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INTEREST OF ELECTRICAL IMPEDANCE MEASUREMENT FOR MONITORING THE CONTROLLED AIR-DRYING OF WATERLOGGED ARCHAEOLOGICAL WOODEN WRECKS

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Summary:

There is no suitable NDT technique available for controlling the drying of waterlogged wrecks during conservation treatment. To permit such real-time assessment, an original non-destructive approach has been developed, involving a frequency analysis of the electrical impedance of the wood. Consequently, it is theoretically possible through a calibration curve to associate the impedance value with the amount of humidity inside the wood, even in the case of waterlogged wood containing more than 40% by weight of water.
After recalling certain aspects of impedance measurements, the principle of moisture content measurement is described. Experimental measurements were carried out on oak and beech samples from excavations at a French medieval site. The relationship between water content and impedance value at specific frequency is clearly established. Comparison between the impedance of the water solution containing the wood, the impedance of the wood/electrode contact and the intrinsic impedance of archaeological wood then demonstrates the feasibility and interest of this type of measurement. Examples of a calibration curves established for medieval oak and beech are then described.
At the end of this paper, impedance testing equipment suitable for monitoring wood drying is proposed. This apparatus, which is simpler than the frequential analyser used in this study, would function only at one fixed frequency; the error resulting from this assumption is estimated to validate this approach.

I. INTRODUCTION

The humidity of an artefact usually has to be monitored accurately from the archaeological excavation through to the conservation workshop, especially when the artefact contains waterlogged wooden parts. Indeed, waterlogged wood is very sensitive to drying effects: if precautions are not taken, artefacts can be irremediably destroyed during uncontrolled air-drying, leading to partial or total collapse of the microstructure. This collapse phenomenon leads to numerous serious deformations, cracks, surface undulations, twisting etc. After such a process the cultural object may lose up to 80% of its initial volume and the transformation of its shape makes it impossible to "read" the artefact intelligently, so that it is totally inappropriate for exhibiting.

To prevent the collapse of waterlogged wood artefacts, Polyethylene Glycol (PEG) permeation treatment followed by freeze-drying generally gives good results [1-2]. Nevertheless when the cultural object is a very large wreck (more than 10m long), no available freeze-drier exists to perform these operations. In these circumstances, only controlled air-drying can be applied. This method consists in drying the wooden object very slowly by controlling both the temperature and the humidity of the environment. The success of the controlled air-drying method depends to a great extent on the moisture gradient through the thickness of the wood, that is to say, the water contents of the surface and of the core. Usually, the surface is drier than the centre of the wood. To avoid a too steep gradient in the thickness of the wood , which may cause serious distortion, the drying operation is conducted very slowly because no efficient testing technique is available to control the moisture gradient perfectly; consequently, controlled air-drying may in fact last between 1 and 2 years, perhaps needlessly.

II. THE ADVANTAGES OF IMPEDANCE MEASUREMENTS

The only real-time and local technique that can be used is electrical resistivity measurement. Although this technique has been well known since the beginning of this century and applied to modern wood [3-6], it has only been applied to dry wood with a moisture content lower than 40% (by weight). Indeed high water contents in wood prevent any direct determination of pure resistance: the signal is not stable due to polarisation/capacitive effects [7-9]. The behaviour of a waterlogged wood is similar to that of an aqueous electrolytic solution containing ions.

Other techniques are available, such as microwave or high-frequency wave methods. But although these methods are suitable for waterlogged wood, they are general and not local, that is to say they do not enable any distinction to be made between moisture content in the core of the object and moisture content near the surface. These methods evaluate a sort of average between surface and volume water. However, this distinction between both types of water is essential to assess the water gradient in the object.

Another technique consists in weighing some parts from the artefact in question before and after drying, but this is not acceptable because it requires numerous samples to be extracted throughout the period of treatment.

Therefore, to avoid the above-mentioned problems, an original method was chosen for determining the pure resistivity of the wood by eliminating the capacitive effect of the electrical component in the impedance. This approach calls for a frequency analysis of waterlogged wood impedance [10-11].

Principle of electrical impedance measurements:

For a given system, the impedance Z is defined as the transfer function between the sinusoidal intensity response to a sinusoidal potential excitation. The impedance is a function of the ω pulsation common to input and output signals.

$$Z = \frac{\Delta V(\omega)}{\Delta I(\omega)} \qquad (1)$$

Amplitude of signal:

Linearity is assumed. If a Taylor development is considered (2), this means that degree terms 2 and upper are negligible. Only the first term, the fundamental term at frequency excitation, is kept. In our case, it is important to keep linearity at high frequencies, which correspond to the most interesting area.

$$\Delta I = (\frac{dI}{dV}) * \Delta V + \frac{1}{2} * (\frac{d^2 I}{d^2 V}) + ... \qquad (2)$$

The signal amplitude was fixed at 1 volt because this gives the best compromise: with higher values, there is a problem of non-linearity and the previous assumption is not valid, whereas a lower value becomes more critical because the signal/noise ratio is too small.

Frequency analysis:

Frequency analysis was chosen to determine the complex impedance of the system [10-11].

For high frequencies, the electronic system can become unstable (welded joint, etc.), whereas at low frequency, the system reacts in stationary mode with non-reversible variation. Classically, electrochemical phenomena are described in the following range: 10^{-2} Hz $\leq f \leq 10^4$ Hz. The frequency range adopted is [1 Hz-60 kHz].

III. EXPERIMENTAL CONDITIONS

Electrical configuration of wood/electrode/generator-analyser system

Two types of waterlogged wood were considered to be archaeological: namely not very degraded oak and very degraded beech from the Charavines-Colletières site near Grenoble (dating from the Medieval period). Two cylindrical needle-type electrodes made of stainless steel (Ø = 2 mm) were used to prevent corrosion problems. The samples consisted of wooden planks (1 cm thick) machined according to the fibre orientations depicted in Figure 1: the distance between both electrodes is set at 2 cm, and testing is performed perpendicular to the direction of the fibres.

To simplify the experimental configuration of this study, the electrodes go through the samples; but for a fruitfull use of this method, it is advised to proceed by recessed holes of different depths to monitor the water gradient in the thickness of the plank. To ensure a very localised measurement, it is possible to use electrodes with insulation membranes and conical shape. In this way, spot contacts are made only at the wood/electrode interface.

Electrode / wood contact

This point has to be emphasised to ensure a good interpretation of the results. Two cases have to be avoided absolutely:

1) Short-circuiting between both electrodes if a liquid film of water exists on the sample surface during the measurement. Figure 2 gives the resistance value of the solution containing the ancient wood: < 5 kΩ. This value is smaller compared with the intrinsic resistance of the wood (>10 kΩ).

2) A gap occurring between electrode and wood. Indeed, due to the natural shrinkage and possible local collapse of the wood during drying, it is possible that perfect electrode/wood contact is lost, leading to an artificial increase in the resistivity of the system. To solve this problem, a silicone oil and graphite powder-based paste is proposed, consisting of 50%-50% mixture by weight. This ensures a permanent and local low-resistance contact. Figure 3 gives the pure resistance of this silicone oil/graphite paste (<100 Ω, which is negligible in comparison with the resistance of wood: > 10 kΩ). Furthermore, if a gap is created at the interface, the fluid paste is able to fill it up, thus preventing the electrode from moving into it.

Results of impedance measurements

The principle of an impedance measurement

For a given sample, a measurement corresponds to drawing a curve -ImZ = f(ReZ) in a Nyquist representation by varying the analysis frequency. A function analyser was used, monitored by an computer. This apparatus enable a direct reading of ImZ and ReZ: ImZ corresponds to the capacitive component of impedance (dephasing of $\pi/2$ between intensity signal and voltage excitation) whereas ReZ represents the pure resistance component without dephasing. All the curves have a double half-circle shape (see Figure 4 with oak example), which is familiar to electrochemistry experts: the first half-circle represents the behaviour of waterlogged wood, the second half-circle depicts the wood/metallic electrode interface. The point M which is common to the two half-circles (ImZ # 0) gives the pure resistance of the wood without any capacitive components. This value can be easily linked to the water content. An equivalent electrical circuit can be drawn (see Figure 5) involving two R//C loops.

Before exploitation of this non-destructive method, it is necessary to assess the reproducibility of the measurement. The curves of Figure 6 demonstrate the satisfactory reproducibility of the measurements for oak and beech species independently of the chosen amplitudes: (50 mV, 100mV, 1V): all the curves can be superimposed for one sample considered.

To check the feasibility of NDT by the impedance method, several trials were performed at different stages of drying, that is to say with different amounts of water inside the wood. The exact quantity of water is determined by weight measurement. It is notable that the more water there is in the wood, the lower its resistivity: water facilitates ions mobility within the wood. Figure 7 despicts the Nyquist curves of beech case for the different steps of drying.

Another feature is the limitation of this method for still dry wood. It is not possible to use impedance analysis when the resistivity becomes too great. The signal/noise ratio becomes unfavourable. On the other hand, however, there is no limitation for a very high moisture content in the wood. There is thus perfect complementarity between conventional resistivity measurements for dry wood assessment and the impedance approach suitable for waterlogged wood with more than 40% water (by weight).

Figure 8 shows the "calibration curves" that can be plotted for medieval oak and beech by collecting all the measured points. It is stressed that a specific "calibration curve" must be established for each sample before beginning real-time monitoring of the moisture gradient during the drying of an ancient wreck. Indeed, the relationship between water content and resistivity depends to a great extent on the species of wood, its state of degradation, shape, dissolved salts in the wood, quantity of polyethylene glycol permeated in the previous conservation treatment, fibre orientation, morphology of the wood and so on.

IV. PROPOSED IMPEDANCE MEASUREMENT USING A FIXED FREQUENCY.

As stated above, the measurement is calculated from the minimum of the curve -ImZ = f(ReZ): point M. This minimum does not always occur for the same frequency, so in theory it is necessary to proceed with a frequency analysis for each resistivity assessment.

To avoid the systematic use of an expensive and complex frequency response analyser, we propose to work through a fixed frequency method to simplify the measurement. By making this assumption, certain errors are made in the measurement.

Considering the oak selected above, it is assumed that the fixed frequency is 5 kHz (this value is a medium one determined from our experiments). The moisture contents calculated with a fixed frequency and previously established calibration curve are compared with correct predictions of humidity by the weighing method (see Table 1).

It is noticed that frequency is not a sensitive parameter for moisture measurement by impedance assessment. Except for the point at 26% humidity, which is a limit for impedance investigation, all errors are less than 5%. Thus a probe using a fixed 5 kHz frequency could be a simple and suitable NDT device for measuring moisture locally in waterlogged oak wood.

With such a device for a given wreck that is to be dried, the experiment begins by establishing a "calibration curve" by using only a single representative sample of the ship, unlike the weighing method which requires numerous samples. Then, various electrodes are stacked in the wood at different spots and different depths. It is important here to maintain consistency in terms of type of electrode, distance between electrodes and fibre orientation. The electrodes will be read throughout the drying process. It will thus be possible to perform all necessary measurements directly on the ship to guide the controlled air-drying process.

V. CONCLUSION

Impedance measurements provide a promising and suitable means for assessing local humidity in waterlogged wood at different measuring depths. In particular, impedance assessment is entirely suitable for the [40-100%] range, as a complement to conventional resistivity measurements performed in the [0-40%] range.

This work paves the way for the development of specific low-tech testing equipment suited to the drying of large artefacts such as shipwrecks in the environment of a conservation workshop.

Further work will consist in using such an NDT method to monitor the drying of some oak parts of an ancient Greek wreck excavated at Marseille (5th century BC). This artefact is currently undergoing PEG impregnation treatment to strengthen it: the drying stage is planned to commence in the year 2000.

Another point to consider is the optimisation of the electrodes shape to diminish as far as possible damages of the wood (with thinner electrodes) and local analysis by using insulation membrane.

Other work will involve setting up a "calibration curves" data base, taking into account different types of degradation, other species, various shapes etc. With the help of such a data base, it is expected that there will eventually be no need to establish further calibration curves, which means sacrificing one piece of the artefact each time.

More generally, these preliminery experiments could demonstrate that impedance ananlysis is well-suited for determining water content in all types of very humid material. In theory, the principle can be implemented to ancient waterlogged leather, bone, rope and porous stones, with specific electrodes.

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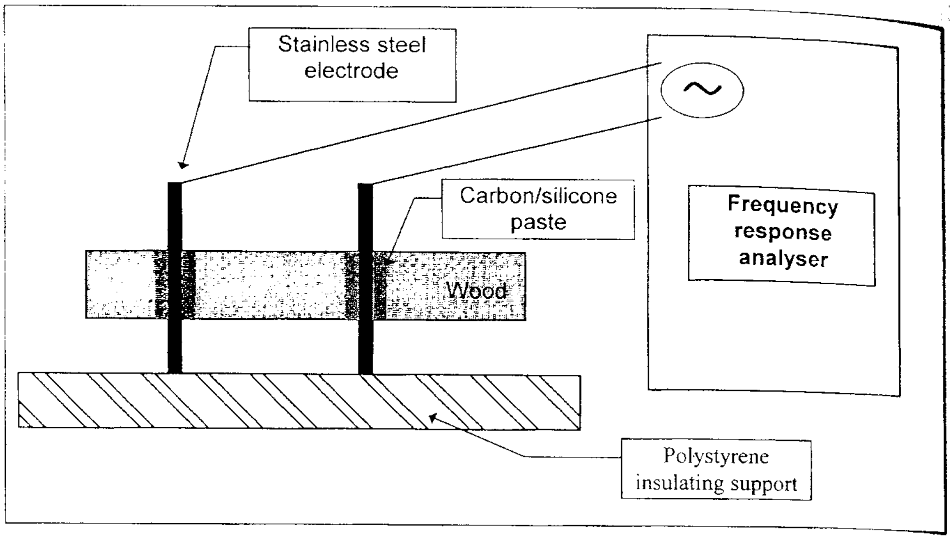

Figure 1 : Description of electrical circuit for impedance measurement.

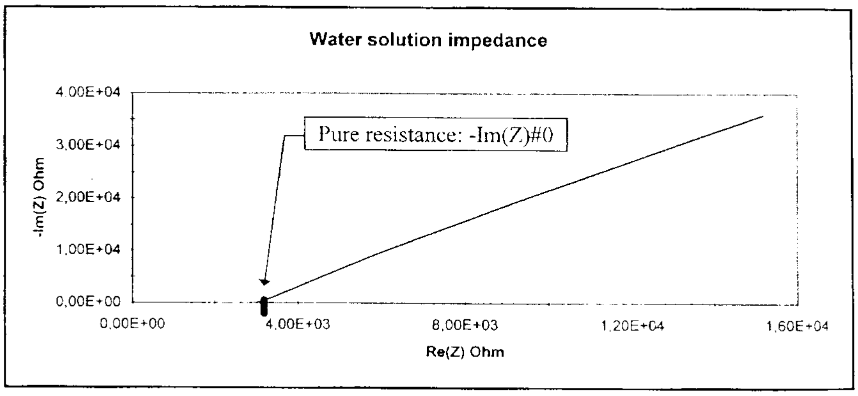

Figure 2 : Impedance measurement of the water without wood (water from the solution containing archeaological oak).

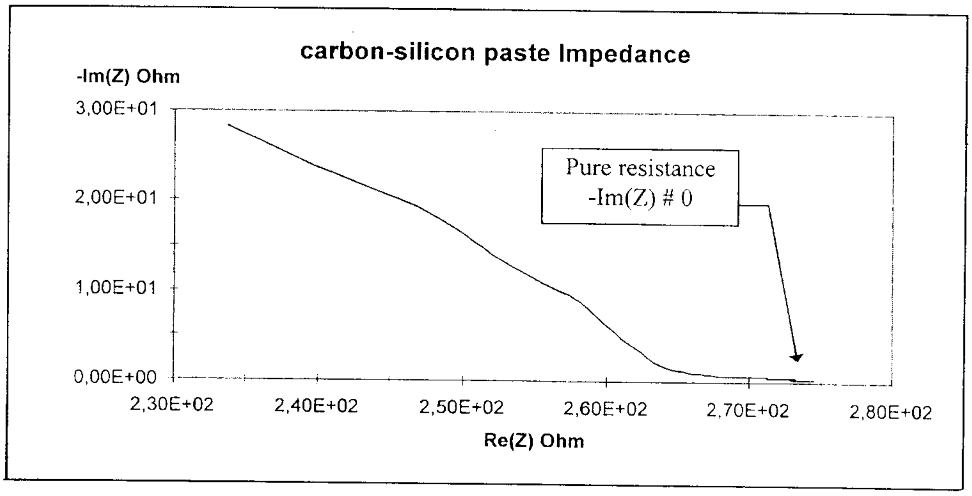

Figure 3 : Impedance measurement of the silicone/carbon paste used to fill up gap occured during the drying at the interface wood/metallic electrodes.

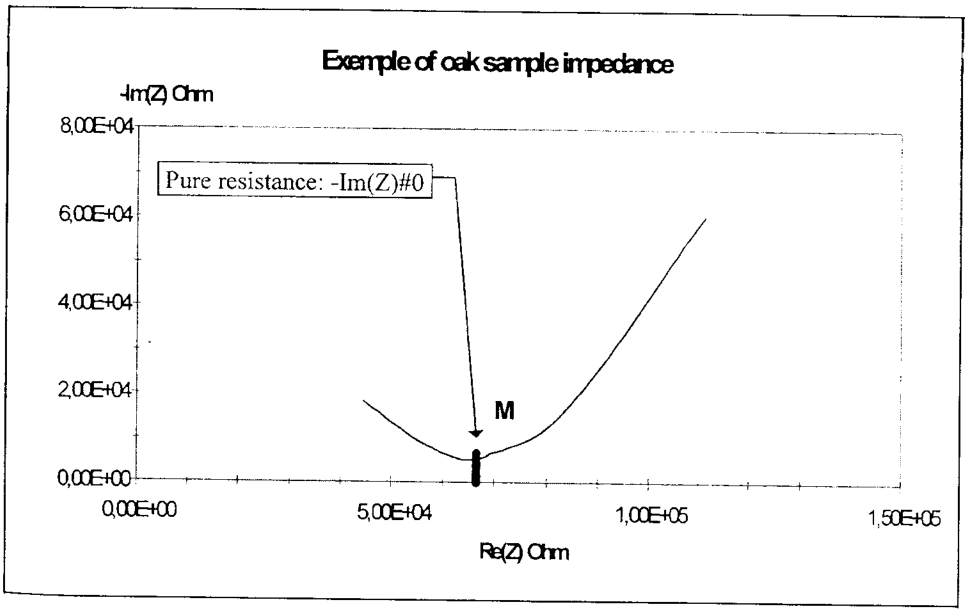

Figure 4 : Typical impedance curve encountered on ancien waterlogged oak of the Charavines site.

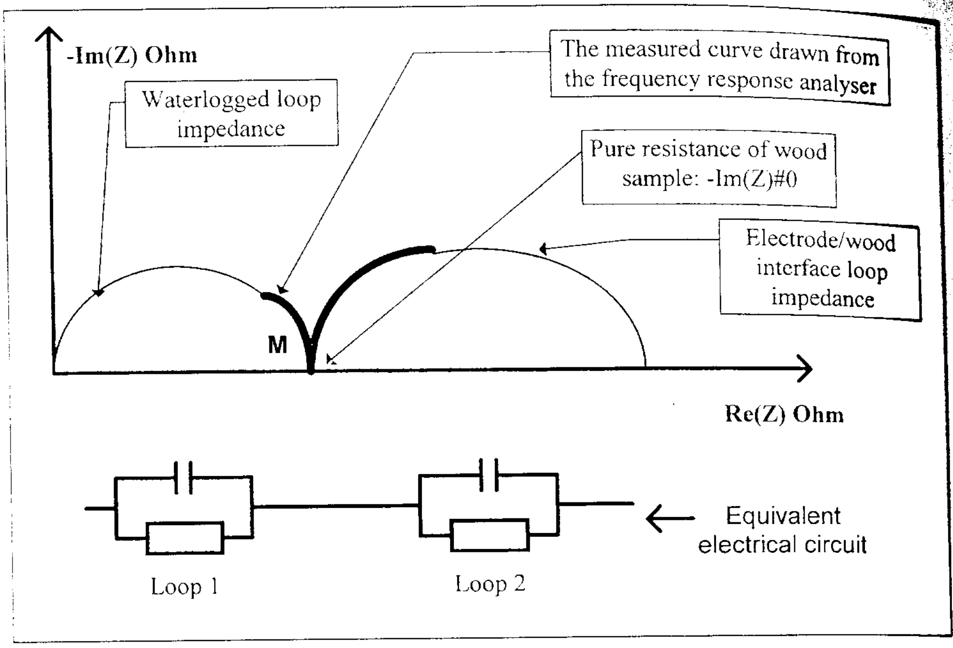

Figure 5: Typical Impedance curve shape obtained for waterlogged wood sample.

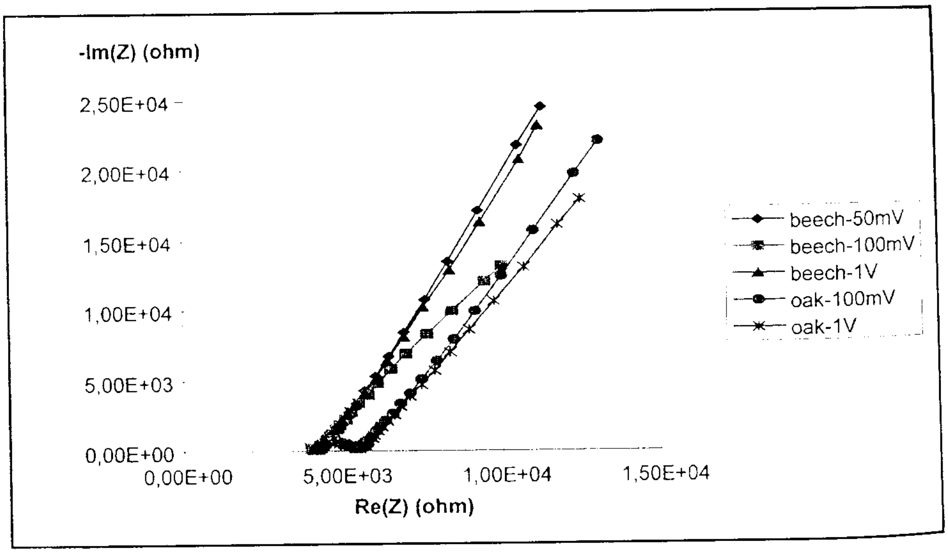

Figure 6: Resistance measurement versus signal amplitude: assessment of the reproducibility.

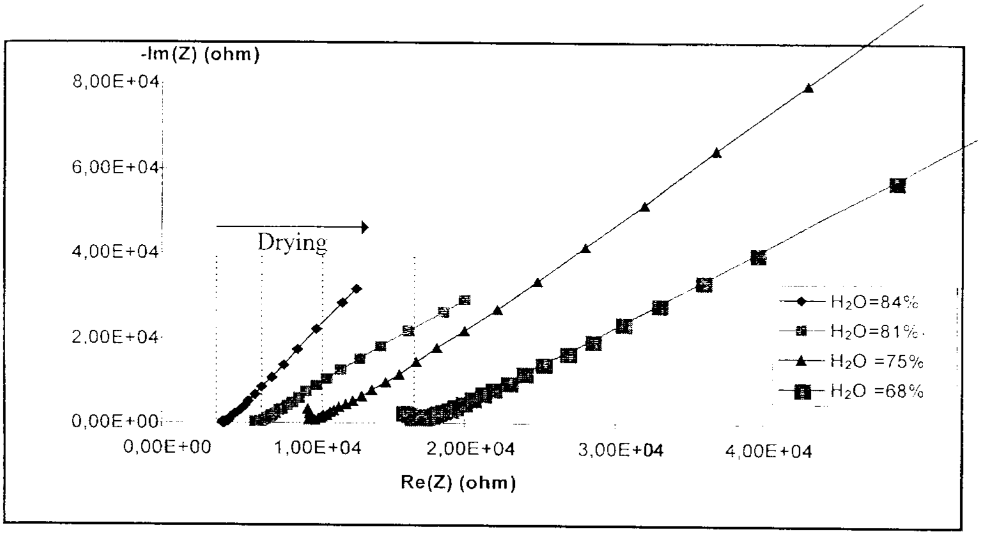

Figure 7: Examples of beech curves impedance versus moisture content.

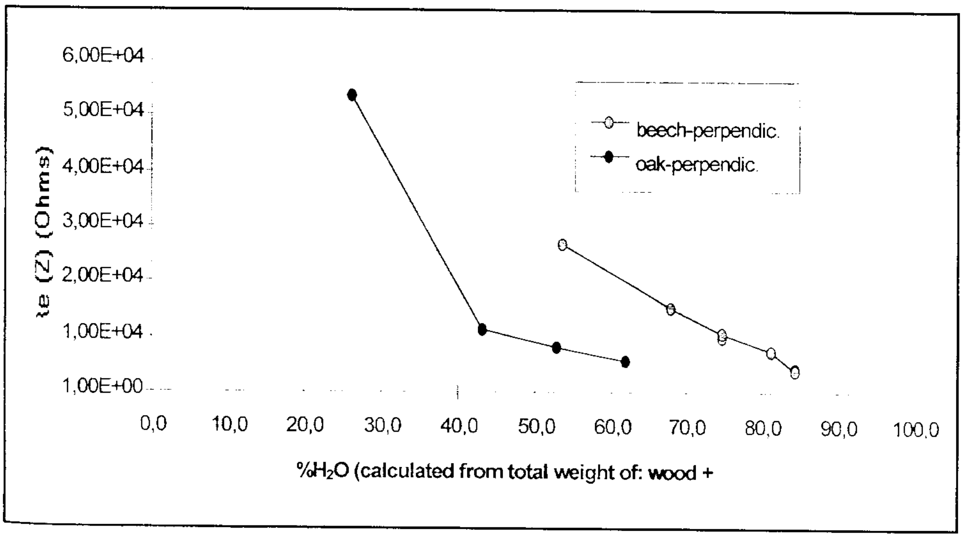

Figure 8 : Calibration curve for medieval oak and beech: moisture = f (pure resistance).

Table 1: Assessment of error made by using a fixed frequency (5 kHz) testing met

Humidity % H_2O by weight	Assessment from fixed frequency and calibration curve % H_2O by weight	Error %
62	63	+1.5
53	54	+1.8
43	43	0
26	28	+7.7