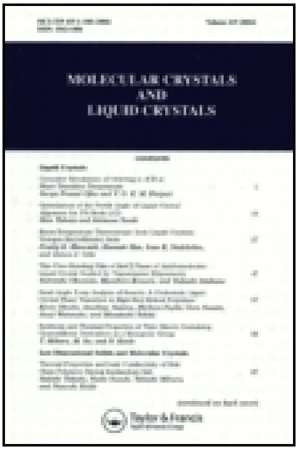

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Behaviour to Accelerate Ageing of Some Natural Biopolymer Constituents of Parchment

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BEHAVIOUR TO ACCELERATE AGEING OF SOME NATURAL BIOPOLYMER CONSTITUENTS OF PARCHMENT

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The aim of this study is testing different new types of parchment materials during thermo-oxidative ageing at 70°C temperature, as well as under UV radiation at 50°C. The changes have been monitored by the physical–chemical tests, UV–VIS and FT–IR spectra, color and thermal analysis.

Based on these experiments the corresponding types of parchment have been selected in order to assure appropriate protection of patrimony materials.

INTRODUCTION

A very important part of the museum patrimony is represented by leather and parchment objects which include precious historical information. Such kind of objects is represented by book bindings, manuscripts, charters, belts, arm covers, sword sheaths, etc.

The most representative parchment Romanian patrimony objects taken into consideration for our study were the following: Romanic Greek Evangelic book, Old Printing by Theodosie Metropolitan from 1693, Bucharest Bible from 1688, Old printing by Serban Cantacuzino Evangelic from 1697, Old printing by Antim Ivireanu Penticostarion from 1742, document on parchment by Simion Moghila 1601, and document on parchment by Mihail Racovita from 1731.

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The restoration of patrimony objects from parchment involves in many cases the replacement of irremediable destroyed parts by new parts. The manufacture of new pieces of parchment for museum purpose assumes realization of special characteristics, adequate for each patrimony object. One of the most important characteristics is the structural compatibility and durability of new parchment materials. Such a structural compatibility of new parchment needs to reproduce the performance of old technologies by using natural auxiliary materials, vegetable extracts and special handmade operations.

The assessment of parchment durability is performed by using different kinds of artificial ageing simulation followed by chemical, physical-mechanical and instrumental analyses.

Our paper presents the procedures for evaluation of durability characteristics of new parchment materials manufactured for museum use.

EXPERIMENTAL

The parchment was obtained from lamb skin, sheep, calf, pig, and bovine skin.

The main steps were the following. Firstly, the skins/hides have been intensively rehydrated. Then, they were dehaired by using hydrated lime ($Ca(OH)_2$ 4–6%), followed by post liming with 1.5–3% $Ca(OH)_2$ for 4–6 days, depending on the type of row material. Next step was deliming by intense washing with/or without ammonium sulphate.

The process is optimized by using enzyme dehairing and degreasing by appropriate detergents.

After drying in toggled state, the parchment was buffed (with special abrasive paper) on flesh side or on both sides, depending on its final usage.

These parchments manufactured were then selected by means of physical characteristics and chemical tests.

The parchment have been submitted at the thermo-oxidative ageing for 7–21 days at 70°C and normal humidity, as well as under UV radiation (240–380 nm) at 50°C for 250 hours, in accordance with standard methodology [1–5].

The changes induced during ageing have been monitored by thermal (Q 1500 D, MOM, Hungary) and spectral analysis (FT–IR 620 and UV–VIS–NIR spectrometers, JASCO, Japan) [6].

Spectral analysis was used to make evident the structure of extractible components; the modification of color surfaces after ageing was objectively monitored by diffuse reflectance system, (CIE-Lab software) and leucometer (white index).

RESULTS AND DISCUSSION

The main characteristics of the new parchment are presented in the Table 1.

Differential thermal analysis of these types of parchment reveals two complex processes for the heating in air, with loss of weight. The first process is endothermal and is produced up to 125°C; it is due to dehydration and loss of some volatiles. The second process is produced at 180–400°C; it is exothermal and consists in thermo-oxidative destruction of parchment.

The main characteristics of the thermo-oxidative process are presented in Table 2.

TABLE 1 Chemical Characteristics of Tested Parchments

	Parchment type				
Characteristics	Lamb	Calf	Pig	Sheep	Bovine
Humidity, %	16.8	17.9	15.5	12.0	13.0
Fatty materials, %	11.4	0.9	1.5	8.9	10.1
Dermic substances, %	52.3	59.1	59.0	51.6	62.2
Organic substances soluble in water, %	7.9	1.3	7.7	1.4	4.7
Mineral substances soluble in water, %	–	–	0.3	0.6	0.3
Combined tannin, %	–	–	15.7	25.8	5.9
Ash, %	6.9	6.1	0.8	0.4	4.2
pH (water extract)	5.2	5.2	4.6	4.6	4.6

TABLE 2 Thermal Characteristics of the Tested Parchments after Thermo-oxidative Degradation

Parchment	T_{max} (DTG)°C	T_{max} (DTA)°C	V(288)	V (T_{max})
Lamb				
Initial	287	266–308	0.37	0.38
21 days	257; 288	244; 279; 319	0.40	0.40
Sheep				
Initial	256; 284	242; 262; 285; 303	0.39	0.40
21 days	266; 313	255; 273; 293; 348	0.40	0.46
Bovine				
Initial	289	261–358	0.43	0.43
21 days	275; 291; 301	256; 280; 305; 319	0.36	0.35–0.37
Pig				
Initial	294; 312	242–278	0.38	0.38
21 days	266; 287; 299	252–304	0.41	0.39–0.42

$V(288) = \frac{d\Delta m}{dT}$ for 288°C; Δ_m – loss of weight at t°C; $V(T_{max}) = \frac{d\Delta m}{dT}$ for DTG.

The obtained data show the following aspects:

- Accelerated ageing of each sort of parchment have produced major changes specific to dehydratation and thermo-oxidative processes.
- For the initial sample the DTG curves exhibit only one maximum for the dehydration processe, while for some aged samples a main maximum was evidenced accompanied by other secondary shoulders (Fig. 1).
- This ensemble of competitive and/or consecutive processes determined by dehydratation and thermo-oxidative stages during accelerated ageing results in crosslinking and scission reactions, able to release free radicals under the action of destructive agents (O_2 and temperature) on the polypeptide chains.

Spectral analysis in FT–IR and UV–VIS domains shows the following aspects:

- Examination before and after thermo-oxidative ageing and UV irradiation of the content in the extractible substances shows the presence of the vibration bands characteristic to υOH ($3450\,cm^{-1}$), υC=O ($1725\,cm^{-1}$) and CONH ($1640\,cm^{-1}$) groups specific to the peptide structures and fatty acids present in the parchment composition.
- Stearic and palmitic acids are the main components of the fatty substances in the parchment.

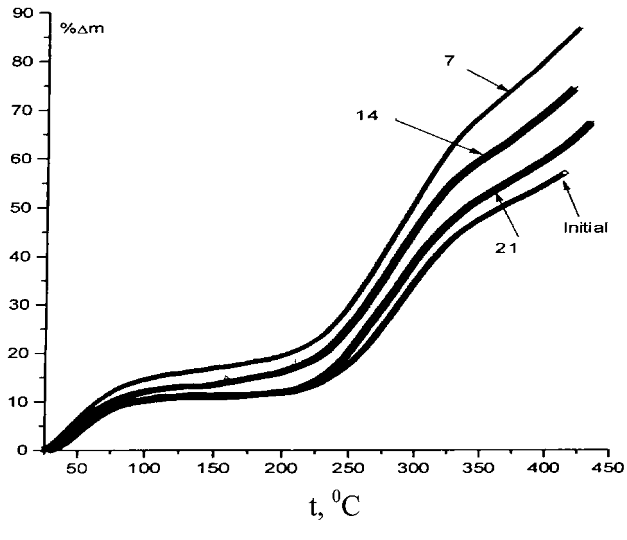

FIGURE 1 TG curves of the age lamb skin at 7, 14, 21 days.

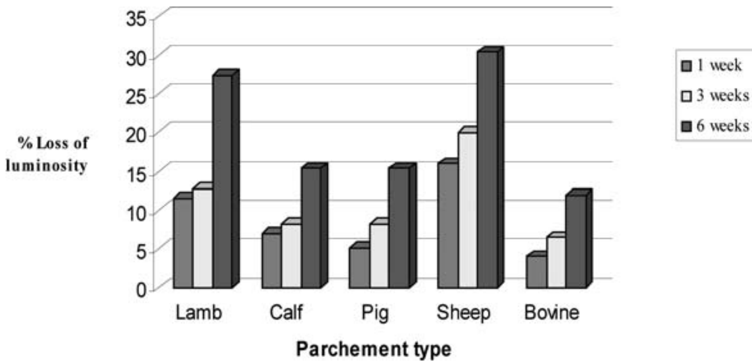

FIGURE 2 Loss of luminosity after thermo-oxidative ageing (%).

- Typical bands for −CONH− structure at 200–280 nm (UV domain), and harmonic and combination bands (NIR domain) at 1182, 1494, 1728, 1936, 2040 and 2157 nm for the CH, OH and NH structure are present in the electronic spectra after UV exposure.
- Corroboration of all these data lead to the conclusion that the basic structure of the parchment matrix is not essentially damaged, as most of changes were noticed in the intensity, not in the position of the bands.

Chromatic characteristics tested before and after thermo-oxidative degradation by means of white index indicated that, during first week, the luminosity loss is increased; after 2–4 weeks the losses are diminished. At the end of the tests, they are higher for lamb and sheep skins, and similar for the other types (Fig. 2).

After UV ageing, the samples were examined by the diffuse reflectance accessory, and using the CIE – Lab programme the main chromatic characteristics: luminosity (L^*), croma (C^*) and shade angle (H) have been registered.

The obtained data (Table 3) reveal 6–14% diminution of the luminosity and increase of the yellow hue of the parchment surface attributed to the formation of some oxygenated structures (carbonyle and carboxyle).

The presence of the ozone in the UV chamber for testing accelerates the oxidative processes.

CONCLUSION

The structural changes induced during thermo-oxidative ageing and under UV radiation has been monitored by physical – chemical analysis and DTA,

TABLE 3 Chromatic Characteristics for Parchment Samples Exposed at UV Radiation

	Chromatic characteristics					
	L*		C*		H	
Sample	Initial	250 h	Initial	250 h	Initial	250 h
Lamb	72.90	72.37	26.01	31.50	88.21	88.58
Calf	68.46	82.06	14.70	25.74	81.08	86.93
Pig	82.40	88.81	7.30	9.76	75.73	87.48
Sheep	76.61	83.17	3.90	10.90	77.22	87.38
Bovine	80.26	88.86	14.24	17.92	80.18	89.84

FT–IR, UV–VIS–NIR spectra, as well as by color analysis by diffuse reflectance spectra.

Based on these experiments the appropriate type of parchment will be selected, taking into account the highest stability in time and under the action of various ageing agents of the tested materials.

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