

Optical Microscopy And SEM-EDX Characterization Of Some Little Micro- Organisms Having Chemical Formulas Similar To Those Of Clays, Deposited On The Turin Shroud Surface

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Abstract—We have studied by optical microscopy and SEM-EDX particles corresponding to small micro-organisms and having chemical formulas similar to those of clays, located on the surface of a small sticky tape triangle corresponding to the Face area of the Turin Shroud. Fifty-two of these particles correspond to individual micro-organisms ; of typical triangular forms, their dimensions are comprised between 2 and 5.5 μm ; they are alumino-silicates, with some contents of iron and phosphorous. These micro-organisms has a tendency to self-associate (between at least two linked individuals in particles comprised between 3.5 and 5.5 μm). We described here in details the special case of some particles (h3-h11) corresponding to a group of micro-organisms linked together. Eleven of such particles correspond to closely linked individuals, micro-organisms ; they are greater (up to 23 μm) than those of individual micro-organisms, and are generally of yellow colour. In some cases (particles b4, b5, e7, e14, e53, f47 and i16) there are visible micro-organisms – or their reliefs (b74, d18, d15 and g37) – on its surfaces. All these particles correspond to some form of phosphorites, similar to those already described in some regions of the Middle-East.

Keywords—Turin Shroud ; Phosphorite particles ; individual or associated micro-organisms ; Scanning Electron Microscopy (SEM) ; Energy Dispersive X-ray (EDX).

1. Introduction

The Turin Shroud (TS) is a well known linen tissue on which a body image is imprinted [1]. We have obtained a small triangular sticky tape that was sampled on its surface (corresponding to the Face of this body) and we concentrated in the past years on the study of microscopic structures located on the surface of this sticky tape [2].

We previously published a preliminary study on the mineral particles found on the triangular surface [3], that included clay particles. More recently we have studied by SEM-EDX analyses the little clays

montmorillonite, illite and kaolinite particles that we have found on the triangle [4].

In the present study we describe in details particles on the triangle detected by SEM-EDX analyses and optical microscopy corresponding to some special little individual micro-organisms, having a chemical formula similar to those of clays. Sometimes these micro-organisms appear as linked together, or constitute coloured particles of greater sizes resulting from close associations between them.

2. Material and Methods

The material is a small (1.36 mm height , 614 μm wide) sticky tape triangle at the surface of which the particles were deposited. For practical reasons, the surface of this triangle was subdivided into 19 sub-samples areas, named A to S (the E area being subdivided again in seven parts Ea-Eg). The positions of each particle were located in a double system of coordinates (in 186 adjacent squares of 50x50 μm).

Particles of the sample were observed, with any preparation, on the adherent part of the surface of the triangle. All the particles described here were studied by optical microscopy (for the biggest ones) and by SEM (Scanning Electron Microscopy) – EDX (Energy Dispersive X-ray) analyses.

Two SEM apparatus (SEM1 and SEM2) were used : 1/ A Philips XL. 30 instrument ; GSE (Gaseous Secondary Electrons) and BSE (Back Scattered Electrons) procedures were used, the last one permitting the better detection of heavy elements. 2/ A FEI model Quanta 25 of FEG, both in LFD (Large Field Detector) and CBS (Circular Back Scattering) procedures. Elemental analysis for each particle observed were realized by EDX, the SEM1 apparatus being equipped with a Bruker probe AXS-EDX and the SEM2 with a probe model X-flash 6/30.

Each elemental analysis is given in the form of a spectrum, with kiloelectrons/Volts (KeV) on the abscissa and elemental peak heights in ordinates . High Resolutive (HR) spectras are those where the ordinate graduations are enhanced, to better see little elemental peaks.

The bigger particles were observed in optical microscopy – to determinate its colours – using a photo-microscope Zeiss, model III, of 1972).

3. Results

3.1. Individual micro-organisms.

Figure 1 shows the appearance and the spectrum of the f24 particle, which is a typical individual micro-organism. It is a particle with vague triangular outlines, of about 2.5 μm of maximal length ; its surface shows morphological peculiarities, notably appendix at some of the corners of the triangle.

In its spectrum the silicium (Si), aluminium (Al) and magnesium (Mg) peaks are in orders of decreasing amounts, a characteristic pattern of aluminosilicate clays [4]. It has three little peaks of iron (Fe) and a little peak of sulphur (S). The phosphorous (P) peak is greater than the Si peak, and it has a peak of calcium (Ca) .

Because of a predominating P peak and of the presence of Ca, the f24 particle can be considered as some form of phosphorite [5]. We ignore the exact nature of the corresponding micro-organism, but we suppose that it could be some form of Cyanophyceae.

Table 1 lists and characterizes the fifty-one micro-organisms – other than f24 – detected in the various areas of the triangle : a9, in the A area ; b74", in the B area ; c6 , c8 and c41, in the C area ; e9, e10, e15 and e21, in the Eb area ; e42, e52 and e62, in the Ec area ; e82 and e97, in the Ed area ; e122 in the Ee area ; f38 and f41, in the F area ; g52, g55 and g55', in the G area ; h15', h22, h29, h32', h33, h38, h39, h43 and h56, in the H area ; i21 , i47, i50 and i51, in the I area ; j42, in the J area ; k18, k19, k25, k43, k71, k72 and k83, in the K area ; l12 and l36, in the L area ; m36, in the M area ; n27, in the N area ; o18, in the O area ; p3, p8 and p28, in the P area ; r5, in the R area ; s26, in the S area.

Although these micro-organisms are of various forms (rounded, ovoid, losangic or elongated), nineteen of them are similar (in form and with appendix) to the f24 type : particles a9, b74", e42, e62, e97, h15', h33, h38, h39, h56, i50, i51, k25, k83, l36, m36, o18, r5 and s26.

Their maximal sizes vary between 2 μm and 5.5 μm . The longest particles (of size between 3.5 μm and 5.5 μm) correspond at least to two tightly linked micro-organisms : particles a9, f38, h22, h29, h32', h33, i47, i51, k18, k83, l36, m36, n27, o18 and r5.

In the spectras of the particles, Si, Al and Mg elements are in orders of decreasing amounts. All these has Fe, but in particles e52, h22, h32 and h38 the iron element is not detectable. All the spectras has the S element, although in the form of traces only (in particles c8, i47, i50, k18, k19 and k71).

The phosphorous element is present in all the spectras, but in the form of traces only in particles a9, c8, e42, h22, h56, i47, k19 and p8. There are little P peaks in spectras of particles c6, c41, e9, e10, e15, e21, e82, e97, f41, g55, h15', h29, i50, k18, k25, o18 and r5, and little more elevated peaks in particles e62, f38, g55', h32', h33, h38, h39 and i51. In the spectras

of particles b74", e52, g52, i21, k71, k72, k83, m36, p3 and s26, the P peak is as elevated as those of Mg ; these P peaks are as elevated as those of Al in particles h43, l12, l36 and n27 and as elevated as those of Si in particles e122 and j42.

The calcium element is present in all the spectras, but in particles a9, b74", c8, e21, e82, e97, h22, h32', i47, k18, k19 and o18, Ca element is detectable only in the calcium mean peak. Calcium excess in particles b74", g52, k39, i21 and i51, is due to contaminations by adjacent calcium carbonate particles.

The potassium element (K) is present in the spectras of numerous particles : a9, c8, c41, e10, e15, e62, e82, e97, g55, h15', h29, h39, h43, h56, i47, i50, k43, k71, k83, l12, m36, n27, o18, p3, p8, p28, r5 and s26. The chlorine element (Cl) is present in the spectras of a greater number of particles : b74", c6, c41, e9, e10, e15, e21, e52, e62, e82, e97, e122, f38, f41, g55, h15', h29, h38, h43, k56, i47, i50, k19, k25, k43, k71, k83, l12, l36, m36, n27, o18, p3, p8, p28, r5 and s26. The sodium element (Na) is present at relatively elevated levels in spectras of particles h56, l12, r5 and s26, that are suggesting some amount of salt (ClNa) in these particles.

Presences of potassium, of chlorine and salt in the spectras are in favour of marine micro-organisms (like some Cyanophyceae). Also in favour of the favour of the Cyanophyceae diagnosis is the ability of these micro-organisms to concentrate heavy metals : copper (Cu), in the e122, l36 and r5 particles, of lead (Pb) in the e62 particle, and of titanium (Ti) – an element frequently associated to iron - in h15 and h43 particles. The manganese element (Mn), in h43 and p28 particles, indicates the "mineral nature" of the corresponding iron.

3.2 Micro-organisms linked together.

We have precedently see that particles of sizes comprised between 3.5 μm and 5.5 μm corresponded to at least two linked micro-organisms. The association between at least nine particles (h3-h11), represented in the photograph of Figure 2, shows a group of micro-organisms closely linked together. Associations between micro-organisms are also characteristics of some forms of Cyanophyceae.

In this photograph is seen some part of the H area showing the h3-h11 group. Surrounding particles are h1 and h12 which are parts of pollens [6], h2 that is a kaolinite [4], h18 that is part of a peridot, and h19 that is a peculiar form of potassium carbonate. Each member of the h3-h11 group are particles of approximate triangular outlines with appendix on corners, and are of about 2.5 μm of maximal lengths.

The below part of Figure 2 shows the h3-h5 spectras ; Figure 3 shows the h6-h8 spectras and Figure 4 shows the h9-h11 spectras. In all spectras Si, Al and Mg are in orders of decreasing amounts. In all the spectras , but in those of the h8 and h9 particles, the main peak of Fe is present. All the particles has P,

S, Cl, K, Ca and Na elements in their spectras. Spectras of h6, h7, h8, h9 and h10 has the Cu element ; spectrum of the h11 particle has the Ti element.

3.3 Particles resulting from close associations between individual micro-organisms.

Some particles, which sizes are greater than those of individual or bipartite micro-organisms, result of a close association between them. Eleven of such particles were found in the different areas of the triangle : b4, b5 and b74, in the B area ; d8 and d15, in the D area ; e7, e14 and e53, in the E area ; f47, in the F area ; g37, in the G area ; i16, in the I area.

The SEM photograph of Figure 5 shows, in some part of the B area, the two adjacent b4 and b5 particles. Neighbouring particles in this area are : b1, that is an hair [7] ; b3, that is a Dinophyceae ; b7 and b9, that are silices ; b8, that is a great clay [8], covered by a triangular (T) Diatom ; b10, that is a portion of a linen fiber [9].

This photograph shows that the b4 particle, of elongated form and of 10 μm of maximal length, results from an association between individual micro-organisms. The b4 spectrum has a greater silicium peak, and Si, Al and Mg peaks are in orders of decreasing amounts. It has iron, traces of phosphorous, and little peaks of sulphur, chlorine and potassium ; calcium value of the mean peak is as elevated as that of the silicium.

The SEM photograph of Figure 6 shows the b5 particle, rounded in form and of 8 μm of maximal size ; it shows some evidence of individual micro-organisms in some parts of its outline. The b5 spectrum is similar to that of the b4 particle, but with a little peak of phosphorous.

The SEM photograph of Figure 7 shows the b74 particle. Adjacent particles are b74", an individual micro-organism, already described; b74', that is a calcite ; C is a Coccolith, and i is an iron lamellae. This photograph shows that the b74 particle is elongated , with a maximal size of about 11 μm ; reliefs of micro-organisms are visible on the b74 surface. The b74 spectrum is similar to that of the b5 particle.

The b4, b5 and b74 are yellow in optical microscopy.

The SEM photograph of Figure 8 shows, in some part of the D area near the right border of the triangle, the two d8 and d15 particles. Surrounding particles in this area are : d3 and d4, that are red blood cells [10] ; d5, d6 and d10, that are rounded calcium carbonates ; d7, that is a glass ; d9, that is a colourant complex ; d11 and d16, that are lapis-lazulis [11] ; d12 that is a Diatom. Both d8 and d15 particles are rounded, of approximately 7.5 μm and 8 μm of maximal sizes respectively. There are evidence micro-organism reliefs on their surfaces. The d8 and d15 spectras are similar to the previous one.

Because of proximity of the border, the d8 and d15 particles cannot be seen in optical microscopy.

The SEM photograph of Figure 9 shows the e7 particle in some part of the Eb area. Neighbouring particles in this area are : b8, that is a glass ; e9, e10 and e15, that are lapis-lazulis [11] ; e41, that is an alumina-silicate of potassium, and some parts of the b14 and b15 particles.

The e7 particle is elongated, with a maximal length of about 15 μm ; individual micro-organisms are clearly visible on its surface. The e7 spectrum is similar to the previous one.

The e7 particle is yellow in optical microscopy.

The SEM photograph of Figure 10 shows, in the Eb area, the particle e14. Adjacent particles in this area are : the e11 particle, that is a characteristic Cyanophyceae ; e16, that is a lapis-lazuli [11] ; e13, that is a gypsum, and e17, that is a montmorillonite [4].

The e14 particle is of a complex triangular form (with a basis maximal of maximal dimension of about 23 μm ; individual micro-organisms are clearly visible on its surface.

The e14 spectrum, in the part of the particle analysed, is similar to the previous one.

The e14 particle is yellow in optical microscopy, with red dots.

The SEM photograph of Figure 11 shows, in some part of the Ec area, the e53 particle. Neighbouring particles are : e51, that is a spore [6] ; e52, that is a lapis lazuli [11] ; e54 is a calcite ; L is a portion of a linen fiber [9], and is a some part of a particle of lead. The e53 particle is elongated, with a maximal length of 7 μm , and with individual micro-organisms on its surface. The e53 spectrum is similar to the previous one.

The e53 particle is yellow in optical microscopy.

The SEM photograph of Figure 12 shows, in some part of the F area near the border, the f47 particle. Neighbouring particles in this area are : f53 and f54, that are Dinophyceae ; f48-f50, that are various parts of an Algae covered with PVC plastic, f38, that is a red blood cell [10] ; f40 and f44, that are calcites ; f43, that is an alumino-silicate of potassium ; f45, that is a dolomite.

The f47 is an elongated particle (of a maximal length of about 24 μm), where individual micro-organisms are visible in some parts of its surface. The f47 spectrum is similar to those of the previous ones, but Cu and with a chlorine excess due to contamination of the PVC of f48-50.

Because of its proximity with the border of the triangle , the f47 particle is not visible in optical microscopy.

The SEM photograph of Figure 13 shows the g37 particle, In some part of the G area. Neighbouring particles in this area are : g32, a dolomite, g33, an alumina-silicate of potassium and g36 and g39, that are silicas. The g37 particle is elongated, with a maximal length of 12.5 µm and with reliefs of micro-organisms on its surface. The g37 spectrum is similar to the previous ones, but it has a higher peak of silicium.

The g37 particle is yellow in optical microscopy, with red glints.

The photograph of Figure 14 shows, in some part of the I area, the i16 particle. Neighbouring particles in this area are : i17, that is a calcium carbonate ; i15, that is a glass ; i19, that is a Dinophyceae ; i18, that is a rounded Diatom ; i22 and i23, that are calcites ; i14, that is an hematite [12].

The i16 particle is elongated, with a maximal length of about 8.5 µm and with micro-organisms on its surface. The i16 spectrum is similar to the previous ones, but with calcium excess (due to the contamination by calcium carbonate particles) and with copper.

The i16 particle is white in optical microscopy, with yellow dots.

Table 2 lists and characterizes these eleven particles which are resulting from close associations of individual micro-organisms, detected in the various areas of the triangle. **Discussion and conclusions.**

In this study we described in details fifty-two peculiar particles that occurred on the TS surface (Table 1) . They are alumino-silicates with some contents of iron, but their mineral compositions is not those of little clays [4]. Of typical triangular forms, their dimensions are comprised between 2 µm and 5.5 µm ; their spectras contain ever some content of phosphorous (and generally of sulphur, calcium , chlorine and potassium).

Morphologically, these particles correspond to (unknown) marine micro-organisms. These micro-organisms has the potentiality to self-associate, in bipartite or of more numerous numbers forms. Particles h3-h11 correspond to nine micro-organisms that are closely linked together.

Eleven greater (up to 23 µm) particles, of mainly yellow colour, are formed by a close association between these individual micro-organisms (Table 2), as the individual micro-organisms already described. They also occurred in the various areas of the TS surface studied.

These two sorts of particles are smaller versions of more greater foms of alumino-silicates found also on the TS surface, more rich in iron and of red colour, which present these micro-organisms on their surfaces [8].

Such a peculiar formation was already described in localities of Oman margins and near the Arabian sea [13, 14].

4. Acknowledgment

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5. Conflict of interest.

The author has declared no conflict of interest regarding the publication of this paper.

6. References

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Table 1. List and characterizations of the individual micro-organisms detected on the various areas of the triangle

Areas of the triangle	Particles	Forms	With appendix	Bipartite	Maximal dimensions (in μm)	Phosphorous contents	Calcium contents	Peculiarities
A	a9	triangular	+	+	4 μm	a very little peak	a little peak	presence of potassium (K) contamination in Ca by the adjacent b74' particle ;
B	b74''	triangular	+		2.5 μm	peak as elevated as that of Mg	A great peak	presence of chlorine (Cl)
C	c6	triangular			2.5 μm	a little peak	two peaks	Presence of Cl
	c8	triangular			3 μm	a very little peak	a peak	Presence of K
	c41	ovoid	+		2.5 μm	a little peak	two peaks	Presences of K and Cl
Eb	e9	losangic			4 μm	a little peak	two peaks	presence of Cl
	e10	losangic			4.5 μm	a little peak	two peaks	presences of K and Cl
	e15	triangular			5.5 μm	a little peak	two peaks	presences of K and Cl
	e21	losangic	+		2.5 μm	a little peak	a peak	presence of Cl
Ec	e42	triangular	+		2.5 μm	a very little peak	two peaks	presences of K and Cl
	e52	ovoid	+		2.5 μm	as elevated as the Mg	two peaks	presence of Cl without iron (Fe)
	e62	triangular	+		2.5 μm	a peak	two peaks	presences of K, Cl, and lead (Pb)
Ed	e82	rounded	+		2 μm	a little peak	a peak	presences of K and Cl
	e97	triangular	+		2 μm	a little peak	a peak	
	e122	ovoid			3.5 μm	peak as elevated as that of Si	two peaks	presence of Cl ; contaminated by copper (Cu)
F	f38	elongated	+	+	5.5 μm	a peak	two peaks	presence of Cl
	f41	losangic			3.5 μm	a little peak	two peaks	presence of Cl
G	g52	rounded			3 μm	peak as elevated as that of Mg	two peaks	contamination of Ca by the adjacent particle
	g55	elongated	+		2.5 μm	a little peak	two peaks	presences of K and Cl
	g55'	rounded	+		2.5 μm	a peak	two peaks	
H	h15'	triangular	+		3.5 μm	a little peak	two peaks	presences of K and Cl ; contamination by titanium (Ti)
	h22	rounded		+	3.5 μm	a very little peak	one peak	without Fe
	h29	triangular		+	5 μm	a little peak	two peaks	presences of K and Cl
	h32'	elongated		+	4.5 μm	a peak	a peak	without Fe
	h33	triangular	+	+	4 μm	a peak	two peaks	
	h38	triangular	+		2.5 μm	a peak	two peaks	presence of Cl without Fe
	h39	triangular	+		2.5 μm	a peak	two peaks	contamination of Ca by adjacent particles
	h43	Losangic			3 μm	a peak as elevated as that of Al	two peaks	presences of K and Cl. Contaminations by Ti and manganese (Mn)
h56	triangular	+		2.5 μm	a very little peak	Two peaks	presences of K, Cl and sodium (Na)	

I	i21	triangular			2.5 μm	peak as elevated as that of Mg	two peaks	very elevated Ca peak due to the adjacent particle
	i47	losangic	+	+	4.5 μm	a very little peak	a peak	presence of Cl
	i50	triangular	+		2.5 μm	a little peak	two peaks	presences of K and Cl
	i51	triangular	+	+	4.5 μm	a peak	two peaks	contamination of Ca by adjacent particle
J	j42	triangular			3 μm	peak as elevated as that of the Si	two peaks	
K	k18	elongated		+	4 μm	a little peak	a Ca peak	
	k19	triangular			2.5 μm	a very little peak	a Ca peak	presence of Cl
	k25	triangular	+		2.5 μm	a little peak	two peaks	presence of Cl
	k71	triangular			2.5 μm	as elevated as that of Mg	two peaks	presences of K and Cl
	k72	triangular			2 μm	as elevated as that of Mg	two peaks	
	k83	triangular	+	+	5 μm	as elevated as that of Mg	two peaks	presences of K and Cl
L	l12	triangular			2.5 μm	as elevated as that of Al	two peaks	presences of K, Cl and Na
	l36	triangular	+	+	3.5 μm	as elevated as that of Al	two peaks	presence of Cl ; contamination by Cu
M	m36	triangular	+	+	4.5 μm	peak as elevated as that of Mg	two peaks	presences of K and Cl
N	n27	elongated	+	+	5 μm	as elevated as that of Al	two peaks	presences of K and Cl
O	o18	triangular	+	+	4.5 μm	a little peak	a peak	presences of K and Cl
P	p3	triangular			2.5 μm	peak as elevated as that of Mg	two peaks	presences of K and Cl
	p8	triangular			2.5 μm	a very little peak	two peaks	presences of K and Cl
	p28	triangular			2.5 μm	a very little peak	two peaks	presences of K and Cl contamination by Mn
R	r5	triangular	+	+	5.5 μm	a little peak	two peaks	presences of K, Cl and Na ; contamination by Cu
S	s26	triangular	+		2.5 μm	as elevated as that of Mg	two peaks	presences of K, Cl and Na

Table 2. List and characterisations of particles of agglomerated micro-organisms detected on the various areas of the triangle.

Areas of the triangle	Particles	Forms	Visible micro-organisms	Maximal dimensions (in μm)	Colours	Phosphorous contents	Peculiarities
B	b4	elongated	+	10 μm	yellow	traces	
	b5	rounded	+	8 μm	yellow	a little peak	
	b74	elongated	reliefs	11 μm	yellow	a little peak	
D	d18	rounded	reliefs	7.5 μm	not visible	a little peak	
	d15	rounded	reliefs	8 μm	not visible	a peak as elevated as that of Al	
Eb	e7	elongated	+	15 μm	yellow	a peak as elevated as that of Mg	
	e14	triangular	+	23 μm	yellow	a peak as elevated as that of Mg	
Ec	e53	elongated	+	7 μm	yellow	a little peak	
F	f47	elongated	+	24 μm	not visible	a little peak	spectrum with Cu, and with Cl excess (due to the PVC plastic)
G	g37	elongated	reliefs	12.5 μm	yellow, with red glints	a peak	spectrum with a higher peak of Si
I	i16	elongated	+	8.5 μm	white, with small yellow dots	a peak	spectrum with Cu ; Ca contamination due to covering calcium carbonate particles

Figure 1. The f24 particle. Above : SEM1 photograph (30 000x), in GSE, of the f24 particle (a : appendix). Below : the HR spectrum of f24 . C : carbon ; O : oxygen ; Fe (three peaks) : iron ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P : phosphorous ; S : sulphur ; Ca (two peaks) : calcium.

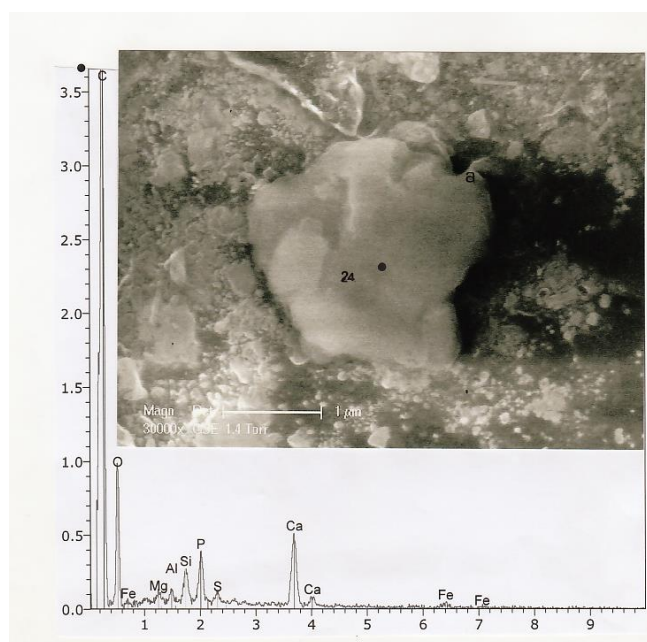


Figure 2. Above : SEM1 photograph (6000x), in GSE, of some part of the H area showing the h3-h11 group, surrounded by particles h1, h2, h18, h19 and h12. Below : spectras of the h3-h5 particles (the lower spectrum is that of h3, the medium spectrum is that of h4, and the upper spectrum is that of h5). C : carbon ; O : oxygen ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P : phosphorous ; S : sulphur ; Cl (two peaks) : chlorine ; K : potassium ; Ca (two peaks) : calcium ; Fe : iron.

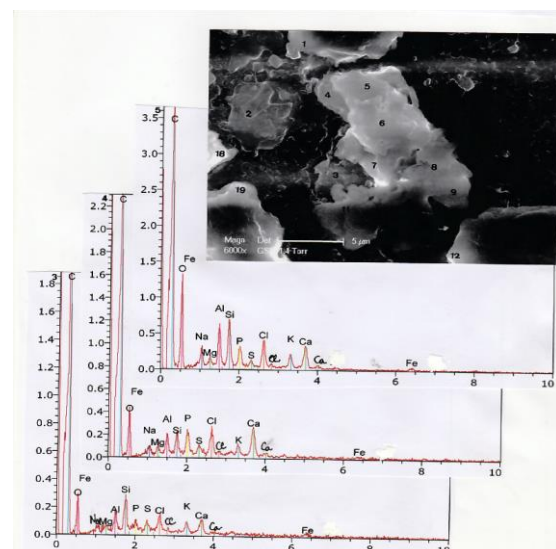


Figure 3. Spectras of the h6-h8 particles (the lower spectrum is that of h6, the medium spectrum is that of h7, and the upper spectrum is that of h8). Cu (three peaks) : copper.

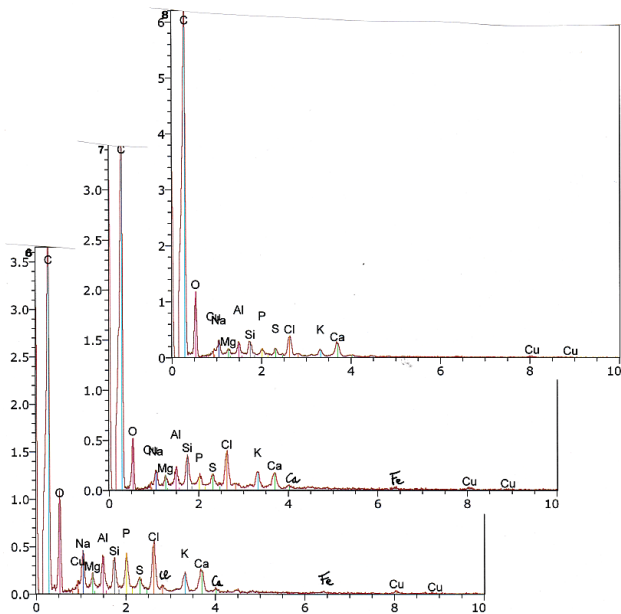


Figure 4. Spectras of the h9-h11 particles (the lower spectrum is that of h9, the medium spectrum is that of h10, and the upper spectrum is that of h11). Ti (two peaks) : titanium.

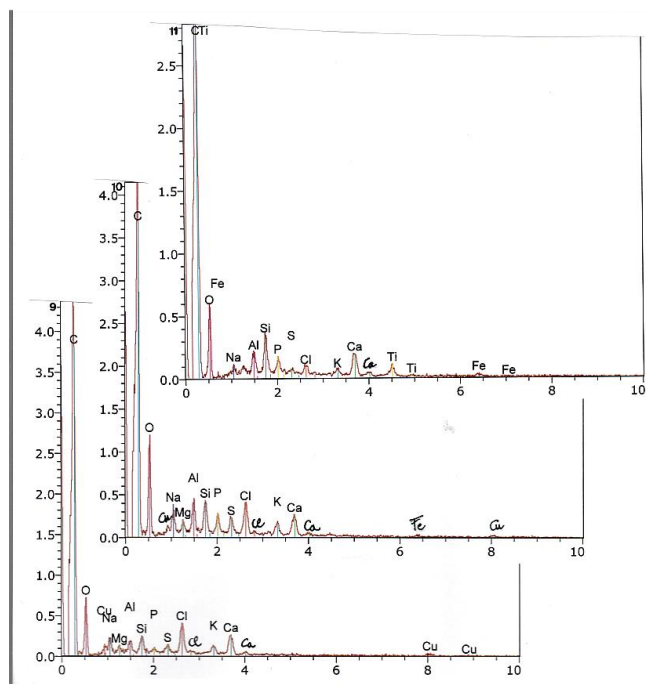


Figure 5. Above : SEM1 photograph (3750x), in GSE, of some part of the B area showing the two adjacent particles b4 and b5. Below : the b4 spectrum. C : carbon ; O : oxygen ; Fe (three peaks) : iron ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P (traces) : phosphorous ; S : sulphur ; Cl : chlorine ; K : potassium ; Ca (two peaks) : calcium.

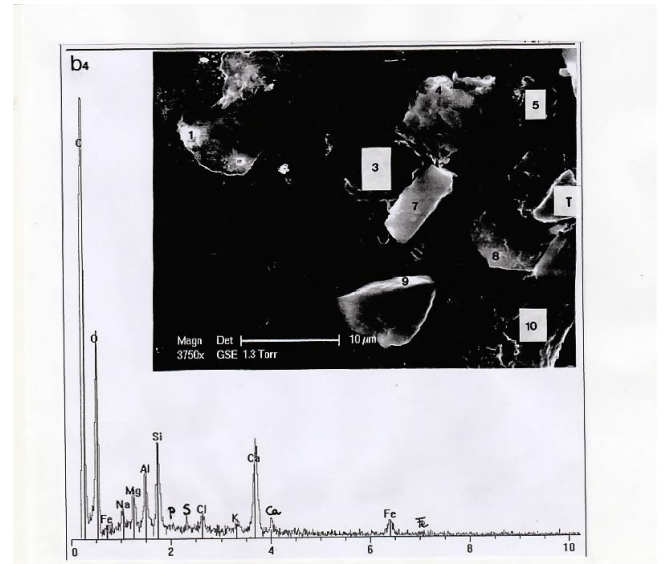


Figure 6. Above : SEM1 photograph (1000x), in GSE, the b5 particle (and some part of b4). Below : the b5 spectrum.

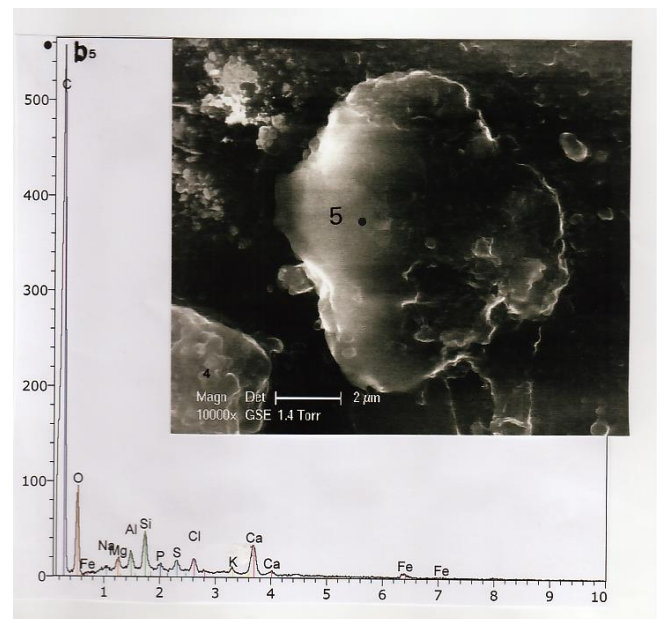


Figure 7. Upper photograph : SEM2 photograph, in LFD, of the b74 particle. Lower photograph : the same, but in CBS. Below : the b74 spectrum.

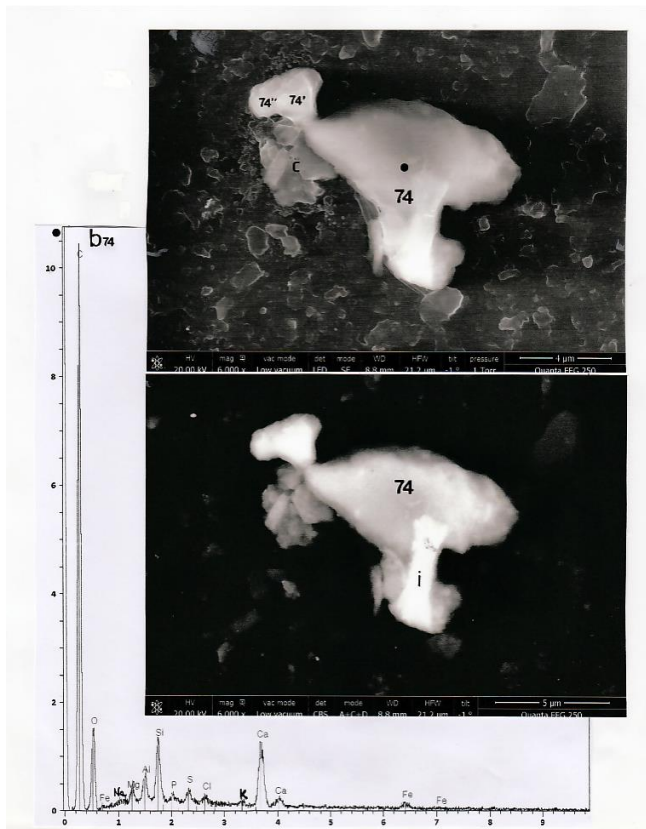


Figure 8. The d8 and d15 particles. Above : SEM1 (1000x) photograph, in GSE, of some part of the border of the D area showing (red arrows) d8 and d15 ; t : holes. Lower spectrum : that of the d8 particle. Upper spectrum : that of the d15particle.

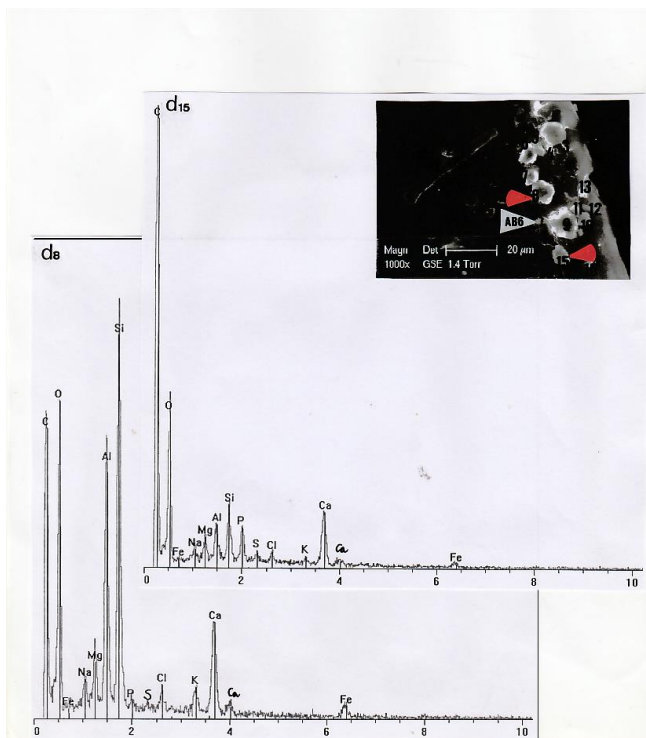


Figure 9. Above : SEM2 photograph (5000x), in LFD, of some part of the Eb area showing the e7 particle. Below : spectrum of the e7 particle.

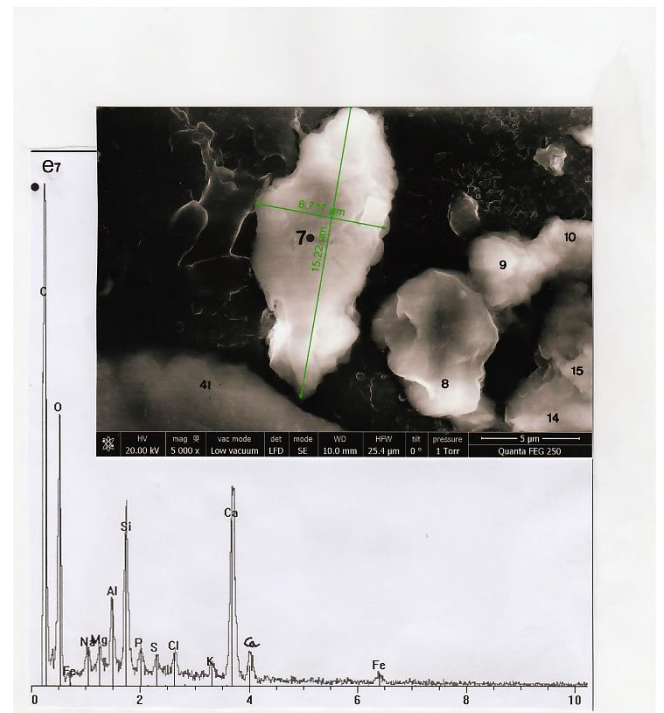


Figure 10. Above : SEM1 photograph (5000x) , in GSE, of some part of the B area showing the e14 particle (the black spot indicates the part of the e14 particle where EDX analysis was realized). Below : spectrum of this part of the e14 particle.

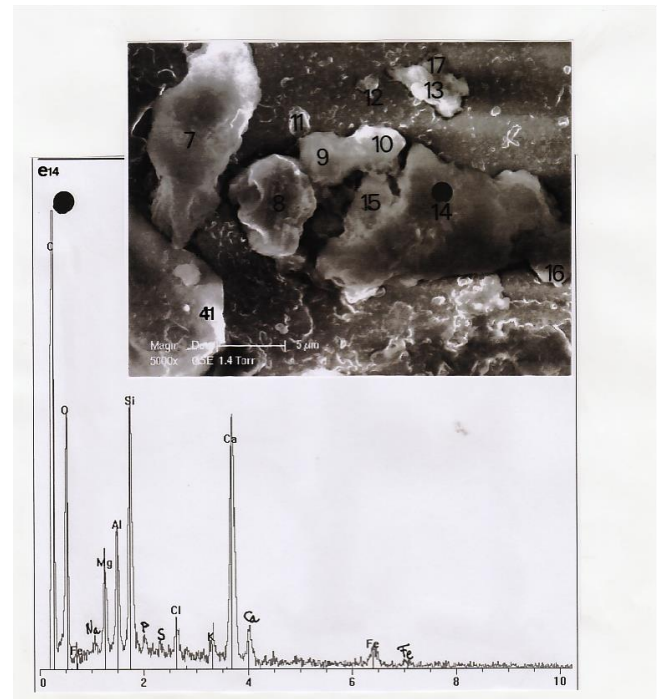


Figure 11. Above : SEM1 photograph (10 000x), in GSE, of some part of the Ec area showing the e53 particle . Below : spectrum of the e53 particle.

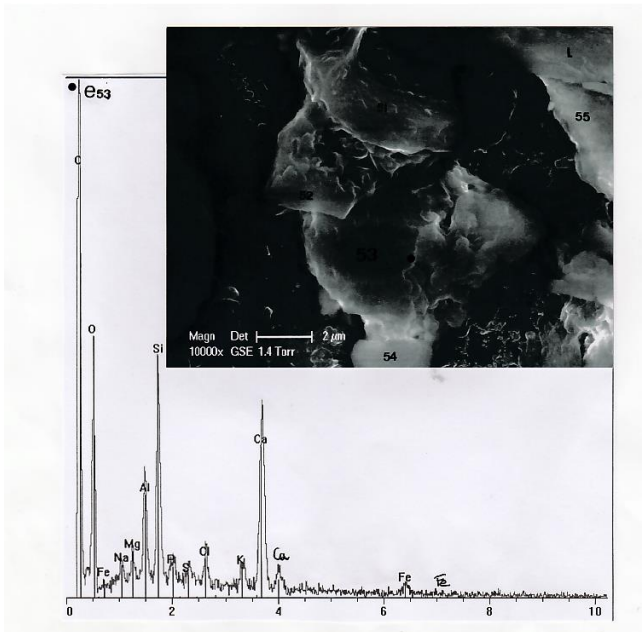


Figure 12. Above : SEM1 photograph (2500x), in GSE, of some part of the F area showing the f47 particle (red arrow). Below : spectrum of the f47 particle. Cu (two peaks) : copper.

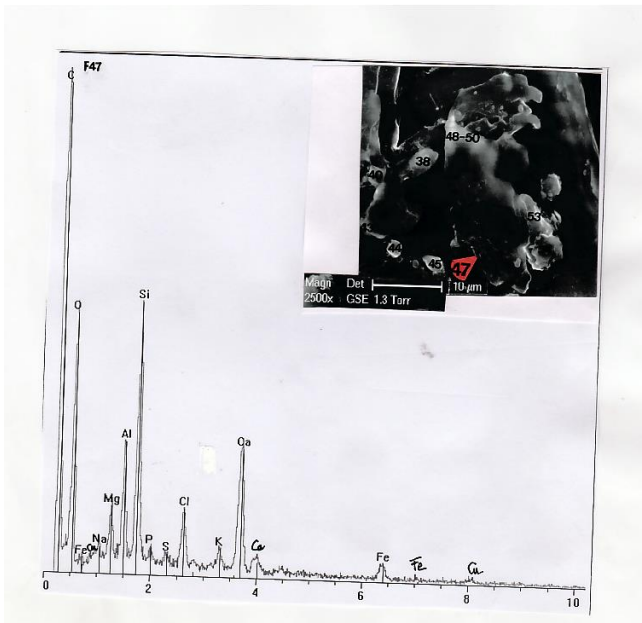


Figure 13. Above : SEM1 photograph (1600x), in BSE, of some part of the G area showing the g37 (red arrow) particle. Below : spectrum of the g37 particle.

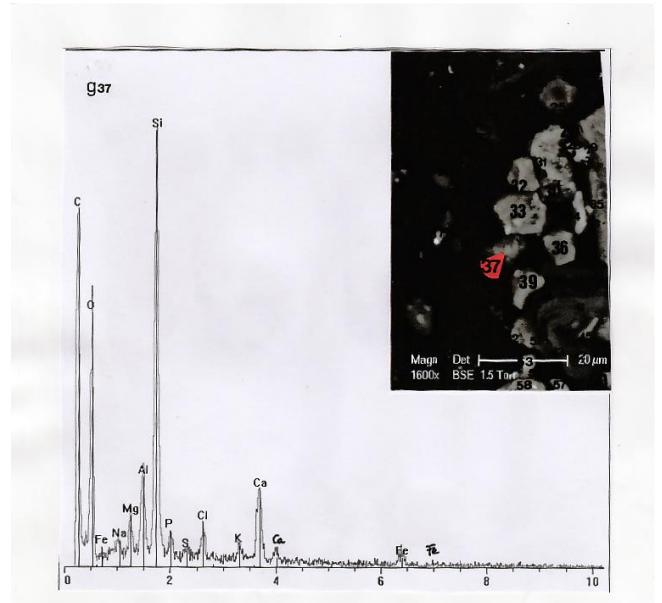


Figure 14. Above : SEM1 photograph (4000x), in BSE, of some part of the I area, showing the i16 particle. Below : spectrum of the i16 particle.

