



## Characterization of mine tailings in their natural state and stabilized with cement, focused on construction

## Caracterización de residuos mineros en estado natural y estabilizados con cemento, enfocada a construcción

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### Resumen

Este trabajo presenta la caracterización físico-química, geotécnica y mecánica de una muestra de residuos mineros provenientes del estado de Zacatecas, México con el objetivo de evaluar su posible uso como material de construcción de capas de soporte de pavimentos o como agregado de un concreto hidráulico. El material analizado fue clasificado como una arena limosa pobremente graduada (SP-SM) según la clasificación SUCS y se compone principalmente de cuarzo y calcita. Con base en los resultados obtenidos se demuestra que la muestra estudiada cumple con los requisitos necesarios, según la normativa mexicana vigente para ser empleados como: i) capa de subrasante en un pavimento, sin la necesidad de añadir agregados vírgenes, o ii) como agregados finos en la elaboración de concretos hidráulicos, habiendo sido previamente cribados para corregir su granulometría. Los residuos mineros estudiados no requieren estabilización debido a que el material transportado en el proceso de lixiviación se encuentra por debajo de los límites permisibles para metales pesados de acuerdo con las regulaciones mexicanas; esto puede deberse al pH neutro de la muestra, demostrando una alta adsorción de los metales pesados que evitan una potencial lixiviación. Sin embargo, se realizó la estabilización de la muestra con cemento Portland con 3 y 5 %, respectivamente, para corroborar la disminución de transporte de metales pesados con base en la bibliografía estudiada. Las concentraciones de los elementos analizados disminuyeron a excepción del cromo debido a su presencia en el material estabilizador y se observa un aumento en el valor de pH.

**Descriptores:** Impacto ambiental, minas, jales, caracterización, materiales.

### Abstract

This work presents the physical-chemical, geotechnical and mechanical characterization of a sample of mine tailings from the state of Zacatecas, Mexico with the objective of evaluating their possible use as a construction material for pavement support layers or as an aggregate of a hydraulic concrete. The material analyzed was classified as a poorly graded silty sand (SP-SM) according to the USCS classification and is composed mainly of quartz and calcite. The analyzed sample satisfies the necessary requirements, according to current Mexican regulations, to be used as i) a subgrade layer in a pavement, without the addition of virgin aggregates, or ii) as fine aggregates in the production of hydraulic concrete, previously sieved to correct its grain-size distribution. The mining residues studied do not require stabilization because the material transported in the leaching process is below the permissible limits for heavy metals according to Mexican regulations; this may be caused by the neutral pH of the sample, demonstrating a high adsorption of the heavy metals that avoids potential leaching. However, the sample was stabilized with Portland cement with 3 and 5 % respectively to corroborate the decrease in heavy metal transport based on the consulted bibliography; the concentrations of the analyzed elements decreased except for chrome due to its presence in the stabilizing material and an increase in the pH value was observed.

**Keywords:** Environmental impact, mine, tailings, characterization, materials.

## INTRODUCTION

The construction industry demands the massive exploitation of natural resources. An alternative to mitigate the impact of these processes and protect the environment is to use some solid waste from mining processes as partial substitutes for raw materials to generate new engineering materials (Ferreira *et al.*, 2015). This contributes to reduce the extraction of virgin materials and to mitigate the impact of the generation of waste that, due to its large scale production, has become a crucial problem to solve regarding with environmental issues (Novais *et al.*, 2015).

Mining activities generate economic benefits worldwide; however, they also produce large amounts of solid and liquid waste that negatively impact the environment if they are not properly managed (Benzaazoua *et al.*, 2008; Sivakugan *et al.*, 2006). Annual global production of mining solid waste is estimated to be between 20 and 25 billion tons, of which 5 to 7 billion tons are mine tailings (Mudd & Boger, 2013). For the disposal of mining waste there are various methods (i.e., storage in piles or deposits, filling of underground mines and open pits, underwater disposal and disposal of slurries); depending on the method, there will be migration of contaminants into the soil, water or air, generating environmental and health problems (Arcos, 2017).

Orozco and Orozco (1992) define a mine tailing or waste as the non-commercial residue of mining processes with characteristics generally similar to a sand, which are deposited in storage dams.

The reuse of mining tailings might help to mitigate the problems mentioned, and could have different applications depending on their physical and chemical characteristics that depend on their mineralogy and geochemistry, type of mining equipment, particle size of the material and moisture content (Lottermoser, 2010). Since the middle of the last decade, the effectiveness of the reuse of mining waste in ceramic products, glass, glass ceramics, cement blocks, bricks, concrete and high performance concrete has been investigated with positive results, resulting in an increase in the tendency to use them for geotechnical and construction purposes (Roy *et al.*, 2007; Choi *et al.*, 2009; Ramesh *et al.*, 2012; Zhu *et al.*, 2015; Zhao *et al.*, 2014). However, Benzaazoua *et al.* (2002) make evident the potential risk of leaching trace elements from untreated tailings, which makes their application in geotechnical engineering as substitute aggregates and fillers for landfills, structural fillers and road construction materials difficult, indicating that stabilization treatment is necessary before their use for construction purposes. The stabiliza-

tion consists of encapsulating the contaminants, making them insoluble in a stable pH environment and propitiating chemical stabilization, particularly by breaking the bonds of the metals, which reduces their potential pollution and toxicity (Voglar & Leštan, 2011). This stabilization can be performed by biological, chemical and/or physical methods, impeding the spread of contaminants in the soil by reducing their leaching and bioavailability.

In regards to this, several investigations (Harter, 1983; Gerriste y Van Driel 1984; Naidu *et al.*, 1994) indicate that there is a direct correlation between soil pH and the retention of heavy metals, resulting that the basic soils present a greater adsorption of heavy metals than those with acidic pH.

In particular, the adaptation of these waste for their use in road construction is easy and appropriate because they can present good engineering characteristics (shear, compressive and tensile strength, compaction, compressibility, permeability and low erodibility by rain) whether or not their properties are modified by the addition of Portland or asphalt cement (Sultan, 1978; 1979). Mahmood and Mulligan (2010) studied the possible use of mine tailings mixed with Portland cement as unpaved road bases, finding that the mixtures produced with the materials from five different mines, of the six sampled, have enough characteristics and are suitable for use as support layers based on unconfined compressive strength tests. Qian *et al.* (2011) used granite mill tailings stabilized with 5 % of cement in a sub-base layer, which presented simple compressive and tensile strengths similar to cemented stabilized stone aggregates, and with higher static and dynamic modulus than them; they constructed a 20.38 km test section using these tailings as a subbase, proving the viability of their use as a substitute to virgin aggregates. Ojuri *et al.* (2017) experimented with the use of mine tailings from an iron mine to form the subgrade layer; they made test specimens with different percentages of lateritic soil, mine tailings and a combination of lime-cement with ratio 1: 2, which were tested for CBR, simple compression, leaching and mineralogical composition; the addition of mining tailings to the layer decreased the consistency limits and the percentage of material passing the no. 200 sieve, as well as increasing the CBR values and simple compression resistance; to guarantee the safety of the mixture, they found that stabilization with cement and lime at a rate of 8 % brought the quantity of leachates to levels accepted by the regulations.

The results of the investigations suggest that the use of mine tailings in the formation of support layers in the pavement structure is feasible, as long as the characte-

ristics of each material are determined and it is placed accordingly within the structure (Oluwasola *et al.*, 2015; Mahmood y Mulligan, 2010; Quian *et al.*, 2011; Ojuri *et al.*, 2017). However, the index, grain-size distribution and engineering properties vary significantly among the samples studied by each author. Due to this discrepancy, it is not possible to generalize the expected results for all materials.

In México, there are hundreds of millions of tons of mine tailings dispersed without an adequate control of storage conditions and environmental affectations, but few resources are assigned to carry out the diagnosis and risk evaluation of the existing tailings dams (Ramos y Siebe, 2006). The sites that are most affected by heavy metal contamination derived from mining are the states of Zacatecas, Querétaro, Hidalgo and San Luis Potosí (Covarrubias y Peña, 2017). Zacatecas is the second largest mining producer in the country. In the period between 1982 and 2014, for the extraction of gold, silver, copper, lead and zinc minerals, 17025 million tons of solid waste were generated, producing 1897.9 million kilograms of cyanide that have contaminated 55.78 million cubic meters of water per year (Guzmán, 2016).

Therefore, this research focuses on studying mining waste from the state of Zacatecas, in order to determine its possible use within the pavement structure or as an aggregate in hydraulic concrete mixtures and to mitigate the regional problem. In contrast to previous investigations, the material extracted from the sampled tailings dam will be used in the pavement structure or as hydraulic concrete aggregate in its natural state, this means without adding virgin aggregates, in an effort to use as much of the waste as possible and minimize the extraction of virgin aggregates.

## METHODOLOGY

In order to characterize the mining waste and determine its potential use as a subgrade material in the pavement structure or as an aggregate for hydraulic concrete, the sampling and nine laboratory tests described below were performed.

### MINE TAILINGS SAMPLE

The mine tailings sampled corresponds to a storage in a dam, consisting of platforms with berms in which the material is thrown, extended and compacted and accumulated in large volumes. The sampling was carried out following the procedure explained in ASTM D420-18 for the case of integral samples which consist of frag-

mented or disaggregated material from different layers, so that the sample extracted reflects the grain-size distribution characteristics and physical-chemical properties of the material in the field in an integral way (ASTM, 2018). The collected samples were transported and preserved according to ASTM D4220 for samples classified as group B (ASTM, 2014a).

### X-RAY DIFFRACTION (XRD)

The chemical composition of the samples was determined by X-ray diffraction in a Bruker D8 Advance diffractometer. The method involves the interaction of electromagnetic radiation with the sample atoms; for this, the sample is bombarded with an X-ray beam; this diffracted beam reflects the planes of the crystalline structure that interfere constructively. Each plane that satisfies Bragg's law will produce a diffraction peak. The X-Ray diffraction patterns presents the intensity of diffraction observed as a result of the angle of incidence, which is characteristic for each sample. The identification of the phases present is made by comparison with the pattern spectrums available in an international data bank (Ramón y Jiménez, 2006; Litter *et al.*, 2009).

### X-RAY FLUORESCENCE (XRF)

Because the X-ray diffraction technique establishes the mineralogical phases of crystalline compounds present in the sample in concentrations greater than approximately 3-5 %, it was necessary to complement the identification of the sample of mine tailings with the X-ray fluorescence technique, using a PANalytical fluoroscope model Epsilon3XLE. This is a non-destructive emission spectroscopic technique based on the emission of X-rays. The process consists of excitation by applying energy to the system; this increase in energy causes the atoms to become excited, making them unstable. To return to its fundamental state the atom produces jumps of electrons from the external levels to the internal levels to fill the gaps generated, emitting energy in form of X-ray radiation which is called X-ray fluorescence. The measurement of the fluorescence intensities is transformed into mineral concentrations using calibration curves (de Pablo, 1977; Litter *et al.*, 2009).

### LEACHING TESTS

Mining waste may contain potentially toxic compounds that on exposure to water present dissolution processes causing their release, representing a risk to the surrounding environment. Therefore, leaching tests were ca-

ried out on the sample in its natural state and stabilized with 3 and 5 % of Portland cement, with the aim of accelerating the release of the chemical components of the sample so that the potential contamination of the environment by these components over a long period of time could be known. For this investigation, the ASTM D4874-95(2014) was followed that is equivalent to (ASTM, 2014b; López & Pérez, 2018).

#### **pH MEASUREMENT**

It is required to measure the hydrogen potential (pH) of the leachates generated to define the acidity or alkalinity levels of the samples according to the conventional pH scale that goes from 1 to 14, being classified from 1 to 6 as acid, 7 as neutral, and 8 to 14 as alkaline or base. The most convenient way to obtain the pH of an aqueous solution is by using a pH meter, which measures the difference in electrical potential between a pH electrode and a reference electrode (Jarpa, 2003). For the test, a HANNA model Combo Grochek instrument was used, based on ASTM E70-19 (ASTM, 2019).

#### **ATOMIC ABSORPTION SPECTROSCOPY (AAS)**

The leachate obtained was analyzed with the technique of atomic absorption spectroscopy that determines the specific concentrations of each element that exists in the analyzed sample. One of the different atomic absorption techniques is atomic optical spectrometry, based on a process called atomization in which the elements present in the sample are converted into atoms or elemental ions in their gaseous state. The most commonly used methods to achieve the atomization of a sample are flame atomic absorption (FAAS), in which the elements copper, cadmium, zinc, lead, chrome and nickel were analyzed in a PerkinElmer Wallac model AAnalyst 100, and the use of electrothermal energy in a graphite furnace (GFAAS), to analyze arsenic using a Varian model SpectrAA. The principle of these methods is based on the absorption of light from an element in an atomic state; each element has a specific wavelength at which the light is absorbed, the number of atoms of the element present in the sample is proportional to the amount of radiation absorbed.

The atomic absorption technique with hydride generation executed by a complement in the PerkinElmer Wallac model AAnalyst 100 equipment allows the quantification of elements such as mercury which has the property of forming the corresponding hydride. The sample is dissolved in an acid with a reducing agent; this reaction generates atomic H that reacts with

the analyzed element (Litter *et al.*, 2009, Gallegos *et al.*, 2012, Torres *et al.*, 2016, Calderón *et al.*, 2016).

#### **SOIL CLASSIFICATION**

The sample was classified by the Unified Soil Classification System (USCS) based on the grain-size distribution analysis and Atterberg limits (liquid and plastic limits) of the materials. According to their size, the forces that will interfere in the behavior of the soils will be different; while in the particles with a diameter lower than 0.075 mm the predominant forces are the electrical ones, in the coarse soils the predominant forces are the own weight and the degree of accommodation of the grains; for this reason, knowing the granulometric composition of a sample is important to define their engineering properties (Atterberg limits, relative density of solids, optimum humidity, maximum dry volumetric weight, expansion and CBR) (Narsillo & Santamarina, 2016).

The granulometric analysis of the residues was carried out according to ASTM D6913M-17 in order to know the particle size distribution (ASTM, 2017a). The determination of the size of the particles smaller than 0.075 mm (Sieve no. 200) was carried out by the sedimentation analysis (hydrometer) regulated by ASTM D7928-17 (ASTM, 2017b).

The consistency limits were obtained by the cone penetrometer test established in the BS 1377-2 standard (British Standards Institution, 1990).

#### **RELATIVE DENSITY OF SOLIDS**

Another property of materials used in engineering calculus is the relative density of solids, as its name suggests, is the density of the material in relation to a blank of distilled water at 4° C expressed by a dimensionless value (Villalaz, 2005). This test was followed based on ASTM C128-15 (ASTM, 2015).

#### **MAXIMUM DRY VOLUMETRIC WEIGHT**

The standard AASHTO test was performed to determine the compaction curve for the sample of mine tailings that will be used as material for pavement support layers. The maximum dry volumetric mass and optimum water content were obtained from this test, performed in accordance to ASTM D698-12e2 (ASTM, 2012).

CALIFORNIA BEARING RATIO (CBR)

The California Bearing Ratio (CBR) is a parameter of special interest for analyzing the feasibility of a material to be employed in a pavement structure (Talukdar, 2014). It relates the shear strength of the soil for given compaction and moisture conditions, and is expressed as the percentage of load required to insert a piston at a given depth into a soil sample compared to that required to insert it at the same depth into a standard material, thereby classifying the material according to its possible uses within the pavement structure (Villalaz, 2005).

To obtain the CBR and the expansion of the analyzed material the manual M-MMP-1-11/08 was followed applying the specific energy of compaction for a subgrade material correspondent to ASTM D1883-16 (SCT, 2008; ASTM, 2016).

RESULTS AND DISCUSSION

The mine tailings sample for this investigation was obtained from a mining deposit from a mine located in Zacatecas, where four main minerals are extracted: copper, silver, zinc and lead by the flotation extraction process. Due to the nature of the work carried out and the sensitivity of some data, the complete confidentiality of the sampled tailings dam company was arranged, as it does not influence the analysis of results.

The chemical composition obtained by X-ray diffraction shows the presence of quartz, calcite, sanidine, microcline, amesite, clinoclhorine and grossular. The X-Ray diffraction pattern of the sample is detailed in Figure 1.

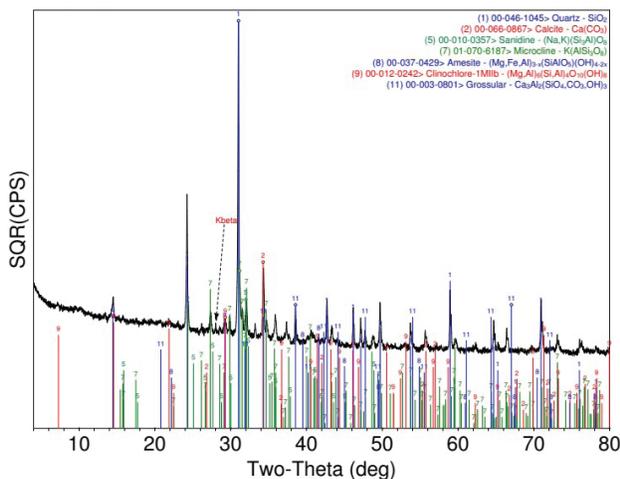


Figure 1. X-ray diffraction pattern resulting from the analyzed mine tailings. Source: self-made

The sample is mostly composed by quartz and calcite, while the remaining compounds present are characteristic of the rock containing the minerals with commercial value (Moreno *et al.*, 2011).

The results obtained from X-ray diffraction show us a general overview of the components that constitute the sample; however, for this investigation it is crucial to determine the amount of heavy metals present in the sample as they are potentially contaminating elements for the environment. The 64 elements that compose the material were analyzed, emphasizing the twelve elements contemplated in the Mexican Official Standard NOM-147-SEMARNAT/SSA1-2004 that establishes the maximum permissible concentrations in contaminated soils by heavy metals, the results are shown in Table 1 (SEMARNAT, 2004). It can be determined that, in its natural state, the sample of mine tailings is not adequate as soil for agricultural, residential or commercial use because it exceeds the permissible limit of arsenic.

Table 1. Identification of heavy metals by X-ray fluorescence compared to Mexican standards

Element	Reference value for agricultural/residential/commercial use (mg/kg) <sup>a</sup>	Sample (mg/kg)
Arsenic	22	238
Barium	5400	No detectable
Beryllium	150	No detectable
Cadmium	37	No detectable
Chromium	280	189
Mercury	23	No detectable
Nickel	1600	19.4
Silver	390	No detectable
Lead	400	44.5
Selenium	390	16.5
Thallium	5.2	No detectable
Vanadium	78	No detectable

Source: self-made and <sup>a</sup> with data of SEMARNAT (2004)

To emulate the transport of heavy metals by exposure to rain, the material was subjected to a leaching test. Subsequently, the pH of the leachate generated was measured, obtaining a value of 7.29, considered to be neutral. The eight heavy metals considered in the Mexican regulations were analyzed to determine the innocuousness of the material through the three atomic absorption techniques. The values are below the permissible limits, so it is concluded that the material does not require stabilization. Even with this, and based on the previously mentioned investigations (Mahmood y Mulligan, 2010; Quian *et al.*, 2011; Oluwasola *et al.*, 2015; Ojuri *et al.*, 2017) the same tests were done for two mixtures of mining waste stabilized with percentages of 1

and 3 % ordinary Portland cement in order to analyze the effect of encapsulation on those elements that were detectable in the leachate. The results obtained with the reference values of the regulations based on NOM-001-SEMARNAT-1996 are shown in Table 2 (SEMARNAT, 1996).

Leachates obtained from cement-stabilized samples show an increase in pH as a result of the percentage of cement, which increases the adsorption of heavy metals from the material, decreasing its leaching potential, as reported in the literature (Harter, 1983; Gerritse y Van Driel 1984; Naidu *et al.*, 1994). The increases in the levels of chromium compared to the leachate in its natural state are caused by the presence of this element in the portland cement (analysed by the atomic absorption spectroscopy technique), so it is recommended in future research to experiment with different alternatives of cements.

The sample of the residues were subjected to geotechnical and mechanical tests to evaluate their possible

use as a subgrade layer in a pavement. The results of the tests, as well as the compliance values for the layer mentioned according to the Mexican Standard N-CMT-1-03/02, are summarized in Table 3 (SCT, 2002b).

Based on Table 3, it is determined that the material has a maximum size, liquid limit, plastic index and expansion below the maximum specifications according to the regulations. It presents a CBR greater than the minimum required to satisfy the characteristics required to be used as a subgrade layer and, due to the fact that it does not run the risk of potential leaching, it is not necessary to apply a stabilization treatment. The characteristics obtained from the tests carried out were compared with the current Mexican Standard N-CTM-2-002/02 for quality of hydraulic concrete aggregates. Table 4 shows the values required for fine aggregate; the liquid limit is in the range of 26 to 35, it does not present plasticity and its fine content is less than 15 % recommended for non-plastic materials (SCT, 2002a).

Table 2. Concentrations of elements in the leachate by atomic absorption

Element	Reference value for natural moisture (mg/l) <sup>b</sup>	Sample in natural state (mg/l)	Sample with 1% Portland cement (mg/l)	Sample with 3% Portland cement (mg/l)
pH	-	7.29	11.87	12.12
Arsenic	0.10	0.0498	0.0411	0.0303
Copper	4.00	No detectable	No detectable	No detectable
Cadmium	0.10	No detectable	No detectable	No detectable
Chromium	0.50	No quantifiable	0.5583	1.4292
Mercury	0.005	No detectable	No detectable	No detectable
Nickel	2.00	0.7069	No detectable	No detectable
Lead	0.20	No detectable	No detectable	No detectable
Zinc	10.00	No detectable	No detectable	No detectable

Source: self-made and <sup>b</sup> with data of SEMARNAT (1996)

Table 3. Summary of particle size, index properties and strength tests compared to subgrade requirements

Characteristic	Requirements for subgrade <sup>c</sup>	M-1
Classification	-	SP-SM
Maximum size (mm)	76	6.3
Minimum size (mm)	-	0.00065
Relative density of solids	-	2.91
Fine content (%)	-	9.839
Liquid limit (%)	40 máx.	27.24
Plastic limit (%)	-	No present
Plastic index (%)	12 máx.	No present
Maximum dry volumetric weight (kg/m <sup>3</sup> )	-	1795.2
Optimum moisture (%)	-	14.50
Expansion (%)	2 máx.	1.14
CBR (%)	20 mín.	33

Source: self-made and <sup>c</sup> with data of SCT (2002b)

Table 4. Fine aggregate characteristics compared to standards

Characteristics of the analyzed mine tailings sample			Quality requirements of fine aggregates for hydraulic concrete		
Sample liquid limit (%)	Sample plastic index (%)	Fine content (%)	Liquid limit (%) <sup>d</sup>	Plastic index (%) <sup>d</sup>	Maximum fine content (%) <sup>d</sup>
27.24	No present	9.839	From 26 to 35	Up to 5	15
				From 5 a 10	11
				From 10 a 15	7

Source: self-made and <sup>d</sup> with data of SCT (2002a)

Figure 2 shows the resulting particle size curve of the mining waste sample compared to the particle size limits for fine aggregate of hydraulic concrete materials. Based on this, it is possible to use the material, after adjusting its particle size as fine aggregate in the fabrication of hydraulic concrete. Due to the fact that there is an excess of material passing through the 200 sieve, two methods of particle size correction are recommended. The first consists of sieving the material through a mesh with an opening of 0.075 mm in order to remove the fine particles from the material, while the second consists of washing the material using industrial methods in mills specifically designed for this purpose. In both cases, the residues must be returned to the dam from which they were initially extracted and which complies with the regulatory requirements for this purpose. It is important to note that additional tests for reactivity with cement alkalis and accelerated weathering are required to confirm the potential use of the waste as fine aggregates in hydraulic concrete mixes. With respect to the grain-size distribution required to comply as a subgrade layer, the material has the necessary characteristics, considering that the regulations that were taken as reference only establish the maximum particle size which is 76 mm and there is no minimum limit of compliance, as shown in Table 3 (SCT, 2002b).

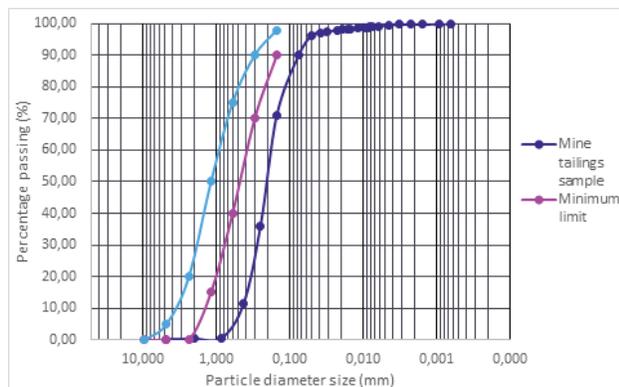


Figure 2. Particle size of the sample compared to standards. Source: self-made with data of SCT (2002a)

## CONCLUSIONS

The sampled residues have the necessary characteristics of grain-size distribution, resistance and consistency limits to be used as subgrade material according to the Mexican Standard N-CMT-1-03/02 (SCT, 2002b). The material can also be used as fine aggregate in the elaboration of hydraulic concrete according to standard N-CMT-2-02-002/02 (SCT, 2002a), being subjected to a method of particle size correction, either sieved through a 0.075 mm mesh or washed in mills, in order to reduce the content of fines in the material and performing the rest of the necessary tests such as reactivity with the alkalis in the cement and accelerated weathering.

The stabilization of mine tailings was not necessary because the material transported by leaching is below the permissible heavy metal limits according to NOM-155-SEMARNAT-2007 (SEMARNAT, 2007). This may be due to the neutral pH of the sample, which indicates a high adsorption of heavy metals that would avoid their migration. On the contrary, there was an increase in the chromium content of the leachates after the addition of different percentages of Portland cement. Therefore, the chemical composition of the cement used for the stabilization and its effect on the transport of contaminants by leaching should be considered before employing it as a building material in engineering works.

The construction industry requires materials and soils for different purposes, which means the massive extraction of virgin resources. The characterization of mine tailings as possible substitutes for these raw materials represents an opportunity to mitigate the environmental impact generated by mining activity and the overexploitation of material banks. However, it is necessary to characterize each mine tailings dam to determine the possible uses of these waste.

## ACKNOWLEDGEMENTS

I want to give special thanks to the Center for Academic Studies on Environmental Pollution (CEACA) of the Autonomous University of Queretaro (UAQ) for allowing the atomic absorption tests to be carried out. This project was financed through the Rectory Special

Projects Fund (FOPER) through contract FOPER 2019-01056 and by a master scholarship of National Council for Science and Technology (CONACYT).

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