Novel Process Control for the Resin and Panel Industries Based on FT-NIR

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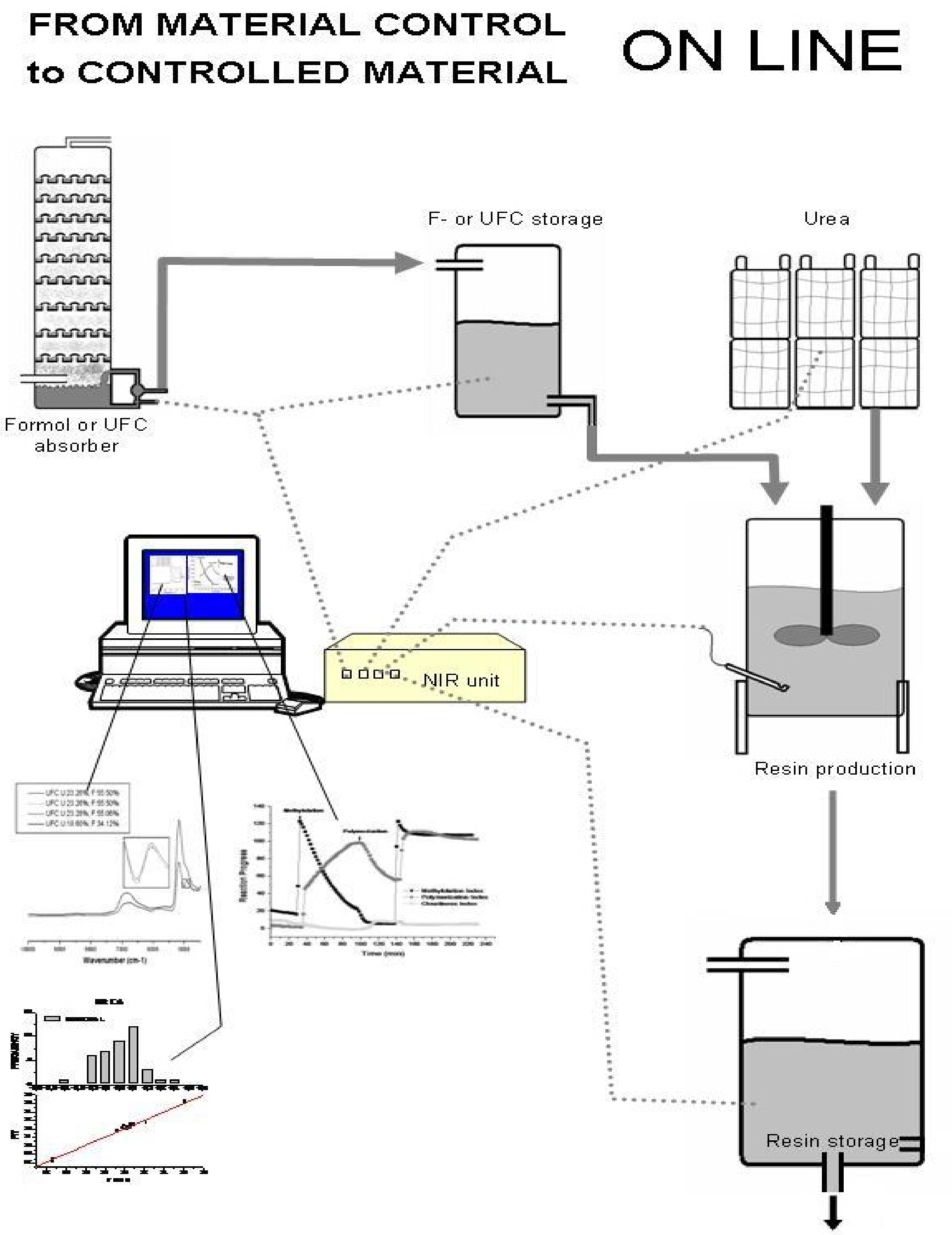
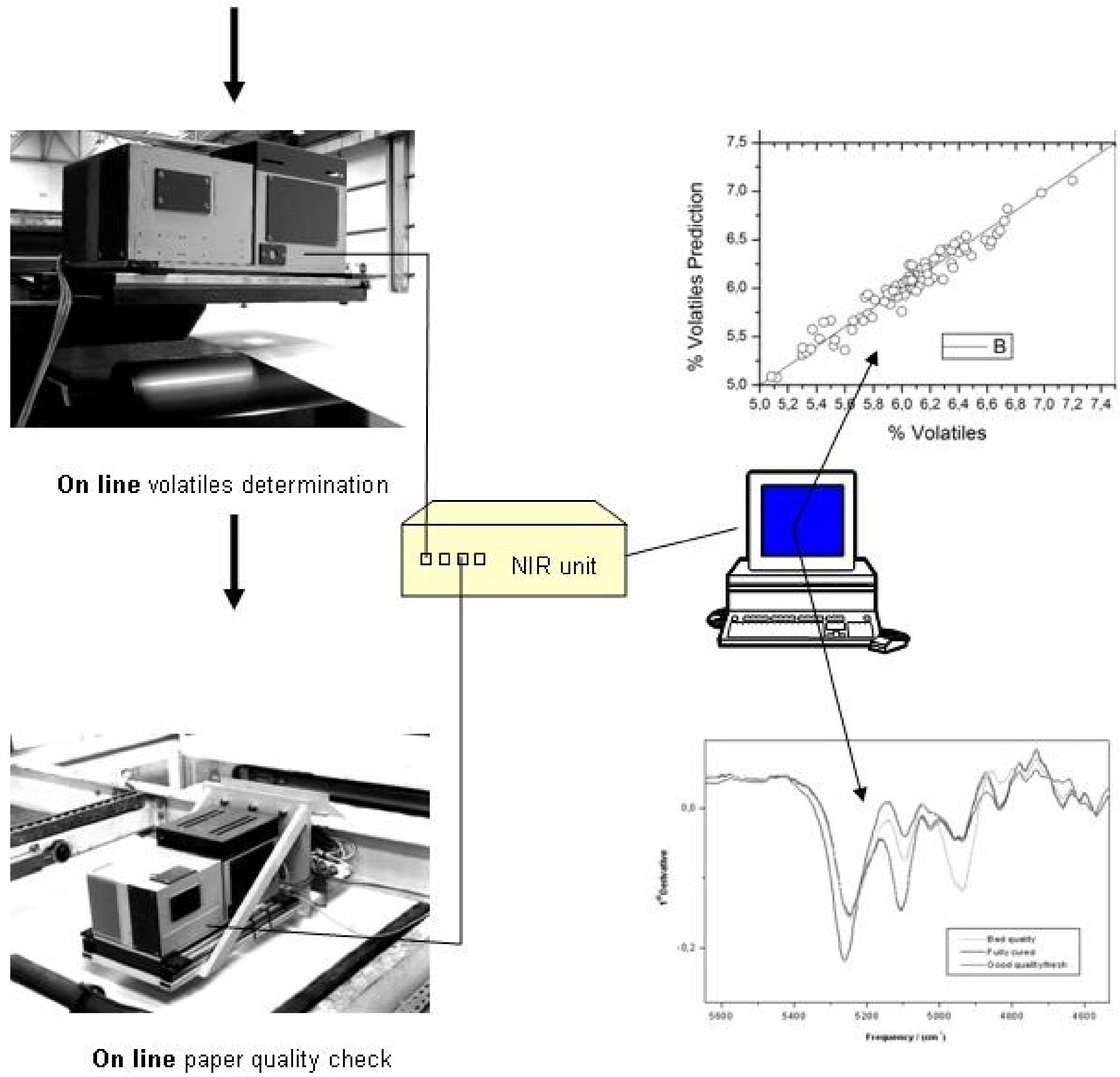
Summary

The present study overviews the progress made by Chimar Hellas on the NIR-based control of aminoplastics production and application processes. Methodologies, based on FT-Near Infrared (NIR) spectroscopy are presented. These methodologies were developed for raw material and final product evaluation as well as in-situ monitoring of formaldehyde-based resin synthesis.





1. Introduction

Over the years, aminoplastic resin systems, widely used as adhesives in the wood panel industry, have been the subject of extensive research. It has been shown that, due to their complex nature, systematic process control mechanisms are essential to ensure the reproducibility and the overall quality of this product. However, in the majority of resin industries, the process control is still based on empirical procedures.

During recent years, FT-Near Infrared (FT-NIR) spectroscopic techniques have been systematically developed, to monitor various processes. They allow spectra acquisition by light transmission through **intact** materials and thus permitting a rapid and non-destructive analysis (no sample preparation is needed). The use of optical fibres enables remote operation. In addition, new advanced computing hardware has enabled the coupling of NIR with chemometrics [1].

In the field of wood adhesives, NIR methodologies have been developed for the on- and off-line characterization of aminoplastic resin systems at laboratory and industrial scales. New methods target the quantitative analysis of urea and UFC solutions [2-4] as well as the on line monitoring of the synthesis of formaldehyde-based resins [2-4, 6]. These methods were proven particularly useful in cases where the reproducibility of production was difficult to assess. Moreover, NIR has been used for the on line measurement of the volatile content in laminating papers during impregnation with formaldehyde-based resins. The usability of laminating paper has also been determined with NIR chemometrics in a semi quantitative manner [4-5].

The above-mentioned methodologies [2-3, 5] are patented by **CHIMAR HELLAS S.A**. and are marketed under the trade name **GNOSSI** (**G**eneral **N**on-destructive **O**n-line **S**pectro**S**copic **I**nterpretation). They have been successfully applied industrially for the **on-line control** of urea–formaldehyde pre-condensate production , the production of formaldehyde based-resins, the determination of the volatiles content in paper impregnating lines and the analysis of impregnated paper prior to lamination. The purpose of this paper is to present the current progress on the NIR applications in the wood resin industry.

2. Experimental

Spectroscopic Techniques

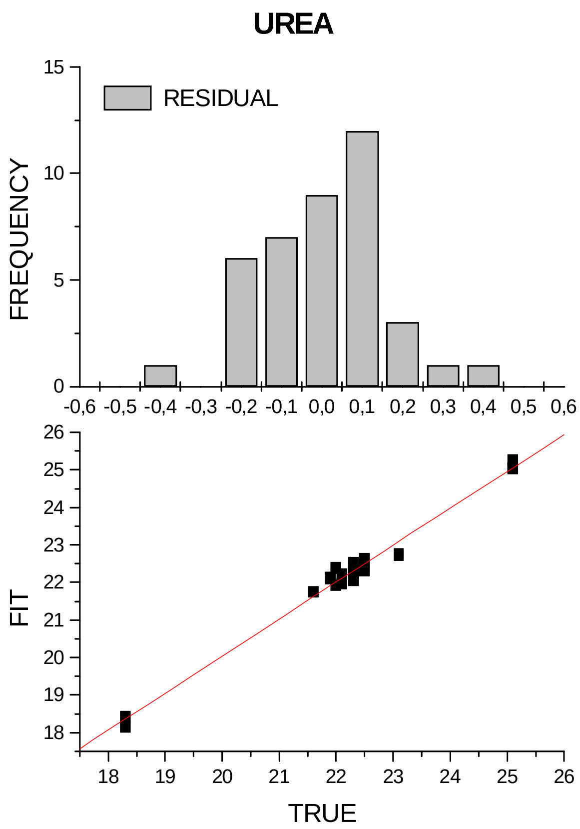
The near-infrared spectra of liquid samples were acquired via a FT-NIR spectrometer (Bruker Vector 22/N) in fiber-optic data acquisition mode. Typically the spectra represent averages of ca. 150 scans (acquisition time 1.5 min). A quartz single fiber optical cable coupled to a transmission probe (Hellma QX, 1.00 mm optical path) was used for liquid samples. The total length of the fiber was 2 m for laboratory measurements and 40 m for industrial measurements.

Impregnated papers were measured in the diffuse reflectance mode via a remote sensing spectrometer (Bruker Matrix E).

Data treatment and chemometric multivariate analysis were based on subroutines available in the Bruker-Opus software.

3. Results and Discussion

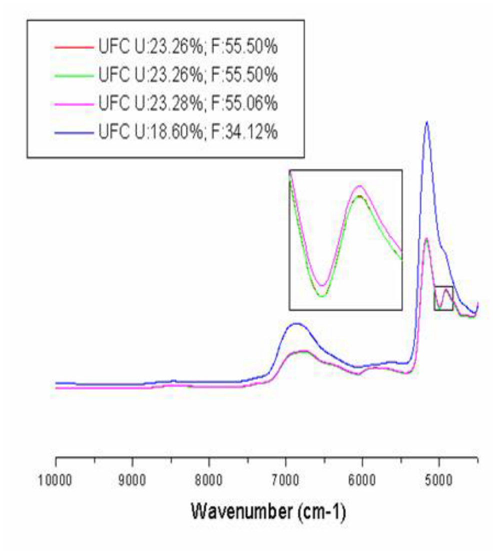
a. Raw Material Characterization



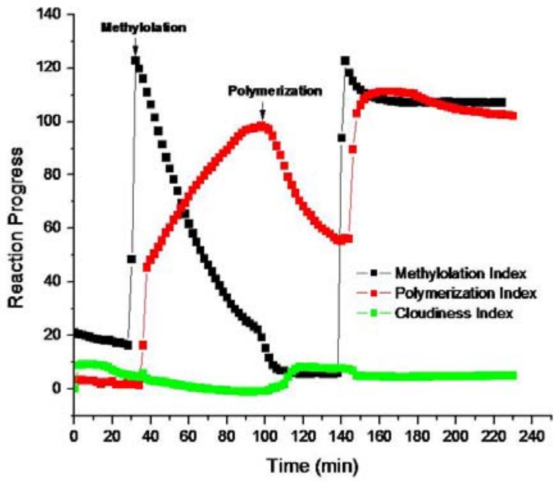
One of the factors affecting the reproducibility of the industrial synthesis of aminoplastics is the quality of the technical urea used. Depending on its origin, solid technical urea contains impurities. NIR chemometric methodologies for the quantitative determination of urea in a wide concentration and temperature range (aqueous urea solutions with 0-50 % w/w urea at temperatures 25-50 ^{o}C) have also been developed resulting in 4-rank models predicting the content of urea with R^2=100.0% and RMS error of prediction equal to 0.04 % w/w (see figure beside) [4]. Application of such models for the evaluation of technical urea, sampled from several industrial sites, gave predictions of the pure urea content ranging from 96.8-99.6 % w/w. Taking into consideration the fact that in industrial calculations urea is assumed to be 100 % pure, it can be easily noticed that a significant error may be introduced in % solids or molar ratio calculations. This fact is very important for the very low molar ratio resins that are used today.

NIR has also been employed in the on- and off-line determination of the **composition of urea-formaldehyde concentrates (UFCs)**. To quantify complex multicomponent systems such as UFC, multivariate calibrations were created. The resulting models required up to 6 ranks to account the chemical richness of this system with RMS errors of prediction equal to 0.7 and 0.17 (R^2=99.9 and 98.4%) for formaldehyde and urea respectively [2-4].

The industrial scale application of the above models has proved to be extremely useful in controlling more accurately the molar ratio and % solids content of aminoplastic resins and related products.



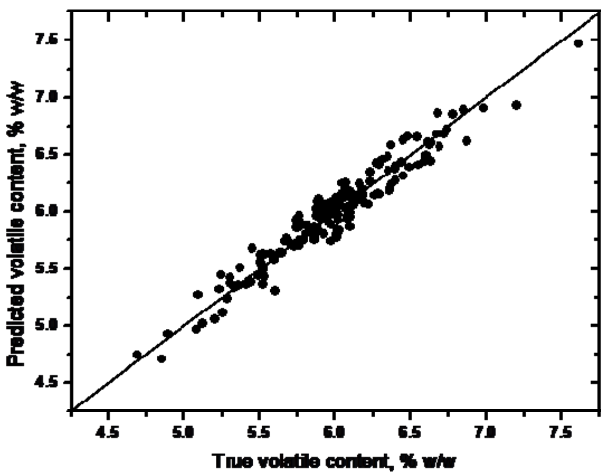
b. Resin Production



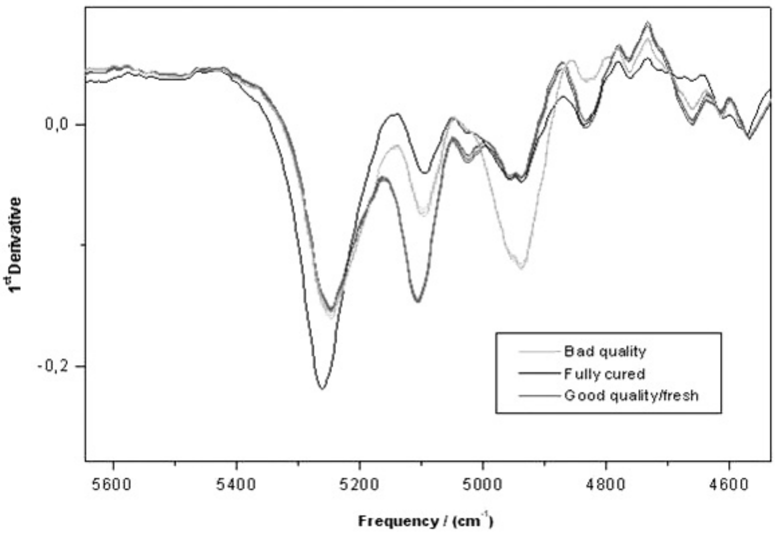
The time evolution of characteristic bands in the NIR frequency range has been employed for the on-line investigation of the reaction stages of real lab and industrial synthesis procedures [2-4, 6]. Monitoring is based on a number of indicators deduced in real time from the NIR spectra. Some indicators (such as the cloudiness index) have a straightforward physical significance. Others, especially those monitoring the kinetics of **methylolation** and **polymerization (condensation)** stages, are considerably more complicated, defining phenomenological reaction pathways that accurately describe the production stages (Euclidean and absolute frequency indices, see figure above). The **rate of methylolation and condensation** can be monitored on line, allowing preventive actions from the side of the resin operators. The on-line monitoring of aminoplastic resin synthesis by NIR spectroscopy indicators has been tested successfully in **industrial applications**.

c. Laminating Paper Impregnation

The NIR chemometrics have also been used for the determination of % volatiles content of laminating paper during production. Multivariate calibration models (8-rank models with R^2=93.3% and RMS=0.12) were created covering a wide range of industrial paper types (340 industrial paper samples of 30 different paper types, see figure beside). Installation of this spectroscopic system in impregnation lines provides real time determination of the % volatiles in the impregnated paper, permitting a faster and more responsive adjustment of the impregnation process.



d. End Products – Laminating Paper



For the evaluation of laminating papers a quick and reliable conformity test has been developed based on NIR spectra acquired from paper samples in the diffuse reflectance mode. An empirical usability index has been built to detect the state of impregnated papers in a semi quantitative manner and report on its ability to be used [4-5].

4. Conclusions

NIR-based methodologies developed by Chimar Hellas S.A. for on- and off- line characterization have successfully been applied in the industrial production, due to their advantages, to ensure its quality and reproducibility.

NIR applications on raw materials concern the determination of urea and formaldehyde in aqueous solutions and the composition of urea-formaldehyde pre-condensate in terms of % w/w urea, % w/w formaldehyde, F/U molar ratio and % w/w solids content, leading to aminoplastic resins and related products with more accurate molar ratio and % solids content.

Real time monitoring of resin synthesis by checking indices of methylolation, polymerization and cloudiness examines the influence of raw materials, heating rate, pH and temperature and allows more responsive adjustments in the process line.

A PLS chemometric algorithm was developed for the prediction of the % w/w volatile content in laminated papers. This algorithm is based on the NIR reflectance spectra of several industrial paper samples. The method serves as an integral part of the production line to provide on-line quality control of the impregnation process. The evaluation of final products includes the usability of laminating papers by detecting the state of impregnated paper and reporting on its ability to be used.

The NIR methodologies introduced by Chimar offer a significant advantage to the resin and panel industries in improving the quality of their products.

5. Acknowledgements

The contribution of :
Dr. E. Dessipri and Dr. J. Prinos of Chimar Hellas and
Dr. G. Chryssikos and Dr. V. Gionis of National Hellenic Research Foundation
is greatly appreciated.

6. References

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