



The origin of black pigmentation in a sample of Mexican prehispanic human bones

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ABSTRACT

The study of black patches and spots in prehispanic bones of Tlatelolco and Tlapacoya is presented. The chosen characterization techniques, X-ray Fluorescence, X-ray Diffraction, Scanning Electron Microscopy, Energy Dispersive X-ray Spectroscopy, Infrared Spectroscopy and Gas Chromatography are complementary and they all conclude that the bone black pigment is constituted, mainly, by amorphous carbon and some mineral inclusions. Gas Chromatography and Infrared Spectroscopy show that the pigment is constituted by organic compounds as aromatic hydrocarbons, mainly bitumen. An explanation on how the spots were formed is proposed.

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1. Introduction

In Mesoamerica, *post mortem* ritual treatment of bodies is very complex; burial habits vary depending not only on the epoch, the site, or the society, but also on age, sex, and social status or condition of the deceased. One of these elaborated ritual practices is, for instance, the post sacrificial processing which comprises disarticulation, mutilation, disaggregation, or de-fleshing of corpses. To color the human remains is a rather frequent habit (Chávez, 2007).

Black patches on skeletons are not unique to prehispanic cultures. Color over ribs and lower arms of a skeleton from Illyria (Albania) has been interpreted as the remnants of a partial bitumen lining of a shroud or some other item of clothing or ornament (Papadopoulos et al., 2007). Also sherds with bitumen have been reported where it seems that bitumen was applied as a viscous liquid after heating. Black-colored bones have been found in

Hayonim cave, Israel; the color is attributed to burning, those that were not burned, are stained naturally by iron and manganese oxides (Shahack-Gross et al., 1997). In most black bones recovered in the paleontological site of Liñares cave (NW of Iberian Peninsula), color is due to manganese coatings (López-González et al., 2006). Of course, weathering, described as overlapping reactions controlled by water, acid, oxygen, and soil, cannot be discarded as shown by Mansilla et al. (2002) and White and Hannus (1983). Black color in teeth, found in archaeological Tlatelolco burials, may be due to diagenesis (Mansilla et al., 2002); however, black teeth in ceremonial deposits have been attributed to asphalt diffusing into hydroxyapatite, such features have been interpreted as the evidence of foreigners coming from the Veracruz Coast (Gulf of Mexico) to Tlatelolco (Mexico City), probably prisoners (Pijoan et al., 2004; Pijoan, 1997).

Hence, if the bone color is black, it may be due to burning, to painting or staining with manganese oxide, graphite, asphalt or bitumen among others. Either color originally on the body (painted skin, artifacts as jewels, etc.) (Papadopoulos et al., 2007) reached the bones after putrefaction, or, color was intentionally dispersed on the bones as it was done in the Mayan burials using red (Tiessler et al., 2004; González Cruz, 1998). A last possibility, not often mentioned, could be the accidental coloring during the *post mortem* rituals.

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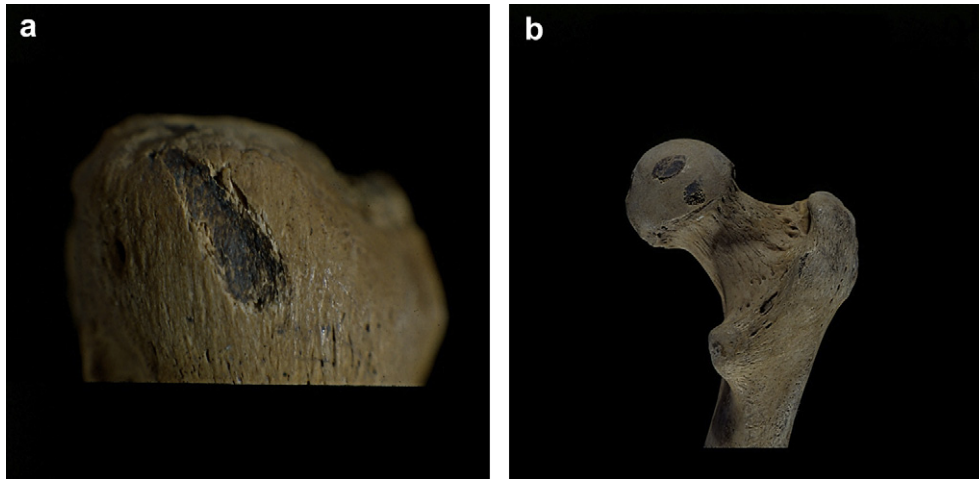


Fig. 1. Black stained bone: a) ulna and b) femur.

Bones with black colored spots are found in ancient burials of Tlatelolco and Tlapacoya. Color is distributed on bone surface as patches and spots. It is generally found on articulation surfaces and it is darker and thicker inside cavities which are man made depressions due to impacts. Periarticular cut marks are present, Figs. 1 and 2.

In this work, small samples of those pigmented bones are analyzed and characterized to find out what are the main components of black color and, then, propose how such color came onto the bone, either by accidental or intentional coloring during the *post mortem* rituals or by staining.

2. Experimental

2.1. Archaeological context

Hernán Cortes and Bernal Díaz del Castillo report that Tlatelolco, now a suburb of Mexico city, accommodated more than sixty thousand people in trading activities. Tlatelolco was a Post-classical (1340–1521 ACE) urban center, it was located on an island in Texcoco Lake. Tlatelolco was the twin city of Mexico-Tenochtitlan. The society, which shared sociocultural traditions with the Mexica group, was characterized by a high degree of

stratification, an extremely complex religion, and a high population density.

Tlapacoya corresponds to an archaeological site located at the foot of the Tlapacoya volcano, southeast of Mexico City, on the former shore of the lake of Chalco. Tlapacoya was a major site for the Tlatilco culture and it is well known because of the Tlapacoya figurines. These sophisticated earthenware statuettes were generally created between 1500 and 300 BCE and are representative of the Preclassic Period. This site was a scattered village community with the longest history of settlement in the Basin (1000–800 BCE). It corresponds to the Preclassical or Formative period (Niederberger, 1987).

Hence, we have two cities, two periods, two very different cultures which both have produced bones with black patches. The difference, if any, between pigmentation mechanism or processes should be evident.

2.2. Samples

Bone samples were selected from the collection of the Instituto Nacional de Antropología e Historia. The specimens are small pieces (*ca.* 0.5 cm) or powders. Two of them (Tlate 1 and Tlate 2) are from burial 14 and sample Tlate3 is from burial 72. The samples

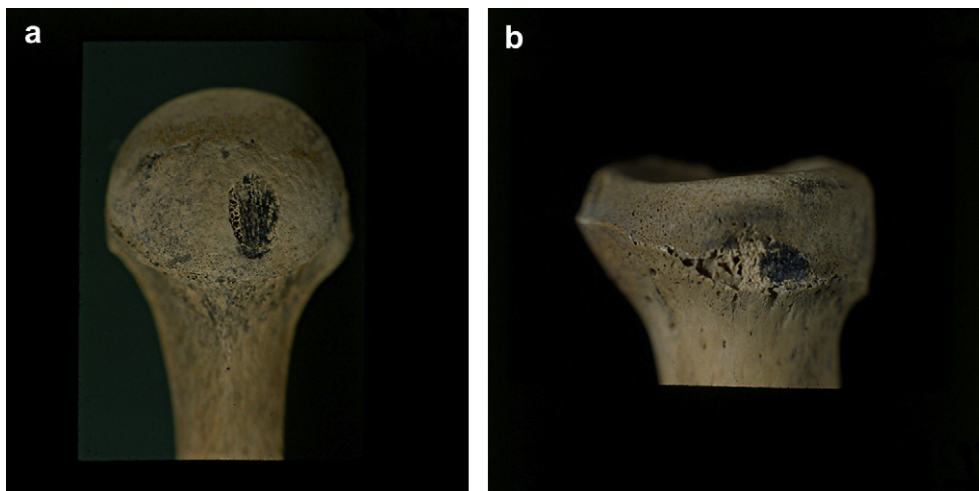


Fig. 2. Black stained bones: a) humerus and b) radius.



Fig. 3. Tlatelolco burial 14, general view.

Table 1

Studied samples.

INAH Classification.	Label
Tlatelolco D.F., burial 14p, humerus 63, Male.	Tlate1
Tlatelolco D.F., M burial 14 M box14	Tlate2
Tlatelolco, D.F., burial 72-H, humerus 19.	Tlate3
Tlapacoya, Edo. burial, IV, A14 layer II 4.20	Tlapa4

were chosen randomly. Both burials were found in the Tlatelolco ceremonial archaeological site in central Mexico.

These burials correspond to two sacrificial deposits, each one containing more than 100 dismembered sacrificed individuals, Fig. 3 (Pijoan, 1997; Pijoan and Mansilla, 2010). A fourth sample from another chronological and geographical area, was chosen to compare the compositions. This sample, Tlapa4, corresponds to a black pigmented bone from Tlapacoya, in the Estado de México, close to Mexico City. The sample comes from an isolated skull which may have been included in a ceremonial rite, Table 1. Of course, in this case, the blackened zone was not specific of articulations.

2.3. Characterization techniques

Samples had to be grinded to be characterized by X-ray Fluorescence, X-ray Diffraction, and Scanning Electron Microscopy. The same samples were sequentially used as X-ray Fluorescence and X-ray Diffraction are non destructive. The last study was by Scanning Electron Microscopy as, in this technique, samples were covered with gold to avoid electron charge. Black color was not scratched from the bones. Sections of black pigmented and non pigmented bone were selected.

The bulk elemental composition was determined by X-ray Fluorescence (XRF) with a Siemens SRS303 spectrometer and a Semi-Quant software. Although this software is only semi-

Table 2

Composition determined by XRF.

Tlate1	Tlate2	Tlate3	Tlapa4
Si	Si	Si	Si
Ca	Ca	Ca	Ca
P	P	P	P
Fe	Fe	Fe	Fe
Al	Al	Al	—
Ti	Ti	Ti	—
K	K	K	—
Mg	S	S	—

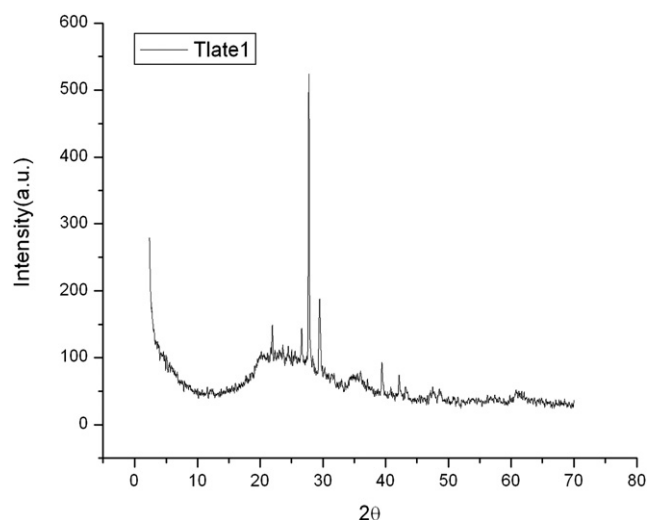


Fig. 4. XRD pattern of Tlate1 sample.

quantitative it presents the advantages that the sample does not need to be put into solution, and no standards are required for semi-quantitative analysis.

To analyze small regions of the colored surface, Scanning Electron Microscopy (SEM) coupled to an Energy Dispersive X-ray Spectroscopy probe (EDS) was used. For EDS studies the beam was focused in order to cover a surface of 1 μm and a depth of 4 μm . The microscope was a Stereoscan 440, Cambridge Leica with Leica Electron Optics software.

A Bruker-axs D8 Advance diffractometer with a Cu anode tube was used to obtain the X-ray Diffraction (XRD) patterns. The crystalline compounds were identified in the conventional way with JCPDS files.

A gas chromatograph coupled to a mass spectrometer Bruker GC Mate was used to identify the organic compounds present on the bone surface. The column was 5% phenilmethylsiloxane (30 m \times 0.25 mm) and the flow was 1 mL/min. The initial temperature was 150 $^{\circ}\text{C}$ and the heating rate 10 $^{\circ}\text{C}/\text{min}$ up to 300 $^{\circ}\text{C}$. Only sample Tlate3 was studied with this technique as it was the darkest. This method is a destructive method in the sense

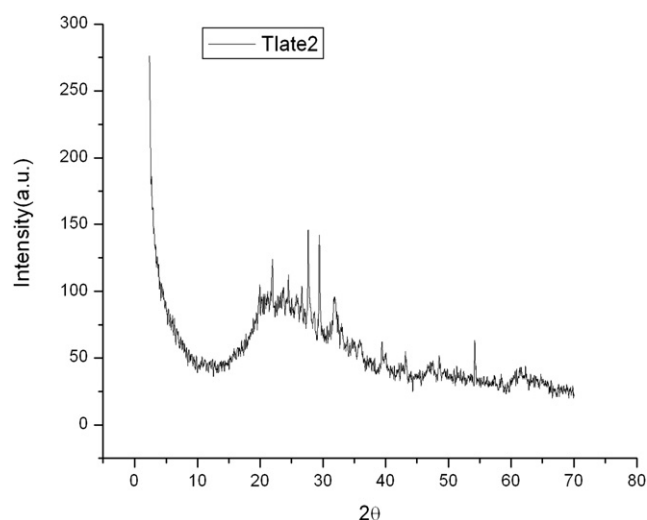


Fig. 5. XRD pattern of Tlate2 sample.

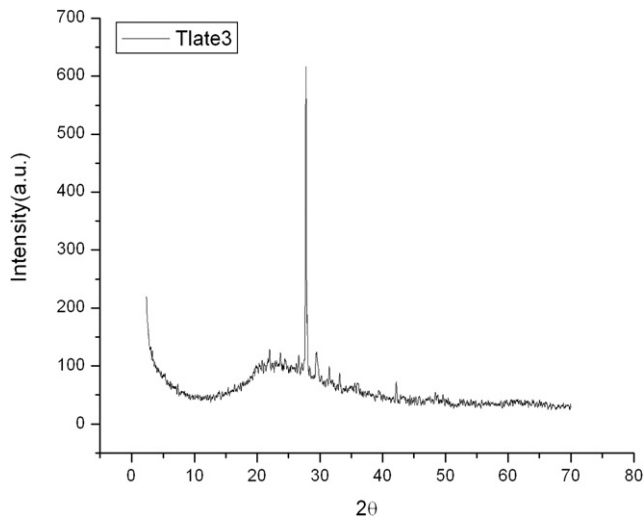


Fig. 6. XRD pattern of Tlate3 sample.

that samples had to be dissolved. Indeed, the sample must be able to be vaporized in the system inlet. The sample can be a gas, a liquid or in some cases a solid (thermally stable or capable of producing a definite pyrolysis pattern). The preparation technique is based on extraction, *i.e.*, again, the sample color was partially dissolved.

Infrared spectroscopy was chosen to determine the chemical species, and in this way to distinguish if the pigment was a bitumen defined as a mixture of organic liquids that are highly viscous, black, sticky, and composed primarily of highly condensed polycyclic aromatic hydrocarbons, an asphalt (a sticky, black and highly viscous liquid or semi-solid that is present in most crude petroleum and in some natural deposits, it is modelled as a colloid, with asphaltene as the dispersed phase, and maltene as the continuous phase) or only crude oil (a naturally occurring, flammable liquid found in rock formations in the Earth consisting of a complex mixture of hydrocarbons of various molecular weights, plus other organic compounds). A Perkin Elmer Series spectrophotometer model 6X was operated in the ATR-FTIR mode. This method has the advantage that the sample does not have to be mixed with potassium bromide.

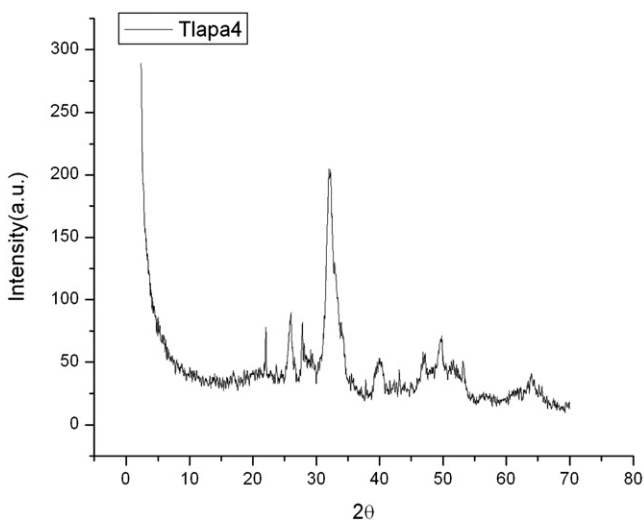


Fig. 7. XRD pattern of Tlapa4 sample.

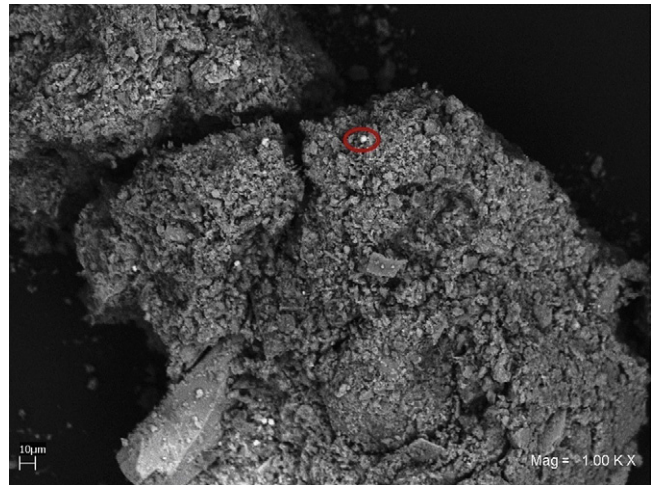


Fig. 8. Surface of the sample Tlate1. The appearance is rough, with cracks and some shining dots (encircled). The particle size distribution is broad as it comprises sizes from 0.1 to 1.5 μm .

3. Results

Table 2 compares the bulk elemental composition determined by XRF. As XRF is a semiquantitative technique, no percentages are reported and, only, amounts higher than 1% are considered. Elements are ordered from highest to lowest content. Presence of calcium and phosphorus was expected as they are due to bone composition. A high amount of silicon is found, probably due to clays and sand adhered to the samples. It has to be emphasized that XRF does not detect low weight elements as oxygen or carbon.

The elemental composition of the various samples is very similar. Only small differences can be noticed. The sample Tlate1 contains a small amount of magnesium whereas the other samples have sulphur. The Tlapacoya sample does not contain aluminum, titanium, potassium nor sulphur or magnesium.

An XRD analysis was, then, performed to determine if the formed crystalline compounds were all the same, Figs. 4–7. In all Tlatelolco samples, the main peaks correspond to crystalline silicon oxide, the small peaks may be attributed to hydroxyapatite and no

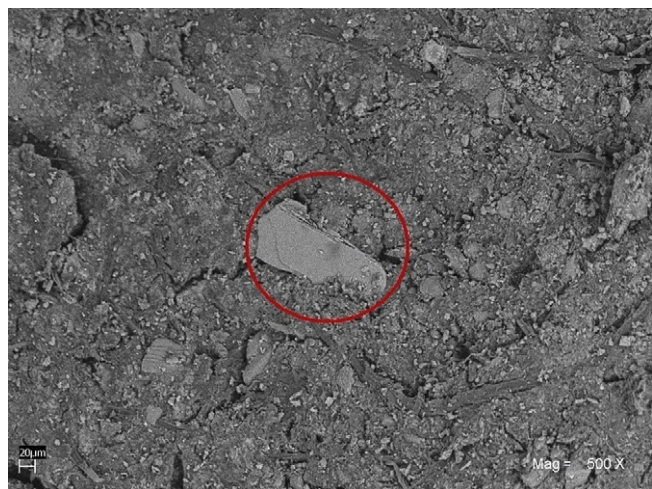


Fig. 9. This image corresponds to the Tlatelolco painted bone Tlate2. It shows a rather homogeneous surface. Some large and smooth particles, as the shoe-shaped one inside the circle, may be observed.

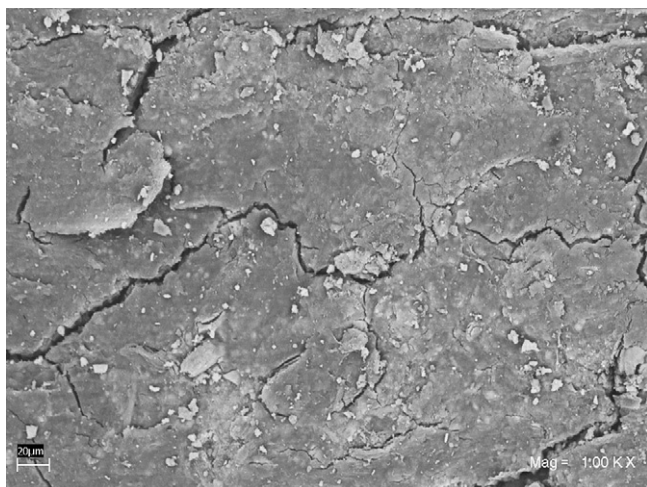


Fig. 10. The Tlate3 surface is smooth and does not present small particles. Only cracks are observed. The surface morphology is definitely different from the morphology of a non-pigmented bone.

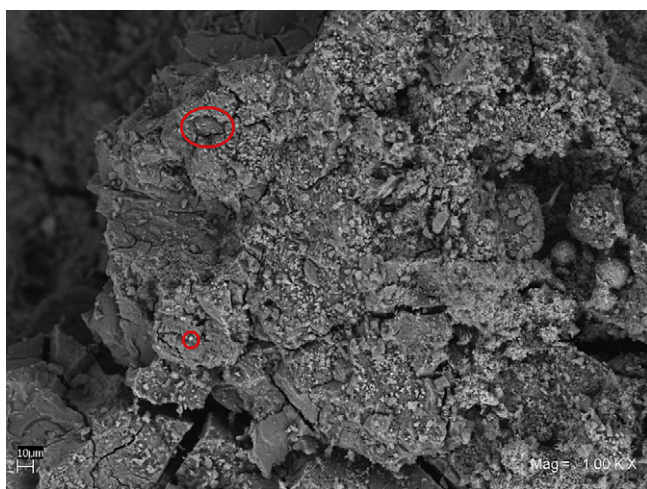


Fig. 11. Surface of the Tlapa4 sample, the morphology is similar to Tlate1. In this image two slimming dots were selected to perform a local analysis.

other crystalline compounds are observed. However, the background presents a bump from 15 to 40° (2θ), this background is typical of an amorphous compound which cannot be identified. The Tlapacoya sample, instead, is more crystalline and the amount of silicon oxide is much lower. None of these crystalline compounds (silicon oxide or hydroxyapatite) is black. Hence, the black compound must be included in the amorphous fraction of the samples (Fig. 7).

XRD is, as well as XRF, a bulk analysis. As pigment is only on bone surface and into small cavities at the bone articulations, a local determination was required. Such a study was made with a scanning electron microscope (SEM) coupled to an Energy Dispersive X-ray probe (EDS). In this way, the elemental analysis of selected surface zones may be obtained. Furthermore, the surface morphology of the black patches, provided by SEM, can be compared to the non-pigmented bone.

Figs. 8–11 present the SEM images of some pigmented zones of the samples; Tlate1, Tlate2 and Tlapa4 are fairly similar, they all present two granulometries: large and smooth particles (10 μm or more) or much smaller ones (1 μm or less) spread over the larger ones. Such morphology is not typical of a healthy and clean bone (Fratzl et al., 2004; Marotti, 1993). The small particles and the disorder in the sample are typical of a calcined bone surface (Pijoan et al., 2007). Instead, the surface of sample Tlate3 is smooth and cracked; a thick layer of a compound which is not hydroxyapatite covers the bone.

The EDS spectra corresponding to each of the SEM images, Figs. 12–15, show carbon which was not detected in the bulk analysis by X-ray Fluorescence. The relative intensity of the carbon peak compared to the calcium peak, follows the sequence:

Tlate1 < Tlate2 = Tlapa4 << Tlate3

The analysis by EDS shows that black color is due to a carbon compound spread over bone surface; the highest amount of carbon corresponds to a thicker and smoother layer of black pigment.

In sample Tlate3, compounds such as o-xylene, dibutyl phthalate or diphenols were identified by Gas Chromatography, Fig. 16. These compounds are polyaromatic hydrocarbons which are derivatives of bitumen (Olah and Molnar, 1995). Bitumen is constituted by variable ratios of terpanes and steranes (Connan, 1999), and those compounds are identified with a column that

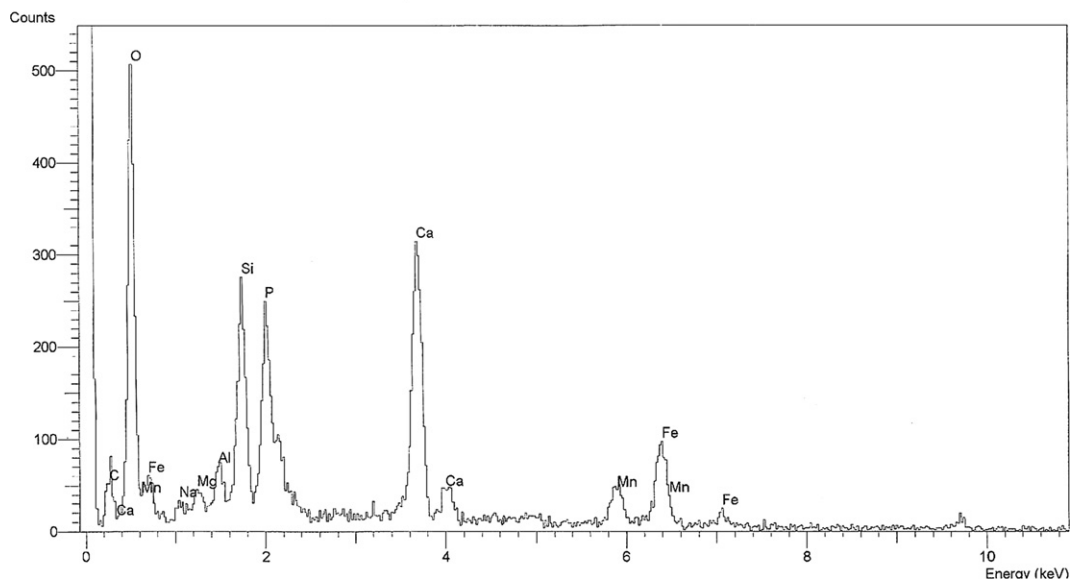


Fig. 12. EDS analysis of sample Tlate1, showing the surface presence of a small amount of carbon.

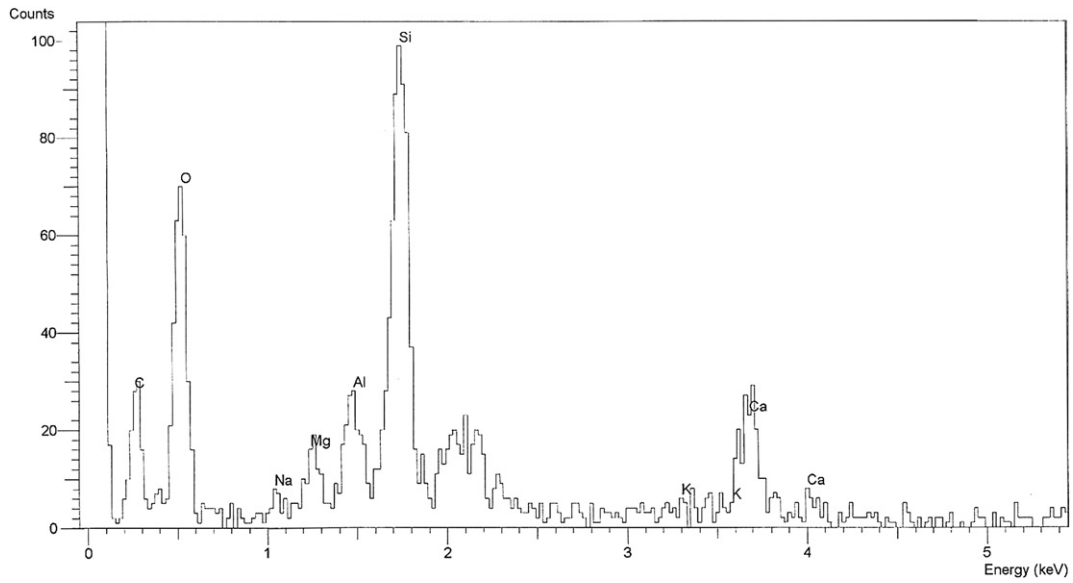


Fig. 13. EDS analysis of sample Tlate2, showing the surface presence of a large amount of carbon.

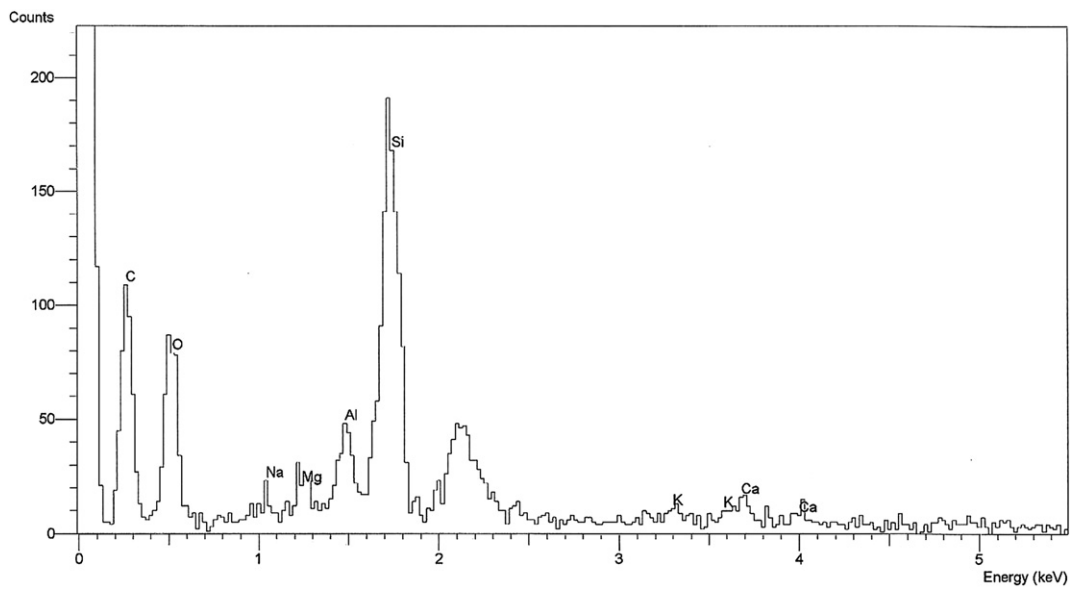


Fig. 14. EDS analysis of sample Tlate3, showing the surface presence of a very large amount of carbon.

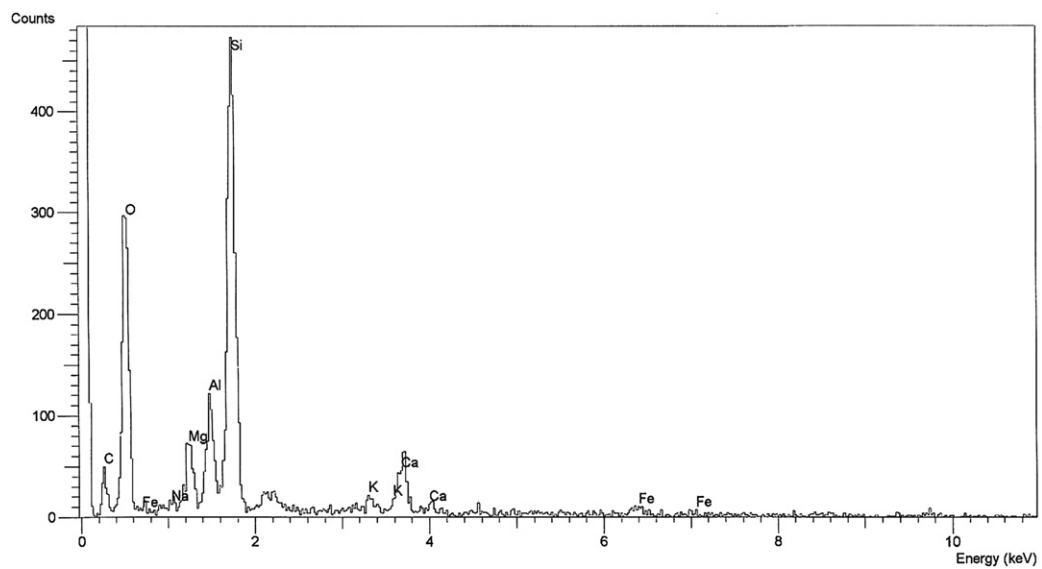


Fig. 15. EDS analysis of sample Tlapa4, showing the surface presence of a large amount of carbon.

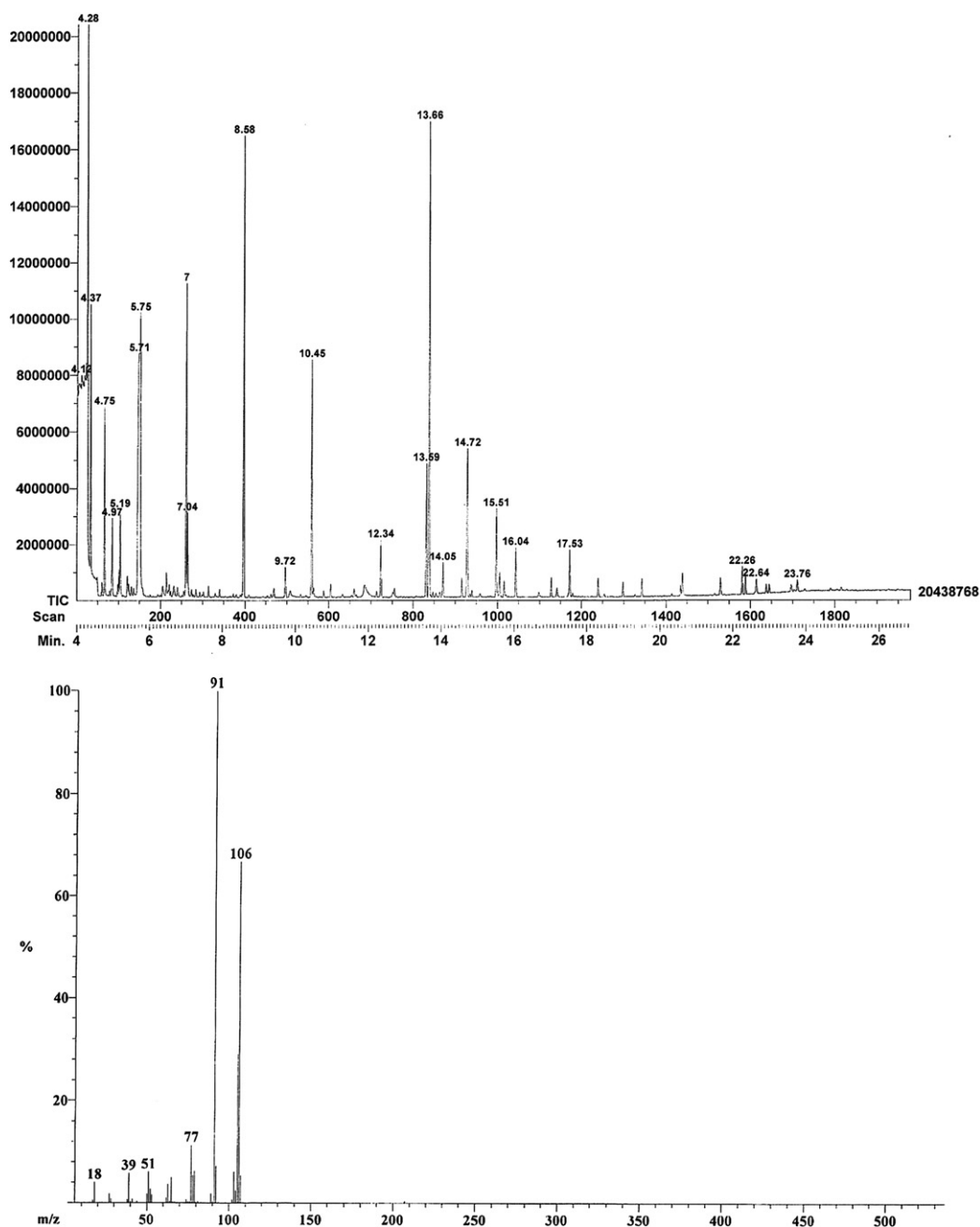


Fig. 16. Gas chromatography patterns of sample Tlate 3, showing the presence of aromatics.

we do not have. Then, although our analysis by Gas Chromatography showed that the pigment was constituted by some bitumen derivative, it did not identify bitumen as such due to experimental limits.

ATR-FTIR spectroscopy can determine the presence of bitumen in the zone 2000 to 700 cm^{-1} (Borrego et al., 1996; Masson et al., 2001). In Figs. 17 and 18 we present the spectra of the samples Tlate1 and Tlapa4. All peaks in sample Tlate1 correspond to bitumen. The peak at 713.97 cm^{-1} can be attributed to rocking bonds of $(\text{CH}_2)_n$, n being lower than 4. The C–H out of plane bending in 1,2,4 and 1,4-substituted aromatics appears at 800 cm^{-1} . The intense peak at 1029.36 cm^{-1} corresponds to S=O stretching and the peak at 1410.43 cm^{-1} to a deformation band of methyl and methylene groups $\delta(\text{CH}_3 + \text{CH}_2)$. Hence, this spectrum clearly shows that the compound is bitumen. These peaks are found in the

spectra of the other samples, therefore, all samples contain bitumen.

The other peaks present in Tlapa4 spectrum can be explained as due to human bone (Kafalak-Hachulska and Kolodziejski, 1999). Peak at 1278 cm^{-1} may be attributed to P=O (free), CO_3^{2-} appears at 1463 and 871 cm^{-1} . Peaks at 1647 and 1540 cm^{-1} correspond to amide I and II in defect bone, respectively. A small peak is found at 1740 cm^{-1} which can be attributed to C=O in intact bone. These results were reproduced by sample Tlate 2.

Apparently the obtained infrared spectra contradict the gas chromatography identifications. However, bitumen can give four bitumen fractions: saturates (paraffins), aromatics, resins and asphaltenes. This remark is important as *o*-xylene, dibutyl phthalate and diphenols are aromatics. Hence the results are complementary and the sample is constituted by bitumen and some of its derivatives.

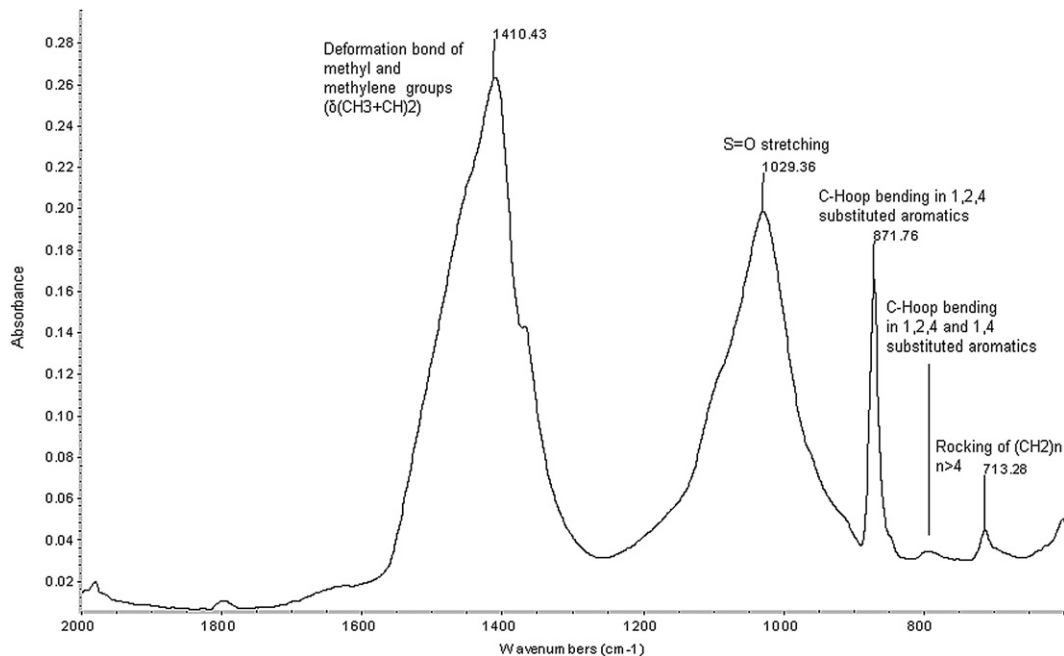


Fig. 17. Infrared spectrum of sample Tlate1, bitumen vibrations are observed.

4. Discussion

Our results may be summarized as follows. Black color present in the articulations of the studied bones is constituted by a surface layer whose morphology varies as it may appear as small deposited particles (Tlate1 and Tlapa4) or as a continuous film (Tlate2 and Tlate3) of variable thickness. Comparing the analyses by XRF and EDS it is established that the black pigment is constituted by carbon, silicon, and aluminum as main elements and magnesium, barium, and iron in lower concentrations. Furthermore, the

compound is not crystalline and, therefore, it cannot be identified by XRD. Gas Chromatography identified hydrocarbons that can be related to the presence of bitumen derivatives. Infrared Spectroscopy showed that the black pigment was bitumen.

Several hypotheses may be advanced. Soot could pigment the bones, probably by a deliberate use of graphite or through the partial calcination of bone. A second possibility could be that black pigments such as iron or magnesium oxides were intentionally prepared for this purpose. The third possibility is that the color on the bone was due to a highly viscous material as a burnt vegetal-

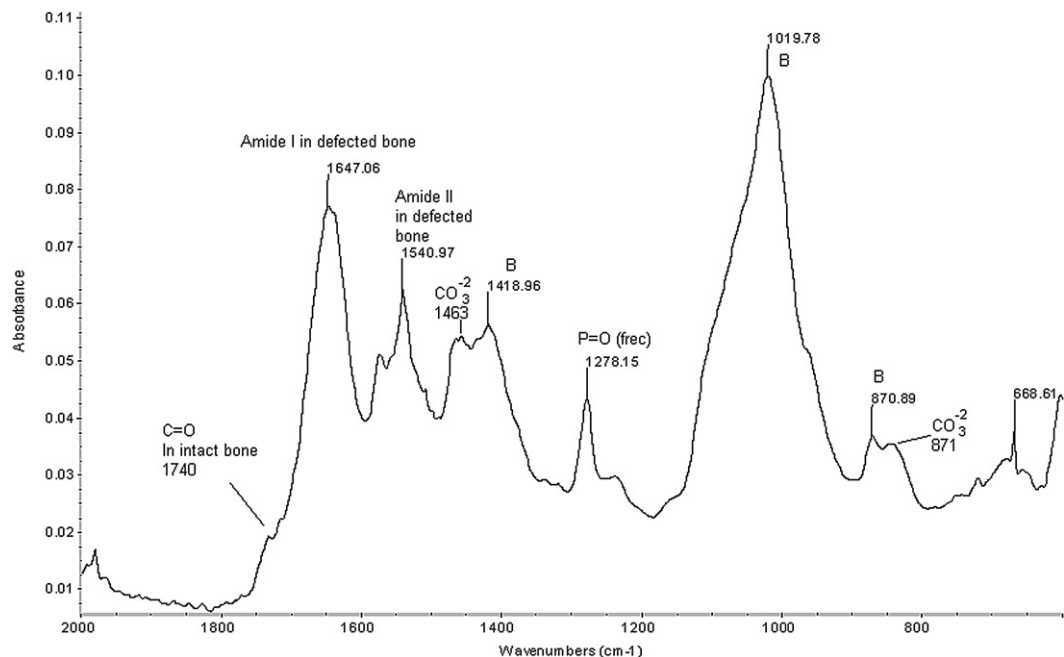


Fig. 18. Infrared spectrum of sample Tlate3, bitumen and bone vibrations are observed. The peaks correspond to the vibrations of bitumen already assigned in the spectrum of Fig. 17.

resin, known as *Tilli ócoltl* (López Luján et al., 2005) or as a petroleum compound.

As pigment is found on the articulation surfaces and it is located in man made cavities on the bone it cannot be due to thermal alterations. Carbon, as graphite, is a crystalline compound which would appear in the X-ray diffractograms, it has, then, to be discarded. Carbon as carbon black can be eliminated because the pigment is a paste and it includes silicon and aluminum.

The second hypothesis, *i.e.* black color due to metal oxides, has to be rejected as Mansilla et al. (2002) have shown that Tlatelolco soils contain high amounts of magnesium and iron, and although they do contain organic compounds (as most soils do) the amount is very low and not enough to cause the observed staining patterns. Oxides of magnesium or iron were not identified in the X-ray diffraction patterns.

As far as the third proposition (a highly viscous material) is concerned, the pigment is shown to be a dried and strongly adhered paste mainly constituted by carbon, silicon and aluminum. If it was a burnt resin, only carbon would be observed with other organic elements. The high contents of silicon, aluminum, and iron in the paste cannot come from a *Tilli ócoltl*.

Hence, the only option left is that black pigments found in bones from Tlatelolco and Tlapacoya were originally a petroleum derivative. This statement is confirmed by the infrared spectra which showed that the color is due to bitumen. From our results it can be concluded that the used bitumen contained or was mixed with high amounts of soil. Such proposition would explain the presence of aluminum and silicon among other elements as vanadium, nickel, barium, iron and magnesium found in X-ray fluorescence. Texture varies as a function of the bitumen amount present in the mixture. The hot bitumen calcined locally the bone, as shown by the SEM micrographs.

It is well known that Prehispanic people collected, processed, and used bitumen, asphalt, and petroleum, for decoration, sealant, or adhesive. Unfortunately, our analyses, due to the small amount of sample, are not precise enough to correlate our results with those published previously for bitumen in the Olmec region (Wendt and Lu, 2006) and to determine the geographical origin of the black pigment.

To summarize, black color is not due to the environmental conditions, it is bitumen, and it came onto the bone during a dismembering process. It is generally found periarticularly in long bones or, as a patch, on skulls. The black color has not been reported previously as part of a ritual, nevertheless it did not come accidentally to the bone. It was left, then, as part of a technique used to dismember bodies. Remember that the Tlatelolco bones correspond to post sacrificial deposits containing dismembered individuals. From this site more than 400 burials were excavated between 1961 and 1968. Many of them were simultaneous multiple burials containing a large amount of people. From the corresponding pictures, Fig. 3, it is clear that they are formed by dismembered body parts. Furthermore, almost all the bones show cut marks, some of which were found at the anatomic location of muscle insertions and others periarticularly, Fig. 19. In the first case, the cut marks must be due to the defleshing process, while in the second they are attributed to the effort to reach the articulation as part of the procedure of dismembering (Pijoan, 1997; Pijoan and Mansilla, 2010).

Hence, due to its location and distribution, black pigment could come from tools used to dismember bodies which were impregnated with hot bitumen to facilitate the task. This procedure was probably required to separate arms, legs and vertebral columns from more than 100 bodies in a short period of time (Pijoan, 1997; Pijoan and Mansilla, 2010). Petroleum derivatives reach high temperatures, they are oily and they maintain heat for a rather long time. This proposition, which includes bones from Tlapacoya and Tlatelolco, fits with all the characterization results and explains that the pigmented bone surface is locally thermally altered.

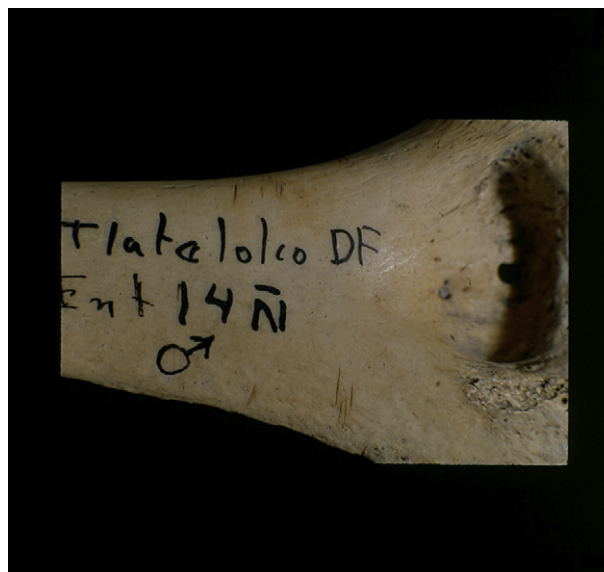


Fig. 19. Male left humerus showing clear periarticular cutting marks.

Figs. 1 and 2 show that the spot size and the corresponding cavity are ca. 0.5–2.5 cm. The shapes of the stain and the cavity are attributed to the shape of the used tool. As the impact trace is different, the tools must have been different depending on the purpose. Such morphology could only be obtained opening, first, the articulation. Then, an instrument coated with bitumen had to be introduced into the articular capsule by pressure, or hitting (impact) to facilitate the dismembering. Furthermore, the heat of the bitumen decomposed the cartilage of the articulation allowing the introduction of an instrument used as a lever. The cutting tools were probably made of obsidian, and the hafts were made of wood as this material is a thermal insulator often used in Mesoamerica (Pijoan and Mansilla, 2004; Pijoan et al., 2010). In the present everyday life, shaving blades work better if they are hot and lubricated by soap. In medicine, hot-knife conization of the cervix is recommended.

5. Conclusions

The direct analysis of human remains is of great value for the identification and interpretation of human sacrifice and ritual body manipulations in Mesoamerica. In our work, complementary analyses have shown that black pigment often present in the articulations is mainly composed by carbon and small particles of aluminosilicates. It is shown that such compound is bitumen. It is suggested that, in Tlatelolco samples, hot liquid bitumen was used to lubricate and enhance the action of tools used to dismember corpses as part of a post sacrificial ritual process exerted on more than 100 individuals. In Tlapacoya the black pigment on the skull may be attributed to post-sacrificial rituals including tools lubricated with bitumen. Such tools have not been found as they were not deposited into the burials; they were most probably made with materials such as wood that desintegrate in a short period of time.

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