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SEM-EDX Characterization Of The Little Clays Particles Deposited On The Turin Shroud Surface

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***Abstract*—We have studied by SEM and EDX the montmorillonite, illite and kaolinite particles located on the surface of a small sticky tape triangle corresponding to the Face area of the Turin Shroud. Twenty-four montmorillonite, twenty-one illite and nine kaolinite particles were detected. Montmorillonite and illite particles came from the different soils on which the Turin Shroud was exhibited. We suppose that kaolinite particles are micro-fragments of pottery which was put down on the Face of the Turin Shroud.**

Keywords—Turin Shroud ; clays ; montmorillonite ; illite and kaolinite particles ; 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 organic structures located on the surface of this sticky tape : linen fibers [**2**], pollens and spores [**3**], red blood cells [**4**], an hair fragment [**5**], skin debris [**6**] and osseous remains [**7**].

The “clay” word is in fact an obsolete term in Earth Sciences ; it corresponds now to all the alumino-silicate mineral materials measuring of the order of the micrometre (µm) of size [**8**]. In the present study we detected three sorts of little clay particles deposited on the TS surface that are montmorillonite, illite and kaolinite. Montmorillonite is an aluminium and magnesium hydrated silicate, of the developed formula $(Na, Ca)_{0.3}\,(Al, Mg)_2\,Si_4\,O_{10}\,(OH)_2.nH_20$. Illite is also an aluminium and magnesium hydrated silicate, of the simplified formula : $Al_2Si_2O_5(OH)_4$.

We previously published a preliminary study on the minerals found on the triangular surface [**9**] that included our initial detections of little clay particles. In the present study we describe in details all the montmorillonite, illite and kaolinite particles detected by SEM-EDX analyses that we have found on the triangle.

2. Material and Methods.

The material is the small (1.36 mm height, 614 µm wide) sticky tape triangle at the surface of which all particles were deposited. For practical reasons, the surface of this triangle was subdivided into 19 sub-samples areas, named A to S [**10**]. The positions of each particle sticking to the triangle surface 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. The observations were conducted by SEM (Scanning Electron Microscopy), using a Philips XL instrument (of the environmental version). GSE (Gazeous Secondary Electrons) and BSE (Back Scattered Electrons) procedures are used, the last one to better detect heavy elements. Elemental analysis for each particle observed were realized by EDX (Energy Dispersive X-ray), this SEM microscope being equipped with a Bruker probe AXS-EDX (the system of analysis is PGT : Spirit Model, of Princeton Gamma Technology).

Each elemental analysis is given in the form of a spectrum, with kiloelectrons / Volts (ke/V) on the abscissa and elemental peak heights in ordinates.

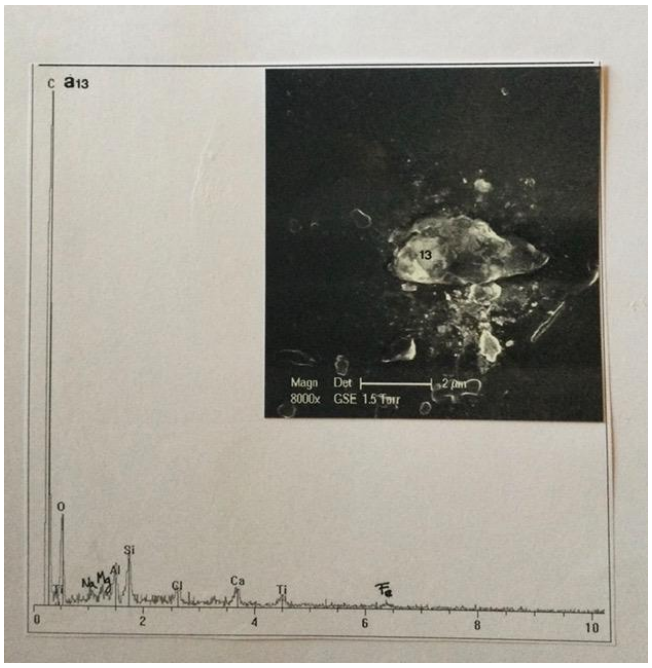
In this study we adopted a similar approach to that adopted by Schuttlefield *et al*. [**12**] for montmorillonite, illite and kaolinite, and for Sengupta *et al.* [**13**] for kaolinite.

3. Results.

3.1 The montmorillonite particles

Figure 1 shows the appearance and the spectrum of the a13 particle , which is a typical montmorillonite. It is an elongated particle, of about 2.5µm of maximal length. In its spectrum the silicium (Si), aluminium (Al), and magnesium (Mg) peaks are in orders of decreasing amounts. It has a little peak of iron (Fe) ; it has also little peaks of calcium (Ca), chlorine (Cl) and titanium (Ti).

Figure 1. The montmorillonite a13. *Above* : SEM photograph (8000x), in GSE, of the a13 particle. *Below* : the a13 spectrum. C : carbon, ; Ti (two peaks) : titanium ; O : oxygen ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; Cl : chlorine ; Ca : calcium ; Fe (little peak) : iron.



This spectrum corresponds well to that of an aluminium and magnesium hydrated silicate, in which chlorine and titanium are external pollutants. Table 1 list and characterizes the thirteen montmorillonite particles detected in the various areas of the triangle : a9 and a 9', a13 and a22 in the A area ; b68 in the B area ; e17 in the E area ; f4 and f50 in the F area ; l33 and l37 in the L area ; m4, m5 and m6 in the M area.

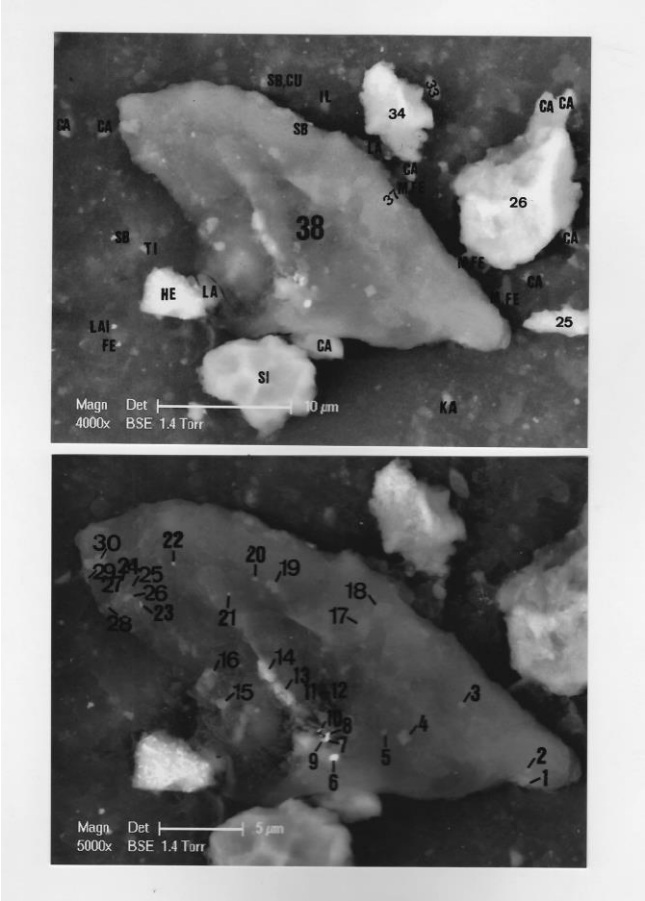
They are particles of various forms, of dimensions comprised between 8 µm (for m4) and 1.5 µm (for l37 and m5).

All of them have spectras similar to that of the a13 type. Most of them have iron (but iron traces only in a9', a13, f4 and m6) ; the spectrum of e17 is the only one without iron. Most of them (a9, a13, a22 and f4) have traces of chlorine, the particles m4, m5 and m6 being massively contaminated by this element. The a13 spectrum shows also titanium traces.

Table 1. List and characterisations of the montmorillonite particles detected on the various areas of the triangle.

Areas of the triangle	Particles	Forms	Maximal dimensions (in µm)	Spectras	Peculiarities
A	a9	rod	4.5 µm	with Cl traces	neighbouring particles
A	a9'	triangular	2.5 µm	with Fe traces	
A	a13	elongated	3.5 µm	with Fe traces ; with Ti and Cl	
A	a22	rounded	4 µm	with Cl	
B	b68	with angular outlines	5.5 µm		partially masqued by the a91 particle
E	e17	triangular	2.5 µm		
F	f4	triangular	2 µm	With Cl traces ; with Fe traces	
F	f50	triangular	3.5 µm		
L	l33	rod	3.5 µm		
L	l37	rod	1.5 µm		
M	m4	elongated	8 µm	with Fe traces ; contaminated by Cl	chlorine contamination is well visible on this particle
M	m5	rod	1.5 µm	contaminated by Cl	
M	m6	rounded	2.5 µm	with Fe traces ; contaminated by Cl	

Figure 2. The l38 particle. *Above* : SEM photograph (4000x), in BSE, showing the l38 particle. This photograph shows also the particles l25 (a gypsum), l26 (an alumina-silicate iron-rich), l33 (a montmorillonite), l34 (a calcium phosphate) and l37 (a montmorillonite) ; the two l38 adjacent particles (located between l26 and l38) are not numbered montmorillonites. IL is the not-numbered illite. KA is the not-numbered kaolinite. LA are the two lapis lazuli particles ; Si is a quartz, and HE an hematite. CA : calcite particles, SB : barium sulphate particles, one of them being associated to copper (CU) ; LAI : a brass particle ; FE : an iron particle ; Ti : a titanium particle . *Below* : SEM photograph (5000x), in BSE of the l38. Sub-particles located on the l38 surface are numbered **1** to **30.**

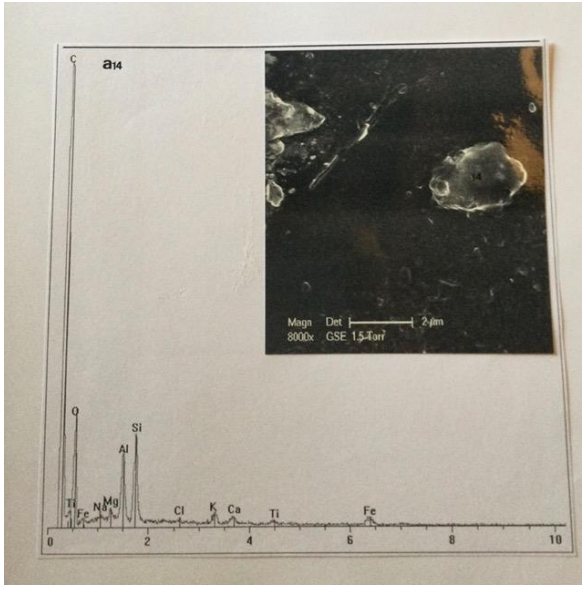


Photographs of Figure 2 shows the l38 particle (located in the L area). It is a relatively great particle (20 µm of basis, on 30 µm of height) of triangular form ; its spectrum establishes that it is a wax. Adjacent to the l38 right corner and located between l26 (an alumino-silicate iron-rich particle) and l38 are two (not-numbered) montmorillonite particles (with iron).

On the l38 surface of an enlarged photograph (Figure 3, below) one can distinguish at least two adjacent imprint parts of linen fibers of the TS. They are thirty sub-particles (numbered 1 to 30) on the l38 surface ; among these sub-particles , nine of them (**1**, **2**, **3**, **9**, **11**, **17**, **18**, **23** and **28**) are montmorillonite iron-rich.

These supplementary studies on the l38 bears to twenty-four the total number of montmorillonite particles (and subparticles) found at the surface of the triangle.

Figure 3. The illite a14. *Above* : SEM photograph (8000x) , in GSE, of the a13 particle. *Below* : the a 14 spectrum. C : carbon ; Ti (two peaks) : titanium ; O : oxygen ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; Cl (traces) : chlorine ; K : potassium ; Ca : calcium ; Fe (two peaks) : iron.



3.2. The illite particles.

Figure 3 shows the appearance and the spectrum of the a14 particle, which is a typical illite. It is an elongated particle, of about 2 µm of maximal length. In its spectrum the silicium, aluminium and magnesium peaks are in orders of decreasing amounts and it contains iron. It has a little peak of potassium and substantial amounts of calcium, chlorine and titanium. This spectrum corresponds to that of an illite, the potassium element being distinctive from a montmorillonite.

Table 2 lists and characterizes the twenty one illite particles detected in the various areas of the triangle : a14, a18 and a19 in the A area ; b85 in the B area ; f25 in the F area ; i64 in the I area ; j29, j53 and j70 in the J area ; k46 in the K area ; l22', the non-numbered particle, l71' and l33' in the L area ; m8, m65 and m66 in the M area ; n22 in the N area, p58 and p59 in the P area ; s24 in the S area.

They are particles of various forms, of dimensions comprised between 7.5 µm (for k46) and 1 µm (for the non-numbered particle).

All of them have spectras similar to that of the a14 type. All of them have iron ; j53 is the only iron-rich illite particle, but they are iron traces for f25, i64, j29, j70, k46, l22', l71', l33' and m66. Traces of titanium are detectable in a14, a18, p58 and p59. Traces of chlorine are detectable in a14, a19, i64, l22', m66, p59 and s14, the particles a18 and m65 being massively contaminated by this element.

The l22' particle is the only one where we detected traces of sulphur (S).

Table 2. List and characterizations of the illite particles detected on the various areas of the triangle.

Areas of the triangle	Particles	Forms	Maximal dimensions (in µm)	Spectras	Peculiarities
A	a14	elongated	2 µm	traces of Cl and Ti	neighbouring particles
A	a18	elongated	6.5 µm	traces of Ti ; peak of cl	
A	a19	squarred	4 µm	traces of Cl	
B	b85	rounded	4.5 µm		
F	f25	triangular	5 µm	traces of Fe	
I	i64	hexagonal outlines	3.5 µm	Traces of Fe ; traces of Cl	
J	j29	squared	4.5 µm	traces of Fe	
J	j53	elongated	2.5 µm	iron-rich	
J	j70	rounded	2 µm	traces of Fe	
K	k46	great spine	7.5 µm	traces of Fe	
L	l22’	rod	2 µm	traces of Fe ; traces of Cl and S	presence of sulphur
L	non-numbered	pentagonal outlines	1 µm		
L	l71’	rod	2.5 µm	traces of Fe	
L	l33’	rod	1 µm	traces of Fe	
M	m8	squared	4.5 µm	traces of Fe	
M	m65	rounded	3 µm	traces of Ti ; peak of Cl	
M	m66	losangic	3.5 µm	traces of Cl	
N	n22	rod	4µm		
P	p58	angular outlines	5 µm	traces of Ti	
P	p59	squarred	4.5 µm	traces of Ti and Cl	
S	S24	angular outlines	6.5 µm	traces of Cl	

3.2 The kaolinite particles.

Figure 4 shows the appearance and the spectrum of the e131 particle , which is a typical kaolinite. It is a little particle of about 3 µm of maximal length, which is with pentagonal outlines. In its spectrum the aluminium and silicium peaks are of about equal height values, and it contains a substantial amount of calcium and little peaks of magnesium, sodium and chlorine. This spectrum corresponds to that of an aluminium hydrated silicate.

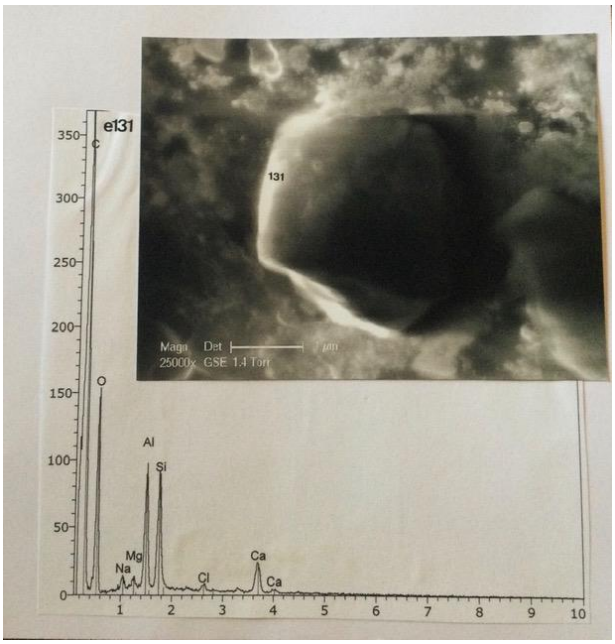


Figure 4. The kaolinite e131. *Above* : SEM photograph (2500x), in GSE, of the e131 particle. *Below* : the e131 spectrum . C : carbon ; O : oxygen ; Na : sodium ; Mg : magnesium ; Al : aluminium ; Si : silicium ; Cl : chlorine ; Ca (two peaks) : calcium.

We have already signalled the presence of a little non-named kaolinite particle, located in the L area near the right angle of the l38 particle (Figure 2, above). Table 3 lists and characterises the eight kaolinite particles detected in the various areas of the triangle : e7 and e131 in the E area ; h2 and h51-3 in the H area ; i12 in the I area ; m58 in the M area ; n26 and S16 in the N and S areas, respectively.

They are little particles of crystals with pentagonal or hexagonal outlines, of dimensions comprised between 7.5 µm (for i12) and 1.5 µm (for n26).

All of them have spectras similar to that of the e131 type. The e7 particle is the only one where we detect some traces of iron (the I 12 particle is iron-rich) ; most of them contain chlorine traces. A little peak or traces of potassium are detected in h51-3, i12, m58, n26 and s16 ; this s16 particle is the only one where we detected traces of phosphorous (P).

Table 3. List and characterizations of the kaolinite particles detected on the various areas of the triangle.

Areas of the triangle	**Particles**	**Forms**	**Maximal dimensions** (in µm)	**Spectras**	**Peculiarities**
E	e7	crystal with pentagonal outlines	2.5 µm	with Fe traces	
E	e131	crystal with pentagonal outlines	3 µm	with Cl traces	
H	h2	crystal with hexagonal outlines	4.5 µm	with Cl traces	
H	h51-3	crystal with pentagonal outlines	6 µm	with Cl traces and a little peak of K	seems located to the other side of the L area
I	i12	crystal of hexagonal outlines	7.5 µm	with Cl and K traces	iron-rich
M	m58	crystal with hexagonal outlines	7 µm	with Cl traces and a little peak of K	
N	n26	little crystal with angular outlines	1.5 µm	with a little peak of Cl and traces of K	
S	s16	crystal with pentagonal outlines	4.5 µm K and P	With traces of Cl,	presence of phosphorous (P)

4. **Discussion and conclusions**.

In this study we confirm and complete the preliminary results we obtained initially [**9**] concerning the presence of little clay particles of montmorillonite, illite and kaolinite on the TS surface. By SEM-EDX analyses, we distinguish between the three sorts of particles : illite particles differ from montmorillonite by the potassium element in their spectras, and in kaolinite spectras the aluminium and silicium peaks are of equal heights.

A total number of fifty-four well characterized of these little clays are so detected on the triangle surface : twenty-four of montmorillonite, twenty-one of illite and nine of kaolinite. It does not seem that there are special distributions of these three sorts of particles in the different areas of the triangle, but we note an abnormal concentration (11/24) of montmorillonite particles around and on the l38 particle , located in the L area.

The montmorillonite and illite clays are largely spread out on the earth surface , where they constitute usual mineral of soils (red montmorillonite - that is iron-rich - is characteristic of arid and dry soils). These dusts are transported by the wind and deposited on various objects. We can explain the observed abnormal concentration of montmorillonite on the l38 particle by the fact that it is a wax, that retained the various microparticles (including these clays) at the surface from the numerous cleanings and washing that suffered the TS during times.

Kaolinite was a clay mineral used since many times for pottery and ceramic. We suspect that the kaolinite particles observed are micro-residues of some vase or recipient put down on the Face area of the Turin Shroud.

5. Acknowledgment.

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6. Conflict of interest.

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

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