

# A New Method of Determining the Normal Range of Hydric-Equilibrium Variation in Wood, with Multiple Applications

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*This paper presents a new method for the determination of the normal range of variation of the equilibrium moisture content ( $\Delta$ EMC) for limewood (lindenwood) on the basis of the variation of the reversible moisture content (RMC) as a result of the isothermal and isobar processes of adsorption/hydration at 100% RH and desorption/dehydration at 10% RH. There have been selected a series of characteristics with archaeometric potentials such as the content of the reversible water and the critical time for the correlation of the two curves and others. On the basis of some preliminary experimental data on limewood, there has been created the premise for the usage of these characteristics for clarifying the mechanism of the adsorption/desorption processes of the reversible water in the impact studies regarding the influence of active preservation treatments. Such aspects as well as their applicability in authentication will be further studied for other wood species in some future papers.*

*Keywords: reversible moisture content, hydrous equilibrium, wood hygroscopicity, adsorption and desorption processes, preservation treatment, archaeometric characteristics, limewood*

Wooden supports, as any organic materials, due to the volume non-homogeneities, have quite a large and complex specific range of the variation of the equilibrium moisture content (EMC), which depends on a series of exogenous factors (e.g. atmospheric humidity, temperature, pressure and illumination) as well as on different preservation and restoration interventions. The specificity and complexity of this range are determined by endogenous factors [1-4].

The EMC is the moisture content (MC) at which the wood is neither gaining nor losing moisture; however, this is a dynamic equilibrium and it changes according to the relative humidity, the temperature and the pressure involved [5-7].

It is known that the water contained in materials is divided into two main types: the physically-bonded water, the so called "hygroscopic water", known as a phenomenon of "material hygroscopicity", and the chemical bound water or the "constitution water", from the OH groups [1,4,8].

The physically-bonded water or the material "hygroscopicity", also known as "material humidity" or MC, can be divided into several types of water:

- the reversible water, named the reversible moisture content (RMC) which changes under the influence of the atmospheric humidity in the range of normal temperatures and pressures;

- the irreversible water, which varies under the influence of some "aggressive" exogeneous factors and which can be found in the structure of the organic materials such as hydro-gels, inner cells or inside other anatomical structures (membrane water, tissue water, capillary water, etc.).

In the case of wood, the "reversible hygroscopicity" is the quantity of water continuously changed with the environment, by means of a dynamic process of sorption-

desorption, water which leads to sensitive dimensional changes of the objects, also known as "the play of the wood", which means the swelling and the shrinkage. In this respect, we talk about the "normal range of the variation of the equilibrium moisture content" ( $\Delta$ EMC), and the "reversible hygroscopicity range", respectively, which change continuously with the environment [9-13].

The  $\Delta$ EMC is specific to each wood species and it is related both to the oldness and conservation state and to the structural and dimensional complexity of the object [13]. For this reason, the  $\Delta$ EMC characteristics can be used for the evaluation of some attributes for the authentication and for the impact of some treatments on the wood conservability. This range varies from the fiber saturation point (30..33% MC) which can be obtained at 100%RH (normal environment pressure and temperature), up to the minimum MC value for wood, obtained at 10%RH (normal environment pressure and temperature) [11,12].

Within the limits of this range of variation the pre-collapse of the wood is avoided because the centers of minimum resistance are not activated [13].

The specialized literature indicates some methods for the MC determination by the electrical resistivity measurements [14-16], the NIR spectroscopy [17], the NMR technique [18], the radio frequency/vacuum technique [19,20], the GC-MS technique [21,22] as well as for the EMC evaluation [23,24].

This paper describes a new method for the  $\Delta$ EMC determination together with two applications for assessing some archaeometric characteristics (the correlation or the equilibrium point between the adsorption/hydration and desorption/drying curves; the necessary time to reach this point; the maximum MC value at 100%RH, the minimum MC value at 10%RH, under normal pressure and temperature conditions; the minimum/maximum values

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**Table 1**  
ORIGIN, CONSERVATION STATE AND DENDROCHRONOLOGICAL DATA OF  
THE LIMWOOD (*TILLIA CORDATA MILL*)

| Sample   | Characteristic                     |                    |                         |                        |                         |     |
|----------|------------------------------------|--------------------|-------------------------|------------------------|-------------------------|-----|
|          | Origin                             | Conservation state |                         | Age of tree<br>(years) | Wood oldness<br>(years) |     |
| New wood | Board stabilised                   | hydrous            | Excellent, anatomical   | without defects        | 76                      | 8   |
| Old wood | Support from a carved frame (1740) |                    | Good conservation state |                        | 86                      | 269 |

from the first and the second derivatives, respectively, of the adsorption curve) and the impact of some treatments on the wood conservability.

### Experimental part

The sampling consisted of preparing 15 new and 15 old (dated from 1740 A.D.) limewood (*Tillia Cordata Mill*) samples with a parallelepiped shape and the dimensions of 10(T)x20(R)x40(L) mm, by taking into account the position of the growth rings.

The origin, the conservation state and the dendrochronological data of the wooden samples are presented in table 1.

From all these samples, 5 of them have been used as reference (non-treated), 5 samples have been treated with red petroleum (aromatic crude oil pumped out from the Câmpeni village, Bacău County) and 5 with alcoholic propolis solution (20%). The great number of samples for analysis is necessary for obtaining a statistical data evaluation because the wood, as a heterogeneous material (a matrix system made of lignin as a dispersion medium and cellulose fibers as the armature or the dispersive phase), has a different behavior on the three directions (longitudinal-L, radial-R and tangential-T, regarding the annual rings and the position of fibers/cells).

The samples have been initially weighted by using an analytical balance with four digits; after that they have been kept into a dessicator until they have got a constant weight, in an atmospheric residual humidity below 10% (R.H.). The dessicator contained CaCl<sub>2</sub> sic and anhydrous silicagel. This dehydration process allows the elimination of the reversible water which is taken over by the environment under normal temperature and pressure conditions. The measurements of the humidity in the dessicator have been done by using an U.S.A. made EXTECH-type digital electronic hygrometer.

After drying, the samples have been introduced in a hermetic vessel as a humidifier, containing distilled water in an open Petri plate, keeping over more than 99% RH inside to allow the hydration process. The measurements of the humidity in the humidifier have been done with the help of the same type of hygrometer mentioned above. Each sample has been weighted each 10 min with the analytical balance for the first 60 min, then each 20 min for the next 60 min, then each 30 min for the next 120 min; after that the weighting has been done each hour up to 8 h of adsorption and then after 2, 4 h, etc., the measurement interval being doubled each time until the weight has remained constant and the fiber saturation point has been reached [11,12]. The *adsorption* or the *hydration curves*, respectively, have been obtained from the graph  $U\% = f(t)$ .

The dessicators and the humidifier have been previously treated against bacterial and fungal attacks.

The hydrated samples have been transferred to a dessicator with a relative humidity below 10%, where the

dehydration process takes place. The same kind of measurements as for hydration have been performed, at the same time intervals until the constant weight has been reached, this meaning that the reversible water has been totally eliminated. Thus, the samples have been situated at the limit between the content of the reversible hygroscopic water and the irreversible water. The desorption or the dehydration curves, respectively, have been obtained from the graph  $MC\% = f(t)$ .

The compatibility studies of the different types of treatments which take into account both the material and the procedure, consider the evaluation of the impact of these treatments on some physico-structural and chemical characteristics which do not imply degradation or deterioration evolutive processes.

Based on our researches [9-13], the evaluation of the  $\Delta EMC$  represents a very efficient method which allows to establish the impact of different types of treatments on wood, and it is recommended for the compatibility studies for interventions on new wood, wood to be used for the first time or old wood which has been exposed to some operations of active preservation and restoration.

In the evaluation of the treatment impact on wood, some characteristics from the adsorption/hydration and desorption/dehydration curves, respectively, are used. They are the following:

- the correlation or equilibrium point, which represents the intersection of the two curves;
- the time necessary to reach the equilibrium point, obtained from the curve  $RMC = f(t)$ ;
- the limits of the variation range of the hygroscopic reversible humidity – the maximum  $RMC = \Delta EMC$  and the minimum  $RMC = 0$ ;
- the minimum and the maximum values of the first derivative curve,  $dRMC/dt = f(t)$ ,
- the minimum and the maximum values of the second derivative curve,  $d^2RMC/dt^2 = f(t)$ ,

From these characteristics, the equilibrium point between the adsorption and the desorption as well as the minimum and the maximum values, respectively, from the first and the second derivative curves have an archaeometric and dendrological specificity, having a chronological evolution which also depends on essence, conservation state, age/oldness, etc. The study of these characteristics can be used for the authentication based on the material and on the related technology of fabrication/elaboration as well as for assessing the compatibility of the preservation treatments.

### Results and discussion

The hydration and dehydration curves for the new limewood (lindenwood) have been drawn, being used as reference in the evaluation of the modifications of the above-mentioned characteristics; afterwards the curves for the same kind of samples treated with organic solutions

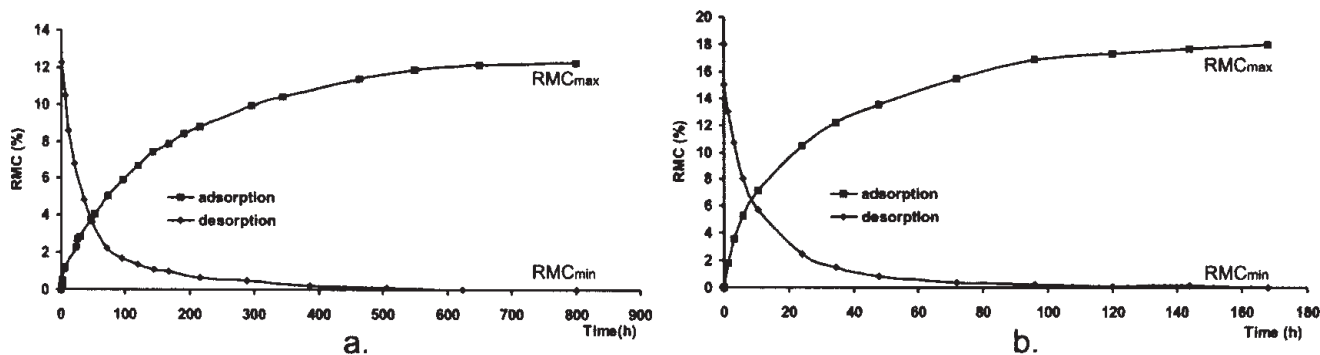


Fig. 1. Adsorption/hydration and desorption/dehydration curves for the limewood specimens: a – new wood, b – old wood (1740 A.D.)

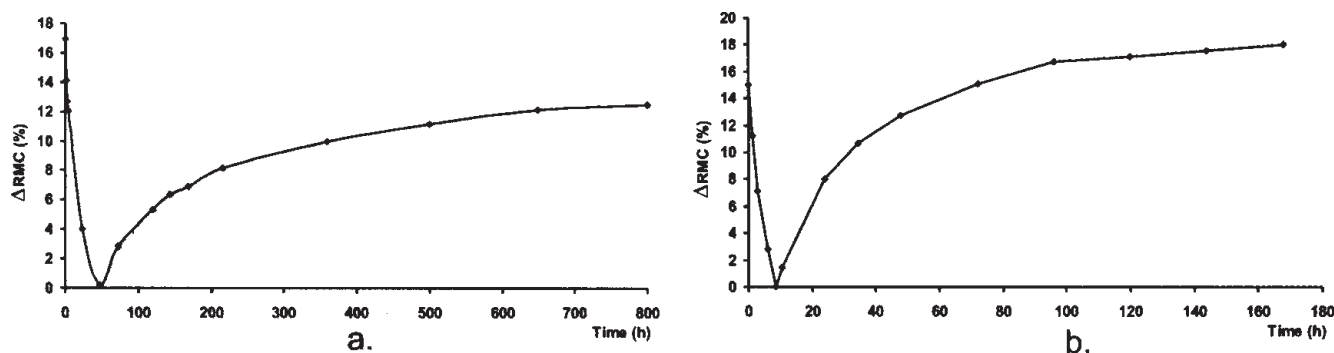


Fig. 2. The curve  $\Delta RMC = f(t)$  evaluated from the hydration and dehydration curves for the limewood specimens: a – new wood, b – old wood (1740 A.D.)

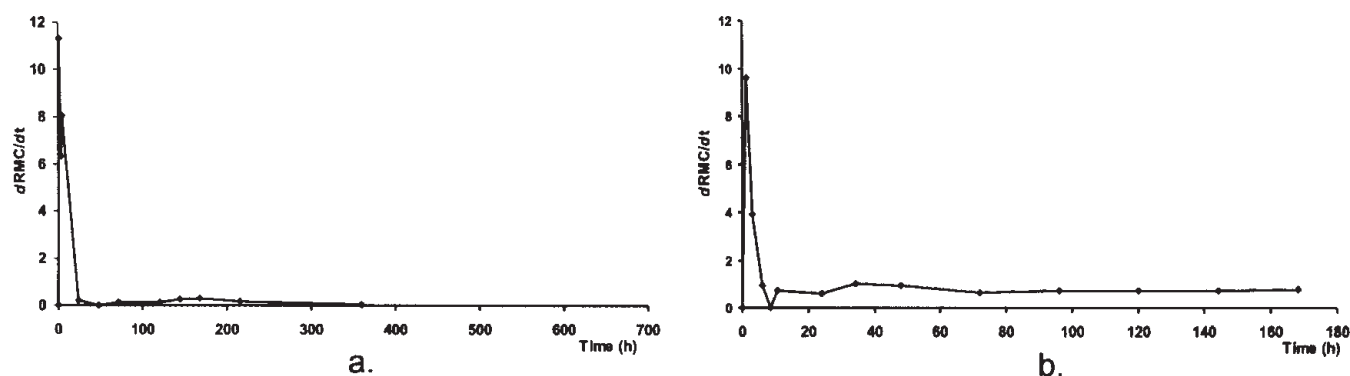


Fig. 3. The curve  $dRMC/dt = f(t)$  evaluated from the hydration curve of the limewood specimens: a – new wood, b – old wood (1740 A.D.)

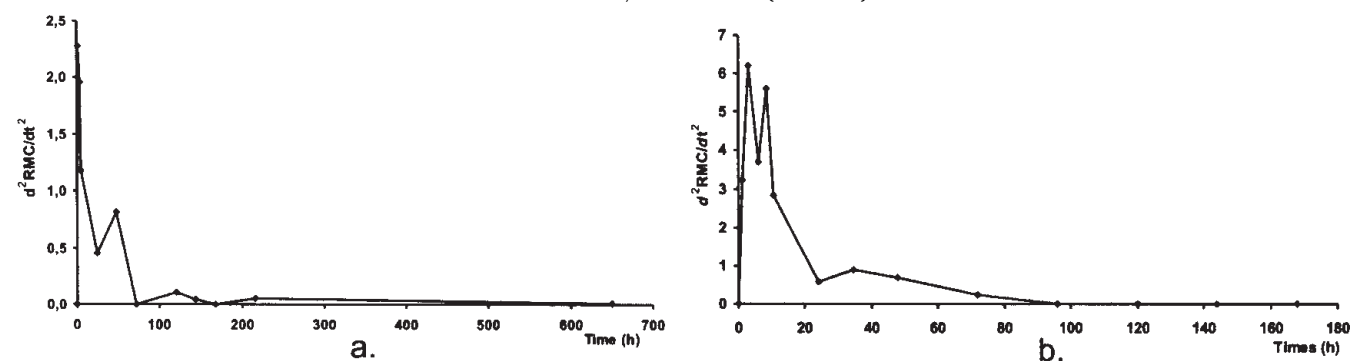


Fig. 4. The  $d^2RMC/dt^2 = f(t)$  evaluated from the hydration curve of the limewood specimens: a – new wood, b – old wood (1740 A.D.)

based on the main components of the recipes described in some patents have been obtained [11,12].

In the figures 1 - 4 the average experimental data of 5 samples from the new and 5 samples from the old limewood (1740 A.D.) are presented, in correlation with the hydration and dehydration curves (fig.1), allowing the evaluation both of some archaeometric characteristics and of those implied in the impact of different treatments of

active preservation on  $\Delta EMC$  (figs. 2 - 4). In this respect, besides the time necessary to reach the critical point of correlation of both processes, namely, the hydration and dehydration evaluated in figure 1, the curves  $\Delta RMC = f(t)$ ,  $dRMC/dt = f(t)$  and  $d^2RMC/dt^2 = f(t)$  have been obtained. The first curve represented in figure 2 allows the evaluation of the time when the two processes intersect, while the others, corresponding to the first and the second derivatives,

respectively (figs. 3 and 4), are evaluated on the basis of the hydration processes for the new and the old wood specimens. These representations allow the evaluation of the archaeometric characteristics which are approached only for the qualitative appreciations of the hydration and dehydration mechanisms stages for the non-treated new and old wood specimens.

In figure 1 the domain  $RMC_{max} - RMC_{min} = RMC_{max}$  represents the  $\Delta EMC$ , which has four specific characteristics, namely, the correlation or the equilibrium point between the adsorption/hydration and desorption/drying curves, respectively, the necessary time to reach this point, the maximum MC value at 100%RH, the minimum MC value at 10%RH, under normal pressure and temperature conditions (isotherm and isobar processes).

From figures 1 and 2 there have resulted that the time characteristics for the intersection of the two processes is 46 h for the new wood and 9 h for the old wood, values which correspond to a humidity of 3.9% for the new wood and 6.4% for the old one. These values demonstrate that the new limewood hydrously stabilized dehydrates harder than the old one. At the same time the maximum moisture content is 12.5% for the new wood and 18% for the old one.

The graphs of the first and the second derivatives of the hydration curve of the new and old wood, respectively, are presented in figures 3 and 4.

Figures 3 and 4 show how the adsorption process takes place in three stages; the first stage takes about 1.5-2 h and is characteristic for the adsorbed water at the external surface of the wooden specimen; the second stage, in the range of 80-140 h, which is a characteristic for the adsorbed water at the internal surface of the micro-fibers in the form of monomolecular layer; the third stage, ranging from 150-250 h when a poly-molecular water layer is formed, which is a characteristic close to the condensed water.

Experimental data, obtained in the same way for another set of 5 new and 5 old (1740 A.D.) wood specimens treated by immersion for 20 min in red petroleum (rp) and propolis alcoholic solution (p), are presented in the form of

correlation graphs of the hydration and dehydration curves via the hygroscopic humidity adsorption variation, and the desorption one, respectively, versus time.

Figures 5 and 6 show the hydration and dehydration curves, as isotherm and isobar processes, of the treated samples with red petroleum (rp) and propolis (p), respectively.

Figures 5 and 6 allow the comparison of the impact of the treatments with red petroleum and propolis on the hydration and dehydration curves, on the basis of three characteristics, namely, the time and the average humidity for the correlation of the two hydrous processes (adsorption and desorption of the hygroscopic water) as well as the maximum reversible humidity ( $RMC_{max} = \Delta EMC$ ) of the reversible hygroscopic water.

Figures 5 and 6 show that the time characteristics for the intersection of the two processes are:

- for the new wood: 7.5% at 48 h for the red petroleum treatment and 7.8% at 46 h for the propolis treatment,
- for the old wood: 6.8% at 26 h for the red petroleum treatment and 7.1% at 22 h for the propolis treatment.

The data presented in table 2 resumes the impact of the two active compounds used for preservation (red petroleum and propolis) upon the modifications of the  $\Delta EMC$ .

This table shows that the time necessary to reach the critical hygroscopicity from the correlation point of the hydration and the dehydration processes of the treated new and old wood specimens as compared to the non-treated wood, is constant for the new hydrously stabilized wood (decreasing with 2 h in the case of the propolis treatment) and increases with 17 h after the red petroleum treatment and with 13 h for the propolis one. The  $RMC_c$  increases on the new wood with 3.6 for red petroleum and 3.9% for propolis ( $\Delta RMC_c$ ) and on the old wood with 0.40 for red petroleum and 0.70% for propolis. These data demonstrate that the both solutions used for the treatment of the wooden specimens do not significantly modify the adsorption and desorption processes of the hygroscopic

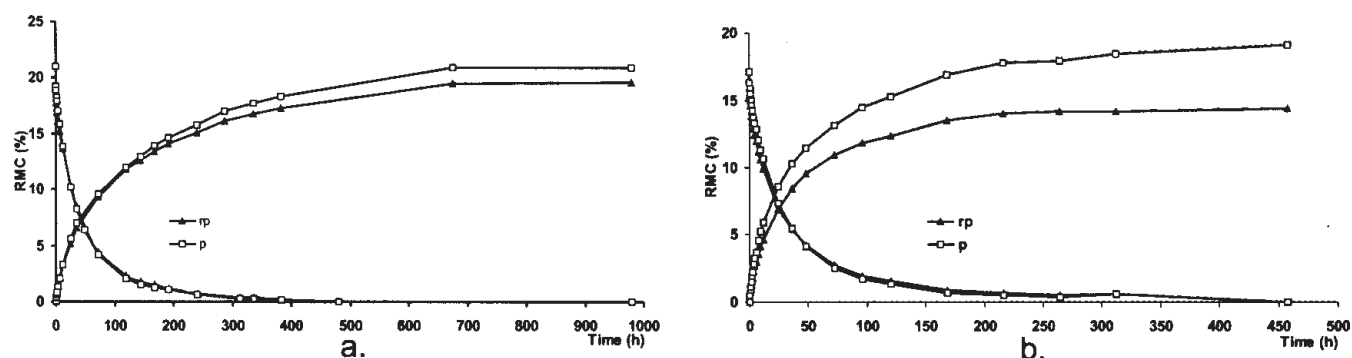


Fig. 5. Hydration and dehydration curves for the samples treated with red petroleum (rp) and propolis (p): a - new wood, b - old wood (1740 A.D.)

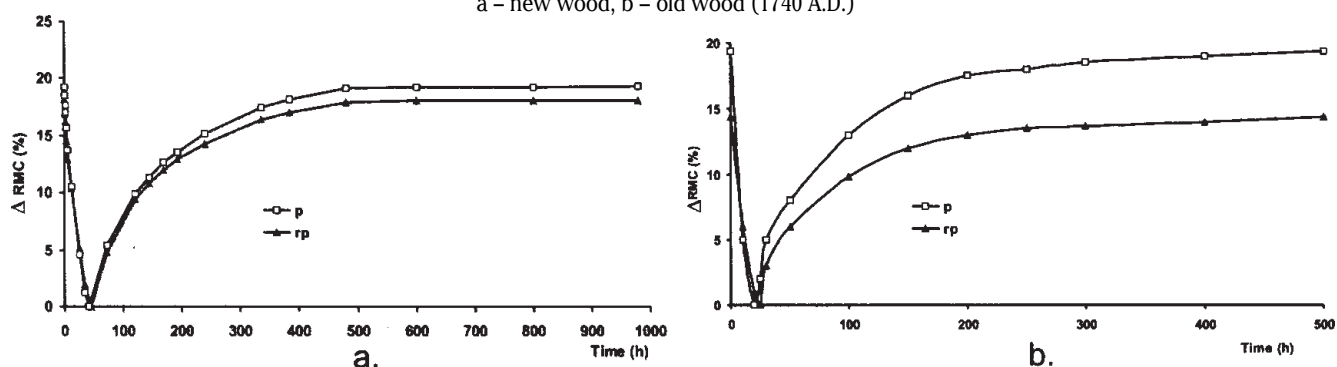


Fig. 6. The curves  $\Delta RMC = f(t)$  for the samples treated with red petroleum (rp) and propolis (p): a - new wood, b - old wood (1740 A.D.)

**Table 2**  
THE VALUES OF THE CHARACTERISTICS IMPLIED IN THE IMPACT STUDY  
ON THE TREATMENTS OF THE LIMWOOD SPECIMENS

| No. | Sample                                  | Specific characteristic |                             | ΔEMC                      |                               | Change or variation:     |                        |                            |                          |
|-----|---|-------------------------|-----------------------------|---------------------------|-------------------------------|--------------------------|------------------------|----------------------------|--------------------------|
|     |   | RMC <sub>c</sub><br>(%) | Time, t <sub>c</sub><br>(h) | RMC <sub>max</sub><br>(%) | Time, t <sub>max</sub><br>(h) | ΔRMC <sub>c</sub><br>(%) | Δt <sub>c</sub><br>(h) | ΔRMC <sub>max</sub><br>(%) | Δt <sub>max</sub><br>(h) |
| 1.  | Non-treated new limewood                | 3.90                    | 46                          | 12.20                     | 800                           | -                        | -                      | -                          | -                        |
| 2.  | New limewood treated with red petroleum | 7.50                    | 48                          | 19.00                     | 800                           | +3.60                    | +2                     | +6.80                      | 0                        |
| 3.  | New limewood treated with propolis      | 7.80                    | 46                          | 14.00                     | 800                           | +3.90                    | 0                      | +1.80                      | 0                        |
| 4.  | Non-treated old limewood                | 6.40                    | 9                           | 18.00                     | 200                           | -                        | -                      | -                          | -                        |
| 5.  | Old limewood treated with red petroleum | 6.80                    | 26                          | 21.00                     | 500                           | +0.40                    | +17                    | +3.00                      | +300                     |
| 6.  | Old limewood treated with propolis      | 7.10                    | 22                          | 19.00                     | 500                           | +0.70                    | +13                    | +1.00                      | +300                     |

$$\Delta RMC_c = RMC_c(\text{treated wood}) - RMC_c(\text{non-treated wood})$$

$$\Delta t_c = t_c(\text{treated wood}) - t_c(\text{non-treated wood})$$

$$\Delta RMC_{max} = RMC_{max}(\text{treated wood}) - RMC_{max}(\text{non-treated wood})$$

$$\Delta t_{max} = t_{max}(\text{treated wood}) - t_{max}(\text{non-treated wood})$$

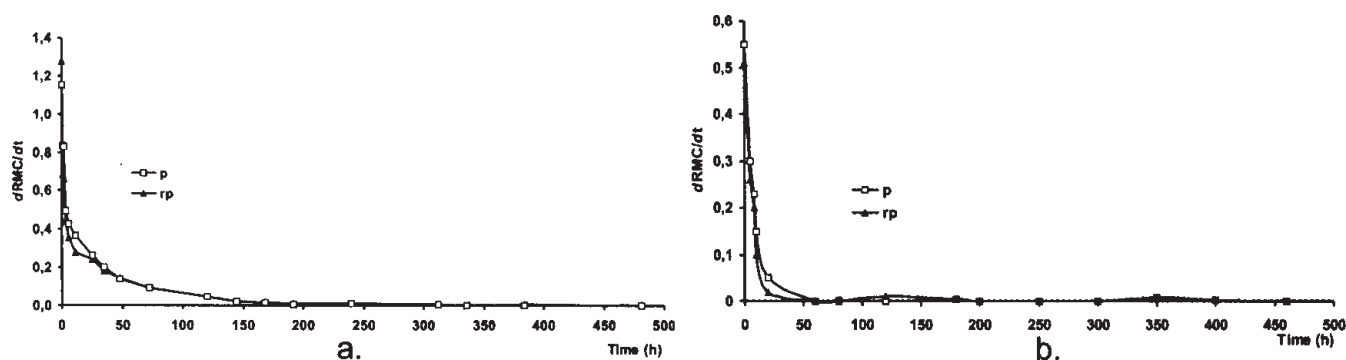


Fig. 7. The curves  $dRMC/dt = f(t)$  for the hydration processes of the samples treated with red petroleum (rp) and propolis (p): a – new wood, b – old wood (1740 A.D.)

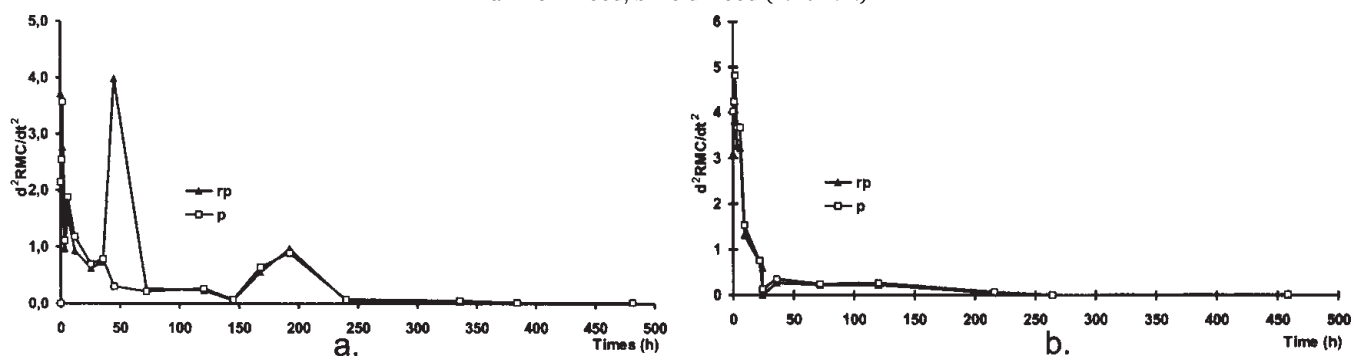


Fig. 8. The curves  $d^2RMC/dt^2 = f(t)$  for the hydration processes of the samples treated with red petroleum (rp) and propolis (p): a – new wood, b – old wood (1740 A.D.)

reversible water. Furthermore, the maximum humidity reached in adsorption, increases for the new hydrously stabilized limewood specimens treated with propolis with 1.8% and for red petroleum with 6.8%. In return, for the old limewood, the two types of treatments decrease the maximum value reached by the hygroscopic humidity with about 1.0% for propolis and 3.0% for red petroleum. Since the two solutions are hydrophobic, a decrease of the

maximum value of humidity with much larger values is to be expected.

Notably, the time necessary to reach the maximum hydration and dehydration is 800 h for the new wood and 500 h for the old one.

It is known that adsorption and desorption can be explained by the mathematical processing of the curves as first and second derivatives, respectively. The maximum

and minimum values obtained from the representation of the derivatives delimit the stages of the isotherm and isobar processes of adsorption and desorption, respectively. Because the desorption processes are slow, the evaluation has been attempted in the case of adsorption/hydration

The graphs of the first and the second derivatives of the hydration curve of the new and old wood treated with red petroleum (rp) and propolis (p), respectively, are presented in figures 7 and 8. These curves for the isobar and isotherm hydration processes of the new hydrously stabilized limewood specimens and of the old ones, allow, as we have remarked before, the evaluation of the different stages of the sorption process of the hygroscopic water.

It is also possible to make an appreciation of the behavior of the treated and the non-treated new and old limewood specimens, respectively, during the two treatments. Thus, the first stage (the adsorption of the hygroscopic water at the external surface of the form of a monomolecular layer) is finished after 2 h, reaches a maximum value between 20 to 40 h, and the last stage (the formation of the multi-molecular layers) reaches a maximum value after 120 h. Some maximum values could be important as archaeometric characteristics and in impact studies. This assertion will be demonstrated in a future study.

## Conclusions

The paper presents a new method for determining the normal range of variation of the equilibrium moisture content, method which can be used in the evaluation of some archaeometric characteristics for the authentication of wooden objects and in the study of the impact of some treatments on the wood conservability.

We have drawn the following conclusions based on experimental data:

- from the hydration and dehydration curves obtained for the new hydrously stabilized and the old limewood specimens, respectively, the normal range of the variation of the equilibrium moisture content ( $\Delta\text{EMC}$ ) has been evaluated on the basis of the reversible moisture content (RMC);

- the graphs  $\Delta\text{RMC} = f(t)$ ,  $d\text{RMC}/dt = f(t)$  and  $d^2\text{RMC}/dt^2 = f(t)$  obtained from the experimental data led to the establishment of both the archaeometric characteristics and of the one used in the impact studies, thus the mechanisms of the adsorption and desorption processes, respectively, of the reversible water was better understood and these characteristics were established, as it follows:

- the time ( $t_c$ ) after which the two curves are intersecting (called the "correlation time" of adsorption and desorption);

- the critical RMC value ( $\text{RMC}_c$ ) at the intersection of the curves;

- the maximum reversible moisture content  $\text{RMC}_{\max}$ ;
- the minimum reversible moisture content  $\text{RMC}_{\min}$  considered 0;

- the normal range of the variation of the equilibrium moisture content is  $\text{RMC}_{\max} - \text{RMC}_{\min} = \Delta\text{EMC}$ ;

- the minimum and the maximum values of the first derivative curve,  $d\text{RMC}/dt = f(t)$ ;

- the minimum and the maximum values of the second derivative curve,  $d^2\text{RMC}/dt^2 = f(t)$ . These parameters allow the quantitative estimations of the two processes, and the minimum and the maximum values of the first and the second derivative curves, respectively, offer a qualitative estimation of both processes.

For the limewood specimens, the difference of approximate 260 years between the old and the new wood leads to characteristics changes (used as archaeometric characteristics):

- increase with 2.50 % for  $\text{RMC}_c$
- decrease with 37 h for  $t_c$
- increase with 5.80% for  $\Delta\text{EMC}$
- decrease with 600 h for  $t_{\max}$

Regarding the parameters for the two active preservation treatments of the old wood samples, the red petroleum increases the  $t_c$  with 17 h and the propolis with 13 h, a value which is closer to that of the new wood improving the hydrous stability (the reversible hygroscopicity varies in a longer time). The hydrous stabilization time ( $t_{\max}$ ) necessary to obtain the  $\Delta\text{EMC}$  increases, being closer to the new wood sample.

The minimum and the maximum values of the curve of the first and the second derivatives, respectively, allow to establish the stages of the adsorption and desorption processes of the reversible water for the new and the old limewood specimens. Thus, the first stage (the adsorption of the hygroscopic water at the external surface of the form of a monomolecular layer) is finished after 2 h and it reaches a maximum value between 20 to 40 h while the last stage (the formation of multi-molecular layers) reaches a maximum value after 120 h.

As a result of the treatments with the two organic components, the wood becomes more stable, not being affected by the sudden changes in the atmospheric humidity.

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