

Barium Sulphate As A White Pigment On The Turin Shroud : Optical Microscopy Studies And SEM-EDX Analyses

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Abstract—I have explored, by optical microscopy and scanning electron microscopy coupled with energy-dispersive X-ray, fifty-seven particles or sub-particles containing barium sulphate located in a sample of the Face area of the Turin Shroud. They consist of five feuil, thirty-two micro-plaques, three micro-plaques, three micro-scales, eight micro-balls and nine deposits of barium sulphate, that are generally of white colours. These particles are of barium sulphate white.

Keywords—*Turin Shroud, Face area, White of barium sulphate, Optical Microscopy, Scanning Electron Microscopy, Energy Dispersive X-ray.*

1. Introduction.

The Turin Shroud 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 a part of the Face of this body) and we concentrated in the past years on the study of microscopic particles located on the surface of this sticky tape [2].

Barium sulphate (BaSO₄) is mainly a white background used in painting since 1810. We have found previously this product in association with gold and silver in the two particles a23 and i57 [3], in lapis lazuli particles g74, j26, j28, j34, j34' and j39 [4] and more recently in twenty-four particles (a18, a19, a20, a33, a40, b27, d9, e50, e61, f38, f53", g72', g78, g79, g88, h32, h32', i65, k41, m2, m42, m51, m52 and g12), containing cadmium sulphide [5].

In the present study I describe, by optic microscopy and SEM-EDX analyses, some other particles containing barium sulphate found on the triangle surface.

2. Material and Methods.

The material is the small (1.36 mm height, 614 m wide) sticky tape triangle at the surface of which all particles were deposited. For practical reasons, the surface of this triangle was subdivided into nineteen subsample areas, named A to S ; because of its complexity, the E area was subdivided into seven (a to g) sub-areas.

The sample of reference is a powder of barium sulphate ("charge neutre de sulfate de baryte naturel") from the firm Sennelier, Paris.

All the particles containing barium sulphate were previously observed (to determine colour) by optical microscopy, using a photo-microscope Zeiss (Model III, 1972).

These particles were also observed, with any preparation, on the adhesive part of the surface of the triangle. These observations were conducted by SEM (Scanning Electron Microscopy), using a Philips XL instrument (of the environmental version). GSE (Gaseous Secondary Electrons) and BSE (Back Scattering Electrons) procedures were used, the second one to better detect heavy elements. Elemental analyses for each particle observed were realised by EDX (Energy Dispersive X-ray), this SEM1 microscope being equipped with a Bruker probe AXS-EDX (the system analysis is PGT : Spirit Model of Princeton Gamma Technology).

Particle m43 was examined with another SEM apparatus (SEM2) : a FEI model Quanta 25 of FEG, both in LFD (Large Field Detector) and CBS (Circular Back Scattering) procedures. The barium sulphate sample of reference was studied by a third apparatus (SEM3), an Auriga FEG-FIB (Zeiss), images being performed in secondary electrons.

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

3. Results.

3.1. Barium sulphate sample of reference.

Figure 1 shows the results of the study of the barium sulphate powder of reference. The SEM3 photograph shows that this powder is constituted of angular particles of up to 10  m of maximal length ; colour of the powder (insert) is white. The powder spectrum contains mainly oxygen (O), sulphur(S) and barium (Ba) elements.

3.2. Feuils of barium sulphate.

SEM photograph of Figure 2 shows the a39 particle in some part of the A area of the triangle. Other particles in that area are : a38 (which is organic) ; mo : a montmorillonite ; ca : a calcite ; t : a titanium micro-scale. The a 39 particle is twisted and of about of 5 μm of maximal length. Its spectrum contains mainly O, S and Ba elements. Its colour is white in optic microscopy.

Other feuils of barium sulphate detected on the triangle surface are : g72' (an elongated particle of about 7 μm of maximal length) in the G area : k26 (a rectangular particle of about 7 μm of maximal length) and k54 (a triangular particle of about 4 μm of maximal length), both located in the K area ; l35 (a triangular particle of about 4 μm of maximal length) in the L area. Both g72' and k26 are pale-yellow, k54 is white and l35 is grey.

3.3. Micro-plaques of barium sulphate.

The maximal concentration of micro-plaques of barium sulphate occurs on the surface of the h20 particle of Diatom (Figure 3). The SEM photograph of this figure concerning some part of the H area shows that it is covered by at least twenty-four (h20.S1-S4) greater barium sulphate micro-particles at the edges. Other particles in that area are : h19 (a calcium carbonate) ; h24, h26 and Q (parts of Diatoms). The spectrum of this figure contains the O, S and Ba elements. All these sub-particles appear as white micro-spots.

Figure 4 shows HR spectras of sub-particles h20. S1-S4. All contain O, S and Ba elements in their spectras.

Figure 5 shows the SEM photograph of b70-1 sub-particle, located on the b70 particle (an illite), on some part of the B area (i are three sub-particles of iron ; b81 is PVC plastic). The b70-1 spectrum contains the O, S (traces) and Ba elements ; its colour is white.

Other micro-plaques of barium sulphate detected on the surface of the triangle are : g20-1 (on the Diatom g20) in the G area and j32-1 (on the illite j32) in the J area. Both are white.

3.4. Micro-scales of barium sulphate.

The SEM photograph of Figure 6 shows the e116 micro-scale, located in the E.e area. Other particles in that area are : e117 (a calcium carbonate) and e118 (a double carbonate) ; it is a quadrangular particle of about 3 μm of maximal length. Its spectrum contains the O, S and Ba elements. Its colour is white.

The two other micro-scales of barium sulphate detected on the surface of the triangle are j16 and j17, in the J area. Both are white.

3.5. Micro-balls of barium sulphate.

The SEM photograph of Figure 7 shows micro-balls h54, h55 and h> of some part of the H area. Other particles in that area are : h12, a pollen ; h20

and h28, two Diatoms ; Q : a Diatom part ; h30, a calcium carbonate ; h49, a PVC plastic ; h50, a feldspath ; h51, which is organic ; h52, a marble micro-fragment. The two micro-balls h54 and h55 are of about 4 μm of diameters and the microball h> of less than 2 μm . Their spectras contain the O, S and Ba elements. Colour of h54 is pale-yellow and those of h55 and h> are white.

Other micro-balls of barium sulphate detected on the surface of the triangle are : sub-particle i(i), in the I area ; j5 and j(i) in the J area ; k2 in the K area ; sub-particle r11-1 in the R area. The sub-particle i (i) is white, as j5. The sub-particle j(i) is not visible, because of its proximity to the border of the triangle. The particle k2 and the sub-particle r11-1 are white.

3.6. Deposits of barium sulphate.

Simple deposits of barium sulphate occur on the following particles : a22', on a montmorillonite, in the A area ; e12, on a gypsum, in the E.b area ; sub-particle e65-1, on the right extremity of an altered spore, in the E.c area ; e118, on a double carbonate, in the E.e area ; g59, on a Diatom, in the G area ; m13', on a Coccolith and m40 and m43, on calcium carbonates, in the M area ; n17, on an iron micro-ball, in the N area. Particles a22', e12, e65-1, e118, m13', m40 and m43 are white ; m59 is grey and n17 black.

3.7. Particles where barium sulphate is associated to copper.

In three particles, other than h20, barium sulphate is associated to copper : the micro-plaque e57', in the E.g area ; the deposit g12, in the G area ; the micro-plaque i25', in the I area. The three particles are white.

4. Discussion.

The **table** summarizes and characterizes the sixty-seven particles with barium sulphate detected in the present study. In the three particles e57', g12 and i25' the barium sulfate is associated with a copper pigment; so, the barium sulphate product is probably used in these three particles as a white background, like in particles of cadmium sulphure [5].

Barium sulphate alone is present in the other fifty seven particles and sub-particles consisting of thirty-two micro-plaques (b70, g20-1, h20.1-24, h20.s1-s4 and j32-1), of nine deposits (a22', e12, e65-1, e118, g59, m13', m40, m43 and m17), of eight micro-balls(h54, h55, h>, i(i), j5, j(i), k2 and r11-1) of five feuils (a39, g72', k26, k54 and l35) and of three micro-scales (e116, j16 and j17).

Barium sulphate is present in all these particles and sub-particles, but on traces only in a22', e12, g59, h54, h55 and m13'.

All the areas of the Triangle (but areas C, D, F, O, P, Q and S) has at least one particle with barium sulphate, like the B, L, N and R areas ; the A area has two such particles, the K and M areas has three, the G area four and the E area five. It seems that there is

a local concentration of these particles in the H area, that is due to the numerous micro plaques on h20.

When visible, the colour of the particles with barium sulphate are generally white. It is grey in g59 and l35 and pale-yellow in g72', h54 and k26 ; because of its very little size, the iron oxide micro-ball is black.

5. Conclusions.

You have detected on the triangle, by SEM-EDX analyses, a total number of fifty-seven particles or sub-particles with barium sulphate. They are free (feuil and micro-scales) or loaded on various objects adhering on the triangle surface, like minerals (clays, gypsum, calcium carbonates, double carbonate), iron oxide micro-ball and PVC plastic, or living remains (Diatoms, Coccoliths and spores). Their colours are mainly white.

So, these particles are those of the barium sulphate white pigment "blanc fixe" used since 1810.

This pigment can be added to the list of other white pigments [6] detected on the surface of the Turin Shroud.

Conflicts of interest

The author declares no conflicts of interest regarding the publication of this paper.

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Table : Nomenclature and characterization of the barium sulphate particles and sub-particles.

Numbers of particles	Nomenclature of particles and sub-particles	Areas	Particles	Formations	Barium sulphate	Colours
1	a22'	A	montmorillonite	deposit	traces	white
2	a39			feuil	+	white
3	b70-1	B	illite	micro-plaque	+	white
4	e12	E.b	gypsum	deposit	traces	white
5	e57'	E.g	copper pigment	micro-plaque	+	white
6	e65-1	E.c	spore	deposit	+	white
7	e116	E.e		micro-scale	+	white
8	e118	E.e	double carbonate	deposit	+	white
9	g12	G	copper pigment	deposit	+	white
10	g20-1		Diatom	micro-plaque	+	white
11	g59		Diatom	deposit	traces	grey

Numbers of particles	Nomenclature of particles and sub-particles	Areas	Particles	Formations	Barium sulphate	Colours
1	a22'	A	montmorillonite	deposit	traces	white
12	g72'			feuil	+	pale-yellow
13	h20(1-24)	H	Diatom	micro-plaques	+	white
14	h20(S1-S4)		Diatom	micro-plaques	+	white
15	h54			micro-ball	traces	pale-yellow
16	h55			micro-ball	traces	white
17	h>			micro-ball	+	white
18	i25'	I	copper pigment	micro-plaque	+	white
19	i(i)			micro-ball	+	white
20	j5	J	montmorillonite	micro-ball	+	white
21	j16			micro-scale	+	white
22	j17			micro-scale	+	white
23	j32-1			micro-plaque	+	white
24	j(i)		illite	micro-ball	+	not visible
25	k2	K		micro-ball	+	white
26	k26			feuil	+	pale-yellow
27	k54			feuil	+	white
28	l35	L		feuil	+	grey
29	m13'	M	Coccolith	deposit	traces	white
30	m40		calcium carbonate	deposit	+	white
31	m43		calcium carbonate	deposit	+	white
32	n17	N	iron oxide micro-ball	deposit	+	black
33	r11-1	R	PVC fiber	micro-ball	+	white

Figure 1 : The barium sulphate of reference. Above. SEM3 photograph (1 800x) of some part of the powder. Insert : colour of the powder. Below. Global spectrum of the powder. S (three peaks) : sulphur ; C : carbon ; O : oxygen ; Ba (seven peaks) : barium ; Si (silicium).

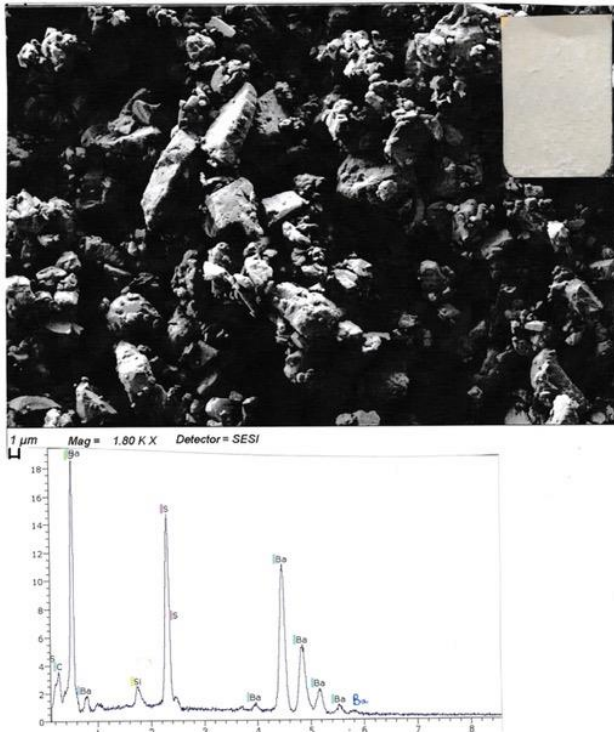


Figure 2 : The barium sulphate feuil a39. Above. SEM1 photograph (10 000x), in GSE of some part of the A area containing a39. Below. The a39 spectrum. C : carbon ; O : oxygen ; Ba (five peaks) ; Al : aluminium ; S : sulphur ; Ca (two peaks) : calcium.

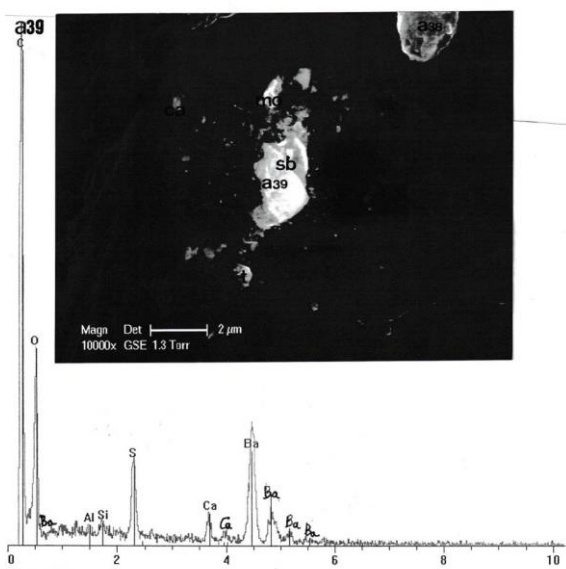


Figure 3 : The diatom h20. Above. SEM1 photograph (6250x), in BSE, of some part of the H area containing h20 (SB : barium sulphate ; S : three particles of salt). The twenty-four sub-particles h20.1-24 and the four subparticles h20.S1-S4 are shown. Below. HR spectrum of h20. C : carbon ; O : oxygen ; Na : sodium ; Mg : magnesium ; Al : aluminium ; S : sulphur ; Cl (two peaks) : chlorine ; K : potassium ; Ca (two peaks) : calcium ; Ba (three peaks) : barium ; Fe : iron ; Cu : copper.

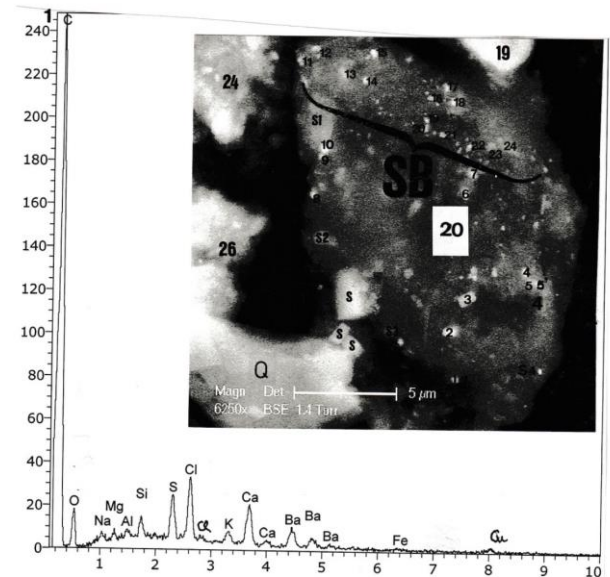


Figure 4 : HR spectras of sub-particles H20.S1-S4. C : carbon ; O : oxygen ; Fe (two peaks) : iron ; Cu (two peaks) : copper ; Na (sodium) ; Mg : magnesium ; Al : aluminum ; Si : silicium ; P : phosphorous ; S : sulphur ; Cl (two peaks) : chlorine ; K : potassium ; Ca (two peaks) : calcium ; Ba (two peaks) : barium.

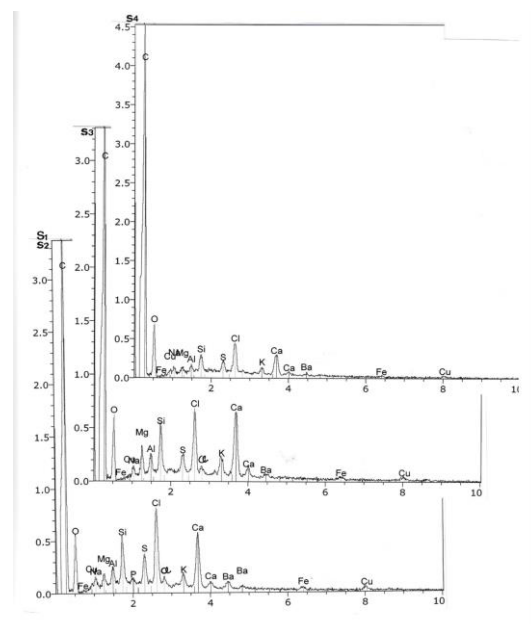


Figure 5. The b70-1 sub-particle. *Above.* SEM1 photograph (10000x), in GSE, of some part of the B area showing b70-1 (70 : b70). *Below :* spectrum of b70-1. C : carbon ; O : oxygen ; Fe (three peaks) : iron ; Na : sodium ; Mg : magnesium ; Al : aluminium ; S (traces) : sulphur ; Cl : chlorine ; Ca (two peaks) : calcium ; Ba (two peaks) ; barium.

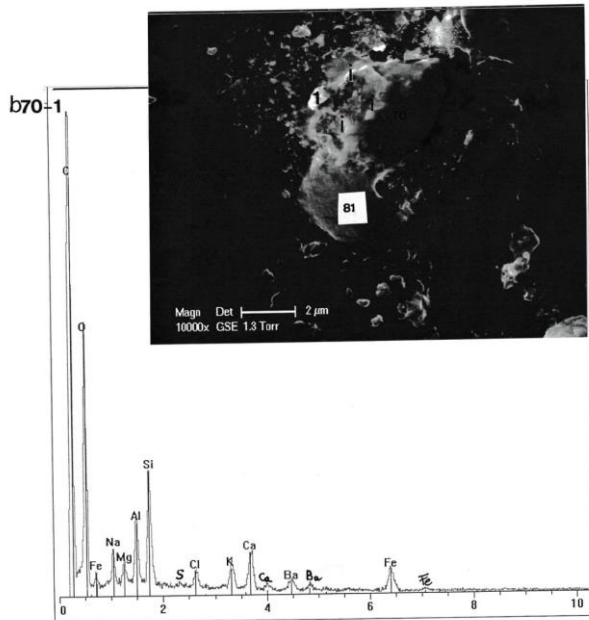


Figure 6. The e116 micro-scale. *Above.* SEM photograph (3000x), in GSE, of some part of the E.e area showing e116. *Below.* The e116 spectrum. C : carbon ; O : oxygen ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P : phosphorous ; S : sulphur ; Cl : chlorine ; K : potassium ; Ca (two peaks) : calcium ; Ba (two peaks) : barium ; Fe : iron.

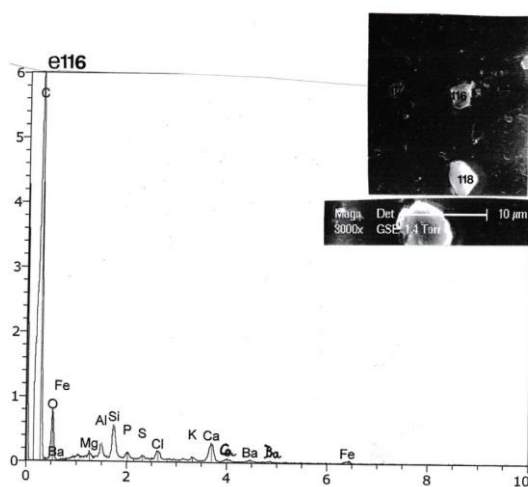


Figure 7. The h54, h55 and h>micro-balls. *Above :* SEM1 photograph (1000x), in GSE, of some part of the H area showing these three micro-balls. *Below :* spectras of these three micro-balls. C : carbon ; O : oxygen ; Fe (three peaks) : iron ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P (traces) : phosphorous ; S : sulphur ; Cl : chlorine ; K : potassium ; Ca (two peaks) : calcium ; Ba (two peaks) : barium.

