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Mineralogical and Elemental Characterization of Conventional Asphalt of Peruvian Roadways by X-Ray Diffraction and Energy Dispersive X-ray Fluorescence

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Abstract: This research deals with the characterization of a sample of conventional asphalt, which is used for the manufacture of roadways in the city of Trujillo, Peru. The sample has been designed by using the Marshall method, through which the optimal content of the asphalt mixture was determined for an adequate combination of the aggregates. This method includes the determination of the optimum content of asphalt cement, stability, unit weight, air voids, flow, voids in the mineral aggregate, and flow stability. The Marshall test was carried out after dosing for the traditional mixture. We have used two techniques: energy dispersive X-ray fluorescence and X-ray diffractometry. The identification and quantification of the chemical elements present was carried out by using energy dispersive X-ray fluorescence, and the mineralogical analysis, by using X-ray diffractometry. This work is useful as a source of reference information. Subsequently, it will allow the implementation of procedures for modifying asphalt mixtures to optimize their performance.

Keywords: Asphalt, Marshall method, X-ray diffraction, Energy dispersive, X-ray fluorescence

Introduction

All over the world, there is no human activity that does not require the use of communication routes, whether road, rail, sea, lake, river, or air. The importance and improvement of these routes keep close parallelism with the cultural, commercial, and industrial development of each country; they are the means by which religion, culture and commerce are introduced into the regions of primitive civilization of a country (Vignolo, 1943). There are two concepts closely related to roadways: bitumen and asphalt. Bitumen or bitumen asphalt is a black to dark brown, semi-solid organic material that gradually liquefies when heated. It is usually obtained as a residue from petroleum distillation by using atmospheric or vacuum distillation units. Asphalt is a natural or commercial mixture of bitumen and inert mineral matter. Natural asphalt occurs as surface deposits, or as a colloidal crude oil system near the surface. In America, the term *asphalt* is often synonymous with the material known in England as asphaltic bitumen or bitumen (Whitehead, 2005).

Trujillo is the third most important city in Peru; however, due to various factors, it has a road infrastructure in poor conditions. This could improve with a detailed knowledge of the constituents of roads, which would allow defining asphalt modification mechanisms, and consequently, provide better performance to the roads.

The durability of asphalt pavements is directly influenced by the climate of the place where they are located. In addition, other parameters must be considered, such as the magnitude and frequency of the traffic loads, the properties of the materials that compose it, the characteristics of the subgrade, humidity, and the construction process, among others. Together, these parameters greatly affect pavement performance as well as increase its potential to develop permanent deformation failure (Warrior and Chang, 2015).

Due to the importance of pavements in population dynamics, all its components must comply with technical specifications that guarantee their good performance: from their preparation to the different tests to which they are subjected, depending on the function for which they are intended. In Peru, these tests are regulated by the Ministry of Transport and Communications. Thereupon, it is necessary to delve into the different aspects that are associated with the lifespan of pavements. In this work, we carried out elemental and mineralogical analyses of a conventional asphalt sample obtained by the Marshall method. We expect that the information obtained can serve as a reference for the elaboration of modified asphalts.

Materials and Methods

Before the analysis of the sample, we proceeded as indicated below.

Preparation of the Asphalt Mixture

The aggregates to be used in the asphalt mixture manufacturing are dried at a constant weight, that is, the sample is heated at $105 \pm 0.5\ ^{o}C$ for a certain time, after which it is weighed and then the procedure is repeated until no variation in mass is seen. After drying, the aggregates will be separated by dry sieving into suitable fractions. A detailed procedure for obtaining the asphalt mixture can be found in the Peruvian technical standard MTC E 504.

Method and Physical Techniques Applied

We have used the Marshall method to design the asphalt mixture. Also, the modified asphalt sample has been characterized by using the two techniques indicated below.

Marshall Method

We applied the Marshall test to the conventional mixture because it is the standard method of asphalt mixture design. The asphalt design content in the final paving mixture is determined by the following parameters: stability, unit weight, air void, flow, VMA, and stability/flow; VMA means Voids in Mineral Aggregate. The method is as follows: First, the asphalt content is determined for which the void content is 4%, and then the other parameters calculated and measured for this asphalt content are evaluated and compared with the design criteria. If all the criteria are met, the asphalt design content is optimal. Otherwise, we should make some readjustments or redesign the mix.

Energy Dispersive X-ray Fluorescence (*EDXRF*)

This technique allows detecting the presence of chemical elements with atomic number Z equal to and greater than 13 by detecting the characteristic X-rays emitted by atoms. The energy of these rays increases with Z, and they can be detected as long as they have enough energy to penetrate the window in the detector. Due to this limitation, the Na (Z=11) and Mg (Z=12) peaks cannot be recorded in the EDXRF spectrum. The source used emits X-rays in two components: one spectrum with a continuous distribution from 0 to 30 keV, and the other containing the L and M characteristic X-rays from silver atoms, which are produced by the bombardment of the silver anode by energetic electrons. Consequently, the EDXRF spectrum have three main components: a continuous component provoked by the scattering of the x-rays from the continuous component of the source, a discrete spectrum produced by the scattering of the silver characteristic X-rays from the source, and a discrete spectrum of the characteristic X-rays emitted by the sample according to the elements it contains.

In the Archaeometry Laboratory of the Faculty of Physical Sciences (UNMSM), we determined the chemical composition of the sample by using a portable EDXRF equipment, Amptek brand, with a silver anode that operated at 30 kV and 15 μA. Irradiation was carried out with the following setting: (a) incidence and exit angles of around 45º, (b) distance from sample to X-ray source: 4 cm, and (c) distance from sample to detector: 2 cm approx. The operating voltage and the applied current were regulated so that the counting rate did not exceed 5000 cts/s.

X-ray Diffraction (*XRD*)

This technique allows for the identification of the mineralogical phases in the sample. It is also known as X-ray crystallography and is based on the diffraction of X-rays by matter, especially when it is crystalline. This diffraction is an elastic scattering, which originates interferences. This technique uses a beam of X-rays which, upon encountering a crystal, are reflected in specific directions determined by the wavelength of the X-rays, lattice parameters, and orientation of the crystal.

Thus, we conveyed the sample to the Bizalab private laboratory, where the measurements on the sample carried out by using an Endeavor D4 diffractometer, Bruker model, with a copper tube that operated at 40kV and 40 mA. Likewise, a 2θ angular scan from 5^o to 70^o and a step of 0.02^o/s was used. After obtaining the data, we identified the mineral phases by using the JCPDS-ICDD database. JCPDS means Joint Committee of Powder Diffraction Standards, and ICDD, International Center for Diffraction Data.

Discussion and Results

Table 1 shows the corresponding results of applying the Marshall method. Therein it is specified the parameters of Optimum Asphalt Content, stability, Unit Weight, Air Voids, Flow, VMA, Stability/Flow. The data are suitable so that the asphalt design content is optimal.

Table 1: Data from the Marshall test for the conventional asphalt mixture

Characterıstıcs of the Asphalt Mıxture	
Optimum Asphalt Content (%)	5.30
Stability (kg.)	1215
Unit Weight (kg/m^3)	2.342
Air Voids (%)	4.00
Flow (mm)	3.35
VMA (%)	14.78
Stability/Flow (kg/cm)	3626.87

In the EDXRF spectra, we observe that the peaks are positioned at certain energy values which indicate the presence of the chemical element in the sample. Symbols for the corresponding chemical elements are identified on top of the peaks. The height of the peaks is an indicator of the concentration of the chemical element in the sample. The EDXRF spectrum of the sample is presented in Figure 1. Table 2 details the corresponding quantitative elemental analysis. Figure 2 shows graphically the results of elemental concentration for the sample studied.

Figure 3 shows the X-ray diffractogram of the sample, which. concerning its mineralogical composition, registers the presence of quartz (SiO_2), clinochlore $(Mg,Fe^{2+})_5Al_2Si_3O_{10}(OH)_8$, albite $Na_{0,986}(Al_{1,005}Si_{2,995}O_8)$, and muscovite $KAl_3Si_3O_{10}(OH)_2$. The presence of Muscovite is not desirable, since, from the point of view of road construction, the presence of clay minerals results in greater use of bitumen. In addition, the lifespan of the pavement is reduced due to the low mechanical resistance of clay minerals (Geber and Gömze, 2015). An additional and important consequence of the small particle size is the high surface area to volume ratio (Parker & Rae, 1998). An indicator of the relative importance of surface effects is the specific surface area of a grain, which increases as the particle size decreases (Kaliakin et al., 2014). Thus, the presence of albite is not convenient either, since feldspars increase the specific surface area and decrease the mechanical resistance of the pavement (Geber & Gömze, 2015).

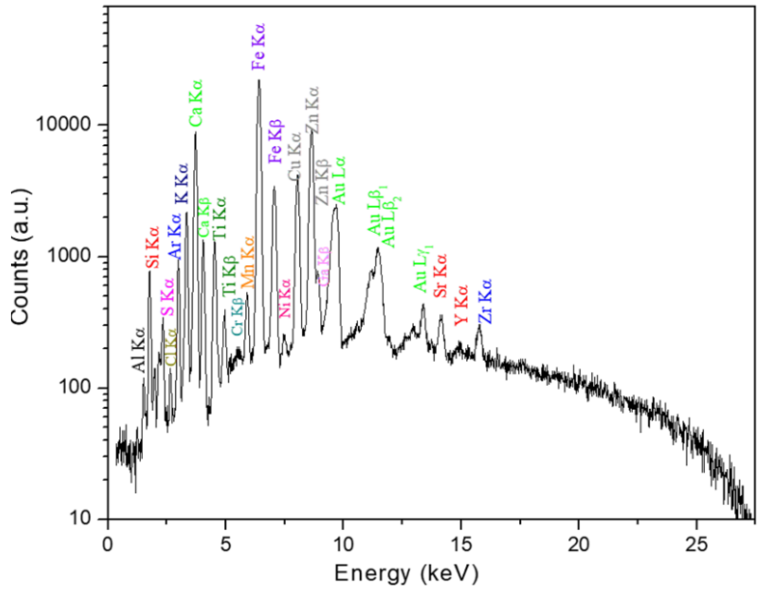

Figure 1. Energy dispersive X-ray fluorescence spectrum of the sample studied.

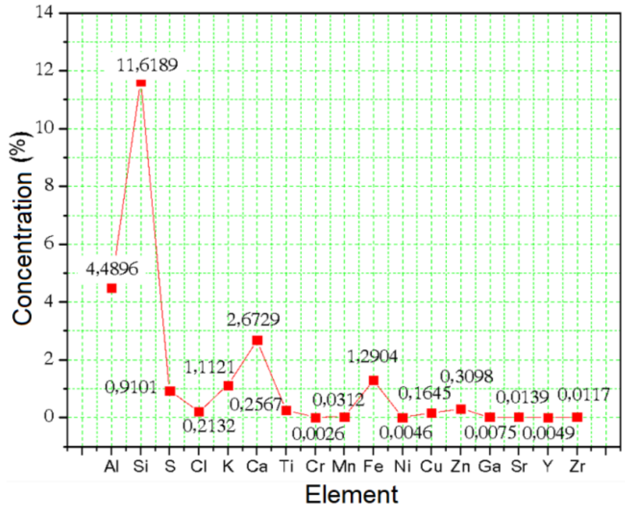

Figure 2. Graphic representation of quantitative analysis of the sample studied.

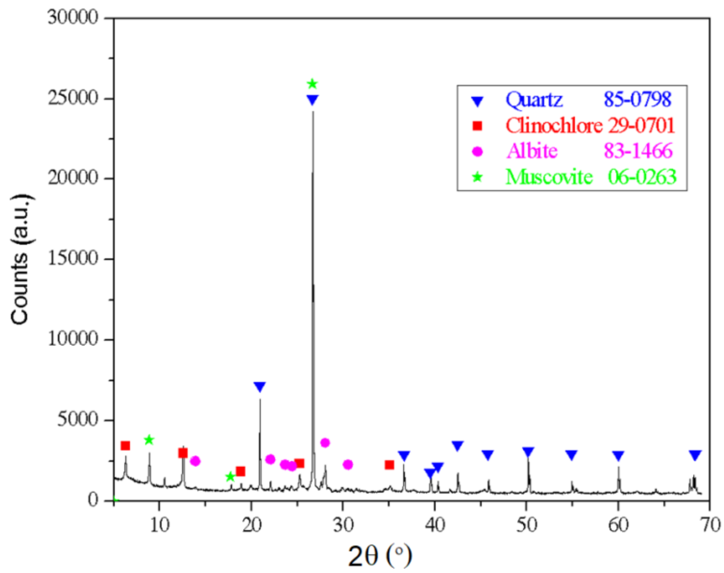

Figure 3. X-ray diffractogram of the sample. We noted the mineral composition and the codes from the JCPDS.

Conclusions

The sample studied presents aluminium, silicon, and calcium as major elements; to a lesser extent, sulphur, potassium, and iron are observed. Chlorine, titanium, chromium, manganese, nickel, copper, zinc, gallium, strontium, yttrium, and zirconium are minor elements. The mineralogical composition of the sample is formed by quartz, clinochlore, albite, and muscovite. We expect that the information obtained is useful so that modification procedures for asphalt mixtures are subsequently implemented to optimize their performance.

Recommendations

This work can be extended with the manufacturing of additional samples by the Marshal method, which have other percentages of optimum asphalt content and VMA.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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