A new automated method for **macrocontaminant analysis**

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Abstract

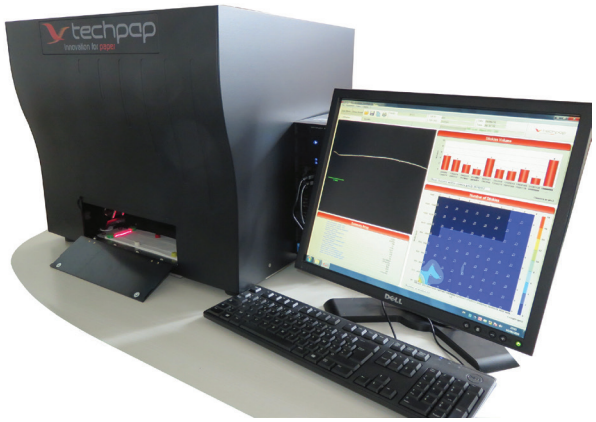
In recycled paper processes, stickies are at the origin of many production disturbances. In this paper, we present an automated macro-contaminant measurement method which allows to (i) determine the three-dimensional morphology of screened particles (without any deformation) and (ii) classify the particles as stickies among contaminants. This is achieved by a combination of laser triangulation and local near-infrared (NIR) spectroscopy.

Note - Some of those results have been reported in the NPPR Journal: Huber, P., Borel, P., Soysouvanh, D., Ossard, S., & Delagoutte, T. (2015). Macrostickies measurement by an automated method using laser triangulation and near-infrared spectroscopy. *Nordic Pulp & Paper Research Journal,* 30(2).

Introduction

In recycled paper processes, stickies are at the origin of many production disturbances, such as machine breaks, defects in paper and converting problems. Macrostickies are typically evaluated by measuring their concentration in the pulp after screening. Monitoring of macrocontaminants is an essential task for most recycling mills, yet tedious and laborious with typical measurement methods. Macrocontaminants are separated from the pulp by screening, then submitted to various inspection and classification methods.

The most recognised method in Europe is INGEDE#4 [1]. It gives useful information about stickies particles count and size. The INGEDE#4 method is based on contaminants isolation by pulp screening followed by measuring their size distribution over a paper filter. Tacky particles (stickies) are specifically coloured and quantified by image analysis. However the successive pressing and drying steps cause deformation of the stickies. Furthermore, the measurement is time-consuming, because of the manual preparation involving numerous steps.



The chemical nature of stickies makes them suitable for analysis by near-infrared spectroscopy (NIRS). The technique is rapidly gaining acceptance in the industry, with applications in paper recycling such as paper waste stream sorting [2,3,4], raw material quality assessment [5,6,7] or quantification of stickies embedded in handsheets [8,9].

The objective of this work is to describe a new measurement method for stickies in recycled pulps. The method discriminates stickies among all contaminants and provides 3 dimensional information of the stickies morphology. That is achieved through a combination of laser triangulation and near-infrared spectroscopy inspection of contaminants deposited on a filter paper. The contaminants are classified by analysis of their NIR spectra. The developed sensor (3DStick) offers a fully automated characterisation of stickies.

Material and methods

Contaminated pulp samples

Model stickies were prepared with rolls of label (acrylic PSA adhesive, E115 Jackstadt, 3.5% on pulp) laid on bleached hardwood kraft pulp sheet (BHKP) and pulped in a Helico pulper (10%, 20 min, 45°C). Besides, the "industrial pulp" sample was simulated by fully deinking sorted office waste (SOW) raw material on the CTP deinking pilot plant.

Preparation of filter papers with contaminants

Each contaminated pulp sample was screened (Somerville, 0.1 mm slots, 25 g, 20 min). The rejects were deposited on a filter paper, and air-dried. The filter paper with deposited contaminated was then submitted to the 3DStick analysis.

After this non-destructive analysis, the same filter was then submitted to conventional characterisation (INGEDE #4 method).

Proposed method for macro-stickies measurement

Step 1: Characterisation of contaminants in their native state (3DStick method)

Three-dimensional characterisation of the stickies population was performed with the developed 3DStick device. The contaminants deposited on the filter are scanned with a red laser sheet illumination (Fig 1). A visible CCD camera (2048 pixel field width) placed at 90°, analyses the deformation of the projected laser line caused by the presence of an object.

As the couple laser/camera is moved horizontally relative to the filter paper, all profiles scanned are stacked in order to reconstruct a 3D image of the surface of the filter with all deposited contaminants (resolution *dx= dy=20 µm, dz=3 µm*). Each detected contaminant is then analysed through blob image analysis. An equivalent cuboid object is identified (*L x w x t)*, where length L corresponds to the major axis of the equivalent ellipsis, width w is calculated from the projected surface S of the detected stickies (*w=S/L)* and thickness t is calculated from the total volume V of the detected stickies (*t=V/S)*. Thus, a 3D map of all contaminants is reconstructed and objects counts, lengths, widths and thicknesses may be statistically studied.

The optical system is motorised in both the X and Y directions by high precision axes. The maximum surface that can be scanned is 250x250 mm². The filter paper does not have to be flat, as baseline profile variations are mathematically corrected by a projection algorithm.

Step 2: Classification of contaminants based on their NIR spectra

The chemical nature of the contaminants was assessed by near-infrared spectroscopy. A NIR head, comprising a light source and a light collection system, was travelled over each contaminant to perform a local scan. The backscattered light was sent to a NIR spectrometer, via an optical device (optical gain of 1) connected to an optical fibre (diameter 200 µm). The NIR spectrometer had a spectral range of 1103 to 2197 nm, with 256 channels and an integration time of 4 ms. The unknown spectrum was pre-treated with classical methods. In the following, we use only the 1st derivative spectra for all calculations, plotting and discussion. The spectrum was then cut to a window of interest (1604 to 1840 nm, roughly corresponding to the CH band).

We need to identify macrocontaminants deposited on filter paper, i.e. on a cellulosic background. The developed spectral identification method used point-to-point correlation with reference spectra from a database. The method sought the best possible correlation between the unknown spectrum and linear areal mixes of pure compounds spectra and cellulose spectra. The selected similarity index was Pearson's correlation coefficient *r.*

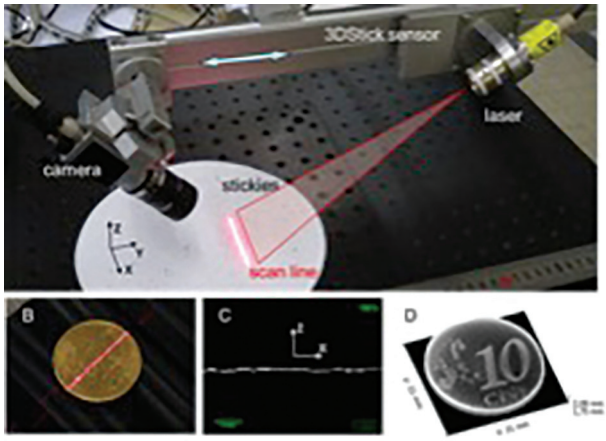


FIGURE 1. Laser triangulation setup. (A), example of profile scanning with a coin (B), detected scan line (C) and reconstructed altitude model (D). For clarity, the NIR head is not shown.

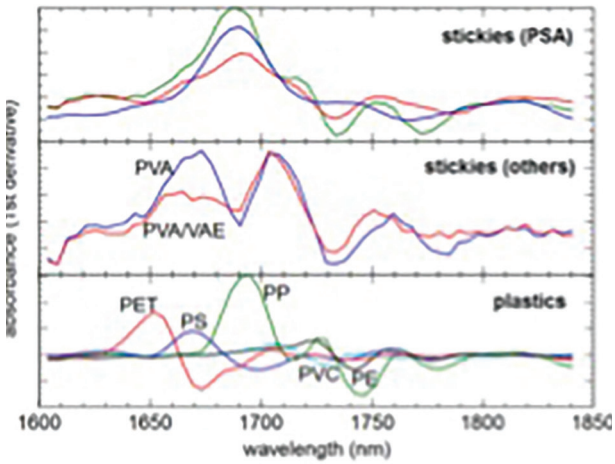


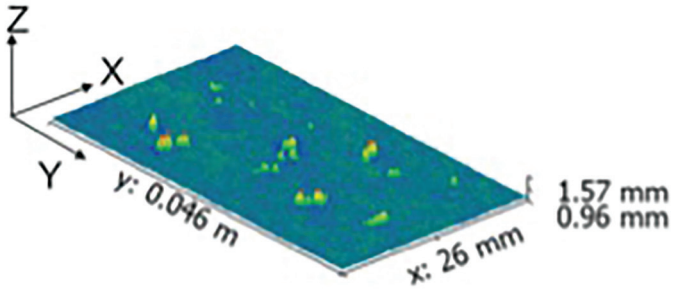
FIGURE 2. Example of first derivative NIR spectra for the main reference pure compounds in the database

Firstly, a database of expected pure compounds was built. Three classes of contaminants were defined for classification: stickies (PSA), stickies (others), and plastics (see example spectra in Fig 2). Within each of these classes, various compounds were included. For each database pure compound, we calculated the areal mix with cellulose that had the highest similarity index with the unknown spectrum (from a table of pre-calculated mixes, for fast real-time analysis).

We selected the database compound that has the highest similarity index *r.* We considered that we had a match when *r>0.975.* Otherwise, the unknown spectrum was left unidentified.

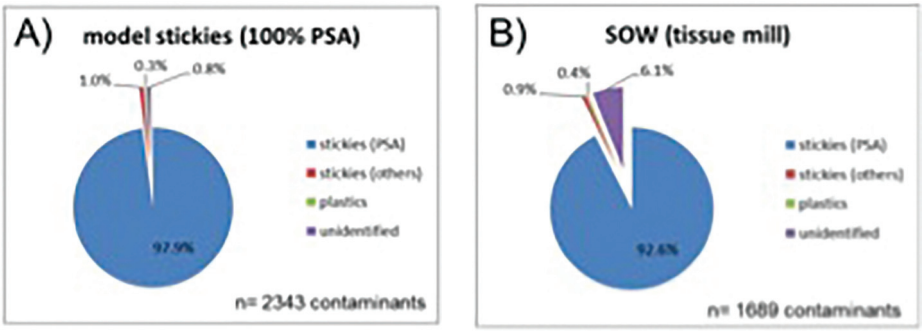
Results

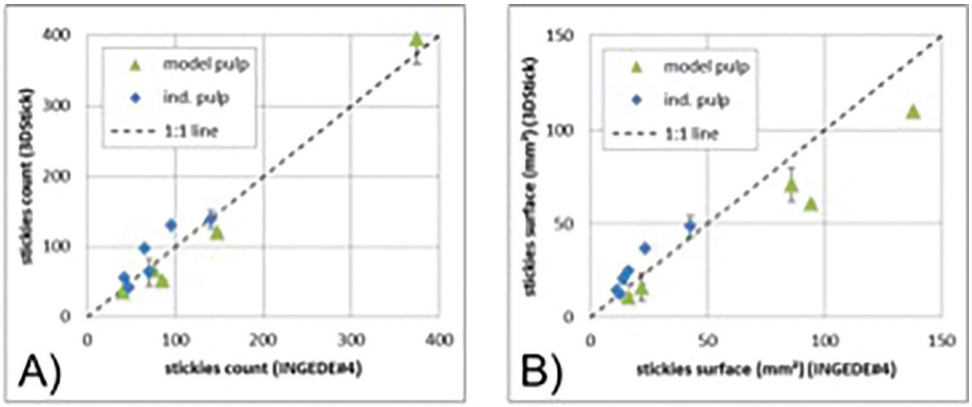
During a 3DStick measurement run, the screened contaminants deposited on a filter paper are firstly mapped by laser triangulation. An example of such 3D image map can be seen in Fig 3. The protruding contaminants are then isolated by image analysis, for location, measuring, and further NIR analysis.



(LEFT) FIGURE 3. Example of reconstructed altitude map for contaminants deposited on filter paper.

(RIGHT) FIGURE 4. Breakdown of contaminants by classes A) virgin pulp with model stickies. B) SOW (tissue mill)





(LEFT) FIGURE 5. Comparison of 3DStick and INGEDE#4 stickies measurement results, in number (A) and in surface (B). (Error bars are two standard deviations)

After the contaminants have been measured and located by laser triangulation, they are classified by a local NIR scan.

With the "model stickies" raw material, the vast majority of contaminants (97.9 %) are recognised as "stickies (PSA)", as expected (Fig 4A). A small proportion of contaminants (0.8%) is left unidentified by the NIR classification algorithm.

In the "industrial pulp" sample, the majority of contaminants (92.6 %) are also identified as "stickies (PSA)" (Fig 4B). Almost no plastics were found (0.4 %). A small but significant proportion of contaminants (6.1 %) were not identified. The "industrial pulp" sample is indeed anticipated to contain mostly residual PSA particles as the raw material comes from an SOW mill, whereas other contaminants such as plastics are likely to be efficiently removed by the extensive deinking performed before contaminants screening.

When gathering results obtained with the five model stickies series, and the six industrial stickies series, an acceptable correlation is found between the 3DStick results and the INGEDE#4 results (Fig 5). The stickies count given by both methods is similar. The stickies surface given the 3DStick was systematically lower than that from INGEDE#4 for the model stickies: the deformation of stickies by the pressing step of INGEDE#4 causes this method to over-estimate stickies surface, compared to their native surface. Stickies deformation was observed with the model stickies series, but was less apparent with the tested industrial contaminated pulp. It is likely that different types of stickies will react differently to deformation upon heating and pressing.

The 3DStick sensor has many advantages over the reference method (INGEDE#4).

- The measurement is automated, and requires no sample preparation, other than pulp screening and deposition of rejects on a filter paper (compared to pressing, drying, dying, revealing and image analysis steps).
- The measurement is fast (20 minutes).
- Information about 3D morphology of stickies is obtained, without any deformation from pressing steps.
- The NIR selection method offers instant classification of stickies among contaminants.

Conclusions

Contaminants deposited on filter paper are firstly mapped by laser triangulation. Secondly, they are classified under different classes (PSA stickies, other stickies, plastics, etc.), through analysis of their NIR spectra.

The stickies count results from the 3DStick are closely correlated with those from the INGEDE#4 method. However, the analysis of the stickies 3D morphology demonstrates that the INGEDE#4 method flattens some type of stickies, which artificially increases their surface.

Besides stickies monitoring, the 3DStick device could be useful to investigate contaminants removal efficiency in recycling lines (at screening, flotation, etc.), identify contaminated raw materials, etc.

The 3DStick device has the potential to be connected to a pulp sampler and screening device, and we plan to use it for on-line characterisation of stickies in recycled pulp mills. ■

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