



# A hyperspectral imaging sensor for on-line quality control of extruded polymer composite products

Ryan Gosselin, Denis Rodrigue, Carl Duchesne\*

Department of Chemical Engineering, Université Laval, Québec (Qc), Canada G1V 0A6

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## ABSTRACT

This study examines the ability of chemometrics methods, namely multivariate image analysis (MIA) and Grey Level Co-occurrence Matrix analysis (GLCM), to extract meaningful information from visible and near-infrared spectral images of extruded wood/plastic composite materials for predicting spatio-temporal variations in their properties. The samples were produced under varying process and feed conditions according to designed experiments. Mechanical properties of the samples were measured using standard analytical methods both during steady-state and dynamic transition periods. A Bootstrap-PLS regression technique was first used for selecting the spectral bands (i.e. wavelengths) that were the most highly correlated with the material properties. In a second step, a more parsimonious PLS regression model was built between the spectral and textural features extracted from the lower dimensional spectral images and the corresponding quality properties of each sample. The imaging sensor was able to simultaneously monitor 7 properties in both steady-state operation and during transitions.

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## 1. Introduction

Polymer extrusion is essentially a steady-state process since its operating conditions are tightly controlled. Common automatic feedback control loops on machine variables (i.e. temperature, screw speed, etc.) provide satisfactory results as long as there are no stringent constraints on product quality (Tadmor, Lipshitz, & Lavie, 1974). On the other hand, raw material properties, such as melt flow index and composition, are seldom uniform in complex polymeric flows. The resulting extrudate may suffer from quality fluctuations that cannot be tempered by the machine variable controllers. Moreover, these fluctuations may never be detected in a timely fashion due to the lack of on-line sensors for key polymer properties, which leads to quality control issues in polymer processing plants (Ohshima & Tanigaki, 2000). In practice, quality control of polymer products is mainly performed by destructive testing in the laboratory using a very small proportion of the production. It is therefore likely that large amounts of abnormal products may be generated before quality issues are detected. This problem may be addressed by taking on-line quality measurements (or estimates) of the samples and including this information in quality control and monitoring schemes. Specifically, an efficient system should rely on in-process analysis of polymer characteristics during manufacturing providing real-time assessment of sample properties such

as structure, morphology and composition (Dumoulin, Gendron, & Cole, 1996). The reader unfamiliar with the polymer processing field is referred to Tadmor and Gogos (2006) for further information.

Spectroscopic soft sensing techniques have successfully been applied in a number of fields such as food analysis (De Temmerman, Saeys, Nicolaï, & Ramon, 2007; Mezreb, Goullieux, Ralainirina, & Queneudec, 2003), pharmaceuticals (Côté & Abatzoglou, 2006; Cournoyer, Simard, Cartilier, & Abatzoglou, 2008) and polymer science (Barnes, Sibley, Edwards, & Coates, 2003; Hansen & Vedula, 1998; Rohe, Becker, Kölle, Eisenreich, & Eyerer, 1999). The appeal of these methods lies in their ability to provide a great amount of information on the nature and structure of the sample through the location of the peaks and the intensity of the spectrum. Conventional machine variable control methods are simply not suited for maintaining molecular-specific properties to set-point, especially in complex or multi-component polymer systems.

Analytical soft-sensing methods such as UV-visible, near-IR, mid-IR and Raman spectroscopy can be applied in four modes of operation depending on sample nature, geometry and property of interest: transmittance, reflectance, transreflectance (light passing through the sample onto a mirror and reflected back through the sample) and diffuse reflectance (light penetrating partway into a moderately opaque sample) (Reshadat et al., 1999). Other on-line soft-sensing methods include ultrasound transit time measurements (Barnes et al., 2003; Chen, Nguyen, Wen, & Jen, 1999), X-ray scattering (Albano, Papa, Gonzalez, Navarro, & Gonzalez, 2003; Pantani, Coccorullo, Speranza, & Titomanlio, 2005; Stark &

\* Corresponding author. Tel.: +1 418 656 5184; fax: +1 418 656 5993.  
E-mail address: [carl.duchesne@gch.ulaval.ca](mailto:carl.duchesne@gch.ulaval.ca) (C. Duchesne).

Matuana, 2007), fluorescence spectroscopy (Barnes et al., 2003), and nuclear magnetic resonance (NMR) spectroscopy (Bergmann, 1981; Pelsoci, 2007). According to Barnes et al. (2003), the polymer industry has so far mainly applied spectroscopic methods in either off-line or near-line (e.g. pellet composition) applications. Nevertheless, several sample properties have now been investigated on-line. These include estimation of the melt viscosity (Broadhead, Nelson, & Dealy, 1993; Hansen & Vedula, 1998; Merikoski et al., 2001; McAfee & McNally, 2006), yield stress (Broadhead et al., 1993), polymer density (Watari et al., 1996), composition (Barnes et al., 2003; Hansen & Vedula, 1998; Rohe et al., 1999), extrudate temperature (Chen et al., 1999) and surface chemistry (Gupta, Reiniati, & Laborie, 2007; Stark & Matuana, 2007).

However, most sensing methods are based on single point or averaged measurements. Estimating sample quality distribution over a larger area using these approaches is difficult to perform on a high-speed production line. At best, a coarse spatial coverage of the surface can be achieved using probe arrays. Nevertheless, fine spatial measurements greatly enhance the probability of identifying local weaknesses that may compromise the quality of the whole. The mechanical, optical and barrier properties of an entire sample may fall below specifications because of local discrepancies that may be overlooked by a coarse spatial analysis.

Historically, modelling the open-loop response of a plasticating extrusion process can be traced back to the 1970s. During this period, research aimed at proposing relationships between screw speed, die pressure, extrudate flowrate and melt temperature (Reber, Lynn, & Freeh, 1973; Tadmor et al., 1974). Later, work was done on the dynamics and control of the extruder pressure in cases where variations in feed quality and feed rates were present (Akdogan & Rumsey, 1996; Costin, Taylor, & Wright, 1982; Chan, Nelson, & Lee, 1986). More recently, Mudalamane and Bigio (2003) studied the transmission of external disturbances through the extruder whereas McAfee and McNally (2006) determined the extruder dynamics using the melt viscosity. More complex modelling techniques were also applied to the extrusion process, for example using finite element methods (Bajimaya, Park, & Wang, 2007; Ha, Cho, Kim, & Kim, 2008; Yuan, Ball, & Edwards, 1994), artificial neural networks (Shihani, Kumbhar, & Kulshreshtha, 2006) and fuzzy logic (Fodil-Pacha, Arhaliass, Ait-Ahmed, Boillereaux, & Legrand, 2007).

This work investigates a new machine vision approach for on-line monitoring of product quality obtained by an extrusion process. On-line visible-NIR spectral imaging is used for rapid and non-destructive detection of spatio-temporal variations in the polymer properties in far greater detail than spectroscopic probes or probe arrays may achieve. Furthermore, rich spatial data would also be useful for precise diagnostic of operational problems in the production line. The proposed methodology uses chemometrics methods for extracting the spatial and spectral features that are most highly correlated with the material properties, and use

this information to predict the properties of samples unknown to the model. To illustrate the concepts, the proposed techniques will be applied to the production of extruded wood/plastic composites (WPC). The rationale behind this approach is twofold: (1) WPCs are complex multiphase systems that are not as well understood and controlled as neat polymers and (2) the production of WPCs has been increasing steadily in response to growing concerns over waste plastic management. In fact, the production of WPCs quadrupled between 1997 and 2000 (Li & Wolcott, 2004) and is expected to reach \$2.4 billion US by 2011 (Wrap, 2003). Nevertheless, these methods are not specific to WPCs. They are intended for any product that may be distinguished by its spectral and/or spatial surface characteristics.

## 2. Materials and methods

### 2.1. Sample production and testing

Extruded high density polyethylene (HDPE)/wood composites were produced and tested under different processing conditions and wood contents. The polymer used was commercial HDPE (Fortiflex A60-70-162) and birch wood fibers were obtained from a local sawmill. These fibers were sieved between 45 and 180  $\mu\text{m}$  and dried in a vacuum oven at 104 °C overnight in order to minimize the amount of volatiles. Compounding of the polymer and wood was achieved using a Haake Rheomix TW-100 dual-feeder counter-rotating twin-screw extruder. This allows proper distribution of fibers in the polymer blend. In this first step the barrel temperatures of the extruder were controlled at 150, 160, and 160 °C for zones 1, 2 and 3, respectively, and 160 °C for the die. The compound was extruded through a cylindrical 5 mm die, air cooled, and pelletized. After this blending phase, the composites were extruded using a flat horizontal 3 mm  $\times$  80 mm die. The following process conditions were investigated in a complete 2<sup>3</sup> factorial design: die temperature (160 and 180 °C), screw speed (70 and 90 rpm), and polymer throughput (500 and 600 g/h) with a fixed wood content of 20 wt.%. Wood content was also varied in a separate set of experiments at three levels (10, 20 and 30 wt.%) under constant nominal process conditions (i.e. centre point of 2<sup>3</sup> design). Steady-states were allowed to run for about 10 min for each condition tested. These processing conditions are presented in Table 1.

Sample quality consists of 6 tensile properties (modulus, stress and strain at yield, stress and strain at break, as well as the energy at break), measured according to ASTM D638 using an Instron 5565 in the direction of extrusion at a crosshead speed of 1 mm/min, measured over a 25 mm gage length. The measurements also include 1 thermal property (enthalpy of fusion) obtained using a Perkin Elmer DSC 7. While enthalpy of fusion and crystallinity are closely linked (the former is used to calculate the latter), crystallinity also requires an estimation of the sample composition. In order to avoid this complication during DSC testing (test sample typically weight

**Table 1**

Randomized 2<sup>3</sup> full factorial design for machine variables at 20% wood (samples 1–8) combined with 3 additional experiments in which wood content was varied under nominal processing conditions (samples 9–11).

Sample	Wood (%)	Temperature (°C)	Throughput (g/min)	Screw speed (rpm)
1	20	160	600	70
2	20	160	500	90
3	20	180	500	70
4	20	180	600	90
5	20	160	600	90
6	20	160	500	70
7	20	180	600	70
8	20	180	500	90
9	10	170	550	80
10	20	170	550	80
11	30	170	550	80

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