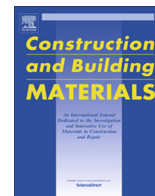




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Estimation of physical and mechanical properties of timber members in service by means of infrared spectroscopy

Anna Sandak, Jakub Sandak*, Mariapaola Riggio

CNR/IVALSA, S. Michele all'Adige (TN), Italy

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ABSTRACT

On site characterization of wood members is a very challenging task, after consideration of all the variables affecting the material used for the construction and the complexity of the structure itself. Current procedures are limited to few characterizations; in general based on visual inspection supported by the localized drilling resistance analysis and moisture content estimation. The goal of this work was to highlight the potential of the infrared spectroscopy as a tool capable of providing complementary information for the expert inspector assessing the timber structures. The paper presents several examples of successful application of mid and near infrared spectroscopy as used for prediction of physical and mechanical properties of wood. Requirements for implementation of mid and/or near infrared spectroscopy in routine assessment protocols are also provided.

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1. Introduction

The common method for evaluation of integrity in timber structures is based on the visual assessment. Numerous researchers have proposed the use of non-destructive and/or semi-destructive methods for timber characterization [1–6]. Some spectroscopic methods, such as X-ray fluorescence, time-of-flight secondary ion mass spectrometry (MS), reflectance spectroscopy in the UV–Visible range and Raman spectroscopy, can possibly support traditional assessment procedures. In particular, the recent availability of a wide range of portable instruments makes it possible to perform various spectroscopic measurements directly on site [7].

Infrared spectroscopy, in both near and mid ranges, is a further technique with a great potential for characterization of materials. It is especially useful for quantification and identification of various organic compounds, due to their selective absorption of the infrared radiation. Different chemical functional groups (possessing dipole momentum), such as $-\text{CH}$, $-\text{OH}$ or $-\text{NH}$ are excited to vibration states depending on the molecular configuration, chemical composition or the physical properties of the (sub)surface measured [8]. Consequently, the infrared radiation absorbed/reflected from the surface can be used for assessment of the physicochemical properties of the surface.

The preparation of samples to be measured with infrared spectrometers has been rather complicated and included solubilization of the sample or preparation of potassium-bromide (KBr) pellets. Such approaches, even if superior due to homogenization of the heterogeneous materials (such as wood) affects significantly the microstructure and micro morphology, changing dramatically the original properties of the material. It is especially noticeable in case of wood, as the long chains of wood cell wall polymers (such as crystalline cellulose) are destroyed often during milling or solvation. As an alternative to transmittance IR, modern Fourier transform infrared instruments equipped with attenuated total reflectance (FT-IR-ATR) accessory measure the infrared light multiply reflected between the sample surface and the ATR crystal. This technique allows measurement of wood powder or even thin wooden block surfaces without time consuming KBr sample preparation.

Some newly introduced infrared instruments are equipped with an external reflectance module and allow spectral measurement of large objects without the need for contact. This technique might be particularly useful during on-site analysis of timber structures.

Near infrared (NIR) spectroscopy offers even simpler measurement approach and its applicability for on-site measurement has been already investigated. The most important difference when compared to IR is in different spectral bands covered by both instruments ($4000\text{--}400\text{ cm}^{-1}$ (2500–25,000 nm) in case of IR and $12,000\text{--}4000\text{ cm}^{-1}$ (830–2500) for NIR), with the NIR region being located close to the red end of the visible spectrum (hence near infrared). NIR spectrometers equipped with fiber optic probes

* Corresponding author.

E-mail addresses: anna.sandak@ivalsa.cnr.it (A. Sandak), sandak@ivalsa.cnr.it (J. Sandak), riggio@ivalsa.cnr.it (M. Riggio).

allow direct solid surface measurement of samples up to a finite distance from the instrument (typically a few meters). The technique does not require particular sample preparation or hazardous chemicals for solvation, making it quick and reliable for quantitative and qualitative analysis. After suitable calibration, it is ideal for rapid material identification and is also a powerful analytical tool capable of accurate multi-component quantitative analysis.

A review on mid infrared studies (IR) on wood was published by More and Owen [9]. Several successful applications of near infrared spectroscopy (NIR) within wood science and technology have been recently summarized in two review papers [10,11].

Several researchers have investigated the evaluation of the physical and mechanical properties of wood, including prediction of moisture content, density, tensile strength, mechanical stresses, bending MOE and MOR. A particular focus of this manuscript is directed towards the practicality of in-situ measurements, despite most of experimental work to date being conducted in the laboratory environment. The specific objectives of this report were to:

- introduce the infrared spectroscopy as a tool capable of providing complementary information for the expert inspector in assessing timber structure;
- highlight the potentials and limitations of the proposed techniques;
- list key important issues to be considered when implementing infrared spectroscopy in routine assessment;
- define recommended measurement protocols for the estimation of selected physical and mechanical properties of timber members;
- provide examples of infrared spectroscopy application for estimation of physical and mechanical properties of timber members in service.

2. Strengths and limitation of infrared spectroscopy

Early IR spectroscopy was usually used to analyze solids, liquids or gases by means of transmitting the infrared radiation through the samples (transmittance mode). The sample preparation was time consuming and required (in case of solid wood) fine milling of the sample (destructive testing) followed by careful sieving and preparation of KBr pellets containing a small amount of the wood to be analyzed. The advantage of such approach was high homogenization of material and high quality of IR spectra, although it also meant very low sample representation as only a few milligrams from a large wood sample were used. Alternatively, the attenuated total reflectance (ATR) allows measurement without necessity of such particular sample preparation, while still allowing measurement of reasonable quality spectra. Some other advantages of IR-ATR technique comparing to other analytical techniques when applied to wood characterization are:

- little or no sample surface preparation (limited to refreshing, smoothing and/or cleaning);
- possibility to analyze samples that are too thick and/or too opaque for measurement in transmission mode;
- relatively fast measurement;
- measurements of solids, powders or liquids;
- possibility for determination of many chemical components simultaneously;
- high degree of precision and accuracy;
- spectral information covering the chemical fingerprint range;
- direct measurement with low cost;
- possibility for on-site measurement with portable instruments.

The restrictions of the IR-ATR technique are related to the limited dimensions of the window of the ATR crystal, pH constraints of

the ATR crystals themselves (possible chemical reaction with the experimental materials), the necessity for good contact/coupling between the sample and the crystal and again the relatively small amount of sample that is analyzed compared to the full sample, although multiple measurements on the sample surface are possible.

The near infrared spectroscopy (NIR) covers spectral range between IR (mid infrared) and visible light. Unfortunately it does not include the finger print region. The fundamental molecular vibrations appear in the spectra as overtones. However, NIR possesses some important advantages (in comparison to other analytical methods, such as IR):

- little (or no) sample preparation (conditioning, refreshing and surface finish);
- non-destructive or semi-destructive testing;
- fast screening capacity for on-site assessment (alternatively, more accurate characterization possible in the laboratory after sample conditioning);
- relatively fast measurement;
- no residues or waste solvents;
- possibility for determination of many properties/components simultaneously;
- high degree of precision and accuracy;
- direct measurement with very low cost.

The most important limitation of the FT-NIR is that the spectra are rather complex. Consequently, chemometric data evaluation is necessary for proper interpretation and understanding of results. The spectral resolution of the NIR spectrometers are limited (compare to mid infrared), thus limiting the spectra interpretation even more. The high sensitivity of the NIR instrument to the wood moisture variation is an important concern. Other factors, such as surface preparation, aging, weathering and roughness may influence the spectral outline as well.

Precise sampling criteria must be defined in case of assessment of timber structural members. It has to be optimized depending on the structure topology, member size, state of preservation and the purpose of measurement. It has to be also highlighted that any measurement of mid or near infrared spectra may provide information related only to the local wood characteristics and is limited to the subsurface of the member.

3. Methodology and protocols for routine assessment of wooden members with spectroscopy

3.1. Sampling and sample preparation

Representative reference sampling is essential when characterizing heterogeneous materials, such as wood/timber. Any sampling approach will cause further measurement errors and will limit the generality of assessment. It is impossible to totally eliminate such errors, but these impacts might be minimized by optimal selection of the number of samples/repetitions/measurement points as well as proper selection of the measurement locations upon the sample surface. Sample preparation and presentation may significantly affect as well the reliability of infrared spectroscopy, and particular the overall “quality” of the raw spectra (signal-to-noise ratio, scatter effect, variability between/within sample sets). Three alternative approaches for sample preparation while characterizing timber structures are as follows:

- on-site measurement with a portable device (non-destructive testing);
- laboratory measurements of representative solid wood samples collected from the wooden members, assuring due sample

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