² g-1) (Ruggieri, Marín, Fernandez-Turiel, Gimeno, García-Valles & Gutíerrez, 2008) and very small compared to other synthetic inorganic minerals, such as zeolites with a SSA of 800 $m^2\ g^{-1}$, (Jiménez-Molero, Soto-Camino & Villaescusa-Alonso, 2006) or activated carbon, with SSA 700 $m^2\ g^{-1}$-1402 $m^2\ g^{-1}$ (Giraldo, García & Moreno, 2007). The fact that the studied mineral shows such low specific surface values explains its high density and the moderate P rates. This suggests the presence of closed pores in the volumetric material (Hodson, 2006).

The SSA value of the 297 μm fraction was 9.7 $m^2\ g^{-1}$; almost twice of the 425 μm fraction, due to the smaller size particle. On the other hand, the pores presented the average volume and a mean diameter value similar to the ones obtained from the 425 μm fraction, since it concerns the same material and, therefore shows the same pores.

Cation exchange capacity (*CEC*)

The experimental *CEC* resulted 4.04 Cmol(+) kg^{-1} for all fractions. This is a very low value, according to the standard classification (NOM-021-SEMARNAT-2000, 2000). Moreover, a CEC value below 10 Cmol(+) kg^{-1} indicates the absence of weathered primary minerals and the buildup of secondary minerals.

Although CEC depends on the method used for its determination, this value is very low, as expected for a volcanic rock with no organic matter (García-Rodeja, Nóvoa, Pontevedra, Martínez-Cortizaz & Buurman, 2004). However, values of 20 Cmol(+) kg^{-1} - 44 Cmol(+) kg^{-1} have been reported for volcanic soils (Yatnoa & Zauyahb, 2008), since they depend on weathering and soil formation (Shoji, Dahlgren & Nanzyo, 1993).

Low *CEC* might be linked to the tiny SSA value. The small *CEC* value is contradictory to the high cation removal efficiency of red tezontle from Cerro de la Cruz previously reported (Ortiz-Polo *et al.,* 2007; López-Muñoz *et al.,* 2010). Ion exchange can be caused by isomorphic substitutions in the surface structure of the studied mineral and little negative charges derived from the bond rupture, bringing hydroxyl groups to the surface. But this hypothesis must be proved and further structural and composition studies should be planned.

X-ray diffraction (XRD)

Due to the low *CEC* values, in contrast to the reported high cation removal efficiency of tezontle reported (Ortiz-Polo *et al.,* 2007), a diffraction pattern was recorded for fraction < 425 μm (figure 3) in order to get a first glance of the phases present in the rock. The preliminary mineral composition was proposed, based on the main peaks observed and previous reports on the composition of volcanic rocks in the studied area.

The most abundant primary mineral found in the diffractogram was quartz, whose presence is demonstrated by the peaks: 3127 Å, 3206 Å, 3267 Å and 3215 Å (Besoain, 1985). This is in some way surprising and merits a deeper insight into the structure of this volcanic rock.

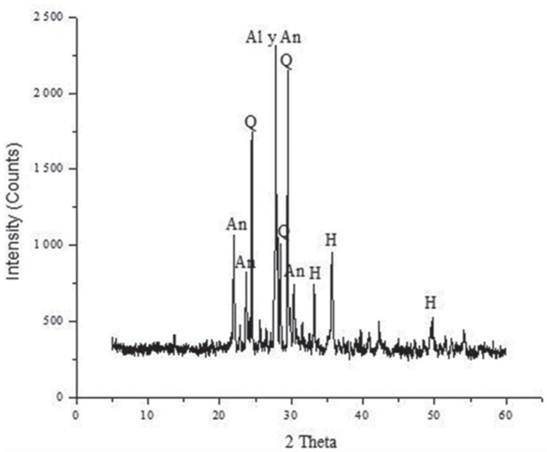

Figure 3. X-rays diffractogram of tezontle. Peaks from: Quartz (Q), anorthite (An), albite (Al) and hematite (H).
Source: Authors own elaboration.

The corresponding quartz phases (SiO_2) peaks, followed by sodium and calcium feldspar (plagioclase) were previously reported for albite Na ($AlSi_3O_8$) and anorthite Ca ($Al_2Si_2O_8$) (del Potro & Hürlimann 2008; Hecker, der Mejide & van der Meer, 2010; Karaguzel, Gulgonul, Demir, Cinar & Celik, 2006) and the peaks were confirmed.

Feldspars structures could be considered to be derived from SiO_2, where tetravalent Si atoms are replaced by trivalent Al atoms. The vacancy is balanced principally by Na^+, K^+, Mg^{2+} or Ca^{2+}. Silicates could be considered as both primary and secondary minerals (Bouabid, Nater & Bloom, 1995; Cheng, Ke, Wang, Wang, Shui & Liu, 2012; Kurama & Ozel, 2009).

Secondary mineral peaks are also observed; Fe oxides (1693 Å, 1452 Å and 1485 Å) like hematite (Fe_2O_3), are considered an impurity within feldspars. Silva, (1997) reported high percentages of SiO_2 and Al_2O_3 in the volcanic rocks studied and some had high proportions of Fe oxides. They vary from 1.3% to 15.2%. The presence of hemathite explains the red color of tezontle.

Further studies must be done to conclude the structure and composition of red tezontle from Cerro de la Cruz, in order to explain the proper qualities for cation removal and to find out how suitable would be be as an environmental technologies material.

CONCLUSIONS

Despite the many uses and applications of mexican red tezontle, few samples have been reported with regard to their physical characteristics, such as particle density and dry bulk density, pore type and cation exchange capacity, and specific surface area. Rocks from Cerro de la Cruz in Tlahuelilpan, Hidalgo, have been characterized and the densities are influenced by the particle size of the selected fraction, and their values are within the range of other previous reports. The specific surface area is very low and this suggests a poor adsorption capacity, despite the measurement being conducted on the smallest particle size fraction (425 μm). On the other hand, the cation-exchange capacity is also low in comparison with other natural rocks, such as zeolites. These results do not explain the previously reported high efficiency of this rock to remove many cations and further physico-chemical studies should be done conducted.

Tezontle has been classified as a pyroclastic scoria and these samples showed complex structures, according to the preliminary analysis of the X-rays diffractogram, which present high quantities of quarz. This fact is some way surprising and merits a deeper insight into the structure of this volcanic rock. In any case, it can be helpful to record the abovementioned properties for a full characterization of this natural material, which is so useful in constructions and landscape gardening and, so functional in preserving plant moisture.

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