Recognition of chemical changes occurring in wood subjected to treatment with some selected physical factors using near infrared spectroscopy

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Introduction

For some decades, ultrasounds has been used ever more widely and ever more often in many different areas. In industry, ultrasonic treatment is utilized not only for control and measurement purposes but also to cause relative acceleration of a number of physico-chemical processes in a given medium.

Ultrasounds have been applied also in some sections of wood technology, for example, in ultrasonic examination of wood,[1,2] intensification of the extraction process[3] etc.

Wood extraction is carried out in order to recover dissolved activity substances or to eliminate compounds which impede the process of wood digestion, using both conventional methods as well as modern techniques of biotechnology. The extracts obtained, e.g. tannin, can be utilized as components of phenol–formaldehyde resins.[4,5]

Wood is a very common, freely available and self-renewable material and, therefore, exceptionally reliable as a resource material.

The main objective of this study was to ascertain the usefulness of NIR in combination with UV in investigations of the effect of:

- ultrasonic field on the changes in the chemical composition of sapwood and heartwood as well as on isolated cellulose and lignin.
- the extraction process.

Studies recently completed in other laboratories have compared near infrared (NIR) and mid-infrared for analysis of lignocellulose,[6] and have assessed NIR for estimating the kappa number in paper.[7,8]

An earlier study in our laboratory indicated that NIR spectroscopy could be used to estimate the lignin, cellulose, pentosans and moisture content in beech wood, which in its natural condition was subjected to mycological degradation.[9]

Material and methods

Ultrasonic treatment

Oak wood was used to investigate the influence of ultrasound on wood. After separation of sapwood from heartwood, the wood was ground and sorted to obtain 0.5–1 mm fractions. Air-conditioned material was subjected to extraction by means of organic solvents (ethanol–benzene) in order to remove solutes. The material extracted was dried at room temperature until a constant humidity was reached. In both parts, sapwood and heartwood, the content of cellulose (using Seifert method) and lignin (by Tappi T-13m—54 method) was determined. For ultrasonic treatment a microorganism disintegrator with a roller concentrator of 24 kHz frequency was used. Ultrasound exposure was performed in an aqueous medium for the period of 1 hour, applying 32 μm amplitude. Next, the wood material was filtered through a foam crucible and dried at a temperature of 105°C until a constant weight was reached. The percentage concentration of weight losses, as well as lignin and cellulose content, were calculated in relation to absolutely dry material subjected to ultrasonic treatment.

Extraction process

Dried willow and birch wood were ground and then subjected to the extraction process. Water, at a temperature of 90–98°C, was used as the extraction factor. The wood was extracted for 3.5 and 7.5 hours. The obtained solution was thickened in an evaporator and then dried to a powder in a nitro-atomizer spray dryer.

Spectroscopic method

All NIR absorbance measurement (log 1/*R*) were recorded on a NIRSystems 6250 NIR spectrophotometer. Scans were made from 1100 to 2500 nm. To obliterate the effects of surface roughness, a second derivative was taken of the scans.

Absorption in the range from 200–400 nm was determined on UV/vis Beckman spectrophotometer.

Results

Figure 1 shows the 2nd derivative spectra of oak wood and its basic components: lignin and cellulose. While the exact structure of lignin is uncertain, there is known to be a phenolic hydroxyl group and carbohydrate groups present. It is quite evident that the region from 1640 to 1740 nm (strong peak at 1675 nm) allows us to observe changes in lignin content, while the region from 1300 to 1420 nm (peak 1365 nm) changes in cellulose content. If we compare NIR spectra in sapwood and heartwood (Figures 2 and 3) before and after exposure to ultrasound, it is possible to observe in them changes connected with the loss of cellulose and lignin. This was the first evidence that NIR could be used to observe changes in wood content. Exposure to ultrasonic treatment caused most significant weight losses in oak wood. They amounted to 6.71% in the sapwood zone and to 6.24% in the heartwood zone. Continuing the study, we isolated the lignin and cellulose and submitted them to ultrasonic waves. This investigation did not corroborate the high sensitivity of lignin to ultrasonic waves (small weight losses in the range of 1%) as seen with NIR (Figure 4). It was found that lignin of the sapwood zone undergoes degradation more easily than the lignin from the heartwood zone.

The loss of weight in cellulose fluctuated from 0.32% in cellulose isolated from sapwood and 2.6% in cellulose from heartwood. This difference in weight loss of cellulose mass depending on anatomic region, results from different degrees of cellulose polymerization.

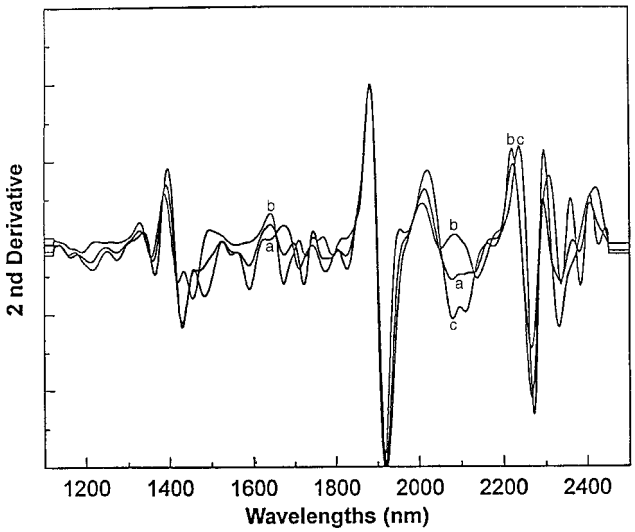

Figure 1. NIR second derivative spectra of (a) wood, (b) lignin and (c) cellulose.

The characteristic feature of ultrasonic treated oak wood composition from the heartwood region was a 3.2% reduction of cellulose in comparison with non-treated wood (Figure 3).

With the aim of assessing tannin content in wood, we performed the extraction process on willow and birch wood. Before and after completing the extraction process on willow and birch, concentrations of lignin and cellulose, as well as pentosans and tannin compounds, were determined (Table 1).

The extraction process turned out to have been too mild: the extraction yield obtained was only 1.6–2.9% for birch and 3.6–5.1% for willow. Powdered extract contained only 20.7% of tannins in the case of willow and 20.9% in the case of birch. The W spectrum (λ_{max} = 275 nm) indicates that substances that passed into hydrolizates were of the phenolic type. The extract obtained in

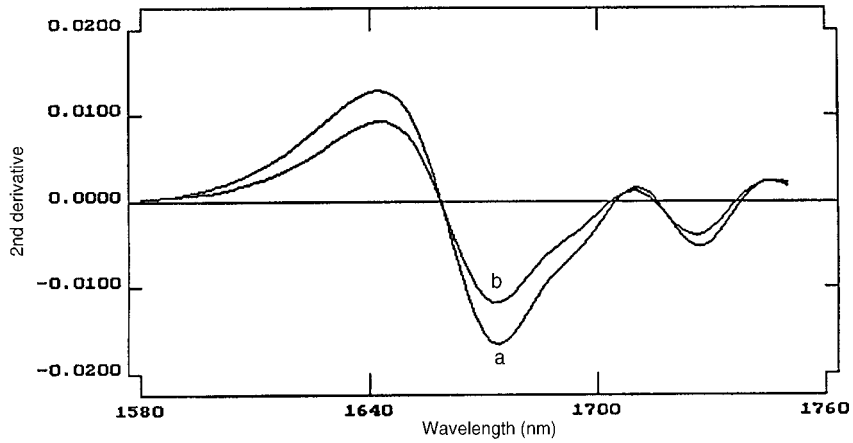

Figure 2. NIR spectra of the sapwood: (a) before and (b) after ultrasonic treatment.

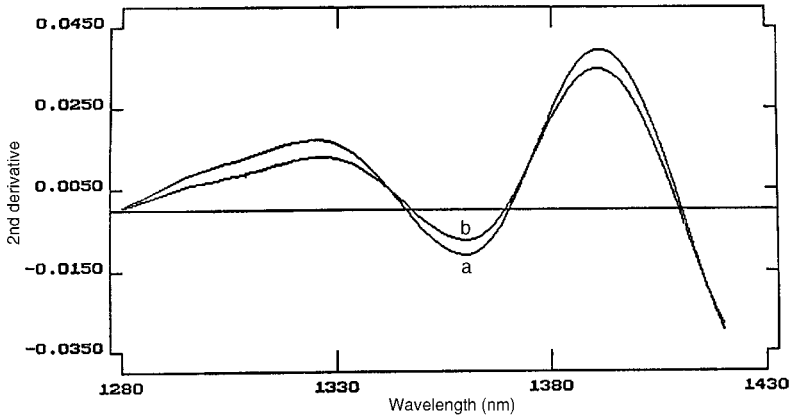

Figure 3. NIR spectra of the heartwood: (a) before and (b) after ultrasonic treatment.

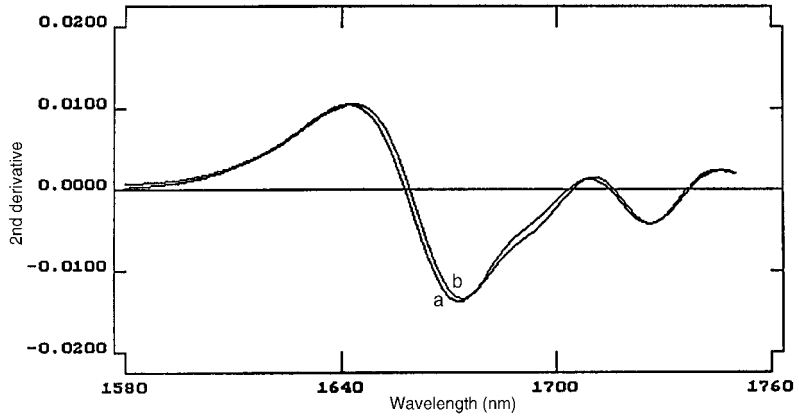

Figure 4. NIR spectra of lignin: (a) before and (b) after ultrasonic treatment.

this process did not appear to be the most economic substrate for the production of phenolic tannin–formaldehyde resins.

Poor efficiency of the extraction process was confirmed by NIR and UV spectra. No changes were observed in the basic constituents of wood substances, i.e. in the holocellulose and lignin systems.

The extraction process must be modified in such a way as to obtain a higher total yield. At the same time, care must be taken not to alter the concentration and structure of cellulose, the main wood component.

Conclusion

It was shown that it is possible to monitor quantitative and qualitative changes occurring in wood subjected to physical factors using NIR and UV techniques. Furthermore, the application of the above techniques allows, on the one hand, to optimize the process of ultrasonic treatment and extraction and, on the other, to recover tannins, which in conventional techniques, contami-

Table 1. Concentration of main components in wood before and after extraction.

	Willow (%)		Birch (%)	
	Before	After	Before	After
Cellulose	41.5 ± 0.8	41.4 ± 0.8	38.6 ± 0.8	40.6 ± 0.8
Lignin	22.6 ± 0.5	23.0 ± 0.5	27.6 ± 0.5	24.8 ± 0.5
Pentosans	17.2 ± 0.4	17.3 ± 0.5	20.4 ± 0.4	19.5 ± 0.4

nated the environment. Fast analysis time allows ultrasonic and extraction processes to be controlled.

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