ON THE EFFECT OF SALT AND ITS REMOVAL FROM THE FIBERS OF THE WOOL WEAVINGS FOUND WITH THE SALT MEN IN CHEHR ABAD SALT MINE, ZANJAN

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1. Introduction

Chehr Abad salt mine in Zanjan has preserved valuable organic and inorganic objects, in addition to mummies, in a good state of conservation, because of its particular salt bed. Today, many researchers and explorers have been able to obtain valuable information from these objects that have remained buried for many years in the heart of these salt mines.

Major mining objects are made of organic materials, which, due to the disinfectant properties of salt, have been biologically immune to damage such as mold-fungus formation and vermin attacks. Due to environmental conditions in the mine, most of the damage to the objects, especially organic ones, is physical; the effects can be found in the mummified bodies and other organic objects, such as woven artifacts, those made from wood and so on. The hand-woven artifacts, due to their enormous importance, are of special attention. The discovery of exquisite weavings from different historical eras (Achaemenid, Sassanid and Qajar) have provided the researchers with valuable information regarding textiles and materials from different periods, particularly the Achaemenid era.

In this study, 5 samples of wool weavings belonging to the Achaemenid era were investigated in order to study the effects of salt crystals on wool fabrics. The samples were studied using a light microscope, Scanning Electron Microscope (SEM), and Energy Dispersive X-ray Analysis (EDX); the salt crystal arrangement was observed using the obtained images and comparing microscopic images.

2. History of the exploration and study of the Chehr Abad salt mine of Zanjan

Salt Man 1

In the first year of mine exploration with mechanical machinery in the winter of 1993, miners using bulldozers to extract mine tailings and salt, discovered the half body of

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the salt man shown in Figure 1 [1]. The Salt Man was discovered in the southern part of the Chehr Abad salt mine, in the middle section of one of the collapsed tunnels dug from southwest to northeast and measuring 45 meters in length [1].

Figure 1. Salt Man 1, National Museum, Tehran.

Upon notification by Zanjan Cultural Heritage administration, archaeological research was initiated in the winter of the same year, first led by Dr. Hooshang Sobouti and then by Dr. Ali Asghar Mir-Fattah. As a result of excavations, a number of interesting objects were found: three knives with handles, one of which has a leather sheath, a silver ear cleaner, a grindstone, trousers with a decorative margin, three pieces of patterned cloth, leather and wool rope parts and walnut shell, broken pieces of pottery and some human bones. However, the most important find discovered in the excava-

tions in 1993 was the left leg of Salt Man 1, which was found inside a long boot. The boot was made of leather and its height was 48 cm [2].

According to the style of haircut and clothing, as well as the discovered pottery pieces, Sobouti attributed the Salt Man's body to the 8th century BC. He compared his clothes with the clothes of the Sakai people and believed that the clothing, golden earrings and other objects found with the body were evidence that the salt man was not a mine worker. According to Sobouti, Salt Man 1 was a commissioner and representative of the salt mine's administration [3].

After completing the exploration, the discovered body and objects were transferred to the research lab of the Restoration Institute for further study and investigation. These were carried out in different fields, which included dating tests, osteology studies using tomography imaging, determination of blood group, DNA studies, and so on. Results of the studies and experiments are published in a booklet on the Salt Man.

The result of C14 dating carried out on samples of bone and cloth determined the date of Salt Man 1 to about 1700 years ago, i.e. the early Sassanid period. Based on studies, it was found that the discovered body belongs to a middle-aged man who had a severe blow on the head before his death. His hair and beard were originally brown, which turned white due to oxidation conditions and the presence of chloride ion in the pigment of the hair. The Salt Man's blood group was B. DNA testing was one of the most important studies on the Salt Man. Using these tests, it was determined that the boot and the leg inside it, found in the archaeological exploration, belong to this Salt Man [4].

Restoration Institute experts also believed that, according to his appearance, the Salt Man was not an ordinary worker. Due to his having a beard and long hair, earrings and elegant boots, he was probably a hunter or a prince killed in the mine [5].

Salt Man 2

In 2004, when miners were again working with bulldozers, they encountered human skeletal remains. Unfortunately, they were largely destroyed by the bulldozer. The skull and jaw along with some hair and beard, bones, pieces of clothing and so on were also discovered. The body belongs to a middle-aged man about 180 cm tall [6, 7].

Salt Man 3

Like Salt Man 2, Salt Man 3 was also discovered while working with bulldozers to remove tailings under a large salt rock. On one side of the rock, there are signs of pickaxe digging and carving.

Salt Man 4

Finally, in 2004, archaeologists excavated the most perfect sample of a salt man body. It is clear that the dry environment full of salt and limited microbial activity helped to preserve the body. Some containers made of clay, the knife with the leather sheath, and whole clothing items were also found.

Salt Man 5

During the second season of the explorations, Salt Man 5 was discovered. There was also a pisooz (a kind of lamp that burns animal fat), two pieces of wood, a large cow horn, some rope made of vegetal fibers and pieces of clothing.

Salt Man 6

The skull of Salt Man 6 was discovered at the beginning of the excavation campaign of 2010 and remained in the salt mine until the following excavation season [8, 9].

3. Conservation state of the mummies and weavings extracted from the archaeological site

After excavation and exploration of the archaeological sites of the ancient Chehr Abad salt mine in Zanjan and finding unique objects in the salt tunnels, including natural mummies known as 'salt men', fabrics and weavings, wooden tools, metal objects, earthenware objects and the like, they were removed from the salt bed and classified by the relevant experts according to the material type of the object. Finally, after classifying the objects, they were washed, labeled and numbered after drying, then packaged for transportation to the storage area [10].

After transferring the objects and mummies to Zanjan, glass show cases were prepared to preserve the mummies in, incorporating 24-hour temperature and humidity smart control devices located in the lower part of the cases. The resulting information is recorded in the computer attached to the glass case. Using the existing charts, possible fluctuations in temperature and humidity can be clearly observed for the mummies inside the glass and, in the case of severe fluctuations, conservation and restoration measures can be carried out [11]. The glass cases where the mummies are kept, are monitored using 24 hour devices; room temperature and humidity is also continuously monitored (Figure 2 A, B).

Figure 2. A) Conservation location of Salt Men; B) glass cases containing Salt Men with humidity control device in the forefront; C) hand-woven artifacts inside zipped bags. H. Khamseh, 2016.

Beside the mummies' bodies were a large number of exquisite weavings in varying materials, colors and textures from different historical eras: Achaemenid, Sassanid and Qajar.

These hand-woven artifacts were cleaned and washed after removing the salt, respecting their material composition of wool, cotton, or goat hair and the severity of the damage. They were then placed inside zipped bags according to their size and stored under normal conditions (Figure 2 C).

A number of weavings are of importance with regard to their age, thread and dye. The woven artifacts were cleaned and repaired by the experts of the Research Institute of Cultural Heritage & Tourism in Tehran and are already on exhibition among the weavings of the Archaeology Museum of Zanjan province in the Salt Men section (Figure 3).

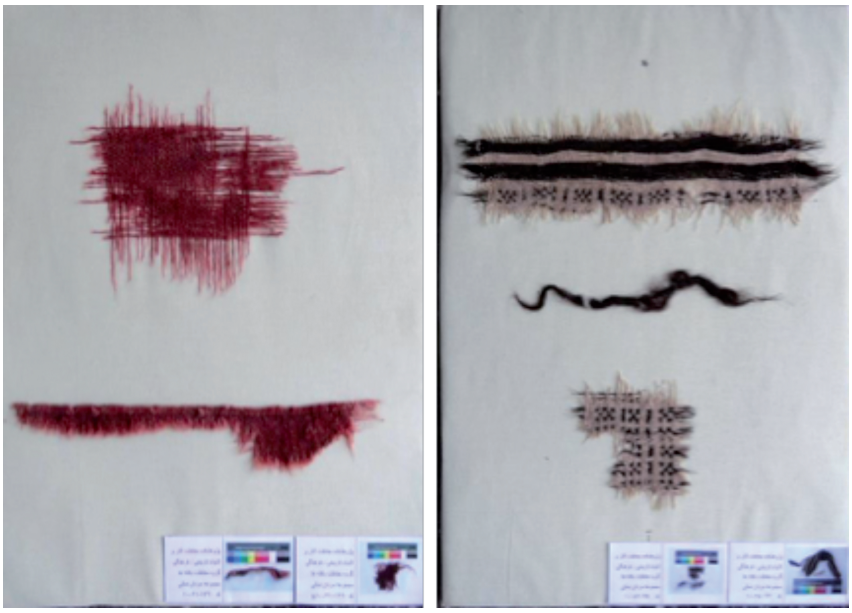

Figure 3. Dyed hand-woven artifact. H. Khamseh, 2016.

4. Laboratory Studies

There are three common ways to identify hand-woven fiber types. They are as follows: burning method, chemical method, and microscopic method. First, the burning method is used. A small amount of fiber, where little damage has occurred, is cut using scissors; it is then placed near a fire and the burning, smell and ash are examined. After visual observation of the burnt fiber and resulting ash and smell, the material of the sample is found to be wool (Figure 4).

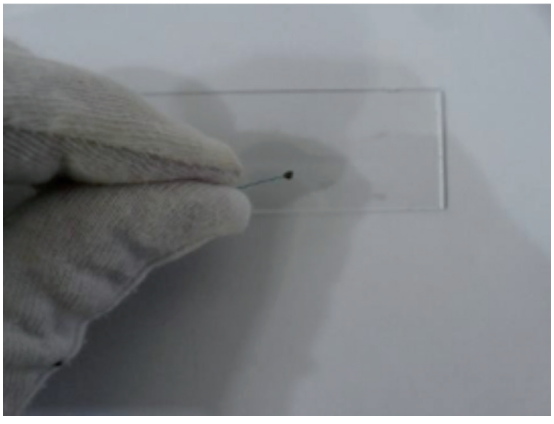

Figure 4. Burning method of fibers. H. Khamseh, 2016.

5. Electron microscopic examination

In order to examine the fibers by SEM electronic microscope, the presented samples were used to compare the microscopic images with different magnifications.

Samples are as follows:

- Sample E1: A piece of cloth from Salt Man 3 from the Achaemenid period (unwashed)
- Sample E2: A piece of cloth from Salt Man 3 from the Achaemenid period (unwashed)
- Sample E3: A piece of cloth from Salt Man 3 from the Achaemenid period (washed)
- Sample E4: A piece of cloth from Salt Man 3 from the Achaemenid period (washed)
- Sample E5 GREEN: Contemporary fine wool fibers
- Sample E6 OLD: Achaemenid era fine wool fibers

Figure 5 shows samples before and after being coated with gold. The samples covered with gold have become discolored. Finally, the prepared samples are placed in the German RONTEC microscope under vacuum and imaging begins with different magnifications.

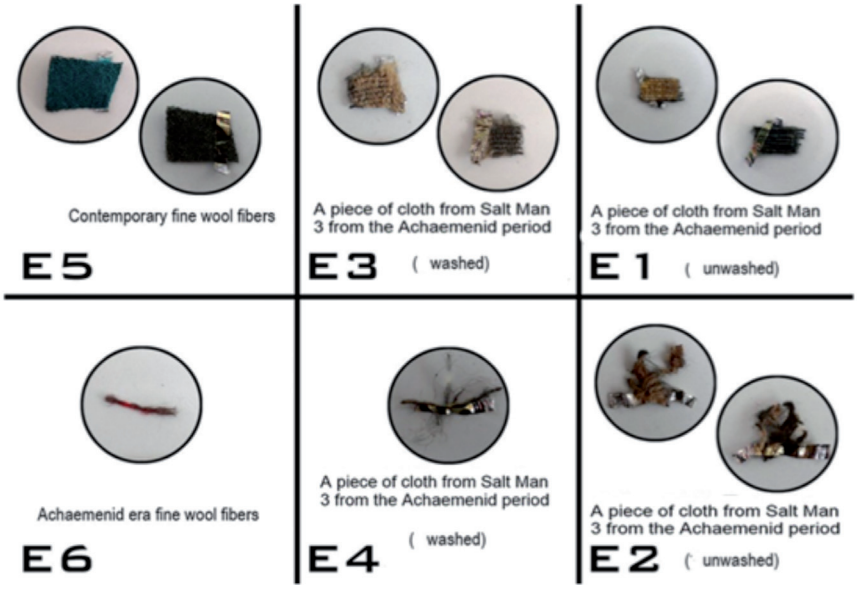

Figure 5. Samples E1-E6 coated with gold.

6. Result of microscopy images

As can be seen in samples E1 and E2, contamination can be detected by the naked eye. Moreover, salt crystals are visible using an optical microscope with a magnifica-

tion of 20× and 40× and SEM micrographs with a magnification of 100×, 2000×, and 5000× (Figure 6 A, B).

As can be seen in Figure 6 A and B below, in the magnification of 2000×, the scales of the wool fibers are damaged and salt crystals have penetrated into the wool fiber.

This damage is also more clearly visible in sample E1 (Figure 6 A). Even in the fibers, the mud and salt crystals can be clearly seen. In this sample, the wool fiber has also been destroyed.

In Figure 6 C of sample E3, with magnification of 20x and 40x, fine salt crystals can be seen in the wool fabric of the cloth from Salt Ma n 3. At a magnification of 100x and 2000x of the SEM, no sign of salt crystals and other contaminants is observed. The fiber scales are less damaged. In Figure 6 D of sample E4, the presence of salt crystals and wool fiber damage are detectable.

In Figure 6 E, showing sample E5, selected as a check sample, the scales are visible with magnification of 20x, 40x, 100x, and 2000x.

Figure 6 F shows a sample of Achaemenid era fine wool fibers.

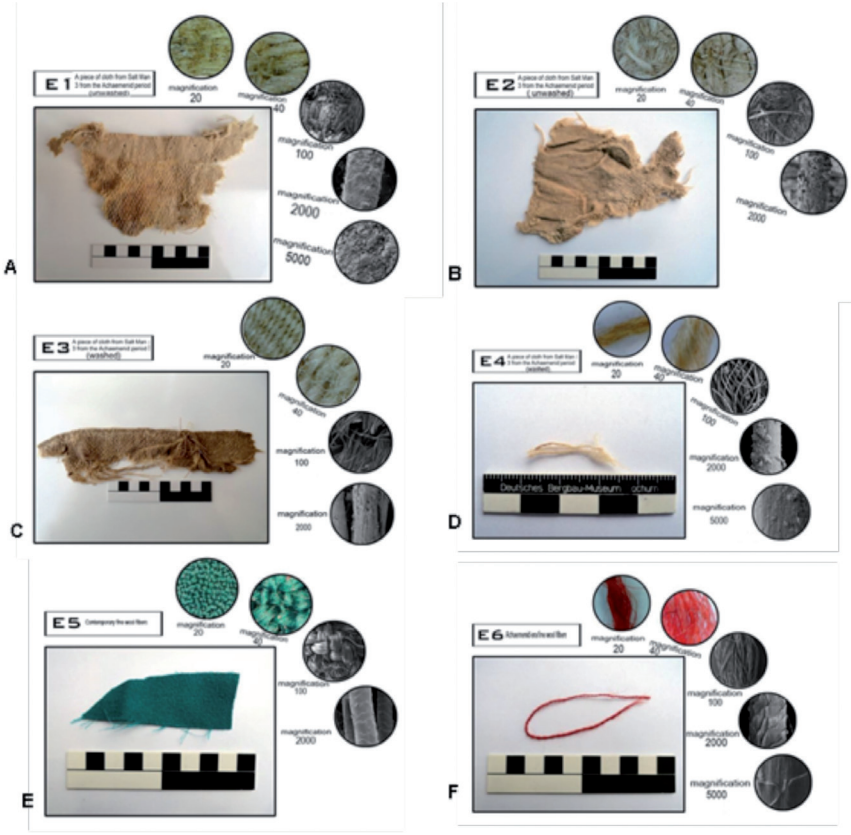

Figure 6. Microscopic examination of samples E1-E6 using an optical microscope and SEM at different magnifications. A-B) Samples E1 and E2, salt crystals are visible at different magnifications; C) Sample E3, no sign of salt crystals is observed at a magnification of 100× and 2000× of the SEM; D) Sample E4, presence of salt on fiber; E) Sample E5, control weave sample; F) Sample E6 of fine wool fibers.

7. Comparison of microscopic images of samples

Figure 7 shows wool fibers with a magnification of 40x and 20x by light microscope and in Figure 8 and 9 electron microscope images are investigated with magnifications of 100x, 2000x, and 5000x.

According to the check samples E5 and E6, salt crystals in samples E4, E2, E3, and E1 are visible.

Check samples E6 and E5; in the case of E4, E2, E3, and E1 salt crystals and damage are clearly observable (Figure 7 B).

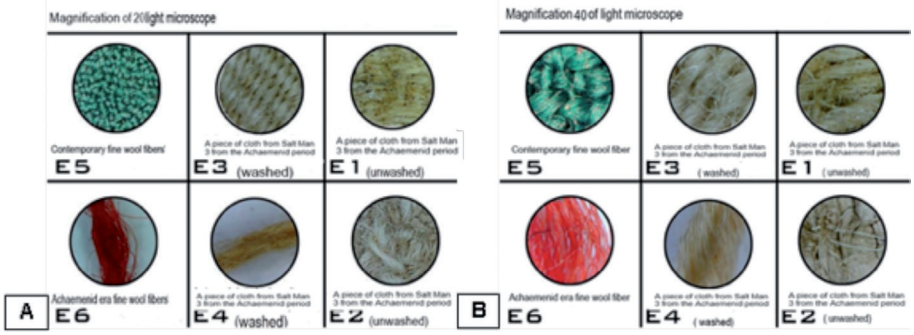

Figure 7. Wool fibers of samples E1-E6. A) Magnification 20× and B) 40× using light microscope. Salt crystals in samples E4, E2, E3, and E1 are visible.

In Figure 8 A, wool fibers can be seen at a magnification of 100× by SEM electron microscope. In Figure 8 B, wool fibers can be seen at a magnification of 2000× by SEM electron microscope. Check samples E6 and E5; in the case of E4, E2, E3, E1, salt crystals and damage are clearly observable. In comparison, we find that sample E3 is better conserved than the other samples of 2800 years. The reason may be due to the washing of the fibers, and weavings and artifacts, after being discovered in the salt bed.

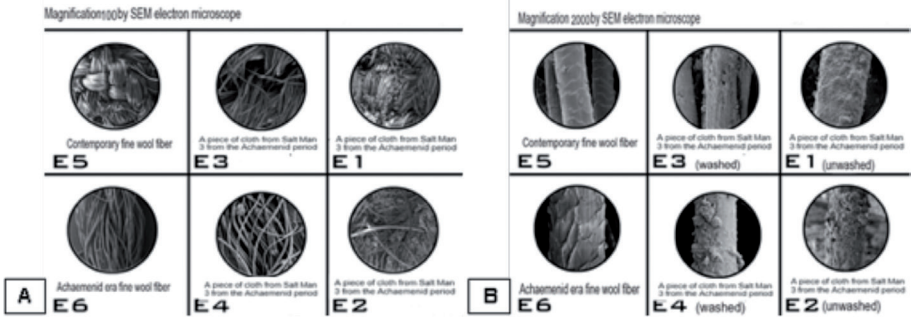

Figure 8. Wool fibers of samples E1-E6. A) Magnification 100× and B) 2000× by SEM electron microscope. Check samples E6 and E5; in the case of E4, E3, E2, E1, salt crystals and damage are clearly observable.

This can also be confirmed in Figure 9 showing a magnification of 5000× by SEM.

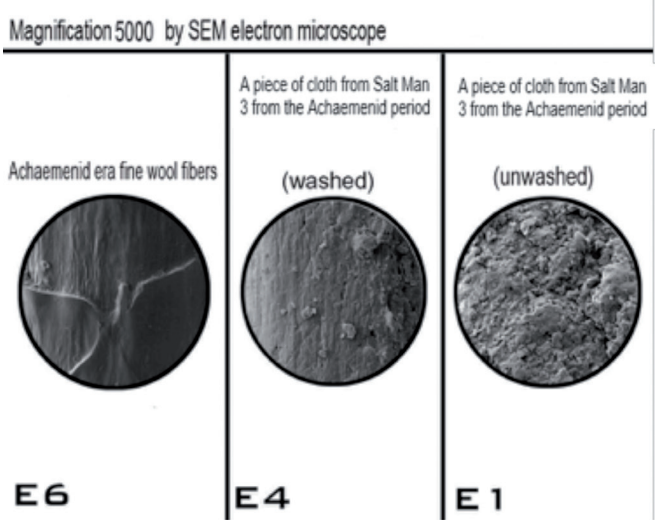

Figure 9. Wool fibers of washed samples E6 Achaemenid era fine wool fibers, E4 washed and E1 unwashed. Magnification 5000× by SEM electron microscope.

8. EDX analysis

Results of the EDX analysis are shown in the following tables and charts. There are no elements of sodium and chloride in sample 3 (Table 1).

Table 1. Elemental analysis of the remains on the fiber material. H. Khamseh, 2016.

Components of sample E1	Compo-nents of sample E3	Compo-nents of sample E2	Components of sample E5	No.
Carbon	Carbon	Carbon	Carbon	1
Oxygen	Oxygen	Oxygen	Oxygen	2
----	----	Sodium	–	2
Magnesium	Magnesium	Magnesium	–	4
Aluminum	Aluminum	Aluminum	–	5
Silicon	Silicon	Silicon	–	6
–	Sulfur	Sulfur	Sulfur	7
–	–	Chloride	–	8
Potassium	Potassium	Potassium	–	9
Calcium	Calcium	Calcium	–	10
–	Iron	Iron	–	11
–	Copper	–	–	12
Gold	Gold	Gold	Gold	13

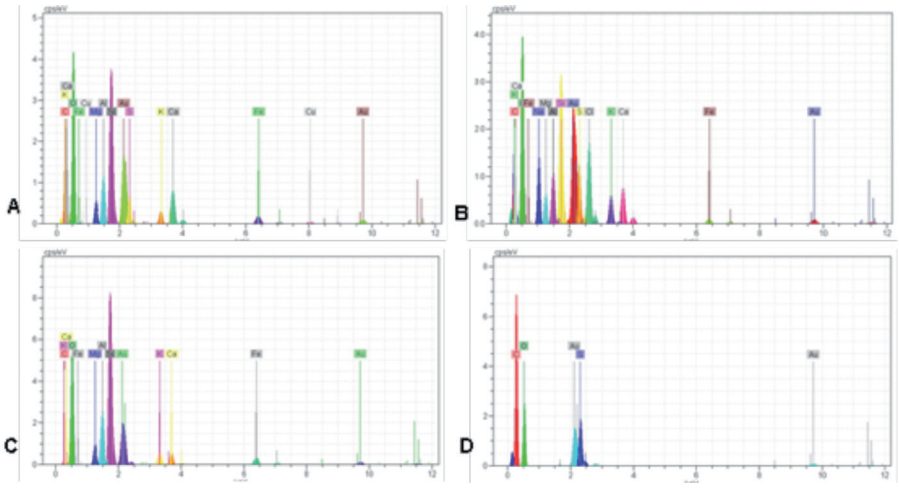

Figure 10. Elemental analysis of E3 (A), sample E2 (B), sample E1 (C), and sample E5 (D) by SEM EDX.

Table 2. Elemental analysis of sample E3.

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	7.56	9.14	16.33
Oxygen	K series	39.24	47.41	63.59
Magnesium	K series	1.34	1.62	1.43
Aluminum	K series	2.67	3.22	2.56
Silicon	K series	8. 18	9.89	7.55
Sulfur	K series	2.11	2.55	1.70
Potassium	K series	1.20	1.45	0.80
Calcium	K series	3.81	4.60	2.46
Iron	K series	3.10	3.74	1.44
Copper	K series	1.26	1.52	0.51
Gold	M series	12.29	14.85	1.62
Total:	82.8%			

Table 3. Elemental analysis of sample E2.

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	3.47	3.92	7.28
Oxygen	K series	42.93	48.54	67.57
Magnesium	K series	1.77	2.00	1.84
Aluminum	K series	4.53	5.13	4.23
Silicon	K series	14.06	15.90	12.61
Potassium	K series	2.20	2.49	1.42
Calcium	K series	2.25	2.55	1.42
Iron	K series	4.47	5.05	2.01
Gold	M series	12.75	14.42	1.63
Total:	88.4 %			

Table 4. Elemental analysis of sample E1.

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	9.98	11.52	21.41
Oxygen	K series	33.20	38.31	53.45
Sodium	K series	3.17	3.66	3.55
Magnesium	K series	1.27	1.47	1.35
Aluminum	K series	2.12	2.44	2.02
Silicon	K series	5.91	6.82	5.42
Sulfur	K series	3.27	3.77	2.63
Chloride	K series	5.07	5.85	3.68
Potassium	K series	2.23	2.58	1.47
Calcium	K series	3.53	4.08	2.27
Iron	K series	1.68	1.93	0.77
Gold	M series	15.23	17.57	1.99
Total:	86.6 %			

Table 5. Elemental analysis of sample E5.

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	49.28	49.28	61.25
Oxygen	K series	38.93	38.93	36.32
Sulfur	K series	3.93	3.93	1.83
Gold	M series	7.87	7.87	0.60
Total:	100.0 %			

9. Results and Discussion

As the salt mountain where the objects were buried is high and steep, water flows quickly down and away from the mountain surface and consequently they have remained in a dry environment, immune to humidity and atmospheric rainfall. As a consequence of this dry environment, salt has played a protective role. In the presence of salt, organic weavings undergo biological changes slowly over time, including both chemical degradation (molecular changes) and biological degradation (degradation by microorganisms). However, when weavings are removed from the place of their archaeological discovery, any salt contained in the fibers rapidly combines with the moisture in the air, creating an ionized environment according to the following formula:

$$NaCl + H_2O \longrightarrow NaOH+HCl \longrightarrow Na^+OH^- + H^+Cl^-$$

These free ions in the environment have a strong affinity with the organic molecules of fibers. Hence, wool fibers in an acid-alkaline environment degrade, resulting in fiber destruction in the hand-woven object. Therefore, to preserve the mentioned fabrics, there are two solutions:

1) Fabrics should not be washed but be kept in a dry and controlled environment. For example, clothing for Salt Man 4 in the smart museum exhibit case, under controlled conditions of temperature and humidity, has remained well conserved.
2) Fabrics should be washed and the salt removed entirely, then kept under controlled conditions of temperature and humidity. As observed, sample E3 washed in the year 2010 and kept in normal conditions, is better conserved than sample E4.

Moreover, these two samples are more well-conserved than the unwashed samples, E1 and E2.

Salt Man 5, who was buried close to the surface of the ground, is more damaged than other samples due to the influence of moisture and ionization (acid-alkaline) in the environment. On the contrary, Salt Man 4 has remained better conserved due to being buried in-depth in the salt mountain and away from moisture.

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