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Comparative analysis between asphaltites and extra heavy oil asphaltenes

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Abstract. The asphaltite material receives many names depending on the country, or area of knowledge, for this reason it is also known as bitumen, asphalt and gilsonite, its composition is a mixture of high molecular weight organic substances, mainly polyaromatic compounds characterized by a black color, and they appear in a semi-solid or solid state. Asphaltenes correspond to the oil fraction with the highest molecular weight, this characteristic provides a high viscosity to hydrocarbons, which contain, in their composition, a concentration equal to or greater than 10%, although the exact structure of this is not known. molecular family, it has been possible to elucidate the functional groups present, such as carbonyl acids, thiols and pyridines, implying the presence of heteroatoms, such as nitrogen, sulfur and oxygen, but the most characteristic part of this type of molecules is their nucleus polyaromatic, which can contain up to 20 or more benzene rings. A sample of Colombian asphaltite was taken and compared with a sample of asphaltenes from a Colombian extra-heavy crude, solubility tests, a spectroscopic characterization by Ultraviolet-Visible, infrared, a thermal evaluation by Thermogravimetry and Differential Scanning Calorimetry were carried out. , in addition to an analysis by X-ray diffraction, in order to determine if the behavior and composition of both The objective of this investigation is to be analyzed by comparing a traditional asphaltite and an asphaltite, if the latter has the same properties and composition, with a view to being used as an energy source .The results obtained reveal that despite the fact that both substances present a similar composition, their glass transitions are different, as well as the crystalline phases present in each of the materials.

1. Introduction

The scarcity of conventional hydrocarbons, added to the high energy consumption of society, forces the search for new resources, which allow to preserve the current quality of life, one of the alternatives as energy of the future, are heavy crude oils, which have viscosities above 1,000,000 cP, thanks to the presence of a high molecular weight and high concentration asphaltenic fraction, another resource that can fill the void of conventional oil are asphaltites, natural solid materials with mainly organic.

The asphaltite material receives many names depending on the country, or area of knowledge, for this reason it is also known as bitumen, asphalt and gilsonite, its composition is a mixture of high viscosity organic substances, mainly polyaromatic compounds of high molecular weight. they are characterized by a black color, and are presented in a semi-solid or solid state

The expression "Asphaltene" originated in 1837 when Boussingault defined them as the Bitumen distillation residue: insoluble in alcohol and soluble in turpentine [1-3]. Currently, asphaltenes are



defined as the heaviest components of petroleum, insoluble in light n-alkanes such as n-pentane (nC5) or n-heptane (nC7) but soluble in aromatics such as toluene.

In order to determine if the behavior and composition of Asphaltite and Asphaltenes are similar, since both have mainly polyaromatic hydrocarbons in their composition, a sample of Colombian asphaltite was taken and compared with a sample of asphaltenes from a Colombian extra-heavy crude oil, solubility tests, a spectroscopic characterization by ultraviolet-visible (UV-Vis), infrared, a thermal evaluation by thermogravimetry (TGA) and differential scanning calorimetry (DSC), as well as an analysis by X-ray diffraction were performed on both samples.[1-3].

2. Materials and methods

The asphaltite samples were collected, in the municipality of Esperanza, department of Norte de Santander, Colombia, in an outcrop of the La Luna formation. The collected samples correspond to solid, organic, bright black, light weight and conchoid fracture material. The asphaltenes were obtained, from precipitation with heptane, making a 1:40 oil-heptane mixture, applying the ASTM D-6560 standard [4], the crude used comes from the eastern plain's region of Colombia.

The solubility of samples was evaluated in two characteristic solvents for hydrocarbons, toluene, and heptane, the first aromatic, while the second aliphatic. 10 grams of sample in 30 ml of solvent was taken. The samples were characterized by infrared (IR) spectroscopy using Bruker Tensor 27 equipment with ATR cell, and the Shimadzu UV-240 IPC equipment was used to perform the characterization by UV-Vis spectroscopy. The determination of the concentration of carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) in the samples was carried out, by combustion gas analysis using the Vario CUBE equipment. The samples thermograms of 30 °C to 1200 °C was obtained at a heating rate of 10 °C/min, this was done using Netzsch 449 F1 equipment. The samples were characterized by X-ray powder diffraction (XRD), using Bruker D8 Advance equipment with DaVinci geometry, and the PDF-4 database [5].

3. Results and discussion

3.1. Solubility test

The solubility of both samples is shown in Table 1, as is to be expected from its way of obtaining, the asphaltene fraction of the extra heavy crude oil is insoluble in heptane, and due to its polyaromatic structure, soluble in toluene, on the other hand the Asphaltite has complete solubility in toluene, and a small fraction is soluble in heptane, probably some type of aliphatic molecules present in its composition.

Table 1. Solubility test.

Sample	Toluene	Heptane
Asphaltite	Soluble	Partially soluble
Asphaltene	Soluble	insoluble

3.2. Spectroscopy characterization

Figure 1 shows the infrared spectrum of asphaltenes and asphaltite, both spectra are very similar, and in them it is possible to detail the signals for the aliphatic and aromatic groups, this result reveals that the organic composition of both samples is very similar, aspect This is verified with the UV-Vis spectrum, shown in Figure 2, where a small signal is observed at 400 nm, corresponding to a polyaromatic nucleus, which, having conjugation of the π bonds, behaves like a chromophore [6,7].

3.3. Elemental quantification

Table 2 shows the elemental composition of both samples, it can be seen that both samples are mostly organic, the rest of the composition of both samples must be oxygen and some inorganic species, in that sense it can be detailed that the Asphaltite has a higher inorganic composition than asphaltene, on the

other hand the content of heteroatoms in both cases is very similar, although it is probable that asphaltene contains more pyridine groups and asphaltite more theopheny groups [8].

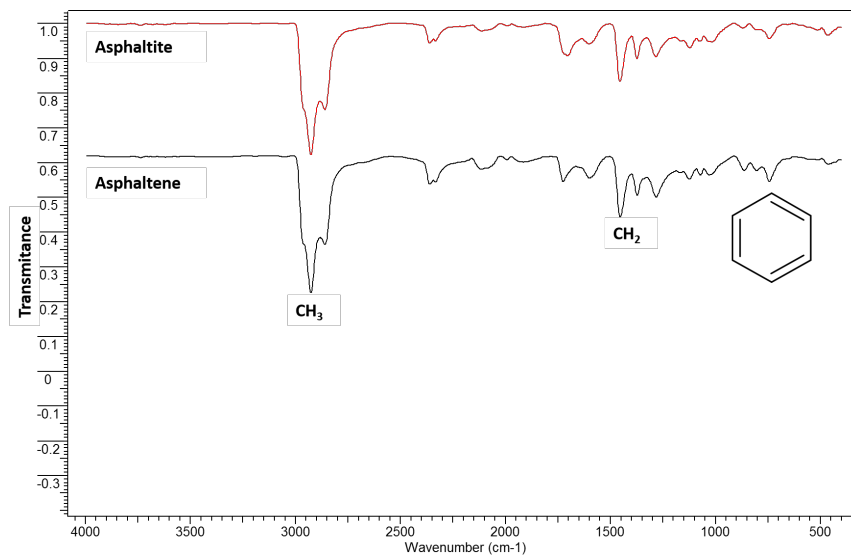


Figure 1. IR spectrum of samples.

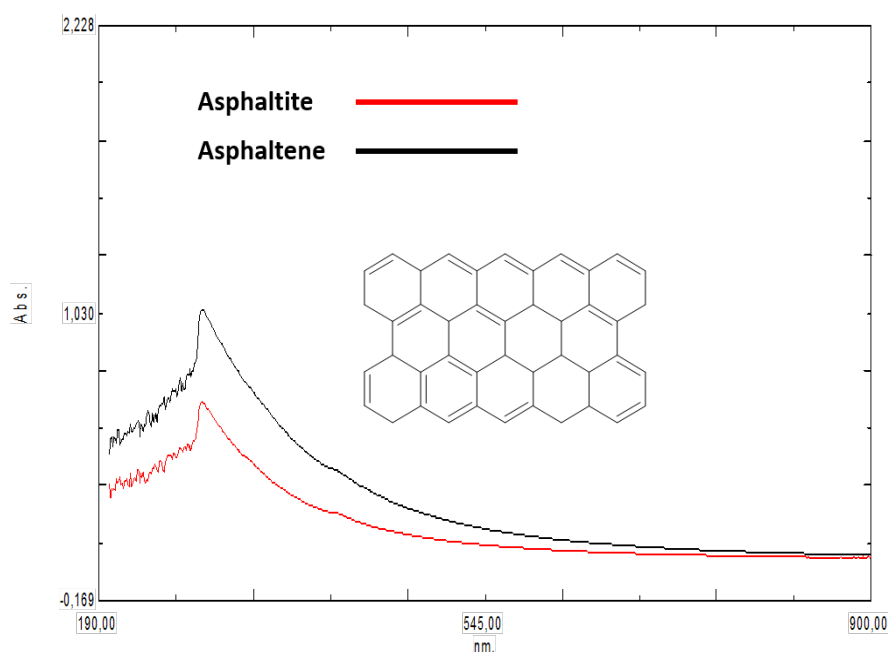


Figure 2. UV spectrum of samples.

Table 2. Concentration of C, N, H and S in samples.

Element	Concentration (%)	
	Asphaltite	Asphaltene
C	78.98	86.1
N	0.86	1.12
H	7.783	9.35
S	4.576	0.175

3.4. Thermogravimetry and differential scanning calorimetry

Figure 3 shows the thermograms for both substances, the behavior of both is expected for two hydrocarbon samples, of organic composition, asphaltite presents a large loss between 200 °C and 5500 °C, equivalent to about 70%, at from this temperature up to 1200 °C the losses are less, since most of the sample is charred, on the other hand the asphaltene, begins to lose mass near 100 °C to 550 °C, but in this case the losses are only 55%, and the subsequent behavior is very similar to that of asphaltite, the residual mass of asphaltenes is 35% while that of asphaltite is 25%, these differences in behavior can be explained, because a low molecular weight fraction still remains in the asphaltites, as observed in Table 1, which contributes to the mass losses, said fraction in the asphaltenes is not present. It is now likely that in the residual mass of asphaltite, the inorganic content is higher than that of asphaltene. [9,10].

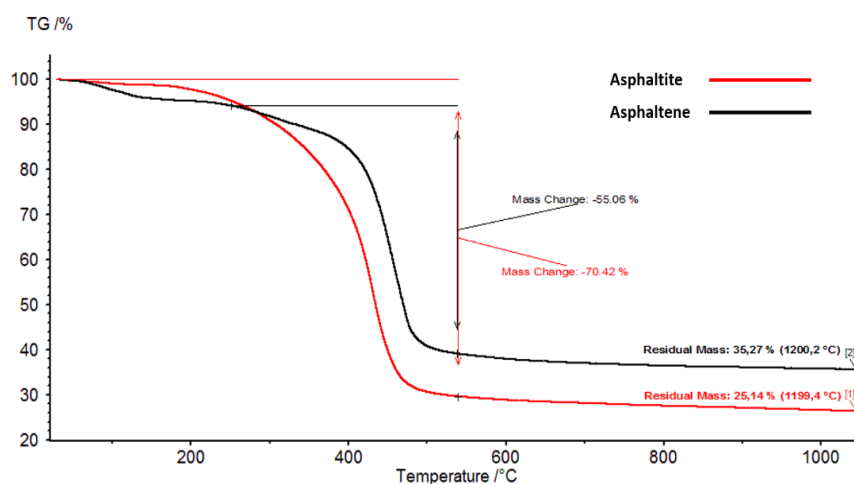


Figure 3. Thermographs of samples.

Figure 4 shows the DSC behaviors, for both samples, different thermal transitions are observed for each sample, asphaltite mainly presents exothermic type transformations, while asphaltene presents both types of transactions.

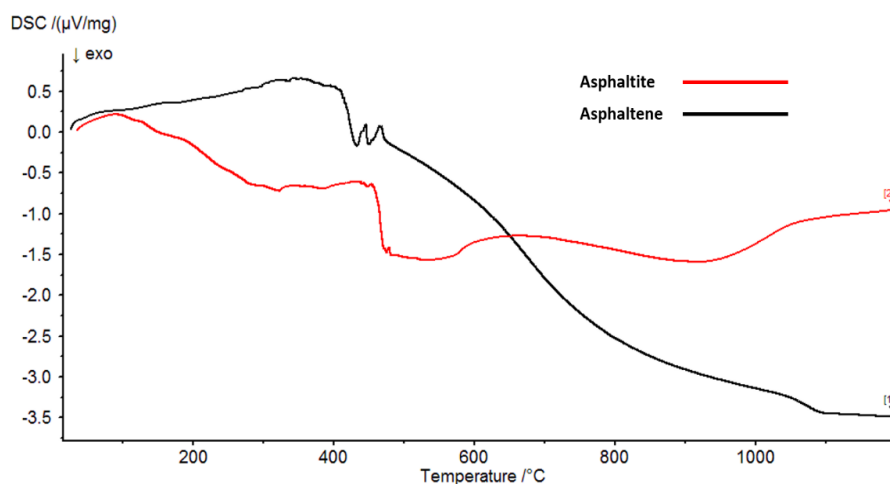


Figure 4. DSC of samples.

3.5. X-Ray diffraction characterization

Figure 5 shows the diffraction patterns for both samples, in which it can be observed that asphaltenes are totally amorphous, characteristic of organic molecules, while asphaltite, in addition to having a certain amorphousness, has crystalline phases, derived from inorganic components. mentioned above.

Figure 6 corresponds to the analysis of the crystalline phases of asphaltite, using the PDF-4 database[5], managing to identify the inorganic compounds present in the sample, see (Table 3), these minerals accompany the sedimentary rock of the La Luna formation, whose deposition environment is related to a shallow marine basin, close to the continent, which explains the presence of minerals such as calcite, Gypsum, pyrite, anatase. These are minerals typical of marine environments, mixed with characteristic minerals of the continent such as kaolinite, muscovite and quartz and other detrital minerals [11,12].

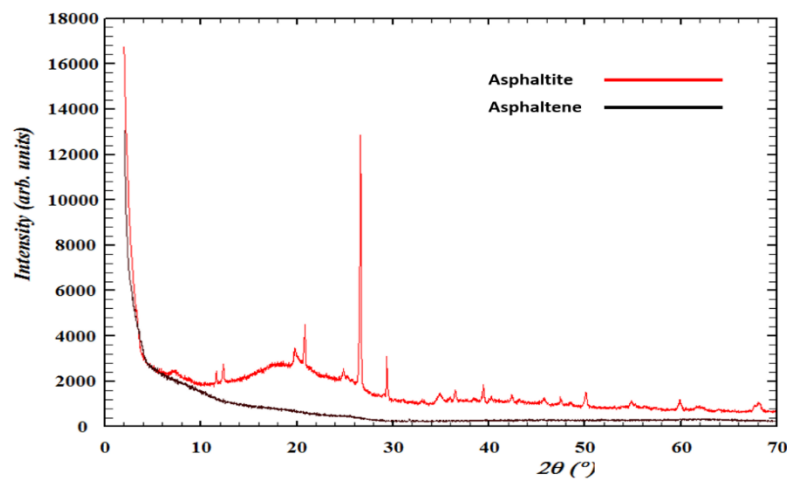


Figure 5. X-ray diffraction pattern for Asphaltite and Asphaltene.

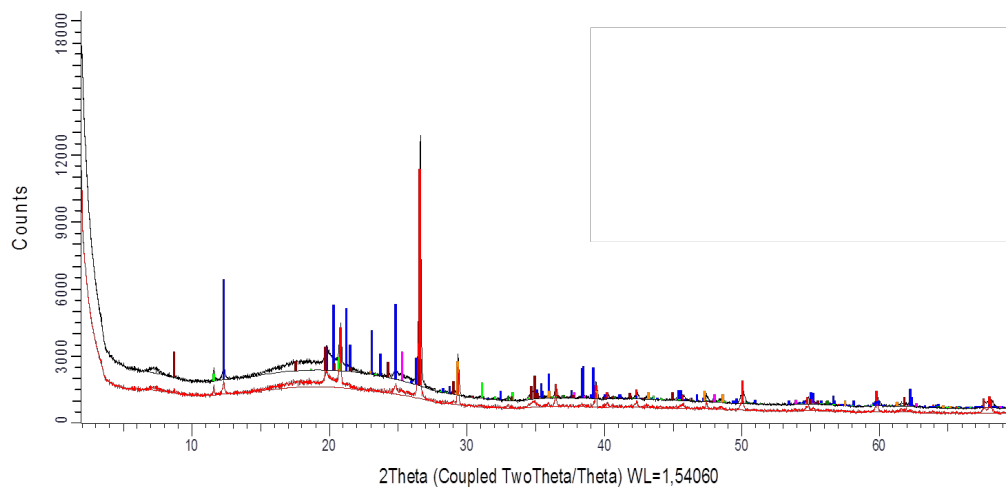


Figure 6. X-ray diffraction pattern for Asphaltite with analysis PDF 4 database from the 2017 International Centre for Diffraction Data.

Table 3. Crystal phases analysis with PDF 4 database from the 2017 International Centre for Diffraction Data.

Molecular formula	Pattern # PDF-4	Compound Name
TiO ₂	PDF 01-070-7348	Anatase, syn
SiO ₂	PDF 01-075-8320	Quartz
KAl ₂ Si ₃ AlO ₁₀ (OH) ₂	PDF 00-007-0025	Muscovite-1M, syn
Fe _{0.987} S ₂	PDF 01-074-8366	Pyrite
CaSO ₄ *2H ₂ O	PDF 00-021-0816	Gypsum
CaCO ₃	PDF 00-001-0837	Calcite
Al ₂ (Si ₂ O ₅) (OH) ₄	PDF 01-079-6476	Kaolinite-1A

4. Conclusions

After conducting a comparative analysis, between the asphaltenes from extra heavy crude and an asphaltite, both of Colombian origin, it was observed that asphaltite, in addition to the high molecular weight polyaromatic components, also contains smaller molecules, as well as a series of inorganic compounds derived from the origin of the sample. Although both substances behave like hydrocarbons, their composition and behavior are not necessarily the same. For that reason, if want to use the asphaltite, like energy source is necessary to consider the inorganic fraction in the material.

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