

Identification of bio-plastics by NIR-SWIR-Hyperspectral-Imaging

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Abstract Bio-plastics are characterized by the highest rate of growth in the plastics industry. In connection with the recycling chain they constitute the so-called “oxo-biodegradation” and drop-in problems. The present study tries to clarify possibilities of automatic recognition and sorting of conventional fossil-based plastics against similar “oxo-biodegradable” plastics and drop-ins by means of NIR-SWIR-Hyperspectral-Imaging (HSI). The spectral structures of the most important plastics (conventional fossil-based plastics and bio-plastics) have been incorporated in a database as references for different plastic types to be subject to identification by NIR-SWIR-HSI. In addition to widespread chemometrical methods (PLS-DA), artificial neural networks (ANN) and support vector machines (SVM) are estimated for classification. For “oxo-biodegradable” plastics it turns out that a decision tree is the most reliable procedure for identification. Different decision trees are passed to an industrial NIR-SWIR-Hyperspectral-Imager for generating chemical images of different plastic mixtures. The mixtures consist of conventional fossil-based plastics and bio-plastics. The aim of the tests was to find bounds for sorting throughput and purity. Results of an industrial sorting trial are finally described.

1 Introduction

Despite there are significant doubts about the environmental sustainability of bio-plastics [1], currently this class of materials has the highest rate of growth in the plastics industry [2–4]. In the context of

the recycling chain they constitute on one hand the so-called “oxo-biodegradation” problems and on the other hand the so-called drop-in problems. The first problem is in connection with the use of oxidation-enhancing additives in the polymer matrix. The second occurs when the chemical structure of a bio-plastics is similar to its pure fossil-based homologous. A mixture of different types of similar plastics within the input stream of a recycling chain can be in some cases useless for reprocessing due to not strict similar physical properties.

The present study attempts to clarify the possibilities of automatic recognition and sorting of bio-plastics, in particular conventional fossil-based plastics against similar “oxo-biodegradable” plastics, by means of NIR-SWIR-Hyperspectral-Imaging. After a brief explanation of bio-plastics material, Hyperspectral-Imaging (HSI) is shortly explained as the state-of-the-art procedure for sensor-based sorting of plastics waste. This involves the three main procedures for extracting useful information from Hyperspectral-Images (PLS-DA (partial least squares discriminant analysis), ANN (artificial neural networks), and SVM (support vector machines)).

Furthermore, the spectral structures of the most important bio-plastics are depicted. Of particular interest are the fingerprints of the chemically similar plastics. The results have been incorporated in a database as references. For classification it turns out that a decision tree is the most reliable procedure for sorting of “oxo-biodegradable” plastics. Different decision trees passed an industrial NIR-SWIR-Hyperspectral-Imager for generating chemical images of different plastic mixtures. The mixtures consist of conventional fossil-based plastics and bio-plastics. The results of an industrial sorting trial are finally described.

2 Bioplastics

The word “bio-plastics” is an expression of leading astray because it primarily suggests just materials that are bio-degradable and therefore supposed environment-friendliness. In the literature the term “bio-plastics” is considered further. It comprises organic materials, in particular organic polymers, that are either bio-based, bio-degradable, or both [5, 6]. A bio-plastics material can be distinguished in one of the follow-

ing three main groups: (1) (partially) bio-based and non-bio-degradable such as bio-PE, bio-PP or bio-PET (so-called "drop - ins") as well as technical polymers such as PTT (poly-trimethylene terephthalate) or TPC-ET (thermoplastic co-polyester elastomers), (2) bio-based and biodegradable such as PLA (polylactic acid), PHA (polyhydroxyalkanoate) or PBS (polybutylensuccinate), and (3) fossil-based (oil, coal, natural gas ...) and bio-degradable such as PBAT (polybutylene adipate-co-terephthalate). For a fully review on present industrial available bio-plastics see [3,4].

The so-called oxo-biodegradable plastics are a subgroup of bio-plastics. Terms such as "degradable", "biodegradable", "oxo-degradable", "oxo-biodegradable" are used to promote products made with traditional plastics supplemented with specific additives. The "oxo-biodegradable" additives are typically incorporated in conventional plastics such as polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET). These additives are based on chemical catalysts, containing transition metals such as cobalt, manganese, iron, etc., which cause fragmentation as a result of a chemical oxidation of the plastics' polymer chains triggered by UV irradiation or heat exposure. In a second phase, the resulting fragments are claimed to eventually undergo biodegradation. But oxo-fragmentable products can hamper recycling of post-consumer plastics. In practice, these plastics are traditional plastics. The only difference is that they incorporate additives which affect their chemical stability. Thus, they are identified and classified according to their chemical structure and finish together with the other plastics waste in the recycling streams. In this way, they bring their degradation additives to the recycle feedstock. As a consequence the recyclates may be destabilized, which will hinder acceptance and lead to reduced value [7]. As a consequence, some public and private organizations therefore militate against oxo-degradable additives.

Drop-in bio-plastics are bio based or partly bio based non-biodegradable materials such as (partly) bio based PE, PP or PET. Drop-ins seem to feature the same properties and functionalities as their fossil counterparts. It is assumed that the value-added chain only requires some irrelevant adaptations in processing. Use, recycling and recovery follow the same routes as conventional PE, PP or PET products do. But

this must not be the full truth. A mixture of both types can be in few cases useless for reprocessing due to not strict equal physical properties.

3 NIR-SWIR-Hyperspectral-Imaging

3.1 NIR-SWIR spectral range

Due to different views related to the NIR-SWIR spectral range, it is necessary to make some comments in this regard. As a start the term Infrared (IR) refers to the full portion of the electromagnetic spectrum between visible light and microwaves. This large range is generally divided into Near Infrared (NIR, 0.78 - 1.4 μm), Short-Wave Infrared (SWIR, 1.4 - 3.0 μm), Medium-Wave Infrared (MWIR, 3.0 - 8 μm), and Long-Wave Infrared (LWIR, 8 - 15 μm). The classification of infrared spectral ranges was initially based on chemical/physical phenomena like the types of molecular vibrations or the atmospheric windows for the radiation, respectively. Latterly more technical aspects divide up the IR-band based on the response of various semiconductor detectors. The industrially most advanced semiconductor detector materials for NIR-SWIR-detection are InGaAs, ext-InGaAs and HgCdTe, respectively [8–10].

For NIR imaging, $\text{In}_x\text{Ga}_{1-x}\text{As}$ is one of the widely used detector materials due to its low dark current. InGaAs thin films typically using an alloy composition of $x = 0.53$. The spectral response covers at room temperature 0.9 - 1.7 μm . Thus, the 0.9 - 1.7 μm spectral ranges is called NIR-range by most of the detector providers. By increasing the composition to $x = 0.82$, InGaAs is able to extend its cut-off to 2.6 μm (so called ext-InGaAs). However, the crystal defects due to epitaxy and the decreased shunt resistance, due to a smaller band gap, degrade performance at the longer cut-off wavelengths [11].

An approach to accomplish SWIR imaging under low light level conditions is HgCdTe. $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ (MCT) is currently the widely used detector materials for SWIR imaging. The ability to tune the cut-on and cut-off wavelengths in SWIR by changing the stoichiometry of the substrate enables application optimizations. The alloy composition x can be fixed to provide an energy band gap equal to the longest wavelength to be measured in the SWIR band [12]. For SWIR imaging mostly a

spectral range from 0.9 – 2.5 μm is chosen. The permission of two different systems (InGaAs and MTC as semiconductor material) based on spectral resolution requirements for the respective application cases.

3.2 Hyperspectral-Imaging

NIR-spectroscopy is currently the most common method for automated sorting of post-consumer plastics. This technique involves irradiating the unsorted (unidentified) plastics with near-infrared waves. When the infrared light reflects off the surface of the plastics, each resin's characteristic infrared absorption band can be measured. These measurements can then be compared directly to known polymer values (correlation analysis) or indirectly by signal transformation and applying of chemometrical procedures to determine the resin type.

NIR-spectroscopy has many advantages. Probably the most significant advantage is the speed of identification. A second advantage is the lack of specimen preparation. Labels or other obstructions like dirt, do not relevant interfere with readings. Another advantage of NIR-based optical systems is that colour does not interfere with proper resin identification. Except for black, the readings are independent of the surface colour.

The special requirements in plastics sorting call for cost-effective, robust NIR spectrometers capable of macroscopic imaging. The best answer to this problem is the application of spectral imaging systems, which are capable of measuring both the spatial and the spectral information in quasi one step. Such devices are called Hyperspectral-Imaging cameras or short Hyperspectral-Imager.

Hyperspectral-Imaging is a combination of spectroscopy with digital image processing. Other than with standard imaging, where only the spatial information plus the three RGB values for each single image pixel are recorded, HSI provides full spectral information (a few hundred values) for each surface pixel over a selected wavelength range.

In instrumentation there are basically three different approaches (**Fig. 6.1**). The first approach is the so called “whiskbroom scanning” (**Fig. 6.1 left**). This sampling method was introduced in the industrial practice of plastics waste sorting in the mid-nineties. A whiskbroom imager scans the sample point-by-point in the x - y -spatial direction and measures a full spectrum of adjacent regions sequentially. The need of using rotating or swinging mirrors to generate a HSI makes such systems mechanically susceptible to faults.

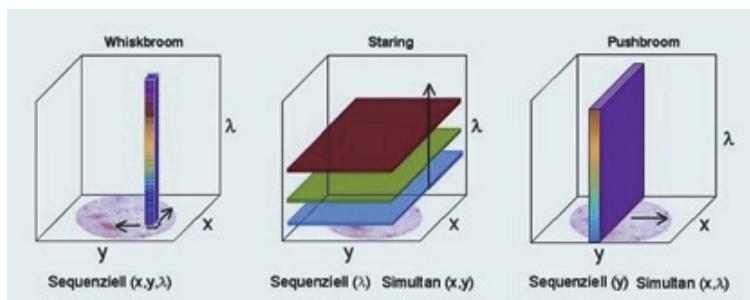


Figure 6.1: Different methods for data acquisition [13]

The second approach is the wavelength scanning method, in remote sensing also called “staring” (**Fig. 6.1 middle**). The sample is kept stationary under the HSI camera and single images are recorded for each different wavelength. The spectral information is provided either by (a) a number of discrete filters, (b) tunable filters or (c) by combination with an imaging Fourier-Transform spectrometer. The images recorded for the different wavelengths are combined in the computer and the spectra calculated. Staring imagers are not been implemented so far for automatic sorting of waste.

The third method, also known as “push-broom scanning” (**Fig. 6.1 right**), requires a relative movement between imager and sample to scan over the surface. Push-broom scanning is currently the state of the art according to advanced plastics waste sorting machines. The spectral imaging system records the spatial information line wise and the spectral information for each pixel along the line by projection of the spectral information along the second axis of a two-dimensional camera chip (focal plane array, FPA). The spectral encoding is provided either by linear

variable filters, by a DMA (digital micro-mirror array) in combination with grating or by dispersive optics forming an imaging spectrograph. A computer combines the slices, derives the second axis and thus reconstructs the full Hyperspectral-Image.

4 Classification Methods

In connection with plastics sorting, classification refers to the assignment of spectral information to a particular source type. Procedures are used that run under the scientific name "chemometrics". The most widespread classification methods used in chemometrics are supervised and unsupervised trainings [14, 15]. The list of such methods is large: linear and quadratic discriminant analysis, partial least squares discriminant analysis (PLS-DA), soft independent modelling of class analogy, hierarchical cluster analysis, k nearest neighbor's and k-means methods to name only the most important. Increasingly also nonlinear methods such as ANN (Artificial Neural Networks, [16, 17]) and SVM (Support Vector Machines, [18]) are used, especially for the so-called Chemical-Imaging. In this paper the performance of PLS-DA, Feed-Forward-ANN and SVM are examined with the main focus on PLS-DA.

4.1 PLS-DA

Partial least squares discriminant analysis (PLS-DA) includes a partial least squares regression of a set Y of binary variables describing the categories of a categorical variable (here types of plastics) on a set X of predictor variables (here wavelength-dependent intensity of NIR reflection) [19]. Partial least squares models are based on principal components (eigenvectors of the covariance matrix) of both the independent data X and the dependent data Y. The central idea is to calculate the principal component scores of the X and the Y data matrix and to set up a regression model between the scores (and not between the original, not rotated data) [20, 21]. This technique is especially suited to deal with a much larger number of predictors than observations and with multicollineality, two of the main problems encountered when analyzing data of NIR reflection spectra (regarding to plastics sorting tasks particular multicollineality).

4.2 FF-ANN

Artificial neural networks (ANN) are a very flexible class of statistical regression techniques. The essential advantage of the application of neural networks is that no explicit assumptions about the functional form of the unknown link need to be made. The most popular ANNs are multi-layer-feed-forward neural networks (FF-ANN) that are trained with a back-propagation learning algorithm [22, 23]. As a drawback of FF-ANN the lack of an exact rule for setting the numbers of neurons and hidden layers for best performance must be mentioned. Also, the computational costs for modelling are extremely high compared to PLS-DA modelling.

4.3 SVM

A Support Vector Machine (SVM) is a set of related supervised learning methods which can be used for classification (and in modified form also for regression). Given a set of training examples, each marked as belonging to one of two categories, an SVM classification training algorithm tries to build a decision model capable of predicting whether a new example falls into one category or the other. If the examples are represented as points in a hyperspace, a linear SVM model can be interpreted as a division of this hyperspace so that the examples belonging to separate categories that are divided by a clear gap that is as wide as possible. New examples are then predicted to belong to a category based on which side of the gap they fall on [24]. A common disadvantage of non-parametric techniques such as SVMs is the lack of transparency of results. SVMs cannot represent scores as a simple parametric function, since (among other things) its dimension may be too high.

5 Application

5.1 Hyperspectral-Imager

For the HSI research a special Hyperspectral-Imager called RTT-HSI 2.2 was employed in push-broom scanning mode. The main system parameters (hereinafter referred to in brackets) are the spectral range (1.2 ...

2.2 μm), the full frame pixel counts (320 spatial \times 256 spectral, also a reverse arrangement is possible), the digital measuring depth (14 bit) and the frame rate (330 Hz). Under the assumption of a conveyor belt width of 2.00 m and a belt speed of 2.00 m/s follows a resolution of less than 7 \times 7 mm² for one surface pixel of an object to be identified on the moving conveyor tape.

5.2 Spectra Library

Identification of spectra is a task similar to pattern recognition. The base is a library of patterns. For the investigations a set of plastic films delivered by the GAIKER co-authors was used. On the one hand, it was a mix of films made of conventional polymers derived from fossil raw materials and on the other hand, it was made of bio-plastics and plastics including oxo-degradable additives (Fig. 6.2).



Figure 6.2: Samples to create the spectra library

Fig. 6.3 shows the pattern spectra of bio-plastics regarding to Tab. 6.1 plus the spectra of LDPE-films, office paper and wood. Office paper and wood were included to illustrate the clear differences against bio-plastics spectra, although they consist likewise of organic material as basic. Also for illustration Fig. 6.4 contains the pattern spectra of LDPE and OXO_P1 in more detail. OXO_P1 is an oxo-degradable PE-based film that is equivalent to LDPE according to the chemical ground structure.

Table 6.1: Codes for used Bio-plastics

Code	Plastics Type
BIO_P0	Potato starch
BIO_P1	Corn starch
BIO_P5	PLA
OXO_P1	PE (oxo-degradable)
OXO_P2	PE (oxo-biodegradable)

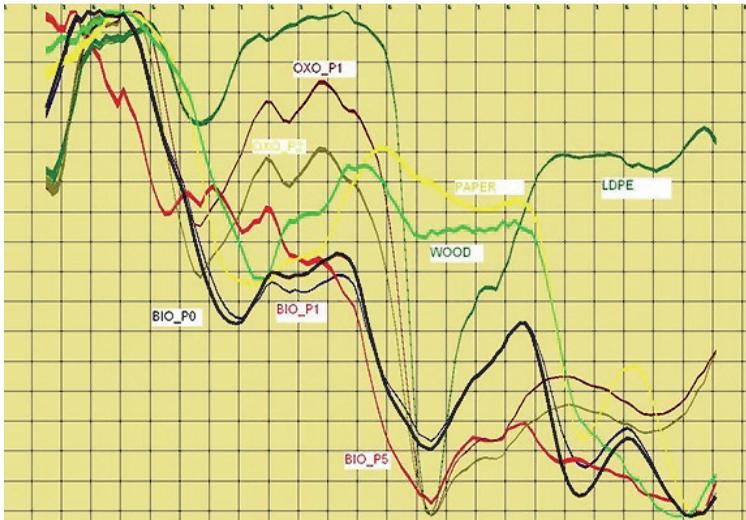


Figure 6.3: Pattern spectra of different bio-plastic films, LDPE films, office paper, and wood; spectral range 1.2 ... 2.1 μm , Savitzky-Golay smoothed (window 15, polynomic-order 3), min-max normalized

5.3 Spectra Identification Results

Three questions were target to be answered: (1) are novel bio-plastics distinguishable from the established conventional ones?, (2) is it possible to discover additives for “oxo-biodegradation”?, and (3) are drop-ins recognizable from fossil based similar plastics?

Spectra shown in **Fig. 6.3** can be easily divided into different classes by all of the three classification methods (PLS-DA, FF-ANN, SVM). The



Figure 6.4: Spectra of fossil based PE-films (LDPE) and oxo-degradable PE-films (OXO_P1); min-max normalized regarding ROI (region of interest, yellow background)

differences are so serious that even mixed spectra can be disassembled into so-called endmembers by unconstrained demixing procedures (here not discussed, see [25]).

The codes OXO.P1 and OXO.P2 label two types of “oxo-degradable” and “oxo-biodegradable” PE-films of different providers, respectively. The used incorporated additives are unknown to the authors and there are no references in the delivered data sheets of the films. According to **Fig. 6.3** (OXO.P2) and **Fig. 6.4** (OXO.P1 and LDPE), all of the characteristic bands of PE are well pronounced, but enough differences exist for a classification (**Fig. 6.4, green oval**; this area has been also identified by calculating the VIP (Variable Importance in the Projection, see [26,27]), here not discussed). To identify the type of spectra by PLS-DA, in the case of OXO.P1, OXO.P2 and LDPE so-called derivative spectra of second order must be generated. This is not necessary when using FF-ANN and SVM as classifiers. But the search for appropriate system parameters for FF-ANN and SVM is very time consuming. Because these parameters are case-specific it should at this point not be subject to any in-depth discussion. However for classification of “oxo-biodegradable” plastics a two-stage algorithm should be used (decision tree), where the second classifier should be either a SVM or an ANN.

To answer the third question, whether drop-ins are recognizable from similar fossil based plastics, the specific case of PET and bio-PET was considered. A conventional PET plastic is made from two components known as mono-ethylene glycol (MEG) and purified terephthalic acid (PTA). MEG makes up for 30 % of the PET weight while the remaining 70 % is made up by PTA. It is possible to replace the fossil-based MEG portions by a bio-based MEG share (bio-MEG). Ethanol from sugar cane and sugar cane by-product-molasses is used as basis of bio-MEG as one of the key plastic ingredients for bio-PET. Aspirations are known to produce also the second base fabric (PTA) from renewable resources, i.e. as a result completely bio-based PET can be produced in the future. In addition there are efforts to develop degradable bio-PET (oxo-bio-PET). Details on the last mentioned procedures are not known from the literature. The procedures are still in the development state.

In the present research the authors tried to explore whether the plastics of ordinary, full fossil based PET bottles and that of so-called PlantBottles™ (brand name of an American soft drink manufacturer) can be distinguished by NIR-SWIR-HSI. As result of extensive series of tests in a laboratory scale is to determine that the products can be assign to the different manufacturers. It is however not been ascertained, whether MEG is fossil-based or has sugar cane as a base. In fact the spectra are more sensitive with regard to different process conditions during plastics production as the origin of the MEG monomers. To elucidate the relationships further investigation are envisaged by exploitation of MWIR and LWIR spectroscopy.

5.4 Sorting Tests

The Hyperspectral-Imager RTT-HSI 2.2 has been used in industrial scale with regard to the separation of different types of plastics including bio-plastics. As basic machine a RTT sorter of type UNISORT PR has been used. The input stream was a waste mix of bottles and containers consisting of PET, PP and PLA. The decision for such a composition is therefore justified that PLA bottles will be in future a serious competitor to PET bottles. PET bottles and PLA bottles are hard to distinguish with the naked eye. However within the recycling chain these two plastic types shall not be mixed, because PLA has a significantly lower melting point [28]. Already 0.1 % PLA in a PET recycling process makes the PET recyclate unusable for many end-products.

For the Identification a two-step classification method was used: (1) distinction into classes of conventional plastics by PLS-DA and (2) two-class-grouping by SVM.

After the material is identified pixel by pixel (Chemical-Image), object recognition must be performed based on digital image processing to blow out an item regarding to its waste fraction. The exploited digital image processing is described in [29] and based on a gray scaled image of the HSI. The object reconstruction is then ultimately carried out by means of connected component labelling.

To sum up, the sorting tests have shown that the considered bio-plastics can be exact extracted from the input waste stream in a two-stage cognition procedure. The resulting throughput corresponds well with ordinary tasks regarding to automatic plastics waste sorting.

6 Conclusions

Hyperspectral-Imaging is a powerful tool and state of the art in optical characterization of plastics waste. In conjunction with digital image processing and discriminant analysis, the method allows automatic sorting in real time under industrial conditions. In the present work, three questions were target to be answered: (1) are novel bio-plastics distinguishable from the established conventional ones?, (2) is it possible to discover additives for “oxo-biodegradation” in the polymer matrix?, and (3) are drop-ins recognizable from fossil based similar plastics? The first question can be answered fully with Yes. Typically, the NIR spectral range is enough already. With regard to the oxo-degradable plastics (2), the full NIR-SWIR spectral range must be exploited to be able to detect additives, which activate or enhance plastics degradation. For this sorting task a two-stage algorithm of classification should be used (decision tree), where the second classifier should be either a SVM or an ANN. The third question is on the basis of the research results to answer with No. The NIR-SWIR spectra seem more sensitive with regard to different process conditions during plastics production as the origin of the monomers (fossil based or bio-based). To elucidate the relationships further investigation are envisaged by exploitation of MWIR and LWIR spectroscopy.

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