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ΘΑΛΗΣ: Ενίσχυση της Διεπιστημονικής ή/και Διιδρυματικής έρευνας και καινοτομίας με δυνατότητα προσέλκυσης ερευνητών υψηλού επιπέδου από το εξωτερικό μέσω της διενέργειας βασικής και εφαρμοσμένης έρευνας αριστείας

Τίτλος υποέργου:
Διερεύνηση των επιδράσεων των περιβαλλοντικών παραγόντων στα οργανικά υλικά τεκμήρια φυσικής και πολιτιστικής κληρονομιάς
(MIS 376986)

ΠΕ.1: Σύνθεση - Αξιολόγηση των αποτελεσμάτων - Δημοσιότητα

Υποδράση 6.3: Αξιολόγηση συστημάτων τεχνητής γήρανσης τα οποία χρησιμοποιούνται για δοκιμές και έλεγχο

Παραδοτέο: 6.3α Επιστημονικές δημοσιεύσεις/ανακοινώσεις

Δημοσίευση στο περιοδικό «ΜΟΥΣΕΙΟ»

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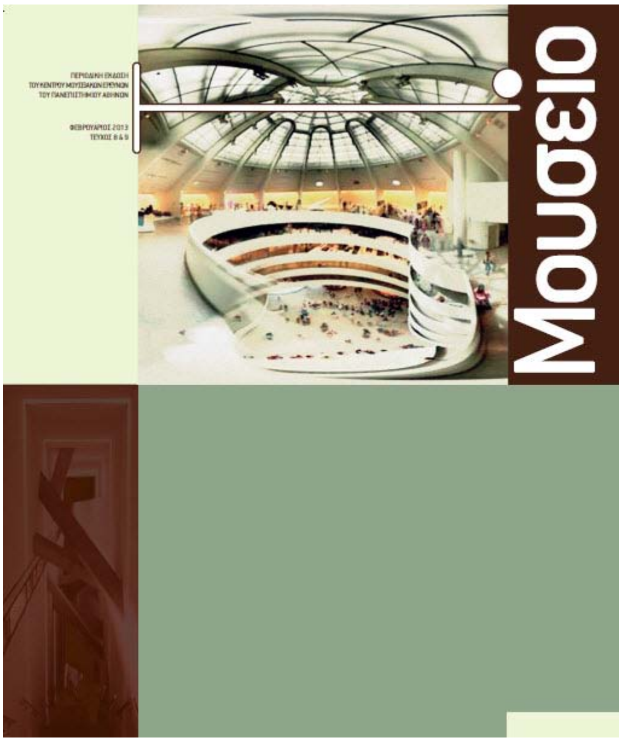
<u>Εισαγωγή</u>

Στο παρόν παραδοτέο παρατίθεται το κείμενο που υποβλήθηκε και εγκρίθηκε προς δημοσίευση στο περιοδικό «Το Μουσείο», το οποίο αποτελεί περιοδική έκδοση του Κέντρου Μουσειακών Ερευνών του Εθνικού και Καποδιστριακού Πανεπιστημίου Αθηνών.



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Development of an ageing protocol to investigate the degradation the non-treated organic materials constituting the cultural heritage

Δημιουργία πρωτοκόλλου γήρανσης μη επεξεργασμένων υλικών με στόχο την περιγραφή λειτουργικών μοντέλων φθοράς τους

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1. INTRODUCTION

In general, during artificial or accelerated ageing extreme environmental conditions are used, in order to simulate the natural ageing process in shorter periods of time (Porck, 2000). The purpose of this experiment is to produce artificially aged samples using specific environmental conditions (temperature, relative humidity and air pollutants) that simulate naturally aged samples, so then to assess the impact of specific environmental conditions on the physical and structural integrity of the organic materials.

The current paper presents the ageing protocols for accelerated ageing of bone and wood samples, textile and parchment samples as well as paper samples, which were conceived as part of the INVENVORG project (Thales Research Funding Program – NRSF).

2. The Ageing Protocols

2.1 BONE and WOOD Ageing Protocols

Factors and levels

According to the literature there have been many studies conducted regarding the aging and thermal effects on bone(Cronyn, 1990; Weintraub and Wolf, 1995; Hedges, 2002). Nevertheless, the behavior of bone objects in a museum environment or storage rooms have not been studied, and a correlation between real-time degradation mechanisms and museum environment conditions has yet to be attempted.

Furthermore, In the case of wood, the main abiotic factors in a museum environment affecting wooden artifacts preservation are heat, radiation, relative humidity, and particulate and gaseous pollution (Rivers and Umney, 2005; George *et al.*, 2005; Yidiz *et al.*, 2006). Literature on the effect of gaseous pollution on wood and its synergistic action with other environmental factors is scare. Gaseous pollutants, such as O_3, NO_2 and SO_2, have been studied mostly for surface treated wood (Grontoft & Raychaudhuri, 2004) or for wood coatings and it has showed that pollutants surface deposition velocities are highly dependent to relative humidity (RH). In order to have a common reference in keeping with the experiments performed under the PROPAINT project, the following procedure shall be conducted.

The preparation of the aged sample will be addressed using the following conditions (factors and levels):

	Factors		**Levels**
1.	Relative Humidity (%)		45, 70
2.	Gaseous pollutants concentration (ppmDay)	(SO_2)	100, 300 *
3.		(NO_2)	100, 300 *
4.	Duration (Days)		15, 30

** Following the PROPAINT Dose concentrations (Dahlin, 2010) for these pollutants and magnifying the alarm levels for Greece by 2000.*

In addition, the order of pollutants is evaluated, as a fifth factor, in order to decide on whether it is important or not.

Concluding, there are five factors with two levels each (low – high) to be investigated. The fractional factorial experimental design (Box et al., 2005) is going to be used and sixteen experiments are going to be performed, as shown in the table below. The number of experiments is therefore reduced significantly leaving more space for repeating analysis tests.

No of experiment	RH	SOx	NOx	D	O
1	Low	High	Low	Low	N
2	High	Low	High	High	N
3	High	High	Low	High	N
4	High	Low	Low	High	S
5	Low	High	High	Low	N
6	High	Low	High	High	S
7	High	Low	Low	Low	N
8	Low	High	High	High	N
9	Low	High	Low	High	S

10	High	High	High	High	S
11	Low	Low	Low	High	N
12	Low	Low	High	Low	N
13	High	High	Low	Low	S
14	Low	High	High	Low	S
15	Low	Low	Low	Low	S
16	High	Low	High	Low	S

RH = Relative Humidity (Low=45% - High=70%)
D = Duration (Low=15days - High=30days)
SOx = Pollutant1 (Low=100ppm - High=300ppm)
NOx = Pollutant2 (Low=100ppm - High=300ppm)
O = Order of the pollutants Sox, NOx. Specifically, the notation S means the sequence SOx-NOx, while the notation N means the opposite, NOx-SOx.

Bone samples

Figure 1 (Vukičević *et al.*, 2012)

For the experiments metapodial bones of roe deers (*Capreolus*) are going to be used. The metapodial bone of a full-grown roe deer is almost equal in length to this of a fallow deer (*Dama*), (Al. Van der Geer 2006). As shown in Figure 1 a typical metapodial bone of a fallow deer is shown, and its mean length appears to be C = 160 mm, while the diameter of the epiphysial joints is Bp = 27 mm and Bd = 26 mm (Vukičević *et al.* 2012).

Each sample is going to be a bone divided in two equal parts transversly, in order to have an epiphysial joint and cortical and cancellous bone in both specimens coming from the same bone. This way the accelerated aging effects are going to be evaluated in both cortical and cancelous bone. For each experiment 3 samples are going to be used due to financial limitations concerning the post processing analysis.

Wood Samples

Both softwood and hardwood specimens will be used in the experiment. Sample dimensions: 10x10x 100 mm

The samples will be cut from a light color coniferous species and a dark color hardwood species.

2.2 TEXTILE and PARCHMENT Ageing Protocols

<u>Factors and levels</u>

According to the literature review on textile ageing, a lot of experimental procedures have illustrated the detrimental effect of visible and UV radiation (Rutherford & Harris 1941, Phillips & Arthur 1964, Gordon 1968, Lennox & King 1968, Harris 1984, Holt & Milligan 1984, Karpovicz 1989, Feller 1994, Davidson 1996, Seli *et al.* 1998, Timár-Balázsy & Eastop 1998, Korenberg 2007, Zhang *et al*. 2008a). The deterioration types have been highlighted whereas optical and mechanical properties have been measured and chemical composition have been analysed.
Although there are a lot of implemented experiments on the thermal degradation of wool and the yellowing as a result of it, there is not clarified whether the synergy of relative humidity and temperature that affect the wool Tg alters also the physical and chemical properties of wool fabric. Moreover, there have been investigated the effect of some gaseous pollutants in the morphology of the fabrics tested but it is not examined whether optical and mechanical properties as well as chemical composition were altered.
Artificial ageing of new parchment samples was performed to induce deterioration similar to naturally aged parchment exposed to atmospheric pollutants. In the framework of this project it has been decided to examine four factors affecting the ageing (Relative humidity, NO2, SO2 and time of exposure). The order that samples are exposed to the pollutants will be evaluated, as a fifth factor, in order to evaluate whether the sequence of the pollutants is important or not. Temperature was decided to remain constant at 25^0C during the process of the experiment while the four above mentioned factors will be ranged in two levels (low -high).

Therefore, for textile and parchment samples relative humidity was decided to range at two levels: one low (45%) and one high (70%). Gaseous pollutants concentration was set at two different levels: one low (10ppm) and one high (25ppm). Finally, two durations of the experiments were chosen: one short duration (15 days) and one long (30 days).

	Factors	**Levels**	
1.	Relative humidity	45 %	70 %
2.	Gaseous Pollutant (NO_2)	concentration: 10 ppm*****	concentration: 25 ppm
3.	Gaseous pollutants (SO_2)	concentration: 10 ppm	concentration: 25 ppm

4.	Time	15 Days	30 Days
5.	Order	(2 levels)	

**industrial and urban areas have gaseous pollutants maximum 0.5ppm. Kobayashi and Yoshizumi (1994) have calculated the urban concentration in order to achieve accelerated ageing relevant to many years of exposure. Therefore, 10ppm corresponds to almost 20 years of exposure while 25ppm corresponds to 50 years.*
Concluding, there are five factors with two levels each (low – high) to be investigated.

The fractional factorial experimental design is going to be used and sixteen experiments are going to be performed, as shown in the following table:

No of experiment	**RH**	**SO2**	**NO2**	**Duration**	**Order**
1	Low	High	Low	Low	N
2	High	Low	High	High	N
3	High	High	Low	High	N
4	High	Low	Low	High	S
5	Low	High	High	Low	N
6	High	Low	High	High	S
7	High	Low	Low	Low	N
8	Low	High	High	High	N
9	Low	High	Low	High	S
10	High	High	High	High	S
11	Low	Low	Low	High	N
12	Low	Low	High	Low	N
13	High	High	Low	Low	S
14	Low	High	High	Low	S
15	Low	Low	Low	Low	S
16	High	Low	High	Low	S

<u>*Parchment samples*</u>
The parchment hides used in this experiment have been acquired by the National Research and Development Institute for Textile and Leather (I.N.C.D.T.P.), Bucharest Romania. The material has been produced by traditional methods and it was chosen for this purpose because it is similar to historical materials met in collections and museums. For this experimental process, 96 samples from goat parchment have been used in total. They have been cut in the middle so forty eight of them have been subjected to artificial ageing with the protocol described here above while the rest forty eight remained as reference samples. This happened because parchment's

characteristics and properties may vary a lot in the same hide topography. Thus it was necessary to have a reference sample for each artificial aged sample, not from a near location but from the exactly adjacent place. For each cycle of artificial ageing, three samples have been used in order to have enough material for statistically safe results and in addition to have spare samples for the Bank of samples for future use.
The parchment samples have been hanged in specific inox construction during their artificial ageing The parchment hide used in the experiments and the method for labeling the samples.

Textile samples

Due to the duration of the current program, the scientific equipment availability and the financial limitations concerning the post processing analysis one type of textile will be used. The chosen textile is wool because woollen fabrics have been extensively used for production of ethnographic artefacts found very often in ethnographic museums (Schaffer 1981). The fabric that will be used is merino wool, because its fibers have bilateral divided cortex (half and half orthocortex and paracortex. The fabric will be wool worsted twill flannel that is natural scoured, of approximate weight: 241 grams/m^2 and approximate width of 152 cm and have product item code 526 from the Testfabrics, Inc©., 415 Delaware Avenue, West Pittston PA 18643 USA, Tel: 1 (570) 603 0432, Fax: 1 (570) 603 0433 and email: info@testfabrics.com

Samples	Natural scoured wool (merino wool) The sample size* for mechanical properties: Tensile strength measurements following standards requirements need 500X50mm KAWABATA: 200X200mm, requires 3 repetitions ELMERDORF: 300X410mm requires 5 repetitions *preliminary tests on testing of the mechanical properties will be done in order to parameterized the dimensions of the samples as the standards dimensions are addressed to industrial tests and not to samples obtained from authentic ethnographic objects and due to the size of the tube that samples will be exposed to gaseous pollutants (7X12cm).

For each experiment 2 samples are going to be used due to financial limitations concerning the post processing analysis.
The number of samples according to the above mentioned will be:
For the 1st accelerated ageing procedure: 8 (2 repetitions, 2 factors, 2 levels)
For the 2nd accelerated ageing procedure: 16 (5 factors and 2 levels)
If the 3rd accelerated ageing procedure will be implemented: 8 (2 repetitions, 2 factors, 2 levels)

2.3 PAPER Ageing Protocols

Factors and levels

Previous studies discussed above showed that ageing paper at medium to relatively high humidity levels (50-80%) and at temperatures around 80-90°C, simulates natural ageing sufficiently well (Erhardt & Mecklenburg 1995). Thus, for the preparation of the aged samples, the following conditions are suggested:

Temperature (T) in °C 80
Relative humidity (RH) % 65 or 75% (depending on the standard used)
Duration (Days) 100
Standards: ISO 5630-3 or an adaptation of the ASTM D 6819-02 2002

The selection of the conditions is based on the principles discussed in the literature review and on the ease of implementation. The application of ISO 5630-3 requires a humid oven operating at 80°C and 65% RH. ASTM D 6819-02 2002 prescribes the use of sealed tubes and can be conveniently adapted for the ageing of large quantities of paper. The latter standard requires the use of an arid oven, since the humidity can be adjusted to the required levels by preconditioning the samples or by use of saturated salt solutions. For example, a level of 75% RH can be achieved by a saturated NaCl solution.

For the Whatman paper, ageing will last 100 days. As seen from the next figure in the two graphs at the bottom, showing two of the mechanical properties of Whatman paper, namely folding endurance and tensile strength, there is a turning point between 90 and 120 days of ageing, where the decline of the properties accelerates. Our target is to age paper up to that point.

Thus, paper will be sensitized towards ageing and there is a good chance that the aged samples will show property changes after the exposure to the museums environments (Feller 1977; Feller et al. 1986; Feller 1994).

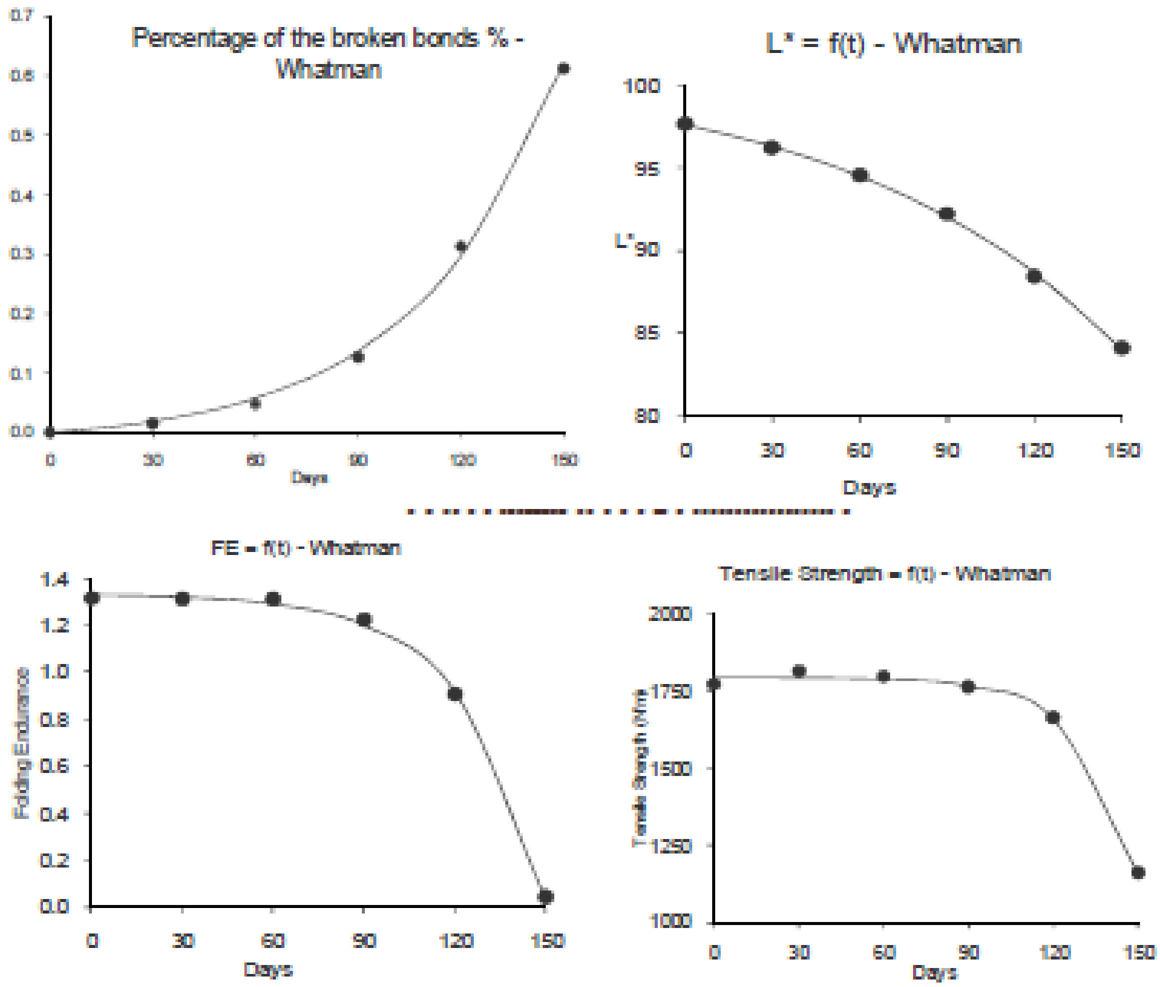

Figure 2. Mechanical properties of Whatman paper.

Paper samples

It was decided that pure cellulose paper should be studied, since it would represent the simplest possible chemical system. The existence of additives, lignin and other components of commercial paper may introduce many unknown factors and would add to the complexity of the system, making very difficult or even impossible to determine and isolate trends. Thus, Whatman no 2 filter paper will be used for the preparation of samples, a standard paper with repeatable properties which has been extensively studied and widely used to model pure cellulose paper, since it comprises pure cotton with no additives, fillers or sizing (Daniels 1976; Moropoulou & Zervos 2003; Zervos & Moropoulou 2005; Zervos 2007). An industrial historic alum/rosin-sized lignin containing paper, similar to historic paper stored in museums, libraries and archives will also be used in its virgin state.

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