

Using NIR Spectrometry for Direct Control of Recovered Papers

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(Received September 4, 2007: Accepted November 15, 2007)

ABSTRACT

This paper sums up all the different steps broached in this project :

The NIR spectroscopy technique has been studied and implemented at CTP using a mobile spectrometer device and different optical materials.

Methods, based on statistical data analysis (in particular PLS regressions), have been investigated.

A laboratory "prototype" using these techniques and methods has been developed in order to control the recovered papers quality, in terms of humidity percentage and sample composition (paper, board, contaminants).

Keywords : *NIR spectroscopy, recovered paper, composition, humidity*

1. State of the Art

For papermaking, the use of recovered papers and boards as raw materials is increasing. As a consequence, we assist more and more at an all-out paper collection and the quality of the recovered paper bales is often very variable. In order to standardize these collections, it exists an European norm called EN643. Its aim is to classify precisely the deliveries of recovered papers and boards using several criteria, essentially based on their content: sorts of papers / boards, respective ratio, origin, aspect, presence of contaminants and quantity. And an other crucial interest for papermakers is the measurement of humidity, because of economic reasons. Indeed, the

price of a recovered paper bale depends on its EN643 classification and its weight, taking into account the humidity percentage. And to pay water instead of raw material is not very interesting for papermakers.

Nowadays, the control of the recovered paper bales at delivery, in order to measure humidity, presence of contaminants and more generally for coherence with EN643 standard, is essentially done by an operator using visual and/or manual procedures (see the procedure proposed by PTS to control the sorts 1.02 and 1.04 at delivery). For instance, to control the compliance with specifications and conditions (EN643 standard) and the presence of contaminants, only visual appreciations of general aspect of a bale are often used. Very seldom, they done a bale bursting and

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a very long manual sorting. Another example is for humidity measurement: they perform a core sampling of the bale (using a core-drilling device for instance) in order to use then classical manual procedures to obtain the humidity percentage (gravimetric method). A capacitive gauge can be also used to control moisture content.

In conclusion, generally speaking, current procedures for control of recovered papers are manually made and therefore time consuming, tiresome and furthermore poorly objective. Soon only few controls are possible and these "methods" have a very low efficiency. As a consequence, papermakers would be certainly very interested to own an easy-to-use device allowing direct controls at delivery, rapid, objective and reliable.

2. Objectives

In order to develop a "sensor" able to do all these direct controls, the CTP is working on recovered paper bales characterization using a new (at least in papermaking) technique called "Near Infra-Red (NIR) spectroscopy". More precisely, the aim of this project is to determine, for each recovered paper bale at delivery, its composition in term of raw materials (paper / board), contaminants (metals, plastics, etc.) and to measure its humidity content. And our final purpose will be to integrate all these developments into an industrial sensor based on a coring device.

The objectives are at different levels. Firstly, the possibility to control efficiently the bales at delivery should allow paying raw material at its right price. Secondly, it's possible that the anticipated knowledge of the raw material quality should allow a better optimisation of the treatments and the productivity. Finally, the using of an "objective method" should allow assuring better relationships between the suppliers (sorting centers) and the customers (papermakers).

3. Perspectives

First trials have been carried out in laboratory and the results are very encouraging. As a consequence, the next step will be to automate these treatments in order to be able to implement these trials to an industrial sensor based on the instrumentation of a coring device.

Finally, the last idea will be to use together the results given by this project and the results obtained with others works currently done at CTP (image processing in visible spectrum (1), volatile contaminants analysis using mass spectrometry (2)) in order to obtain a real "identity card" as precise as possible of each recovered paper bale: ratio of papers, newspapers, magazines, boards, flats, corrugated, brown, grey, contaminants, water, and so on.

4. Results and Discussion

4.1 NIR spectroscopy and data analyses: generalities, principles and main advantages

4.1.1 Generalities

The Near Infra-Red spectroscopy has been already used since several years in different sectors like food processing, chemical or drugs. Indeed, it is a very powerful technique to study precisely and quickly the composition of samples. In particular for humidity measurement, it is well known that NIR spectroscopy is really fine-tuned. That's the principal reason why we have decided to invest in this technology.

As for papermaking sector, it is really an "emerging" technique since two or three years (3), and it is definitely a very "promising" technology for the future.

4.1.2 Some principles

This technique is based on the measurement of reflected light (in our case) given by a sample. First of

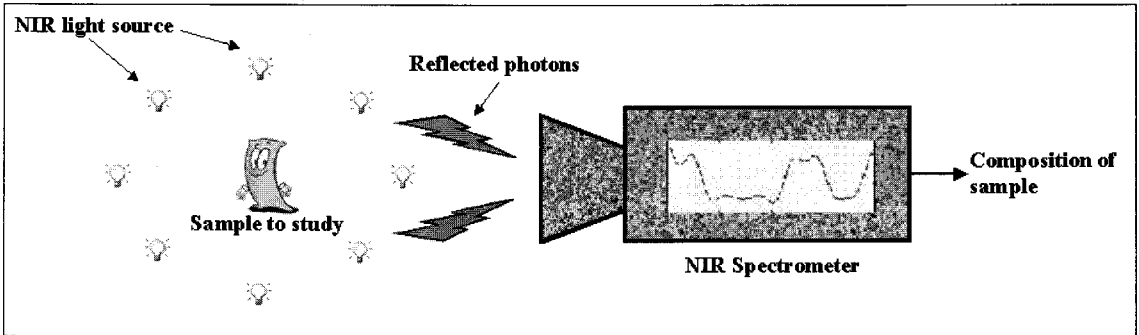


Fig. 1. General principle of NIR spectrometry.

all, the sample to study must be enlightened by a light source with a wavelength (λ) range between 800 and 2500 nm. Then, the light reflected by the sample is collected by a detector and transformed into a spectrum. It is the role of the spectrometer (Fig. 1).

A particularity of the NIR spectrometry (compared to other spectrometries) concerns the exploitation of the spectra. Indeed, it is very difficult to use them directly and often we need data analyses methods in order to develop multi-linear statistics models to predict wanted outlet data (Fig. 2).

Finally, these methods need to do a very important step of calibration in order to obtain the coefficients of the model. To do this "learning", we need to know, for some particular samples, theirs spectra and the outlet data corresponding. Using together the inlet and the outlet data, we can build the prediction model.

4.1.3 Main advantages

The main advantages of the NIR spectrometry are:

- to give data of the composition of samples
- no need of sample preparation
- non destructive technique and, above all for our

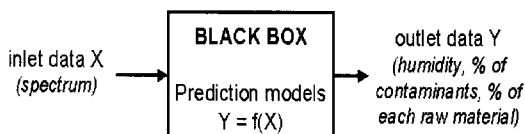


Fig. 2. Prediction models.

project, very rapid measurements.

4.2 The equipment used at CTP

For most of our applications (that is to say the integration of a spectrometer into an industrial sensor), we have decided to look for a mobile spectrometer device, relatively cheap and with the most extended possible λ range. The Fig. 3 shows our spectrometer. It is a portable one, with an USB interface (available in ISA card format too) and with a module diodes detector InGaAs extended (1100 – 2200 nm).

Then, the light source is a simple Halogen-Tungsten source, with a TTL shutter which can be controlled by software.

Finally, we also use an optical fibre with 6 lighting fibres covering 1 fibre collecting the reflected signal



Fig. 3. Our portable spectrometer.

(400 μm cores).

Furthermore, for the data processing and to build our prediction models, we have decided to develop our own algorithms using Matlab before implementing them in C language. We use essentially factors analysis methods like PLS regressions (4) and more precisely, PLS1 algorithm for humidity prediction and PLS2 algorithm for contaminants and raw materials. Finally, for each model, we have done a "samples database" for calibration, with one part of these data to learn the model and the second part to test its precision.

4.3 Humidity study in laboratory

The learning procedure of model needs to have samples with different humidity. In this view, we have used fluted roll for corrugating paper and we have created different humidity point (7 in all) from a lab coater (a Dixon available at CTP). Then, for each 7 samples obtained, we have done an acquisition of NIR spectrum using our spectrometer, and an accurate traditional measurement of humidity (weighing, complete oven drying, re-weighing).

The spectra given by our spectrometer and the corresponding humidity percentages obtained by the standard method are on Fig. 4.

Two remarks on this graph. Firstly, we have done a vertical re-adjustment of spectra on the λ range [1150 nm; 1350 nm]. So, we avoid problems depending on the variation of the distance between the sample and the measurement head. Secondly, to reduce the noise during prediction, we have decided to keep only λ upper than 1350 nm. Furthermore, we can notice that two "bands" seem interesting (around 1450 and 1900 nm), and according to the literature, they correspond to absorption water band.

Using factors analysis methods based on PLS1

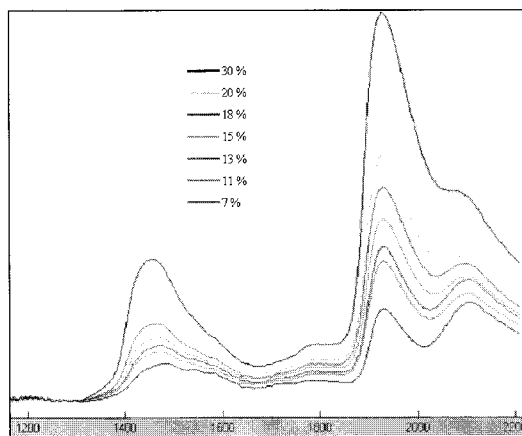


Fig. 4. Spectra of humidity.

algorithm, a prediction model for humidity percentage has been developed. The results given by this model are specified on Table 1, and the mean of the absolute values of the prediction errors is = 0.5 %.

Finally, to test again our model, we have used other raw materials different from fluting and at different humidity by using a tank with constant humidity, and the mean obtained may stay down 1 % (but lower test variability : between 7 % and 13 %).

4.4 Studies in laboratory of contaminants and raw materials

As for humidity, to build this prediction model we need different learning samples containing different contaminant rates and/or raw materials. So the "ideal" would be to have:

- a sample with X1% aluminium, Y1 % plastic, Z1 % paper ...
- a sample with X2 % aluminium, Y2 % plastic, Z2 % paper ...
- etc.

But, practically, how defining, and above all how

Table 1. Prediction of humidity percentage

H measured	11 %	13 %	15 %	18 %	20 %
H predicted	11.19 %	12.60 %	14.56 %	17.09 %	19.39 %

obtaining such samples? In fact, we have decided that the simplest would be to have a sample of 100 % of each compound (and therefore 0 % of "rest").

After several spectrum acquisitions of different samples, the following "distinct enough" compounds for first trials were retained:

- aluminium
- 3 kinds of plastics (types : mugs, paper-bags and packaging)
- textile material
- paper
- board (brown).

The spectra obtained for these compounds are on Fig. 5. We can notice that, as for humidity spectra (and for the same reasons), we have done a vertical re-adjustment on the range [1150 nm; 1350 nm] and we have kept only λ upper than 1350 nm.

So, for each compound, we have acquired spectra with 100 % of compound (for learning), and "qualitative binary compositions" with other compounds (for tests). This has allowed us to develop a prediction model for these different compounds (based on PLS2 algorithm), and to test it. The results obtained are encouraging because the mean is around 15 %, and we had coherent "qualitative" results. To

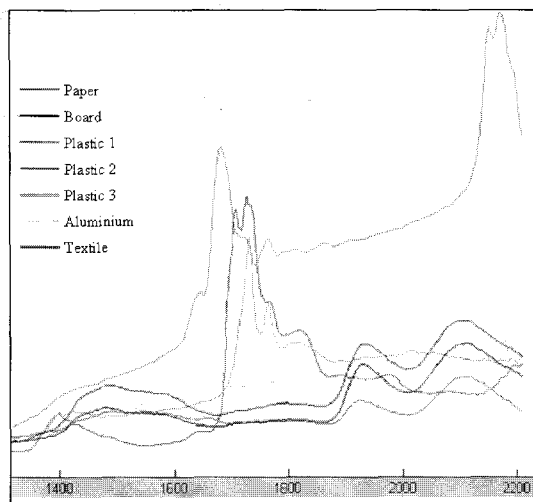


Fig. 5 Spectra of different compounds.

finish, note that a prediction error of 10, 20 or even 30 % may seem considerable but may seem definitely less by taking into account a large number of acquisitions (for instance 1000) from a same coring sample.

5. Conclusion

The first results obtained in laboratory with the NIR technology have allowed us to check the use and the discriminant capability of this approach for humidity, contaminants, and raw materials that we would like to characterize.

This methods are very powerful, however extremely sensitive to the measurement environment. Indeed, optic (and mechanic) for spectra acquisition, but also statistic models used, have to be really fine-tuned in order to optimize the discrimination. As a consequence, we still have a lot of work to do in order to integrate this approach into a direct and complete industrial sensor based on a coring device.

Finally, the validation of this transfer will be based on the comparison between the results given by statistics models and the results manually made by an operator. Furthermore, several sampling will be done on each bale, in order to control their homogeneity and the efficiency of our methods.

Acknowledgement

The authors would like to thank all the CTP Members and Partners who, involving in this project, have allowed us to do this research work.

Furthermore, we would like to thank too all the CTP staff who have worked with us in this project, and in particular Laurent Lyannaz for his job on the Dixon in order to obtain calibration samples for humidity.

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