Determination of the Density of Small and Irregular-Shaped Samples of Sound and Degraded Waterlogged Woods

Authors: Ines Dorina Donato, Marcella Mulè Cascio
University of Palermo – Dipartimento Di Chimica Fisica, Viale delle Scienze - Parco d'Orleans II - Pad. 17, 90128 Palermo - Italy

Determining the density of wood can often prove to be difficult. Wood is a porous material which may include gases (i.e. air or water vapour), liquids (i.e. water or preservative agents), solids (i.e. salts, reinforcing agents), or substances that cause an alteration either in the sample mass or its volume. Humidity affects all the properties of wood, especially its density, so it is necessary to specify the percent of humidity in the wood submitted for density analysis.

If degradation has been provoked by insects or fungus, it is possible to determine the volume of a sample of known mass and afterwards measure the density of the damaged wood (Donato 2002). Determining density is more difficult if the degraded wood has been impregnated with water from submerged finds or wet sites as the sample undergoes a substantial shrinkage process during the drying phase. The evaluation of the degradation level is a fundamental parameter to start an effective conservation process; the experts in the field used to refer to a parameter named "conventional density," R, expressed in g/cm^3 and obtained by the followings:

$$R = (u_{max}/100 + 1/1.5)^{-1}$$

where u_{max} is the maximum humidity percent incorporated by the sample and 1.5 is the density of the cellular walls obtained by measurement of sound woods (Roger 1990).

Thus defined, the conventional density does not require a knowledge of the volume; it is based on the assumptions that all voids are filled with water, all the cellular structures shrink the same way (corresponding to the amount of water), and, ultimately, the density of the cellular walls is the same for all the wood species. Some experimental observations emphasize that the density of the cellular structures of wet woods is lower than that of sound woods. This is due to the loss of some substances related to the specificity of the residing place. The chemical composition, the percent of lignin, cellulose and hemicellulose and their densities are all meaningful issues.

Although the difference in the density of the cellular walls is generally small for woods showing high humidity levels, obtaining a rapid and accurate "effective" density of a small and irregular-shaped sample is difficult. The aim of this work is to develop a method for accurately determining the density of wood that is either sound or degraded, and of either small size or irregular shape.

The Experiment

The apparent density (d_{app}) is the ratio between the mass of a prismatic core sample in humidity environmental equilibrium condition, and its volume obtained by means of calibration; the density of the cellular structure (d_p) is given by the ratio between the mass of the sample and the volume effectively occupied by the cellular structure. In the past, pycnometers for solid materials used mercury to determine density, but mercury had the disadvantage of being highly toxic.

Using low surface tension liquids, such as ethanol, can lead to false results because of its penetration inside the wood, provoking shrinkage of the cellular cavities. Recently some modifications to the most commonly used techniques have been proposed: a) silica powder has been used instead of liquids and b) the sample has been coated with paraffin to avoid liquid absorption (Park 2000). Either way, the measurements have reflected scarce reproducibility and large mistakes.

Volume and density determinations of wood samples have been performed by the University of Palermo (Department of Chimica – Fisica) using the AccuPyc 1330 from Micromeritics. The AccuPyc

is based upon the gas (He) displacement technique. The sample cell of the instrument can be kept at controlled temperature; and it is possible to perform measurements on small irregular-shaped fragments whose weight is not lower than 0.5 g. Volume and density of the sample can be obtained with reproducibility of $\pm 2*10^{-3}$ g/cm^3. Thanks to the rapidity of the measurement, it has been possible to perform 30 determinations for each sample for a more appropriate elaboration of the experimental data. The samples were adequately prepared accordingly to either their nature (sound or degraded) and to the parameter to evaluate (d_{app} or d_p).

Methodology developed for the determination of the density of irregular-shaped sound woods for which it is difficult to determine the macroscopic volume.

The d_p value for samples of irregular shape is provided automatically by the AccuPyc. The instrument determines the volume and calculates density using the entered sample mass.

In the same way d_{app} can be determined provided the measurement is performed only after filling all the pores and cavities of the wood structures with proper blends of impregnating agents. The samples, previously weighed, were impregnated with a blend, 40% by weight of polypropylene glycol 425 in polyethylene glycol 1500 at 45 °C. Volume determinations were then made at 25 °C as the blend used became a solid. The effect of the impregnation is observed with a microscope, performing a "spot test in situ" with cobalt thiocyanate ammonium, while using an image analyser (Image, Pro Plus). No variation in the size of the sample subsequent to the impregnation is observed. The "real" volume of the impregnated sample was next determined by theAccuPyc. D_{app} is obtained by the ratio between the weight of the wood sample and its volume after the impregnation. In order to have good reproducibility of the measurements, it is necessary to quickly remove, prior to solidification, the external layer of the reinforcing agent so that the volume is only related to that of the impregnated wood sample.

Determinations of d_{app} have been performed over wood cores of various taxon; the values are consistent with the ones obtained by traditional methods, that is, the determination of the macroscopic volume of prismatic cores.

Methodology used for the determination of the density of wet degraded wood cores.

The samples of wet degraded woods are retained in deionised water at 4 °C, in sealed vessels to avoid contamination due to bacteria or spores. These woods are generally very difficult to treat due to their low consistency.

D_{app} is determined by the ratio between the mass of the wood material and the volume of the sample impregnated by water, as determined by the AccuPyc. It is possible to determine the amount of wood material contained in the core under analysis, symbolized by u_{max}. u_{max} is determined by the ratio between the weight loss of the sample due to dehydration (18 hours at 105 °C) and the mass of the dry sample.

Over the same sample, dried using the procedure described above, d_p is determined. In fact, using the mass of the wood sample, the instrument determines the volume and density. The determinations have been performed with samples of wet wood, coming from the lacustrine area of Fiavè-Carera and kindly supplied by Servizio Beni Culturali della Provincia Autonoma di Trento.

Results and Discussion

The volume of the cavities (V_c) and the porosity (Z %) percent are obtained from the values of d_{app} and d_p; Z %, defined as the ratio between V_c and the volume of one gram of wood (V) permits an estimation of the voids in the wood matrix:

$$V_c = (1/d_{app} - 1/d_p)$$

$Z\ \% = 100 * (V_c/V)$ from which $Z\ \% = 100 * (1 - d_{app}/d_p)$

Furthermore, it is possible to determine the effectiveness of the impregnation technique (E %) from the percent of the voids filled by the impregnant agent:

$$E\ \% = 100 * (m_{impr}/d_{impr})/V_c$$

where m_{impr} and d_{impr} are, respectively, the mass and the density of the impregnant.

The values of d_{app}, d_p, V_c, Z % and E %, obtained from measurements on core sound woods of

different taxon, are reported in Table 1.

In evaluating the results, it can be observed that differences between various species of woods are related to either the d_{app} values and the d_p values, confirming that these latter ones depend on the nature and the amounts of the substances of the cellular walls.

The porosity of the wood is not only that of the cellular lumen but also of the inner voids of the walls, of the micro fibril and the elemental fibril. The determination of this parameter is fundamental for predicting the permeability of the wood to liquid products such as preservatives, paints, and glues.

The percent of the voids filled has to be correlated to either the chemical characteristics of the impregnating liquid or to the porosity and the anatomy of the wood (omoxilo, eteroxilo).

Table 2 reports the values of d_{app}, d_p, Z %, u_{max}, R and Δ % relevant to the samples of degraded wood. One sample (not identified) is very much degraded and it has not been possible to identify the species.

In evaluating the results, it can be observed that the values of d_{app} of specimens of degraded wood are functions of the humidity percent values: the higher the values of u_{max} the lower the values of d_{app}. The values of d_p depend on the nature of the wood and on the level of degradation and it is not absolutely correct to consider only one value for d_p. The differences observed, Δ %, between the value of the density experimentally determined and conventionally determined have to be correlated with either u_{max} or the type of wood. Since the determinations require very small amounts of sample, it will be possible to perform them on various points of the body of the find in order to get more detailed information about its state of conservation.

Table 1. Data of sound woods.

Taxon	d_{app} gcm^{-3}	d_p gcm^{-3}	Vc cm^{-3}	Z%	E%
Fir Tree	0.505 ± 0.002	1.433 ± 0.002	1.28	65	41
Chestnut	0.558 ± 0.001	1.398 ± 0.005	1.08	60	48
Oak (not aged)	0.749 ± 0.001	1.354 ± 0.005	0.60	45	70
Oak (aged)	0.724 ± 0.001	1.413 ± 0.003	0.67	49	76
Beech	0.701 ± 0.001	1.427 ± 0.003	0.73	51	52
Beech (evaporated)	0.701 ± 0.001	1.396 ± 0.004	0.71	50	41

Table 2. Data of degraded woods.

Taxon	d_{app} gcm^{-3}	d_p gcm^{-3}	Z%	u_{max}	R	Δ %
Not Identified	0.080 ± 0.001	1.340 ± 0.001	94	1224	0.077	3.75
Oak	0.090 ± 0.002	1.390 ± 01770.002	94	1073	0.088	2.22
Beech	0.088 ± 0.002	1.418 ± 0.002	94	1095	0.086	2.27
Larch	0.168 ±0.003	1.410 ± 0.001	88	548	0.163	2.98
Pine	0,140 ± 0.003	1.441 ± 0.002	90	696	0.131	6.43
Yew	0.159 ± 0.003	1.372 ± 0.001	88	600	0.150	5.66

Conclusions

The results obtained show that the AccuPyc 1330 He pycnometer from Micromeritics is an ideal instrument for analysing those small wood finds. By this method, it is possible to remove only a small piece of material for which precise investigation can be performed.