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May 2023

Marker materials and spectroscopic methods for sorting plastic waste

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Summary

Today, plastic is a ubiquitous material with good mechanical, chemical, and thermal properties and therefore used in many industrial sectors. The biggest challenge in recycling plastic waste is to separate the different types of plastic to a high degree of homogeneity. Waste sorting plants use automated sensor-based sorting systems to separate different plastics from each other. Many engineering plastics, such as polyoxymethylene (POM), end up in mixed waste streams, are not detected by sorting systems, and are therefore currently rarely or not at all recycled. Marker-based sorting is an innovative approach to improving recycling rates and achieving the recycling targets of the EU's Circular Economy Action Plan (CEAP). For this, the marker materials are incorporated into plastics, which can only then be detected by automated sorting systems to subsequently achieve improved homogenisation. However, marker-based sorting is currently not economical because of the expected high implementation costs. Consequently, expensive technical plastics show the greatest potential for marker-based sorting. For example, POM from plastic components of waste electrical and electronic equipment could be separated by type and recycled. This dossier provides an overview of advanced materials with spectroscopic "fingerprints" and suitable detection methods that can be used for marker-based sorting.

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Introduction

The application of plastics as industrial and consumer goods is very diverse as plastics have excellent mechanical, thermal, and chemical properties and can be produced costeffectively in large quantities. Since 2005, global production of plastics has increased from 230 to 367 million tonnes in 2020.[1,2] Plastics are currently used primarily in the packaging sector (40.5%), the building and construction sector (20.4%), the automotive sector (8.8%), and in electrical and electronic equipment (6.4%).[2] The remaining 24.1% can be found in other sectors, with each using less than 5% of the total amount of plastics.[2] The recycling rate of plastic packaging in the EU (EU 27) is 39.7% (based on data from 2020).[3] This means there is still some catching up to do to meet a 55% recycling rate for plastic packaging (70% for all packaging) in 2030. It is estimated that approximately 60% of all plastics produced globally between 1950 and 2015 was improperly disposed of or landfilled.[4,5,6] Furthermore, careless littering of used plastics leads to fragmentation of the plastics through mechanical and chemical environmental influences, such as UV radiation, and thus to the formation of microplastics or nanoplastics. These microparts can in turn release various additives (plasticisers, flame retardants, dyes and others), which can endanger human health and sensitive ecosystems.[7]

As a countermeasure, the EU's Circular Economy Action Plan (CEAP) provides for the monitoring and treatment of plastic waste as well as product labelling and an increase in recycling rates – in addition to banning primary microplastics in products (such as exfoliating granules).[8]

For the material recycling of plastics, it is generally very important that used plastics are separated by type with as much purity as possible to avoid undesirable mixing of the polymers or contamination with contaminants.[9] Moreover, used plastics can sometimes have very different material properties. For the separation of plastics, different collection systems (e.g. "yellow bags" or "yellow bins" in Germany and Austria) are used, including a variety of different techniques in sorting plants. For example, valuable fractions such as polyethylene terephthalate (PET) bottles, polyolefin (PO) mixtures, polystyrene granulate or polyethylene (PE) granulate are separated from separately collected light packaging materials using a combination of air separators, centrifuges, floatswim processes, and near-infrared-based sorting techniques (NIR).[10,11]

The composition of other waste streams, such as waste electrical and electronic equipment (WEEE), is more complex and can contain up to 18 different types of plastic with harmful additives, such as brominated flame retardants.[12] In the case of such waste streams, conventional sorting systems reach their limits, which means only a few types of plastic can be recycled. As a result, many technical plastics end up in mixed waste streams and are thus only thermally recycled or co-incinerated, therefore lost for the circular economy.[13] Tracer-based sorting (TBS) is a promising technique for increasing the sorting efficiency for high-quality waste plastic streams, such as technical plastics from WEEE.[14]

Tracer-based sorting (TBS)

TBS is a technical solution to identify and separate types of plastic that could not be recognised by existing sorting systems until now. TBS refers to a methodology of material sorting in which so-called marker materials are already incorporated into or onto the plastic to be sorted during its production. These marker materials give the material special properties and only serve the purpose of targeted detection by spectroscopic methods (see Table 1). TBS can therefore be used directly for the targeted, automated, sensor-based sorting of a specific type of plastic from waste streams.[15,16] For over 30 years, organic fluorescent dyes have been used as tracers or marker materials. For example, since 1992 S.R. Ahmad and his research team have been working on solutions to optimise the automated detection and sorting of post-consumer plastic waste, where detection is mainly facilitated on the basis of identifying fluorescence signatures of intrinsic chromophores or added fluorophores.[17] Since then, numerous research projects, such as ”BRITE-EURAM” or ”MaReK” (marker-based sorting and recycling system for plastic packaging), have looked at the application of TBS in great depth.[18,19,2]

Tracer or marker materials

The most important component of TBS is the so-called marker. These organic or inorganic substances facilitate the detection in the carrier matrix or in specific polymers. Rare earth elements are one promising class of substances that can be detected using X-ray fluorescence and near-infrared spectroscopy, photon upconversion (UC), and laser-induced breakdown spectroscopy (LIBS).[21,22] Elements of this group are increasingly used in high-tech products because of their fluorescence and semiconducting properties. Rare earth elements are crucial to the EU's economy, and are therefore considered critical raw materials.[23,24] The valence or excitation states of rare earth, such as yttrium and cerium, form the basis of fluorescence.[25]

In addition to the fundamental property of being detectable by already established detection methods, other parameters must be considered when selecting markers. The most important parameters include availability, material costs, service life, environmental sustainability and impact on the environment, and various physicochemical parameters (e.g. thermal stability, density, fluorescence yield, etc.).[21] The possibility of recovery or recyclability of the marker from the plastic matrix should also be considered. An existing authorisation of a substance in accordance with European chemical legislation (REACH and CLP regulations) is also relevant for companies processing plastics, as it simplifies the use of the substance as a marker. Figure 1 shows a possible decision tree for the selection of potential markers, which was developed in more detail within the research project “PLASTMARK”.[26,27]

Detection methods for TBS

Spectroscopic detection methods have long been used to identify and separate different materials. Mechanical recycling facilities for used plastics primarily use NIR sensors. However, sensors that detect in the ultraviolet (UV) and visible range (UV-Vis) or that are tuned to RAMAN spectroscopy, photon upconversion or X-ray fluorescence analysis can also be used.[14,28] TBS requires a detection method or a combination of several sensor techniques capable of detecting the markers that are integrated into the plastic matrix. In this context, it is important for the recycling plant to achieve a sufficiently high throughput capacity or sorting speed. Table 1 summarises the spectroscopic methods that can be used to detect the marker or tracer materials. The applied technological assessment – not taking possible implementation costs into consideration – was carried out on the basis of the so-called technology readiness level (TRL). The TRL is measured on a scale of 1 to 9 and was developed by NASA in the 1970s as an indicator of the stage of development in space technology. By now, it has been adapted for many other sectors. Basically, the higher the TRL of a technology, the more advanced it is.[29,30] In other words, a TRL of 8 is defined as a “system complete and qualified”. TRL 9 is then understood to mean suitability for mass production or industrial-scale application.[31]

It should be noted that not all methods summarised in Table 1 have already been successfully tested or applied on an industrial scale in the sense of TBS. Consequently, there is still a great need for research. In general, the various methods can also be combined with each other to detect different contaminants or recyclable materials in the waste stream. At present, multispectral sensors for hyperspectral imaging and other detection methods, such as RAMAN or LIBS, are being researched extensively.[54,55]

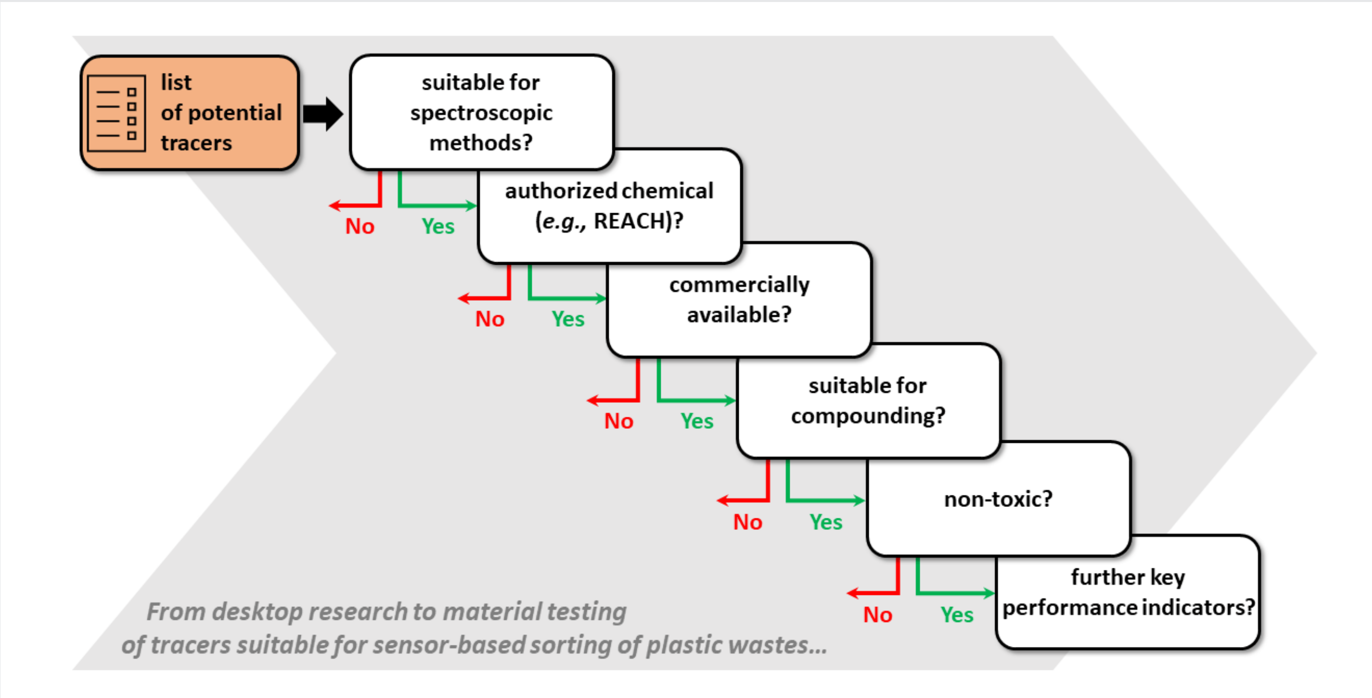

Figure 1: *Proposed decision tree for the selection of marker materials that would be considered for specific plastic streams.*[26]

Table 1: Modified summary of various spectroscopic detection methods that would be considered for tracer-based sorting of certain plastic streams.[26]

Detection method	TRL	Basic principle	Advantages	Disadvantages	Reference
Near-infrared spectroscopy (NIR)	9	NIR measures vibrations of atomic bonds due to the change in dipole moment. These are mainly overtones and combination bands.	Inexpensive; high throughput; hardly any sample pre-treatment necessary	Black polymers cannot be reliably identified; problems with polymer blends	32,33,34
Spectroscopy in the mid-infrared (MIR)	9	MIR also measures vibrations of atomic bonds due to the change in dipole moment. Here, bending and stretching vibrations are measured.	Fewer problems with black polymers; better signal-to-noise ratio than NIR	Additional sample pre-treatment necessary, higher measurement time and sample contact still necessary to date	35,36,37
Spectroscopy in the ultraviolet and visible range (UV-Vis)	9	Measurement of reflection or absorption of ultraviolet or visible radiation depending on the colour of the sample.	High throughput; identification of polymers by colour	No differentiation of types of polymers and additives possible if the same dyes are contained	38,39,40,41
X-ray fluorescence analysis (XRF)	9	Disruption of the electron configuration by high-energy radiation and measurement of the fluorescence radiation that is emitted when the electron equilibrium is restored.	High throughput; inexpensive; can identify brominated flame retardants or heavy metals	Can so far only distinguish well between PVC/PVDC and other polymers; accurate detection is limited to elements with ordinal number > 12; additional radiation protection requirements must be met when using XRF	12,21,42
Fluorescence spectroscopy	7	Measurement of the energy absorption of electrons in the ground state & energy emission of electrons in the excited state as a function of the wavelength and intensity of the excitation radiation.	High throughput; can be used for TBS (organic & inorganic markers)	No characteristic spectra for the different types of polymers possible; high-energy radiation can damage the surface of the target objects	22,38,41
RAMAN spectroscopy	7	Detection of the vibration of atomic bonds due to changes in polarisability.	High throughput; complementary to other methods	Low intensity of the reflected radiation leads to low signal-to-noise ratios	43,44,34
Laser-induced breakdown spectroscopy (LIBS)	5	High-energy radiation is used to generate plasma on the surface of the sample, whose emissions can be detected and assigned to specific elements.	Hardly any sample preparation required; enables most accurate identification of polymers including additives based on specific spectral lines of the elements present	The sample must be damaged to generate the plasma; emission of hazardous gases may occur (e.g. formaldehyde from POM); limited throughput until now; problems in distinguishing between plastics with similar elemental composition	45,46,47,48
Time-gated fluorescence spectroscopy	4	Here, the lifetime of the excitation of the fluorescent dyes is measured against that of the autofluorescence of the matrix materials in order to reduce background noise.	Can improve signal-to-noise ratio when used in addition to other methods	Hardware upgrade is expensive; reduced throughput capacity	49,50,51
Photon upconversion	4	Combination of two or more low-energy photons to cause and detect emission from a higher-energy photon. Composite materials are used in which one substance acts as an activator for the second substance (the emitter).	Enables the use of low-energy radiation; enables targeted identification of types of polymers based on the marker fingerprint; very high signal-to-noise ratio	Marker compounds can be costly; circulation system for markers should ideally be in place	34,52,53

Economic aspects

For the large-scale implementation of TBS concepts, economic aspects regarding material costs for the marker material as well as very high investment costs for the conversion of the recycling facilities are crucial. Using a substance already registered as a marker in REACH has the advantage that the registration costs (including the necessary toxicity tests and documentation) are eliminated. For an annual import or production volume of 100-1,000 tonnes, these costs amount to around €800,000 and more on average.[56] Another economically very important aspect is the high investment costs for the detection and sorting units that would be necessary for large-scale implementation in (existing) recycling plants, especially if the chosen methods have a low TRL. The TRL is also an indicator of the R&D costs for the improvement of a sorting technology up to the large-scale implementation of such TBS systems. In principle, it can be assumed that increases in TRL will also result in strong increases of the R&D costs.[57] Nevertheless, TBS systems that already have a high TRL are advantageous from an economic point of view, as further development costs are saved. These additional costs resulting from the TBS implementation could possibly be offset in view of an "EU plastic tax" of currently €0.80 per tonne of nonrecycled plastic. However, more detailed cost analyses or profitability calculations would have to be carried out for this.

As with almost all new technologies, existing technical, environmental, and social barriers to TBS must also be overcome, and relevant stakeholders must be convinced to invest the necessary resources. For example, relevant stakeholders have already been asked about market drivers and barriers with regard to TBS as part of a study.[58] In this survey, "regulatory and legal barriers", "distribution of costs and benefits", "profitability and competition", "quality and safety aspects", and "procedures and technical issues" emerged as the most critical concerns.[58]

The above-mentioned study also presented two scenarios in more detail: “TBSlight” and “TBScomplete”. In the “TBSlight” scenario, TBS machines are introduced at the end of the already established sorting process to remove problematic materials such as composites. In the “TBScomplete” scenario, TBS machines completely replace conventional sorting technologies to enable single plastic streams with very high purity. The study acknowledges that a future implementation of “TBSlight” (when compared with “TBScomplete”) is much more probable, as the already existing recycling system was considered too inflexible to actually realise the technically “radical” changes for “TBScomplete”. However, it was also mentioned that “TBSlight” is not sufficient to be able to achieve the legally binding recycling targets and the desired reduction of the carbon footprint.[58,15]

Conclusion

Tracer-based sorting (TBS) is a promising concept or technical measure that has the potential to meet the recycling targets of specific waste streams, such as technical plastics from waste electrical equipment, using innovative detection methods. For this, spectroscopy-based methods such as NIR, spectroscopy in the ultraviolet and visible range, photon upconversion or X-ray fluorescence can be used for fully automated sensor-based sorting of used plastics. The marker materials with so-called fingerprints* would be integrated directly into the polymer. The challenge here is to develop a standardised marking code – similar to the Resin Identification Code (RIC) – to enable uniform marking and sorting by several producers, suppliers, and sorters. According to our study[26], rare earth elements such as cerium or yttrium oxide are suitable for marker application; in view of the EU chemicals legislation (REACH and CLP regulations), sound data is available on relevant physicochemical parameters and potential toxicity. Moreover, the organic dye perylene3,4,9,10-tetracarboxylic dianhydride could also be incorporated into plastic products, as this marker has a specific fingerprint in the NIR spectrum. However, successful large-scale implementation of such TBS concepts would have to be investigated in more detail within the framework of feasibility studies. From an environmental, safety, and economic point of view, special consideration should be given to the carry-over or continuation/perpetuation of the marker materials during multiple recycling cycles. In summary, the aim should be to prove that TBS significantly improves the homogeneity of a waste plastics stream and thus increases the material quality and the yield of high-quality recycled materials. During this process, undesirable cross-contamination into other types of plastics must be avoided and the traceability of the markers guaranteed. Finally, if TBS technology is to be implemented successfully in the future, economic efficiency of TBS implementation must also be investigated.

* *Unique combination of markers. By assigning marker fingerprints to individual plastic products with the same composition, the mixing of plastics with different compositions can be avoided and a high degree of purity can be achieved.*

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Owner: Austrian Academy of Sciences; legal person under public law (BGBl 569/1921 idF BGBl I 31/2018); Dr. Ignaz Seipel-Platz 2, A-1010 Vienna

Editor: Institute of Technology Assessment (ITA); Bäckerstraße 13, A-1010 Vienna; www.oeaw.ac.at/ita

Mode of publication: The NanoTrust Dossiers are published irregularly and contain the research results of the Institute of Technology Assessment in the framework of its research project NanoTrust. The Dossiers are made available to the public exclusively via the Internet portal “epub.oeaw”: epub.oeaw.ac.at/ita/nanotrust-dossiers/

NanoTrust-Dossier Nr. 60en, May 2023: epub.oeaw.ac.at/ita/nanotrust-dossiers/dossier060en.pdf

ISSN: 1998-7293