Application Note AN M149

Quality Control of Fumed Oak via FTIR – Fast, Easy, Effective.

Introduction

To improve processability, durability and aesthetics of natural oak a special ammonia based fuming process can be used to tone color, reduce splintering and enhance its resilience against insect or fungal infestation. This makes fumed oak (or smoked oak) an ideal basis for enduring and resilient parquet flooring.

During the 'smoking' procedure, gaseous ammonia or a solution thereof interacts with tannins in the raw oak until the desired color (fig. 1) or properties are achieved. Since the presence of large amounts of residual ammonia in parquet (in the form of volatile ammonium salts) can cause critical complications during its installation, the wood needs to be thoroughly vented and, more importantly, checked for remaining ammonia. A variety of methods can be used for the latter purpose, yet they are time consuming or significantly large amounts of waste is produced during material testing.

One option is to store a piece of smoked oak of a certain size along with water or sulfuric acid in a closed-space vessel for a defined period of time. The time required can vary between 15 minutes and several hours per sample.

Keywords	Instrumentation and Software
Quality Control	ALPHA II FTIR Spectrometer
Natural Materials	Platinum Diamant ATR
Parquet	High Pressure ATR
Wood Industry	OPUS/Quant
Emission Analysis	

Figure 1: A pallet of wood before fuming with ammonia.

Since there is no direct contact between liquid and wood, the ammonia gas emitted by the sample is transported via the air into the liquid medium. By the subsequent use of an ammonium test method, it is then possible to measure the ammonium concentration of the solution and to determine the sample's emission.

Time-saving but unreliable is the attaching of natural wood onto freshly smoked oak. If there is a visible discoloration of the unfumed material at the point of contact, there was insufficient deaerating. Another simple option is the trial-and-error gluing of freshly produced parquet and the subsequent testing of the lift-off resistance. However, this takes several days to complete. Although other time-consuming and photo-based methods such as microchambers or test chambers (DIN 16000) provide good results, they, as well as the aforementioned procedures, are destructive test methods.

The Scheucher-Scan, a new and efficient technique based on FTIR technology and realized via the ALPHA II spectrometer is not only quick and reliable, but goes easy on resources as well.

Experimental

The measurements shown in this application note were performed using the very compact ALPHA II FTIR spectrometer equipped with the robust and dependable Platinum diamond ATR-module. Durable components, low upkeep as well as a low energy profile make the ALPHA II extremely efficient with incomparably small running cost. The ALPHA II shrugs off external disturbances like shocks and vibrations and always provides ideal measuring conditions. Weighing in at only 7 kg (15 ½ lbs.) its toughness and minimalistic size allow the user to move the ALPHA II safely between labs.

Today most routine measurements are performed with the ATR (Attenuated Total Reflection) technique, since it is much more comfortable to use than the conventional transmission mode and does not need elaborate sample preparation. Hereby the IR radiation penetrates slightly (a few microns) into the sample surface and the absorbance is afterwards measured by a detector.
Analysis is further simplified by the beforementioned easy to operate Platinum diamond ATR-module incorporating a hard-wearing and chemically inert diamond that can withstand even the most corrosive substances. With its ergonomic and failsafe one-finger clamp mechanism and 360° hinged pressure applicator, sampling is effortless even for staff without special training.

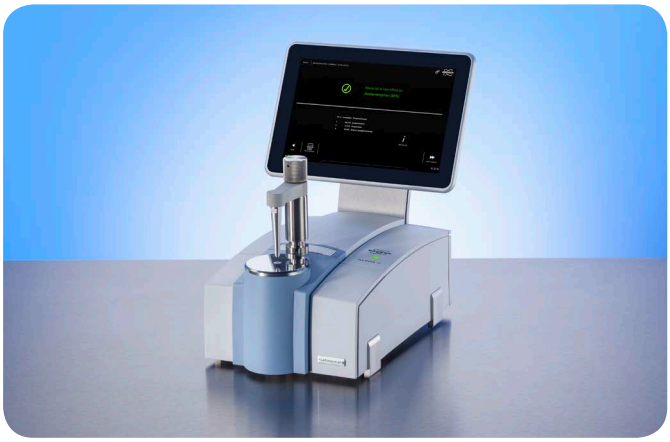

Figure 2: ALPHA II FTIR spectrometer equipped with the platinum diamond ATR and high pressure applicator.

Results

The presented application highlights the advantages of using FTIR to determine the residual ammonia content of smoked oak. While traditional tests destroy large amounts of fumed oak during the test, the loss of material by using the Scheucher-Scan is limited to just a few shavings (Fig. 3). The tested wood batch is kept unharmed and can be fully utilized for later processing. The time required per chip rarely exceeds one minute - including measurement, analysis and evaluation.

Figure 3: Workflow of the „Scheucher-Scan". A) Removal of a single chip of fumed oak B) Fumed Oak chip pressed onto the diamond ATR C) Used chips after analysis (waste).

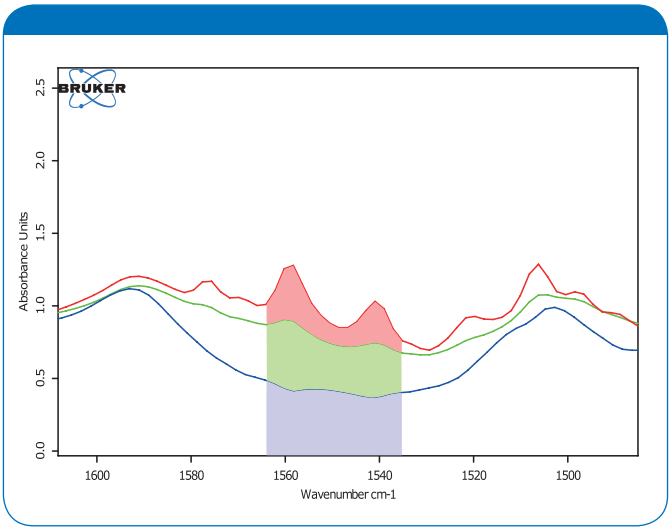

Figure 4: Spectra of unfumed oak (blue), as well as freshly (red) and ventilated (green) fumed oak. The relevant areas for method creation are highlighted.

The recorded spectral data show an obvious relationship between ammonia content and two IR bands at ~1560 and ~1540 cm^{-1}. In Figure 4, spectra of samples with high (red), low (green), and no (blue) residuals are compared. In simple terms: the larger the area under the associated curve, the more ammonia remained after smoking and venting.

To provide a reliable qantitative method, over 490 calibration spectra with different ammonia levels ranging from 0 to 13 mg were used for cross-validation. The residual content of ammonia was checked by measurement with micro chamber and test chamber (DIN 16000). The cross-validation possesses a prediction quality of 88% (R^2 = 87.56), is of good quality and allows reliable information on the ammonia content (Fig.5).

Conclusion

Scheucher, in cooperation with the WOOD K-PLUS, developed a resource-friendly method for the determination of residual ammonia in fumed oak based on FTIR spectroscopy. The analysis with the ALPHA II FTIR spectrometer is not only much faster than traditional methods, it can also be carried out by untrained employees. With an accuracy of almost 88%, the Scheucher-Scan satisfies the demands for extremely fast and uncomplicated analysis of smoked oak.

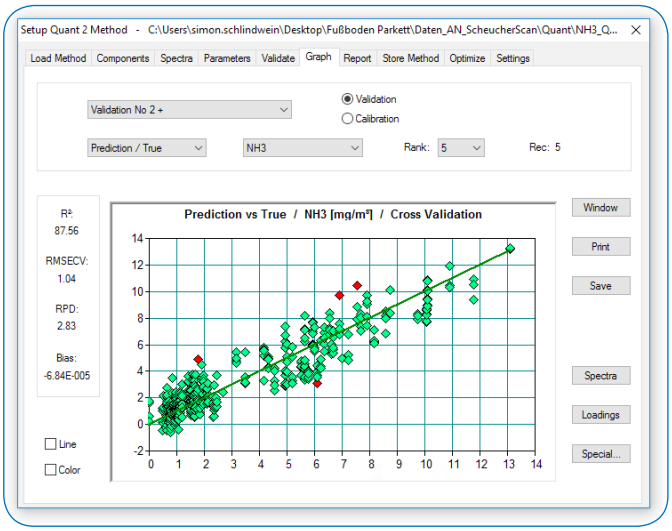

Figure 5: Cross validation of the prediction against the actual residual content of ammonia (determined by micro-chamber).

„Using Bruker's ALPHA II and FTIR spectroscopy, we were able to establish a new method that helps us to better understand the complex process of ammonia fumigation and which also allowed us to significantly speed up the testing of incoming goods. Because the tested product remains intact and it is still normally processable. Since there is almost no loss of raw material, we were able to increase the amount of tests performed, while still saving time.

A complete success thanks to the good support of Bruker!"

Klaus Bauer, Head of Development Scheucher Holzindustrie GmbH

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WOOD KPLUS

Reference

Hogger, E., Bauer, K., Höllbacher, E., et al. (2018). Time-dependent ammonia emissions from fumed oak wood determined by micro-chamber/thermal extractor (µCTE) and FTIR-ATR spectroscopy. Holzforschung, 0(0), pp. -. Retrieved 15 Aug. 2018, from doi: 10.1515/hf-2018-0042

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