# Assessment the effect of thermal aging on efficiency of Chitosan in consolidation of textiles

**Neven.K.fahim**
Lecture, restoration and conservation department, faculty of archaeology, fayoum university, Egypt.
Nkf00@fayoum.edu.eg

## Abstract
This paper present studying of effect of thermal ageing on chitosan for consolidation the archaeological textile. Many researches focused on applying of chitosan in treatment of archaeological organic materials. But none of them talk about the resistance of chitosan against environmental conditions, such as thermal acceleration. Regarding to the aspect of continuity for any materials used in restoration and conservation process. it should be stable in its first state by the time. Chitosan, which dissolved in water in concentration of 5% and10%, was applied on natural wool samples, then were exposed to accelerated aging by heat at 90 °c for two different periods 48-100 h. A stereo microscope, SEM, color measurement, and physical measurements were performed to evaluate its resistance to heat in the future. Stereo images show gradual changes in the morphology appearance of the wool samples. SEM photos revealed that film existed over the surface of the samples was homogenous, in addition to the significant damage in the chitosan layers after ageing for 90 c for 100 hours. Bad effect of thermal aging at 100 c on Mechanical properties of the treated sample was noticeable, and Color measurements are coming to emphasize the last results, concerning the efficiency of chitosan. The study proved the positive effect of chitosan to consolidate the wool textile, but the usage of that material in treatment of archaeological should be studied by taking into consideration the environmental circumstances, the concentration of the nano gel material, and state of damage.

**Keywords:** Chitosan, Textile, thermal aging, Consolidation

**الملخص**

قدم هذا البحث دراسة تأثير التقادم الحراري على الشيتوزان المستخدم فى تقوية النسيج الأثري. ركزت العديد من الأبحاث على تطبيق مادة الشيتوزان في معالجة المواد العضوية الأثرية. لكن لم يتحدث أي منهم عن مقاومة مادة الشيتوزان للظروف البيئية ، مثل التقادم الحراري. فيما يتعلق بجانب الاستمرارية لأي مادة تستخدم في عملية الترميم والحفظ. يجب أن يكون مستقرًا في حالته الأولى مع مرور الزمن. تم اذابة الشيتوزان في الماء بتركيز 5٪ و 10٪ على عينات من الصوف الطبيعي ، ثم تعريض العينات للتقادم المعجل عند 90 درجة مئوية لفترتين مختلفتين 48-100 ساعة. ثم اجراء فحص بالميكرسكوب البصرى ، SEM ، اجراء القياس اللون ، والقياسات الفيزيائية لتقييم مقاومته للتقادم الحرارى في المستقبل. تُظهر الصور الميكرسكوبية تغييرات تدريجية في المظهر السطحى للعينات الصوفية. كما كشفت صور SEM أن الفيلم الموجود فوق سطح العينات كان متجانسًا ، بالإضافة إلى التلف الكبير في طبقات الشيتوزان بعد التقادم لمدة 90 درجة مئوية لمدة 100 ساعة.حيث كان التأثير

السلبى للتقادم المعجل عند 100 درجة مئوية على الخواص الميكانيكية للعينة المعالجة تـأثرا ملحوظًـا ، وتـأتي قياسـات الألـوان للتأكيد على النتائج الأخيرة ، فيما يتعلق بكفـاءة الشـيتوزان. أثبتـت الدراسـة الأثـر الإيجـابي للشـيتوزان فـي تقويـة المنسـوجات الصوفية، ولكن يجب دراسة استخدام تلك المادة في معالجة الآثار مع مراعاة الظروف البيئية ، وتركيز مادة النانو جل ، وحالـة التلف.

**الكلمات الدالة**: شيتوزان، نسيج ، تقادم حرارى، تقوية.

## Introduction:

Archaeological textiles are considered the most organic material exposing to damage by time, due to their brittleness, weakness, and dryness. [1] So lots of textiles are existed in a bad state of deterioration, and need for consolidation[2]. The aging of archaeological textiles can be defined as the accumulation of all physical and chemical changes with time[3]. These changes are irreversible and usually cause loss of mechanical properties[4], [5]. There are several technical methods used for the detection the changes existed in the physical and chemical properties of dyed fabric, resulting from thermal degradation[6].
Within the usage of new materials that replace traditional ones[7]. The risk was starting from the prediction of the properties of new materials and going to the determination of procedures, methods, and materials[8&9]. Many researchers recommended many adhesive

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and consolidated which, its physical and chemical properties depended, according to chemical nature, construction, textile type, and fiber composition[10].

The consolidated materials used in textile treatment should meet many requirements, such as: It must be flexible and transparent for a while, resistant to natural aging, produce damaging products, in addition to never turn yellow. It should be reversed without causing danger to the textile. It shouldn't cause accelerating aging of the textile material or fading of dyes, and it worth to mention that it shouldn't cause biological deterioration [11& 12].Chitosan is proven to be the most valuable alternative for traditional consolidation agents[13&14]. It is considered the only natural polysaccharide with an amino group, which obtained from the de-acetylation of chitin, which exists in nature [15&16]. The preparation of chitosan passes through four stages from crustacean shells to chitosan: (I) demineralization, (II) de-proteinization, (III) discoloration, and (IV) de-acetylation[17].

Chitosan, one of the components of shellfish such as shrimp[18], is one of the most used biopolymers after cellulose, which is considered a non-toxic, biodegradable-biocompatible material. It has many application advantages compared to traditional ones [19].In addition, varies studies on bactericidal activity of chitosan have been carried out[20].

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And proved that there are correlation between bactericidal activity and chitosan molecular weight [21&22]. Recently, many researches efforts have been done to develop and improving chitosan products for safe and long term uses in conservation and restoration field[23].

## 2. Materials and methods:

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### 2.1 materials:

#### 2.1.1: chitosan:

Chitosan, (was supplied by Sigma-Aldrich, USA) is experimented as a consolidated material for the brittle archeological textile. Liquid chitosan is a non-Newtonian fluid that, when made with high molecular weight chitosan reaches extremely high viscosities and requires specialized equipment to dissolve. Chitosan prepared from crab shell had a significantly higher nitrogen content, degree of deacetylation, solubility, and viscosity and antibacterial activity [24](see table.1).

**Table.1 identification of chitosan**

| materials | source | composition | Degree of de acetylation | viscosity | density | pH |
|---|---|---|---|---|---|---|
| chitosan | Shrimp shells | Chitosan oligosaccharide Solvent | 90% | 300 CP | 0.9 GM/ML | 4± |

#### 2.1.2 Wool fibers:

**Table.2 identification of wool fabric**

| materials | direction | Textile structure | color | Counting threads | | source |
|---|---|---|---|---|---|---|
| | | | | Warp/cm | Weft/cm | |
| Wool fiber | S-direction | Plain weave 1/1 | beige | 5 threads | 6 threads | purchase from tex company, Alex, Egypt |

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### 2.1.3 Dye and mordant:

The yellow dye was of turmeric dye. (Curcuma Demestica L) roots were purchased from Heraz stores for dyes and mordant (El azhar, Egypt), its Curcumin material ($C_{21}H_{20}O_6$) was responsible for coloring, .in addition to The potassium–aluminum sulfate (alum), $Al_2(SO_4)^3 \cdot K_2SO_4 \cdot 12H_2O$, and (99,5%), potassium hydrogen tartrate as a mordant.

## 2.2 methods:

### 2.2.1 Preparing samples:

Woolen fibers samples were prepared in the form of Strips of wool fabric previously in dimension 5x5 $cm^2$. Then they were mordant and dyed by dissolving Alum in little deionized water, and added to dying bath through heating to 70 c for 30 m, then scoured wool strips into bath, maintained at this temperature for another 30 min, then were rinsed with deionized water and allowed to dry.

### 2.2.2 Experiment procedure:

Samples were aged thermally at 90 c for 48-100 h, which is equal to 50- 70 years of thermal aging at room temperature. (Sawoszczuk et al 2008; Kerr et al 1986), according to (The American Society for Testing & Materials), then were treated by 5% and 10% of chitosan dissolved in water by brushes, and were accelerated again for the same period to evaluate its efficiency by the time. Their chemical and physical properties were measured before and after ageing.

### 2.2.3 Visual examination:

Samples were examined visually by stereo microscope (at organic material lab, faculty of archaeology, Fayoum University). Type of Carl Zeiss c-2000 (Germany), which operates in the range 380-1100 nm. Invisible, near-ultraviolet, and near-infrared, using a modified Fuji film S5 DSLR camera.

### 2.2.4 Scanning electron microscope:

Raw, aged, pretreated and treated aged Samples were examined by SEM (at faculty of science, Fayoum university) type of Carl Zeiss Sigma 500 VP.

### 2.2.5mechanical and physical measurement:

Tensile and strain measurement properties were performed by tensile and strain tester (STDF labs at faculty of science, Fayoum university). Type of Zwick Roel, Germany. Weight and dimension were recorded before and after ageing.

### 2.2.6 Color measurement:

Changing in coloring aged samples, and the effect of chitosan on coloring textile were evaluated by using Spector densitometer "Exact X-Rite, Switzerland" (Organic materials labs, faculty of archaeology, Fayoum University), according to Commission Internationale de l’Éclairage (CIE), lab colour system "1976".

## 3. Results:

### 3.1. Visual examinations:

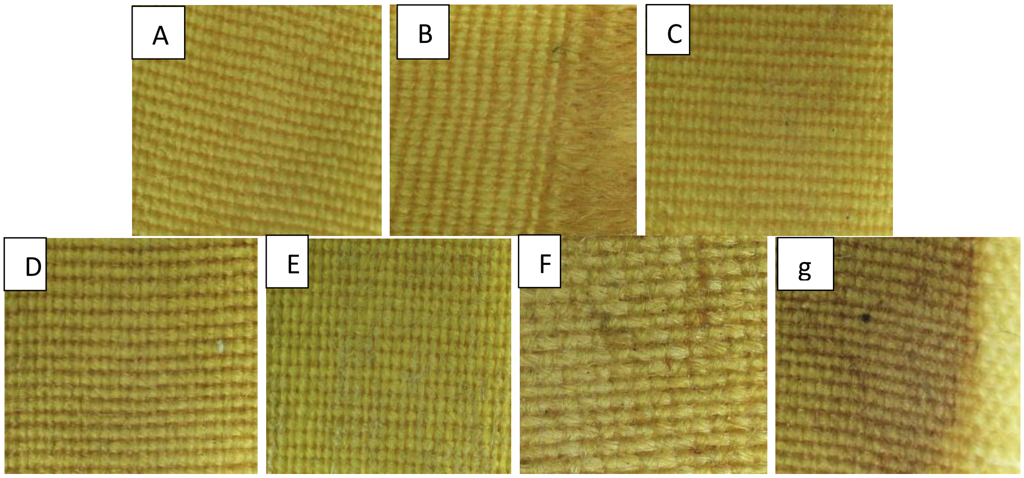


**Figure.1.A show raw woolen sample, 1.b Treated dying wool by 5%, 1.c treated dying wool by 10 %, 1.d aged treated sample 5% at 90 C for 48 h, 1.e Aged treated sample 10% at 90 C for 100 h, 1.f Aged treated sample 5% at 90 C for 48 h, 1.g Aged treated sample 10% at 90 C for 100 h**

### 3.2 Scanning electron microscope:

Photos of SEM prove several changes existed in the morphological appearance of the dyed samples through the stages of the experiment, The photos of treated samples are coming to emphasize the homogeneity , the uniform distribution of fibers, and the alignment in a common direction of the chitosan layers, as shown in fig.2.b,c. Then, the disfiguration of dyed standard sample after accelerated aging at 90 C for 48 h was noticeable, shown in fig.2.d,e. Furthermore, the increased stiffness significantly and disappearing of a part of chitosan layers were existed on the surface of aged dyed samples after ageing for 100 h, as shown in fig 2. f.

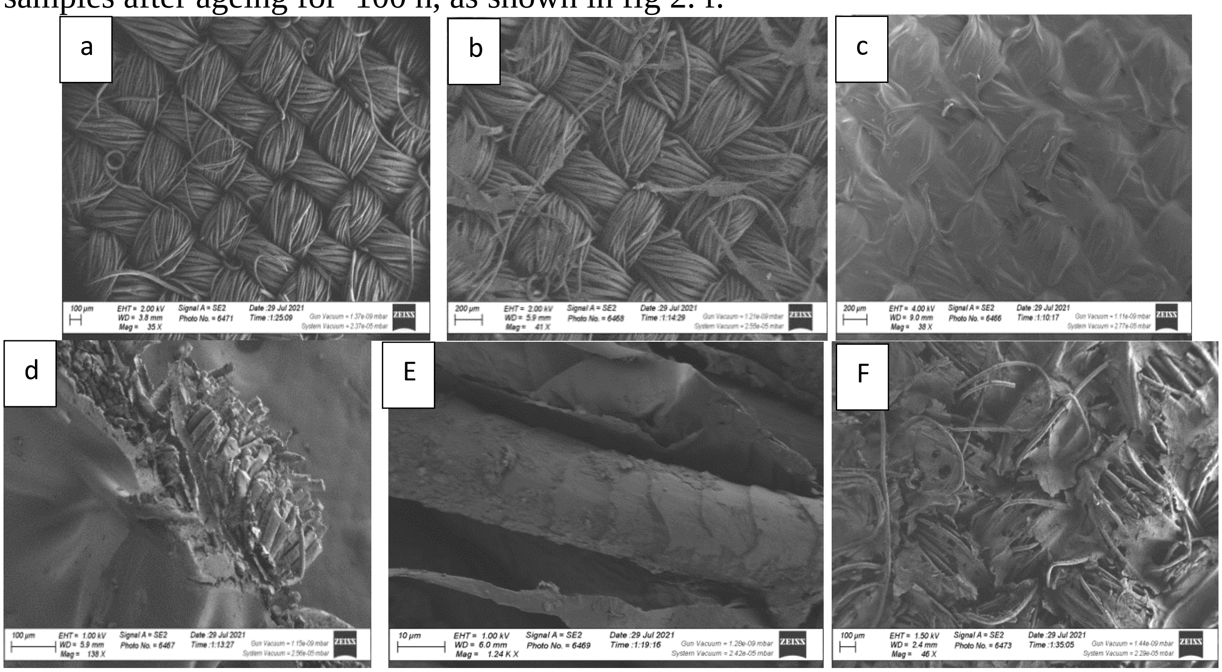


**Figure 2 show the effect of thermal ageing on the efficiency of chitosan in improving the morphological properties of the surface.**

### 3.3 mechanical and physical properties:

Table.3 the mechanical and physical properties of the four samples

| Sample | | Tensile strength (Mpa) | Strain strength (%) | weight (gm/cm3) | Volumetric measurement (cm3) |
|---|---|---|---|---|---|
| Raw material | standard | 14.5 | 29.4 | 21 | 3.75 |
| Control without treatment after the aging to compare the effect of chitosan | Aged for 90 ° 48 h | 12.05 | 27.3 | 20 | 3.60 |
| | Aged for 90 ° 100 h | 10.2 | 22.01 | 18 | 3.18 |
| Treated by chitosan | 5% | 27.4 | 16.2 | 28 | 6.25 |
| | 10% | 29.6 | 20.3 | 35 | 8.75 |
| After thermal aging for 48h | 5% | 23.3 | 13.4 | 25 | 4.416 |
| | 10% | 27.5 | 12.4 | 32 | 2.478 |
| After thermal aging for 100 h | 5% | 22.4 | 7.5 | 21 | 2.09 |
| | 10% | 18.5 | 8.9 | 26 | 1.802 |

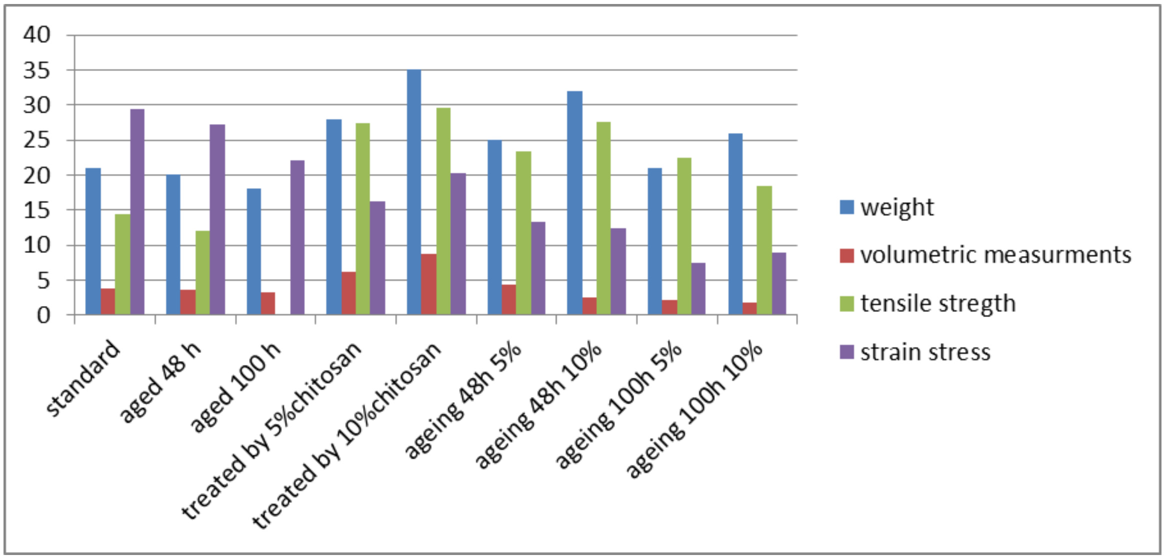


**Figure 3 physical measurement shows the bad effect of thermal ageing on the efficiency of chitosan**

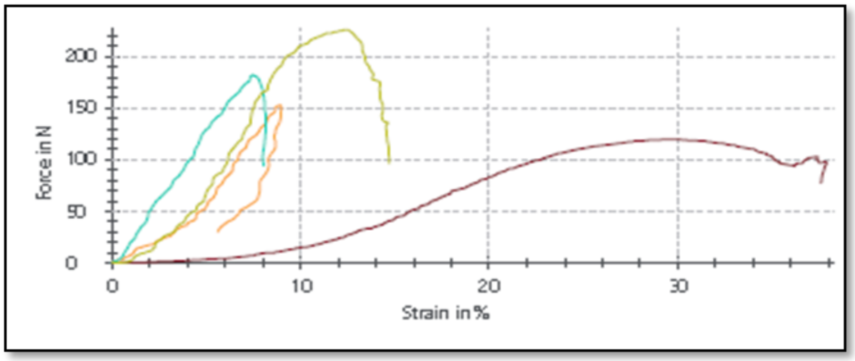


**Figure 4 the measurement of tensile and strain strength of the dyed samples**

### 3.4 color measurement:

Results of color measurements of treated samples show the increasing of "L" readings, as an indicator for darkness of color after treatment. And decreased gradually again after aging of treated samples for 48 h, 100h. The values of "a" and "b" differentiated from standard sample to treated and aged ones. These results revealed the less yellowish color of the treated sample by 10% of chitosan, especially after aging. And keeping the original color for the samples, treated by 5% of chitosan even after thermal aging. (See table.4).

Table.4 color measurements of the samples

| no of sample | kind of sample | L | a | b |
|---|---|---|---|---|
| Raw wool | standard | 59.45 | 18.61 | 57.96 |
| Control without treatment after the aging to compare the effect of chitosan | Aged for 90 ° 48 h | 69.03 | 14.86 | 55.54 |
| | Aged for 90 ° 100 h | 70.30 | 11.72 | 49.02 |
| Treated by chitosan | Treated 5 % | 64.16 | 10.90 | 59.56 |
| | Treated 10 % | 72.30 | 11.72 | 62.10 |
| After thermal aging for 48h | Treated 5 % | 61.63 | 10.3 | 49.54 |
| | Treated 10 % | 45.13 | 10.20 | 24.61 |
| After thermal aging for 100h | Treated 5 % | 56.90 | 16.6 | 44.19 |
| | Treated 10 % | 39.23 | 10.17 | 21.10 |

### Discussion:

The paper proved the bad effect of thermal acceleration on the efficiency of chitosan as a consolidated material of archaeological textiles[25&26]. The changes existed in the surface

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morphology of the samples were indicated to the resistance of the chitosan against thermal acceleration. Although the color of the woolen samples did not change after treatment. It found darkness after accelerating aging, so it is recommended to be used in non-coloring textiles, and on the hidden side of the decorated archaeological textiles.[27]. Physical measurements revealed the improvement happened after applying of 10 % of chitosan on wool fabric[28&29] . But after accelerating ageing for 48, 100 h at 90 c the readings decreased gradually due to rearrangement of polymer chains during ageing time caused changes of flexibility, tensile strength [30&31.] Results of SEM are coming to show the highly homogenous layer[32], formed by chitosan on the surface of woolen fibers. However, separations appeared in this covering layer after thermal aging for 100 h, which led to the disappearance of parts of the homogeneous layer after aging (see fig1.f).[33]. Reinforcement, achieved by chitosan, was clarified by values of tensile strength, as the consolidated material improve the physical properties of the aged samples[34], in addition to the little increasing in weight after treatment, which doesn't present any risk on the brittleness textiles. Furthermore, chitosan helps in getting back the original volumetric shape of the aged fibers (see table.3), but after ageing the reading become more closing to the original sample, which refer to the disappearance of parts of chitosan layer.
Color measurement proved that no noticeable changes existed for the treated samples and aged treated samples (see table.4)[35&36], which indicated the sustainability of this material in the conservation of archaeological dyed textiles. L value after ageing for 100h proved

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the darkness existed on the chitosan layer applied on woolen samples. It is noticed that the treated woolen samples have limited resistance to the accelerating aging.

**Conclusion:**

Archaeological textiles always are in bad need of conservation and consolidation, due to the brittle nature of their raw materials, and the traditional materials used in reinforcement didn't meet all the requirements and have many disadvantages, which affected the fibers and dyes in the future. The paper evaluated the resistance of new materials to consolidate the surface, chitosan has many advantages, such as strength the aged fibers, returns their original volume, improves their morphological appearance, and doesn't change their color and dyes. But after thermal ageing, Chitosan brings limited resistance to thermal degradation, by which the results of SEM, mechanical measurement, and color measurement proved.

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