DETERMINATION OF HYDROTHERMAL STABILITY OF CONSOLIDATED PARCHMENT AND LEATHERS

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Introduction

Since pre-historic times people have known have to make a material that we know as leather. In Egypt, vegetable tanned leather was used since the pre-historic period, but the use of parchment dates back to the Middle Kingdom[(1)]. We may consider that since 1884, with the work of Augustus Schultz, thec hrome tanning process began[(2)]. There are a large number of important parchment manuscripts and tanned leathers (vegetable and chrome tanned leather) in archaeological sites (museums, storages, old libraries and excavation areas). From the moment of its creation, parchment or leather starts to age. Ageing as such is a process resulting from damage brought about as a consequence of production technology, environmental conditions, handling and repair[(3)]. Degradation by heat is probably one of the major factors in the deterioration of a large portion of parchment and leather in museum collections[(4)]. There is no doubt that

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1. Reed, R., Ancients skins, Parchments and leathers, Seminar Press, London, 1972, p. 48.
2. Thomson, R. S., Leather manufacture in the post medieval period withspecial references to Northamptonshire, Post Medieval Arch., London, 1981, p.161.
3. Larsen, R., Micro methods for the analysis of parchment – MAP – preliminary report, in care and conservation of manuscripts 5, Fellows – JENSEN, Gillian and Springborg, the Royal Library, 2000, pp. 28-37.
4. Williams, R.S., Surface encrustation on caribon skin coat, Canadian Conservation Institute, Ottawa, Canada, 1982, pp.1-14.
5. Abdel-Maksoud, G. and Marcinkowska, E., Changes in some properties of aged and historical parchment, Restaurator, Vol. 21, Munich, 2000, p.138.

the first stage of any conservation process should always be a detailed study of the condition of the leather with an understanding of the deterioration process in order to present some solutions for the conservation treatment. Most leather artifacts and parchment manuscripts in Egypt usually suffer because of unsuitable environmental conditions[(5)]. A widly used parameter for measuring the condition of parchment and leather is the shrinkage temperature, representing the temperature of which leather has to be heated in an aqueous environment in order to lose its characteristic tridimensional fibre network construction. The loss of this structure causes the leather or parchment to shrink[(6)].

Heat ageing affected on parchment and leather. This is confirmed by the fact that when one moves a fingernail with not too much pressure over the surface of the leather or parchment, it falls apart as powder. Another indication is the colour of parchment and leather. It changes to be dark in a lot of cases. Leather or parchment in such a state of decay needs a thorough consolidation before it is sufficiently strong[(7)]. The term consolidation is often used to indicate a kind of treatment that will consent the archaeological object to newly a quire mechanical, chemical

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6. Larsen, R. Experiments and observation in the study of environmental impact on historical vegetable tanned leathers, Thermochimica Acta, 2000, p.365.
7. Halleback, P. B., Impregnation of fragile leather objects, In: 6th International Restorer Seminar (restoration of leather and wood training restorers), the National Centre of Museums, Budapest, Hungary, 1987, p. 197.
8. Matteini, M., Montalbanol, L., Rizzi, M., Scarzanella, C. and Schonhaut, G., Experimental testing of different kinds of fixatives used for the consolidation of painted parchment, International Conference on conservation and restoration of archive and library materials, Istituto Centrale per la Patologia del Libro, Roma, April 22nd – 29th , 1996, p.535.

and physical stability. In most cases different and specific treatments and materials are required in order to achieve this objective(8).

This paper aims to:

1. Prepare artificially heat aged parchment and tanned leathers at different temperature to follow the changes in the hydrothermal stability. The changes are compared with samples of selected archaeological parchment manuscripts, vegetable and chrome tanned leather.
2. Refer to the state and reflect the conditions of various parchment manuscripts and leather artifacts at archaeological sites in Egypt.
3. Evaluate selected polymers that will improve the hydrothermal stability in order to choose the best one for the treatment of parchment manuscripts and tanned leathers in museum collections and other sites.

Materials and methods

New samples

Modern parchment: calf sin prepared by the author in accordance with Haines (1999)[(9)] and Abdel-Maksoud (1999)[(10)].

Vegetable tanned leather: Vegetable tanned calf skin (by mimosa), prepared by the author in accordance with Thomson (1991)[(11)], Bickley (1991)[(12)], Haines (1991)[(13)] and Abdel-Maksoud (1999)[(14)].

Chrome tanned leather: Chrome tanned calf skin prepared by the author in accordance with Bienkiewicz (1983)[(15)]

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9. Haines, B.M., The physical and chemical characteristics of parchment and the materials used in its conservation, the Leather Conservation Centre, University college Campus, Northampton, 1999, pp. 7-12.
10. Abdel-Maksoud, G., Preparation of parchment samples for experimental studies in conservation, Journal of Cracow University of Technology, 1999, 1A, p.154.
11. Thomson, R.S., A history of leather processing from the medieval to the present time, In: Leather its composition and changes with time, Great Britain, 1991, pp.12-15.
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15. Bienkiewicz, K. J., Physical chemistry of leather making, Robert E, Krieger Publishing Co. Florida, 1983, pp. 220-236.

Archaeological samples

Tanta Museum: three parchment manuscripts in Hebrew, presenting the Pentateuch of the old testment 18th, 19th A.D. Jewish Cemetary. Tanta City, Egypt.

The Library of the Medical Faculty of Cairo University: Most possessions of the Library of the Faculty of Medicine date back to the time of Klout Pek (19th century) and the time of Ali Basha Ibrahium (20th century). The samples were taken from vegetable tanned leather (the bookbindings of an Anatomy Atlas) and the parchment certificates.

The Rasheed Excavation: the sample of vegetable tanned leather was taken from an unknown bookbinding.

The El-Gharawy Family Group: the samples of chrome tanned leather were taken from three bookbindings dating back to 20th century.

Private bookbindings: the samples were taken from three bookbindings (date back to 18th century) of vegetable tanned leather and three bookbindings (dating back to the 12th century) of chrome tanned leather.

Accelerated ageing

Heat treatment was used in the accelerated dry ageing process (Larsen 1987(16) and Chahine 1995(17)) at 80 °C, 100 °C and 120 °C. The time of ageing was between one week and four weeks (the first accelerated ageing). On the bases of the results obtained, heat treatment for two weeks at 100 °C was chosen to prepare aged samples (parchment and leather) to be used for the conservation treatment (consolidation process). Heat treatment for one week at

16. Larsen, R., Similarities and differences in the amino acid composition of new, historical and aged leather, In: 6th International Restorer Seminar (restoration of leather and wood training restorers), the National Centre of Museums, Budapest, Hungary, 1987, pp. 205-210.
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100 °C was used after the samples treated with selected polymers (the second accelerated ageing).

Selected polymers used for the conservation treatment

Four polymers (Table 1) were examined to test their effectiveness in preventing deterioration. The concentrations used are 1%, 3% and 5%. The latter concentrations are normally applied in the conservation use. Impregnation method was used until the samples were fully saturated. After consolidation process, the samples were allowed to air dry naturally. Before the application of polymer by the impregnation method, the slide of the samples was made clean, free from dirt and dust or, indeed anything that could inhibit the penetration of the polymers.

Table. 1. Polymers used

Chemical name	Trade name	Producer	Solubility
Soluble nylon	Calaton CA	Imperial Chemical industrials (ICI)	Ethyl alcohol
Vinyl acetate / acrylic ester copolymer	Mowilith DM5	Hoechst	Distilled water
Ethyl acrylate / methylacrylate	Plextol B.500	Lascan Restauro	Distilled water
Ethyl acrylate /methylacrylate	Paraloid B.72	Rohm and Hass	Acetone

Preparation for shrinkage temperature measurement

Haines (1987)[18] noted that shrinkage temperature should be measured under controlled conditions which, include: "leather samples need to be thoroughly wet in distilled water…. if the

18. Haines, B.M., Shrinkage temperature in collagen fibres, In: Leather Conservation News, Vol.3, No. 2, 1987, pp. 1-5.

sample skin is untanned, it needs to be brought to a neutral rate". So, the collagen fibres were thoroughly saturated before measurement in several changes of distilled water. It was also felt that the changes of water would help to lessen any contamination due to pH or salts. The samples were heated at a rate of three degrees Celsius per minute.

Measurement of the shrinkage temperature

The procedure for measuring the shrinkage temperature using the hot stage followed that outlined by Young (1990)[19]. Once cut the fibres in the glass depression slides were degreased in acetone for five minute. While still in the glass depression slides, the fibres were then transferred using steel tweezers to a second bath of distilled water for a minimum of forty-five minutes to allow the fibres to fully saturate. Afterwards, the fibres were moved to a microscope slide prepared with a small reservoir of distilled water with glycerine which kept the fibres continually hydrated and unrestrained throughout the heating cycle. Each transfer, from acetone to water and water to slide, was performed quickly to ensure that no drying of the fibres occurred, as this would falsely lower the shrinkage temperature. The slide containing the fibres was inserted into the hot stage, which was placed onto the stage of polarizing microscope. The shrinkage process was observed at 100 – time magnification using a polarizing light microscope.

19. Young, G.S., Microscopical hydrothermal stability measurement of skin and semi-tanned leather, In: ICOM Committee for Conservation, Preprints of the 9th Triennial Meeting, Dresden, 1990, pp. 626-631.

Results and discussion

One property of collagen fibres is that exhibit a sudden shrinkage in length when heated. Characteristic shrinkage temperature are: 60 °C – 65 °C for limed collagen, 75 °C – 85 °C for vegetable tanned leather and 95 °C – 105 °C for chrome tanned leather.

Archaeological and heat aged samples

All results have revealed that the archaeological and heat aged samples suffered from degradation.

Parchment

The percentage loss of the shrinkage temperature of archaeological samples (Table 2) by comparison with the new parchment (before ageing) was as follow:

Parchment certificate No. 1: 23%, Parchment certificate No. 2: 27%, Parchment certificate No. 3: 32%, Parchment manuscript No.1: 34%, Parchment manuscript No.2: 39% and Parchment manuscript No.3: 40%.

Table 2. Shrinkage temperature of Archaeological parchment

Archaeological parchment		Shrinkage temperature (°C)
The Library of the Faculty of Medicine	Parchment certificate No.1	48
	Parchment certificate No.2	45
	Parchment certificate No.3	42
Tanta Museum	Parchment manuscript No.1	41
	Parchment manuscript No.2	38
	Parchment manuscript No.3	37

The aged parchment have also shown a reduction in the shrinkage temperature (Fig. 1). The percentage loss of aged parchment at different temperatures was as follow:

	80 °C	100 °C	120 °C
1 week	2%	15%	19%
2 weeks	3%	27%	40%
3 weeks	6%	35%	48%
4 weeks	10%	44%	52%

It was clear from the results that the percentage loss in the shrinkage temperature of aged sample at 100 °C after 2 and 3 weeks was closed to the percentage loss of archaeological sample (parchment certificate No. 2 and No.3). The percentage loss of the shrinkage temperature of aged sample at 120 °C after 2 weeks is higher than the percentage loss of the shrinkage temperature of archaeological samples studied. It was also noticed that the percentage loss of aged samples at 80 °C was too low during the time of ageing.

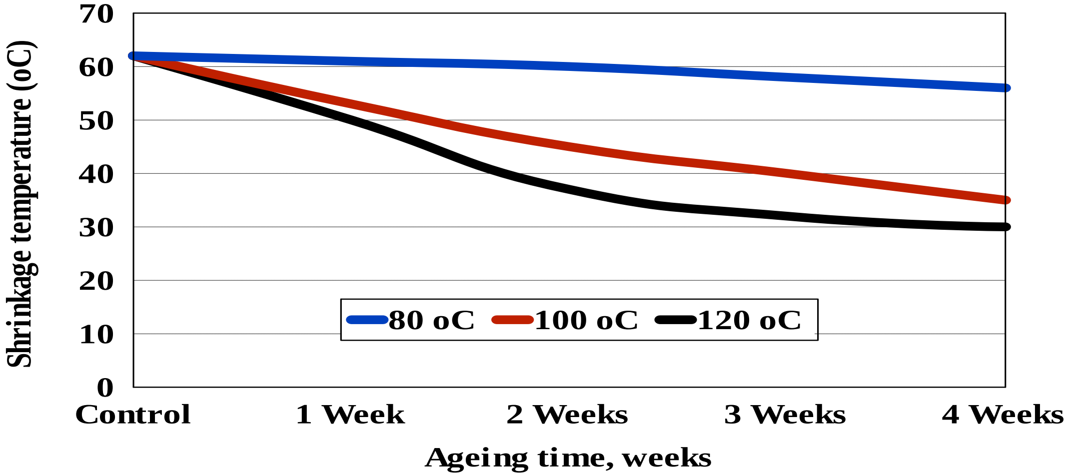

Fig 1. Shrinkage temperature of aged parchment at different temperatures

Vegetable tanned leather

The reduction in the shrinkage temperature of archaeological vegetable tanned leather (Table 3) was compared with the new sample. The percentage loss was as follow: Anatomy Atlas No. 1: 29%, Anatomy Atlas No. 2: 24%, Anatomy Atlas No.3: 25%, unknown bookbinding (Rasheed Excavation): 35%, Private bookbinding No.1: 19%, private bookbinding No. 2: 20% and Private bookbinding No. 3: 21%. The shrinkage temperature of aged vegetable tanned leather was also reduced (Fig. 2). The percentage loss of the shrinkage temperature of vegetable tanned leather at different temperatures was as follow:

Table. 3. Shrinkage temperature of archaeological vegetable tanned leather

Archaeological vegetable tanned leather		Shrinkage temperature (°C)
The Library of the Faculty of Medicine	Anatomy Atlas No.1	60
	Anatomy Atlas No.2	65
	Anatomy Atlas No.3	64
Rasheed Excavation	Unknown bookbinding	55
Private group	Bookbinding No.1	69
	Bookbinding No.2	68
	Bookbinding No.3	67

	80 °C	100 °C	120 °C
1 week	1%	13%	18%
2 weeks	4%	20%	27%
3 weeks	6%	27%	38%
4 weeks	9%	34%	44%

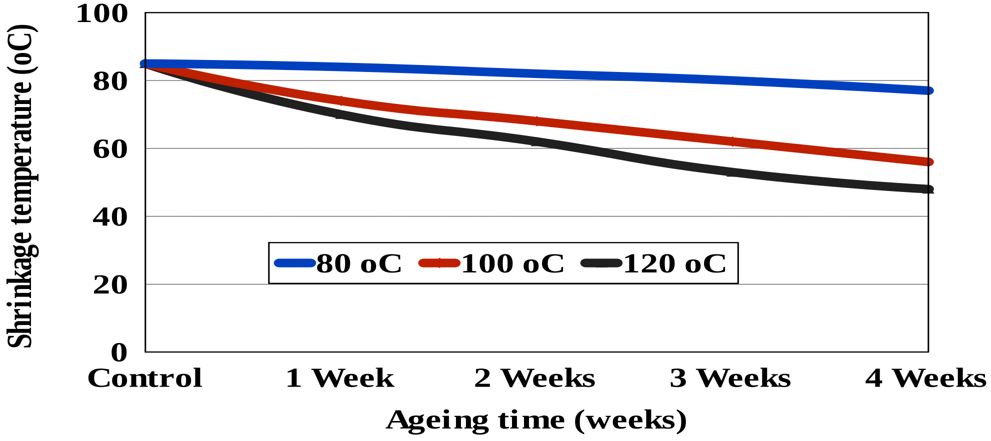

Fig.2. Shrinkage temperature of aged vegetable tanned leather at different temperatures

The results of archaeological and aged samples have shown that the percentage loss of the shrinkage temperature of vegetable tanned leather is lower than the percentage loss of parchment. The reduction of aged samples after 2 and 3 weeks at 100 °C is closed to the most of archaeological samples. The ageing at 120 °C also was too high especially after 3 weeks. The Ageing at 80 °C is too low.

Chrome tanned leather

The shrinkage temperature of historical chrome tanned leather reduced (Table 4). The percentage loss in the shrinkage temperature of archaeological chrome tanned leather in comparison with new sample was as follow: Private bookbinding No.1: 22%, private bookbinding No.2: 20%, private bookbinding No.3: 23%, bookbinding No.1: 18%, bookbinding No.2: 19 and bookbinding No.3: 17%. It was clear from the data obtained that the percentage loss of historical chrome tanned leather was lower than the percentage loss of both parchment and vegetable tanned leather.

Table. 4. Shrinkage temperature of historical chrome tanned leather

Archaeological vegetable tanned leather		**Shrinkage temperature (°C)**
Private group	Bookbinding No.1	76
	Bookbinding No.2	78
	Bookbinding No.3	75
Refaa El-Tahtawy Library	Bookbinding No.1	80
	Bookbinding No.2	79
	Bookbinding No.3	81

The shrinkage temperature of aged chrome tanned leather also reduced (Fig. 3). The percentage loss of aged chrome tanned leather was as follow:

	80 °C	100 °C	120 °C
1 week	1%	10%	14%
2 weeks	3%	18%	21%
3 weeks	5%	24%	30%
4 weeks	7%	29%	38%

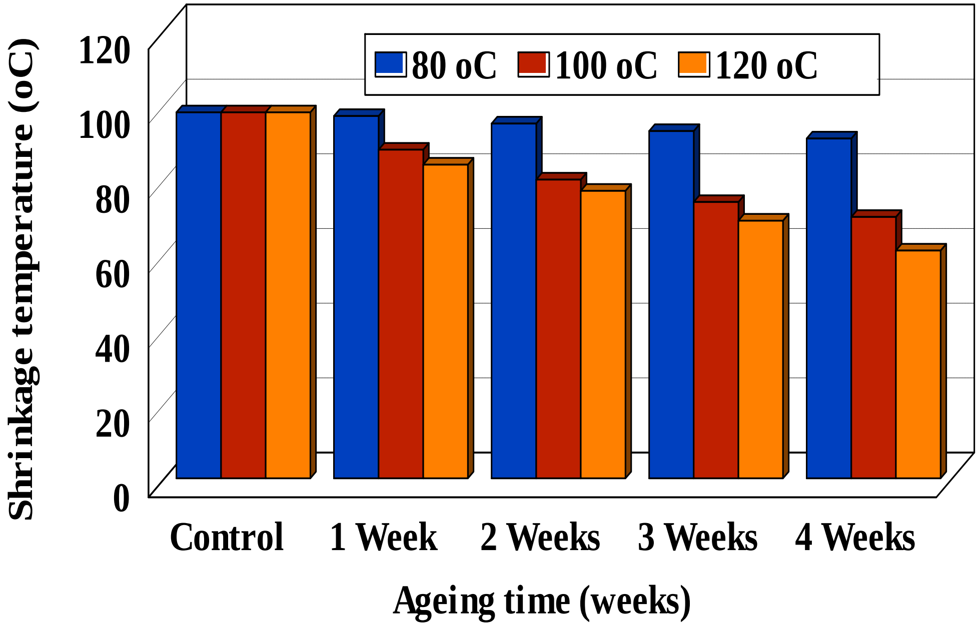

Fig.3. Shrinkage temperature of aged chrome tanned leather at different temperatures

The results proved that chrome tanned leather tolerates heat ageing more than parchment and vegetable tanned leather. It can be also said that the heat ageing at 100 °C for 2 and 3 weeks is closed to all historical samples.

Measurement of the shrinkage temperature of heat aged samples and archaeological samples show that there is a clear connection between the shrinkage temperature and the chemical changes in the studied samples ,which is due to the deterioration. In this study, there are large chemical changes occurred in the main constituents of the leather (the collagen and the tannin structure) so the shrinkage temperature was low and in some cases especially for archaeological samples the shrinkage temperature had been observed to take place at low temperature. From the physical point of view, the most important physical characteristics of collagen are the way in which fibres swell in solutions and the way in which they shrink at a very definite temperature. When collagen is denatured (its helical structures though not necessarily its components ,is destroyed) it froms gelatin, a familiar substance unusuall among protins in that it has no internal orderd stucture.

To draw explanation for the shrinkage temperature process, Southward Southward (1993)[20] explained this process as follows: collagen stability is largely dependent on the highly ordered, semi-crystalline structure of the fibril which is stabilized predominantly by intramolecular hydrogen bonds between peptides of the triple helical collagen molecule. Loss of stability can occur in the following manner:

“When a collagen sample is in aqueous medium, water easily penetrates the collagen molecule through its affinity for the hydrogen bonding sites in the amorphous areas. The presence of water increases the spacing between the collagen’s chains. Macroscopically, this effect is seen as swelling of the sample in question. If the sample is then heated at a certain temperature, the thermal energy will become sufficient to break the hydrogen bonds in the amorphous areas holding the polypeptide chains together, each individual chain then folds into random coils, and the hydrogen macromolecule loses its ordered structure and denatures

20 Southward, J.A., The effects of photodegradation on collagen: an analysis using the shrinkage temperature method, Master of Art Conservation, Queen’s University, Kingston, Ontario, Canada, 1993, p.16-17.

collagen, which has denatured has lost its partially crystalline nature, as can be evidenced under polarized light by a loss of birefringence".

On the basis of the study, it can be noted that the physical disintegration of artificially and naturally aged leather is caused by competitive and interactive oxidative breakdown of both collagen and tannin structure. The cause of the oxidative breakdown is mainly due to environmental factors like heat for artificial aged leather, and several combination factors (heat, light and etc.).

It was noted for the archaeological samples that there is variation in the shrinkage temperature between the samples. this is due to two different main factors which are as follows:

1. The effectiveness of the first conservation treatment (temporary treatment after excavation).
2. The conditions in which is existed before its arrival in the museum collection (after discovered from excavation area).

On the other hand, some regions are apparently less hydrothermally stable than others because of the pertubation or deterioration of crystalline and ordered amorphous structure. The less stable regions begin shrinking at lower temperatures. Whereas the more stable regions still denature at higher temperatures approaching more normal shrinkage temperature values. A similar description applies for fibres. The total of fibril deterioration in each fibre is variable, and therefore, some fibres begin shrinking at lower temperatures than do others.

Larsen et al (1994)[21] have shown the cause of loss in the hydrothermal stability of the old historical samples is due to several

21. Larsen, R., Vest, M and Nielsen, K., Determination of hydrothermal stability (shrinkage temperature), STEP Leather Project (European Commission, Decorative-general for Science, Research and development), The Royal Danish Academy of Fine Arts, School of Conservation, Denmark, 1994, pp.151-160.

combinations of the two main breakdown mechanisms. In general this is:

1. Strong oxidation and strong acid hydrolysis.
2. Strong oxidation and less hydrolysis.
3. Strong acid hydrolysis and less oxidation.

Larsen et al (1997)[22] reported that the differences in the hydrothermal stability of the fibres in sample may be ascribed to three main factors:

1. Difference in stability due to initial treatment of the skin.
2. Non-uniformity in tannage.
3. A non-uniform degree of deterioration.

Many authors have confirmed these results. Young (1990)[23] reported that when the structure of collagen is damaged, by oxidative breakdown, there appear to be fewer and weaker hydrogen bonds maintaining the structure. Under these circumstances, less thermal energy is needed to repture the remaining hydrogen bonds and, therefore fibre shrinkage occurs at a lower temperature. Larsen et al (1997)[24] proved that the shrinkage temperature decreased with an increase in the time of ageing.

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22. Larsen, R., Vest, M., Poulsen, D. V. and Kejser, B., Determination of hydrothermal stability by the micro hot table method, Environmental Leather Project (Deterioration and conservation of vegetable tanned leather, Denmark, 1997, pp. 145-158.
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Consolidated samples

There is no doubt that the polymers used improved the hydrothermal stability of parchment and leathers. The improvement varied with polymers and concentrations used. For parchment, there is an improvement in the shrinkage temperature of consolidated parchment (Table 5). The increasing of the percentage of the shrinkage temperature of consolidated parchment compared with the aged sample after two weeks without consolidation was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	2%	6%	10%
Paraloid B72	4%	8%	12%
Plextol B.500	2%	4%	6%
Mowilith DM5	0%	2%	4%

It was noted by comparison of consolidated parchment after ageing for one week with the aged sample after three weeks without consolidation that the increase of the percentage of the shrinkage temperature was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	2%	5%	7%
Paraloid B72	7%	11%	11%
Plextol B.500	2%	5%	7%
Mowilith DM5	-5%	0%	0%

The shrinkage temperature of consolidated vegetable tanned leather increased (Table 6). The increasing of the percentage of the shrinkage temperature of consolidated vegetable tanned leather compared with the aged sample after two weeks without consolidation was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	3%	6%	7%
Paraloid B72	3%	7%	11%

Plextol B.500	1%	3%	4%
Mowilith DM5	1%	4%	6%

Table 5. Shrinkage temperature of Parchment treatedd with polymers

Polymers	Concentrations	Aged sample (2 weeks)	Sample after consolidation	Consolidated sample after ageing	Aged sample (3weeks)
Calaton CA	1	45	46	41	40
	3	45	48	42	40
	5	45	50	43	40
Paraloid B 72	1	45	47	43	40
	3	45	49	45	40
	5	45	51	45	40
Plextol B. 500	1	45	46	41	40
	3	45	47	42	40
	5	45	48	43	40
Mowilith DM5	1	45	45	38	40
	3	45	46	40	40
	5	45	47	40	40

It was noted by comparison of consolidated vegetable tanned leather after ageing for one week with the aged sample after three weeks without consolidation that the increase of the percentage of the shrinkage temperature was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	6%	9%	10%
Paraloid B72	7%	10%	9%
Plextol B.500	0%	3%	5%

Mowilith DM5	2%	5%	6%

Table 6. Shrinkage temperature of vegetable tanned leather treated with polymers

Polymers	Concentrations	Aged sample (2 weeks)	Sample after consolidation	Consolidated sample after ageing	Aged sample (3weeks)
Calaton CA	1	68	70	66	62
	3	68	72	68	62
	5	68	73	69	62
Paraloid B 72	1	68	70	67	62
	3	68	73	69	62
	5	68	76	68	62
Plextol B. 500	1	68	69	62	62
	3	68	70	64	62
	5	68	71	65	62
Mowilith DM5	1	68	69	63	62
	3	68	71	65	62
	5	68	72	66	62

The consolidation process improved the shrinkage temperature of chrome tanned leather (Table 7). The percentage of increasing the shrinkage temperature of consolidated chrome tanned leather by comparison with aged sample after two weeks without consolidation was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	1%	4%	7%

Paraloid B72	4%	6%	9%
Plextol B.500	1%	2%	5%
Mowilith DM5	2%	5%	7%

It was noted by comparison of consolidated chrome tanned leather after ageing for one week with the aged sample after three weeks without consolidation that the increase of the percentage of the shrinkage temperature was as follows:

	First concentration	Second concentration	Third concentration
Calaton CA	5%	6%	8%
Paraloid B72	8%	10%	9%
Plextol B.500	-3%	-1%	1%
Mowilith DM5	3%	4%	6%

Table 7. Shrinkage temperature of chrome tanned leather treated with polymers

Polymers	Concentrations	Aged sample (2 weeks)	Sample after consolidation	Consolidated sample after ageing	Aged sample (3weeks)
Calaton CA	1	80	81	78	74
	3	80	83	79	74
	5	80	86	80	74
Paraloid B 72	1	80	83	80	74
	3	80	85	82	74
	5	80	88	81	74
Plextol B. 500	1	80	81	72	74
	3	80	82	73	74
	5	80	84	74	74
M ow ilit -	1	80	82	76	74
	3	80	84	77	74

	5	80	86	79	74

It was clear from the results obtained that the consolidation process is very necessary for parchment and leathers, since it make parchment and leather more resistant to ageing.

Conclusions

1. The hydrothermal stability of historical parchment and leathers was low and indicates that most of archaeological sites need to make a plan for monitoring the condition of parchment manuscripts and leather artifacts.
2. The hydrothermal stability of aged leather and parchment reduced as the time of ageing anf temperature increased.
3. Chrome tanned leather tolerates the heat ageing more than vegetable tanned leather and parchment, which is considered the less resistance to heat ageing.
4. The shrinkage temperature of accelerated heat aged parchment and tanned leather was closed to the shrinkage temperature of historical samples taken from parchment and leather. Accelerated heat ageing method proved good in preparation of aged samples for experimental study on the consolidation materials. For preparation of aged parchment and tanned leather, the accelerated heat ageing at 80 °C was found low anf at 120 °C too high. The optimum temperature was 100 °C and the time of ageing of 2 weeks was found sufficient.
5. The measurement of hydrothermal stability by the microscopic method has advantage in that only minute samples of collagen fibres are required as only 0.1 to 0.5 mg. So, the measurement of the shrinkage temperature by polarizing microscope with a hot table technique is considered as a non-destructive method.

6. There are an improvement in the shrinkage temperature of parchment and tanned leathers treated with most polymers studied at different concentrations. It can be said that the shrinkage temperature of the treated samples with polymers reduced after ageing but it was better than the aged sample after three weeks without treatment. This indicates that consolidation process protect parchment and tanned leather from deterioration.
7. The results obtained from the polymers dissolved in organic solvents are better than the results obtained from polymers dissolved in distilled water.
8. Mowilith DM5 gave very bad result with parchment after ageing of consolidated samples especially with the first concentration.
9. The third concentration of Paraloid B.72 with vegetable and chrome tanned leathers was affected by ageing more than the second concentration.
10. The worst results was obtained from Plextol B.500 at all concentrations with chrome tanned leather after ageing.

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