

# EXPLORATION OF THE FACE OF THE TURIN SHROUD. LINEN FIBERS STUDIED BY SEM ANALYSIS

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**Abstract-** We have studied by scanning electron microscopy and energy dispersive X-ray (EDX) the linen fibers loaded on the surface of a small sticky tape triangle corresponding to the Face area of the Turin Shroud (TS). Five linen fibers (FL1-5) were found. They are linen fiber portions (maximal length : 50  $\mu$ ), broken at the basis and thinned at the extremities, and wrenched from flax threads constituting the TS. Some of them present evidence of white painting, by lime or by mineral deposit of calcite particles.

**Keywords :** Face area, linen fibers, optical and petrographic microscopy, scanning electron microscopy (SEM), energy dispersive X-ray (EDX), Turin Shroud (TS).

## I. INTRODUCTION

The Turin Shroud (TS) (Marion and Lucotte, 2006) is an object in which a body image, not yet explained by modern science (Fanti et al., 2010), is imprinted. For this reason, many studies are still open in order to understand better its characteristics. Some of them are devoted to the study of the microscopic aspect of the linen fibers (Raes, 1991) of which the TS is constituted.

In 1978 and in 1988, Giovanni Riggi di Numana took a sample of some specks of dust (Riggi di Numana, 1988) from the back of the TS (at areas corresponding to Hands, Face, Feet, Buttocks and  $^{14}\text{C}$  area) and sampled them on various filters. Different analyses have been done on these dusts at the micrometric level (Fanti, 2006) ; other analyses, using scanning electronic microscopy (SEM) and other instruments (Fanti et al., 2013), have also been performed.

The present work aims at studying, by SEM microscopy and X-Ray (EDX) analysis, the residual linen fibers located at the surface of a small sticky tape triangle (Lucotte, 2012) containing fibers and dusts corresponding to the area of the Face.

## II. MATERIALS AND METHODS

The material (Lucotte, 2012) is a small (1.36 mm height, 614  $\mu\text{m}$  wide) sticky tape triangle at the surface of which portions of fibers, pollen grains and spores, and some plaques of organic matter were deposited. As declared by Riggi di Numana, this sticky tape triangle is one part of a larger piece he applied directly (during the 1978 sampling) to the TS surface, near one "blood area" of the Face.

More than 2500 particles (more than 1  $\mu\text{m}$ ) can be observed at the surface of the triangle ; all of them were studied by optical microscopy, SEM and EDX analysis. For practical reasons, the surface of the triangle was subdivided

into 19 sub-samples areas (areas A to S), containing almost all the particles observed. The positions of each particle sticking to the triangle surface were located in a double system of coordinates (in 186 adjacent squares of 50x50  $\mu\text{m}$  – 2 500  $\mu\text{m}^2$  of the total surface).

Particles of the samples were observed, without any preparation, on the adherent part of the surface of the triangle. The observations were conducted by SEM, using a Philips XL 30 instrument (environmental version) ; GSE and BSE procedures were used, the last one to detect heavy material. Elemental analyses for each particle were realised by X-ray microfluorescence (XRMF), this SEM microscope being equipped with a Bruker AXS energy dispersive X-ray (EDX) ; the system of analysis is PGT (Spirit Model, of Princeton Gamma Technology).

Our SEM studies were completed by the optical observations of fibers (using a photomicroscope Zeiss, model III, 1972) and by studies in cross-polarized light (with the petrographic version of this microscope).

## III. RESULTS

A total number of five linen fiber (according to Bergfjord and Holst, 2010) portions were observed on the triangle (Figure 1). The nomenclature and locations in the various areas of the triangle for each of them are specified in Table 1 (they are numbered and classified among their descending positions along the vertical triangle axis). The following descriptions concern the detailed characterisations of these five linen fiber portions.

### The first (FL1) linen fiber portion (b11).

FL1 (Figure 2) is an isolated portion of linen fiber, broken after a node (nodes, a characteristic of linen fibers, protuberates at regular distances on the surface of linen fibers ; they are the attachment points of the lignin fibers). The total

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length of this fiber portion is about 35  $\mu$ , and its thickness after the node is approximately 6-7  $\mu$ .

The fiber portion frays at the extremity (Figure 3), where individual cellulose fibrillates (fibrillation is a sign that it was worn). In optical microscopy FL1 appears white, with light yellow reflects; there is no birefringence of the fiber when observed in polarized light.

The elementary composition of this fiber-portion (Figure 2) is rich in organic matter (carbon + oxygen) – a composition compatible to that of cellulose – and there is an important peak of calcium ; that possibly represents a linen cellulosic fiber, partially calcified by calcium carbonate ( $\text{CaCO}_3$ ). The compositions of the four round sub-particles CH1 to 4 located at the fiber basis, denser in calcium, are compatible to a composition of lime ( $\text{Ca(OH)}_2$ ). The fifth sub-particle CH5, whiter in BSE (Figure 1) and calcium-rich, is probably a particle of calcium-dense carbonate.

### **The second (FL2) linen fiber portions (b10).**

FL2 (Figures 4 and 5) is a portion of a linen fiber, made of two parts. The upper part (Figure 4) is broken at the basis, after a node ; fibrillation is visible at the other extremity. The lower part of the fiber (Figure 5) is divided from the upper part by a zone of breakage.

The total length of the fiber is about 50  $\mu$  ; the thickness of the upper part of the fiber after the node is approximately 7.5  $\mu$ , just as the largest portion of the lower part. In optical microscopy the upper part of FL2 appears white and the lower part is darker ; there is no birefringence of this fiber when observed in polarized light.

The elementary composition of the upper part is rich in organic matter, with a modest peak of calcium (Figure 4). This calcium peak is relatively much higher in the elementary composition of the lower part of the fiber (Figure 5). On the photography, this lower part appears as completely covered by a dense stratum of little mineral particles ; they are particles of calcite ( $\text{CaCO}_3$ ), some of them (particle number 4) having a characteristic rhombohedral structure.

With these observations, we have the proof that the lower part of FL2 is covered by a layer of white mineral calcite painting.

### **The third (FL3) linen fiber portions (b29).**

FL3 (Figure 6) is a portion of a linen fiber. Its length is approximately 40  $\mu$ , for a thickness of about 5  $\mu$ . The coverage of the fiber in its medium part by particle b23 (an iron oxide as determined by EDX analysis) delineates two parts of the fiber : FL3.1 and FL3.2. At its extremity, the fiber fibrillates. At its basis, the node of the fiber is covered by particle b35. Some twisted elementary fibrils are apparent in the fiber after the node (photography 2 of Figure 6).

Observation in BSE (Figure 7, photography) allows us to observe that the fiber is covered by numerous mineral sub-particles, relatively dense to electrons. The elementary composition of the two parts of the fiber (Figure 7, spectrums) are equally rich in organic matter, but the calcium peak is modest in FL3.1 and relatively high in FL3.2. That may correspond to a fine coverage of the fiber by calcium

carbonate / calcite painting. Particle b35 (located at the lower part of the fiber) has a similar calcium carbonate composition.

### **The fourth (FL4) linen fiber portions (e40).**

FL4 is a tripartite portion of linen fiber (Figure 8). It corresponds to a fiber extremity (part 3), splitted in two (parts 1 and 2) finer filaments ; this bifurcate pattern is characteristic of some linen fiber extremities (such a split-end indicates an intense longitudinal traction of the fiber). The total length of part 3 is about 2.5  $\mu$ , and those of the two branches are approximately 1  $\mu$  only.

There is no apparent deposit of mineral particles at the surface of this fiber (Figure 8, photography). At the surface of some portions (parts 2 and 3) of the fiber, we can distinguish the twisted contour of elementary fibers.

In optical microscopy the fiber appears white, quasi-translucid. In polarized light the fiber (mostly parts 2 and 3) is birefringent, a characteristic of the vegetal textile fibers composed by crystalline cellulose.

The elementary compositions of the three parts of the fiber (Figure 8, spectrums) are equally rich in organic matter (corresponding to cellulose); the individual spectrums show a relatively low content of calcium in the three cases.

### **The fifth (FL5) linen fiber portions (e105).**

FL5 is a portion of linen fiber, rolled up in its medial part (Figure 9). It presents a characteristic node at the upper extremity. The total length of the fiber is 25  $\mu$  approximately ; its thickness is about 1.5  $\mu$  after the node.

The most important part of the fiber after the rolling up is flattened : that represents the crushing zone ; this zone is shaded by a thin film of modern plastic (of the vinyl polychlorure type, chlorine rich). At the periphery of the crushing zone, the fiber defibrillates (Figure 9, photography).

In optical microscopy the fiber, at least between the node and the rolling up, is translucent; in polarized light this zone is birefringent. The elementary composition of the fiber at the rolling up (Figure 9 , spectrum) is rich in organic matter (that corresponds to cellulose) ; in this spectrum the calcium peak has a low value.

## **IV. DISCUSSION**

We know since more than thirty years (Raes, 1991) that the Turin Shroud (TS) is mainly made of flax threads. Any sample taken at the surface of these threads by a sticky tape can include fragile fibers composing these threads. This is the case reported here, because the triangle sample contains five altered portions of linen fibers.

The main characteristics of these five linen fiber portions are summarized in Table 1. They are linen fibers, because all (except the fourth?) of these cylindric fibers show characteristic nodes at the surface. Their colours are white, or translucid (except FL3, not seen in optical microscopy); the two non-covered fiber portions FL4 and FL5 show birefringency in polarized microscopy. All the fiber portion elementary compositions are compatible with that of cellulose. There are no traces of lignin fibers at the nodes, so they correspond to well prepared textile fibers.

They are portions of linen fibers, because broken before the nodes. While the mean length of a full-length linen textile fiber is at least of the order of 1 cm (Reis et al., 2006), their lengths do not exceed 50  $\mu$  (Table 2).

Their natural pointed extremities fibrillate, this indicates that it was worn. It is for these reasons (short thinned fragments of fibers) that these fiber portions of the flax threads are absorbed on the sticky tape.

There are other portions of textile fibers in the triangle (Lucotte, unpublished), including : 1/ A long red silk fiber (g38) ; 2/ three intricate synthetic textile fibers (c12). We have not found any cotton fiber on the triangle surface.

The thickness of the linen fiber portions found here do not exceed 7.5  $\mu$  (Table 2) ; this contrasts with the usual mean diameter of textile linen fibers (Reis et al., 2006), which is about 20-80  $\mu$ . The explanation is that these fiber portions correspond to linen fiber extremities (fiber diameter narrows at the end).

We have some evidence of some mineral overload on the fibers : calcium ion is at significant elevated values in the elementary compositions for all the fiber portions, or for some of their parts (Table 3) ; in some cases it is possible to distinguish in fibers the calcium level due to mineral deposit from that assigned to a calcification process (in the dissolved form).

Calcium carbonate deposits are visible on some parts of the surfaces of fibers 1, 2 and 3. For FL1 and FL2, these deposits are responsible for the white colour of the fibers observed in optical microscopy, and for the absence of birefringence in polarized light. Some particles of lime are located at the basis of the FL1 fiber portion (subparticles CH1, 2, 3 and 4) and on the surface of FL3. A multitude of characteristic calcite sub-particles cover all the surface of FL2.2; possibly CH5 (at the basis of FL1) is a calcite sub-particle.

So, the current observations shown and analysed allow us to conclude that some fibers / flax threads of the TS were painted in white by a mineral composition of some forms of calcium carbonate. One of these forms is lime. Another form is calcite, the painting process with a mineral powder of calcite being evident (Figure 5) for the surface of the second part of the FL2 fiber portion. Painting of the TS in white was necessary for such an old (and necessarily dirty) textile tissue. Painting in white with a mineral powder of calcite is a process known since the Antiquity.

### CONFLICT OF INTEREST

The author has declared no conflict of interest.

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**Table 1. Nomenclature and locations of the five linen fibers**

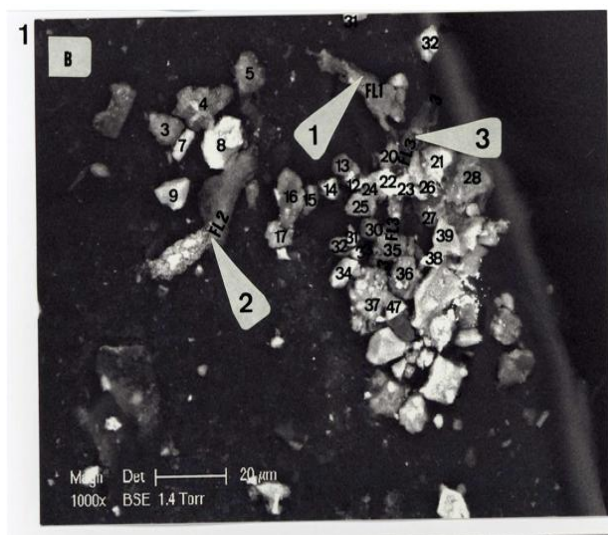
| Numbers | Designations (code) | Locations in the Triangle areas |
|---------|---------------------|---------------------------------|
| 1       | FL1 (b11)           | B                               |
| 2       | FL2 (b10)           | B                               |
| 3       | FL3 (b29)           | B                               |
| 4       | FL4 (e40)           | E.c                             |
| 5       | FL5 (e105)          | E.c                             |

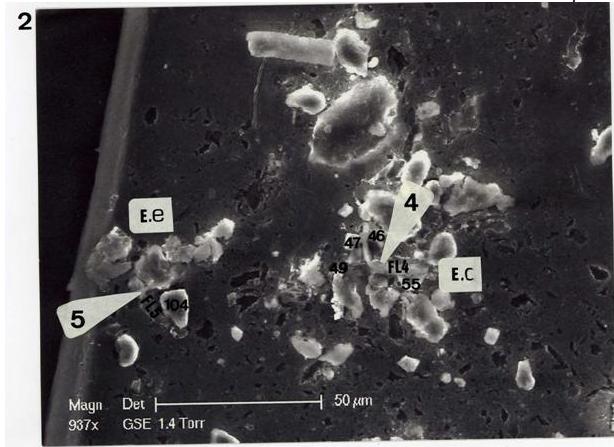
**Table 2. Characterisations of the five fiber portions.**

| Fibers                                     | FL1   | FL2   | FL3 | FL4                  | FL5                                 |
|--------------------------------------------|-------|-------|-----|----------------------|-------------------------------------|
| Nodes                                      | +     | +     | +   | ?                    | +                                   |
| Breakage before the node                   | +     | +     | +   | +                    | +                                   |
| Natural extremity                          | +     | +     | +   | bifurcated           | +                                   |
| Length (in $\mu$ )                         | 35    | 50    | 40  | 25                   | 25                                  |
| Thickness (in $\mu$ )                      | 6-7   | 7.5   | 5   | 2.5                  | 1.5                                 |
| Fibrillation at the extremity              | +     | +     | +   | at the end of part 1 | +, and also around the crushed zone |
| Colour                                     | white | white | ?   | translucid           | translucid                          |
| Birefringency                              | -     | -     | ?   | +                    | +                                   |
| Spectrum compatible with that of cellulose | +     | +     | +   | +                    | +                                   |

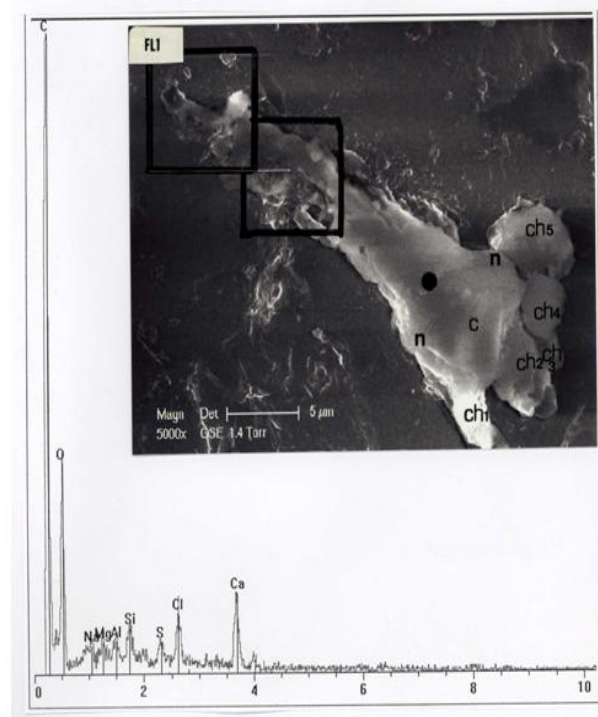
**Table 3. Calcium overload on fibers.**

| Fibers | Elevated values of calcium | Calcium carbonate | Lime                                | Calcite  |
|--------|----------------------------|-------------------|-------------------------------------|----------|
| FL1    | +                          | +                 | At the basis in sub-particles CH1-4 | In CH5?  |
| FL2    | + in FL2.2                 | +                 | -                                   | In FL2.2 |
| FL3    | + in FL3.2                 | +                 | +                                   | -        |
| FL4    | + in FL4.1                 | -                 |                                     |          |
| FL5    | +                          | -                 |                                     |          |



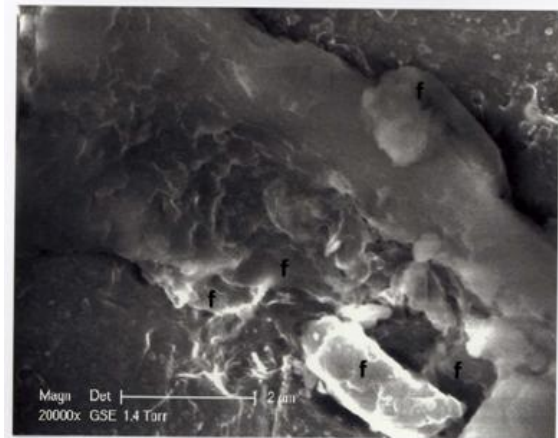
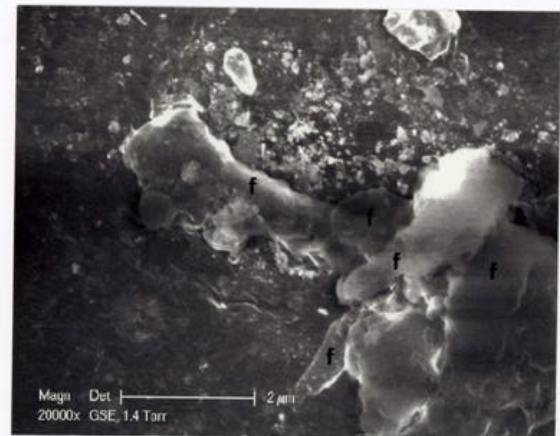


**Figure 1.** Positions of the five linen fibers (FL1=1, FL2=2, FL3=3, FL4=4, FL5=5) in the triangle. Above (1) : SEM photograph (x1000, in BSE) of area B, showing FL1, FL2 and FL3. Below (2) : SEM photograph (x937, in GSE) of areas E.c and E.e showing FL4 and FL5, respectively. FL1 fiber is isolated; particle b8 is at the edge of FL2 ; particle b35 is located at the lowest part of FL3; particle e55 is located below the main part (3) of FL4, particles e46 and e47 are at each side of the first (1) part of FL4, and particles e49 is located at the end of the second (2) part of FL4 ; particle e104 is located near FL5.

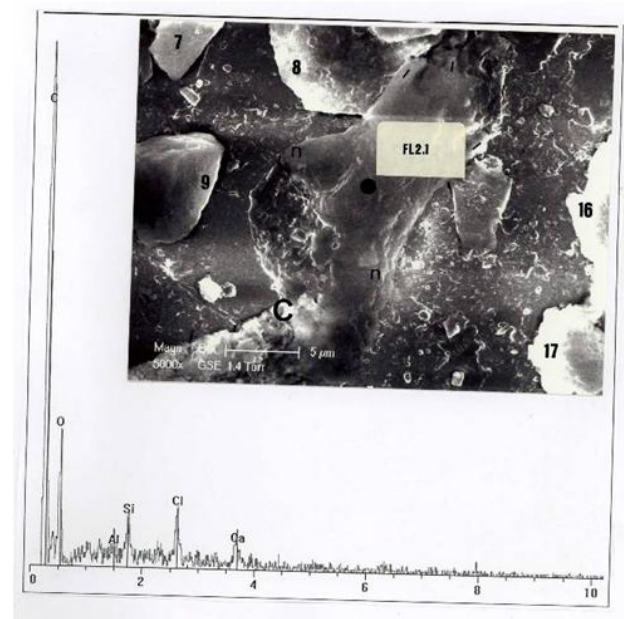


**Figure 2.** The first (FL1) linen fiber (b11), located in area B of the triangle. Above, SEM photograph (x5000) of FL1 (the two square quadrate parts of the fiber located on its top are magnified in the following figure). Below, EDX analysis (horizontal axis is graduated in keV, and heights of the peaks are proportional to quantities of elements in the sample) of FL1 at the black point indicated on the fiber. The

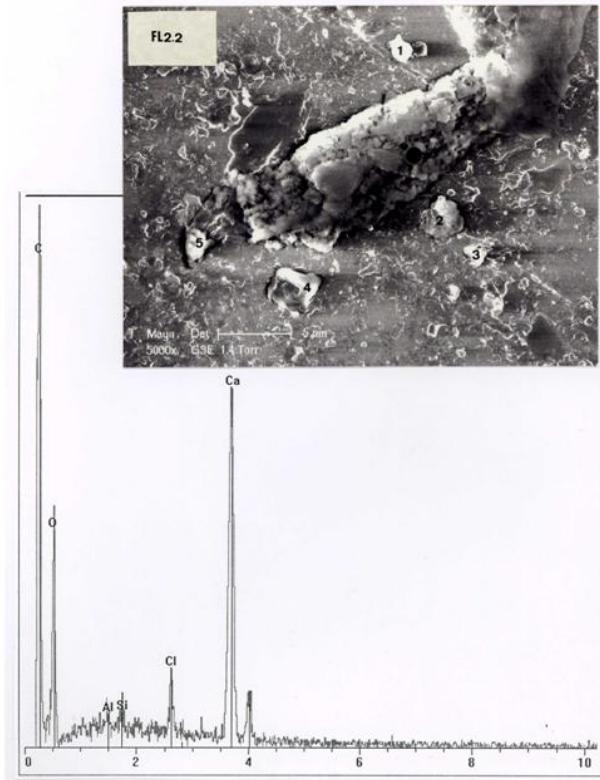
two letters **n** correspond to two borders of the node ; **c** corresponds to the fiber part composed of calcium carbonate ; ch1-5 are round sub-particles located at the basis of the fiber.



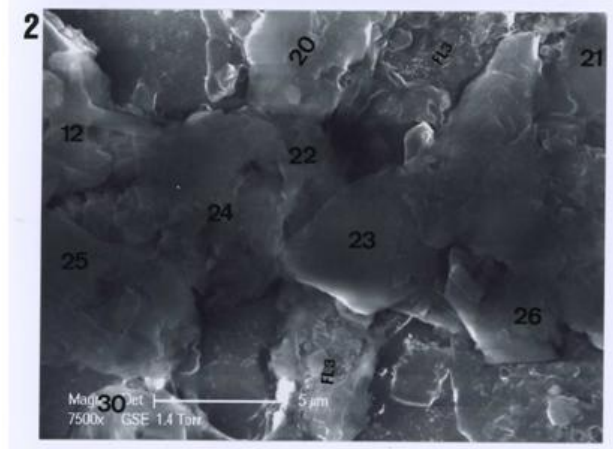
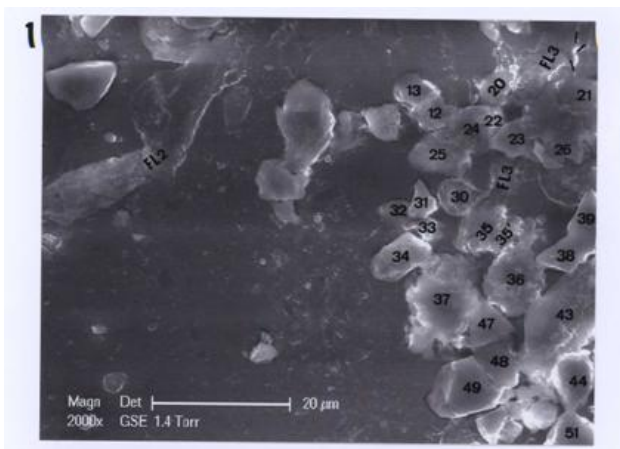
**Figure 3.** Fibrillation at the extremity of the FL1 fiber. Both SEM photographs are x20000 ; letters **f** mean fibrillation. The above photograph represents the point of the fiber ; the below photograph represents the adjacent zone of the first one.



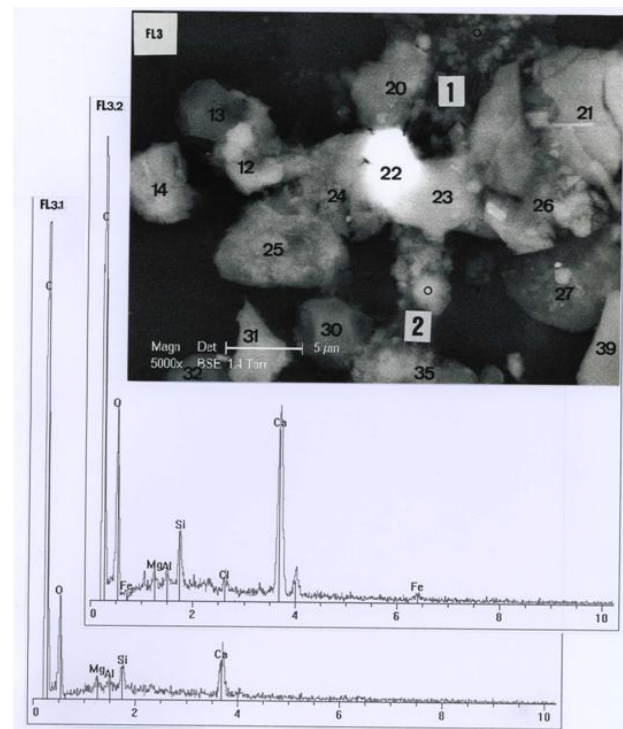
**Figure 4.** The second (FL2) linen fiber (b10), located in area B. Above, SEM photograph (x5000) of FL2.1 (the upper part of FL2). Below, EDX analysis at the black point indicated. The two letters **n** indicate node borders ; the directions of fibrils at the fiber extremity are shown by bars ; C: region of fibre break. Particles b7, 8, 9, 16 and 17 are adjacent to the fiber.



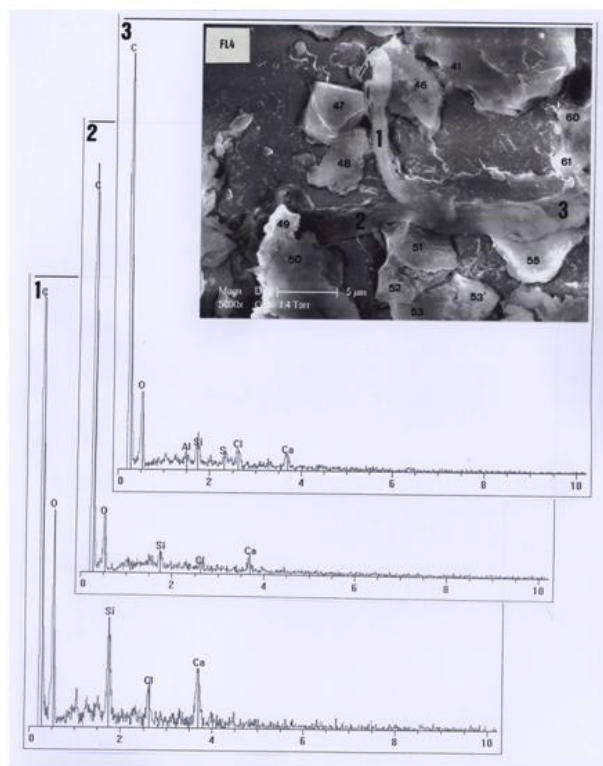
**Figure 5.** FL2.2 is the lower part of FL2. Above, SEM photograph (x5000) of FL2. Below, EDX analysis at the black point indicated. 1,2,3,4 and 5 are little particles of calcite adjacent to FL2.2.



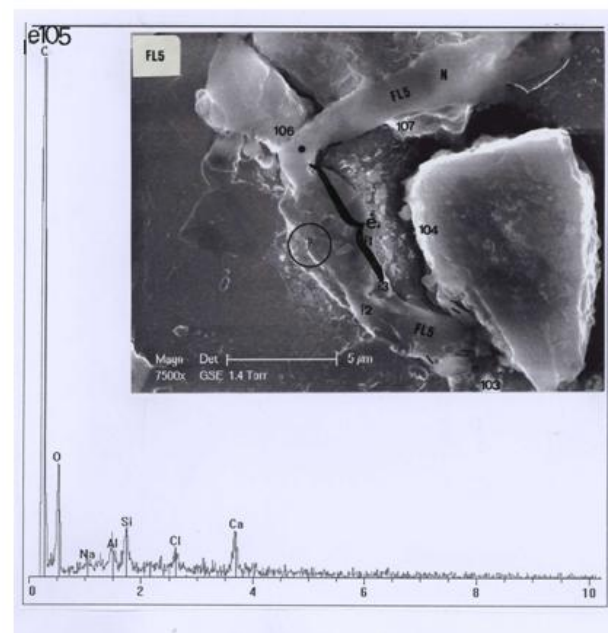
**Figure 6.** The third (FL3) linen fiber (b26), located in area B. Above (1) SEM photograph (x2000) of some part of area B, in the vicinity of FL3, where the FL2 fiber is also shown. The length of the FL3 fiber is divided in two parts by the b23 deposit. Bars indicate fibrillation at the FL3 extremity ; the FL3 basis (node) is covered by particle b35. Particles b20, 22 and 30 are adjacent to FL3. Below (2), SEM photograph (x7500) of the FL3 main part.



**Figure 7.** Above, SEM photograph (x5000, in BSE). Below, EDX analysis of the first (FL3.1) and the second (FL3.2) parts of FL3 at the white points indicated. FL3 is covered in its middle part by particle b23, and limited at the basis by particle b35. Particles b20, 21 and 22 are adjacent to the fiber.



**Figure 8.** The fourth (FL4) linen fiber (e40), located in area E.c. Above, SEM photography (x5000) of FL4 ; 1, 2 and 3 are the three parts of the fiber. Particles e46, 47 and 48 are adjacent to part 1 ; particles e49 and 50 are adjacent to part 2, and e51 and 55 are adjacent to part 1. Below, EDX analyses of the three parts. The directions of fibrils at the part 1 extremity are showed by bars.



**Figure 9.** The fifth (FL5) linen fiber (e105), located in area E.e. Above, SEM photography (x7500) of FL5. Below, EDX analysis of FL5 at the black point indicated. N : node ; f1, f2 and f3 : elementary fibrils,  $\epsilon$  : the crushing zone ; P: zone of

the covering plastic (e103, 104, 106 and 107 are all the particles associated to e105). Directions of fibrils at the extremity are showed by bars.

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