EPJ Web of Conferences **266**, 12004 (2022) https://doi.org/10.1051/epjconf/202226612004
EOSAM 2022

Analysis of size and concentration of microplastics in water using static light scattering combined with PCA and LDA

Mehrdad Lotfi Choobbari[1], Leonardo Ciaccheri[2], Tatevik Chalyan[3], Barbara Adinolfi[2], Wendy Meulebroeck[3], Heidi Ottevaere[3]*

[1]Vrije Universiteit Brussel, Department of Applied Physics and Photonics, Brussel Photonics, Pleinlaan 2, 1050 Brussels, Belgium
[2]CNR-Istituto di Fisica Applicata "Nello Carrara", Via Madonna del Piano 10 - 50019, Sesto Fiorentino (FI) -Italy
[3]Vrije Universiteit Brussel and Flanders Make, Department of Applied Physics and Photonics, Brussel Photonics, Pleinlaan 2, 1050 Brussels, Belgium

Abstract. Quantitative analysis of size and concentration of microplastics is a crucial step for having a better understanding of plastic pollution in the environment. Such information is typically obtained in a single particle mode that significantly increases the analysis time and can be a cumbersome task. Therefore, we demonstrate here a measurement technique based on Static Light Scattering (SLS) combined with chemometric methods such as Principal Component Analysis (PCA) and Linear Discriminant Analysis (LDA) for resolving the size and concentration of multiple microplastic particles in water. Two sets of samples with uniform and non-uniform size distribution of microplastics, called “monodisperse” and “polydisperse”, respectively, are fully investigated. It is shown that a relationship exists between the scattering signals of mono- and polydisperse samples on the PCA space. Hence, a PCA-LDA model that is constructed on the PCA space of monodisperse samples is used to discriminate the size of the microplastics in polydisperse samples. By specifying the size of the particles, their concentration is determined using a simple linear fit.

1 Introduction

Without any doubt, plastics are one of the key materials for today industries with applications in different areas [1]. Notwithstanding, the improper management of plastic waste and the low percentage of them being recycled has led to a new environmental challenge called “Microplastic (MP) identification”. These plastic particles can be found everywhere, even into human placentas [2].

One of the necessary actions for tackling the problem of microplastics is to have a better understanding of their presence in nature through standardized regulatory protocols that are currently lacking in the field. Therefore, different analytical techniques such as Raman spectroscopy (RS), Fourier-transform infrared spectroscopy (FTIR) and static light scattering (SLS) have been proposed for the study of MPs. RS and FTIR have received great attention due to their valuable advantages. Nevertheless, these methods may not be suitable for sizing and enumeration of MPs. It was recently demonstrated that light scattering can be used to study the surface characteristics of MPs. Light scattering is a well-stablished technique that proved to be very useful for the characterization of small particles [3]. Nevertheless, less attention has been paid to this valuable technique for the analysis of microplastics in recent years. Accordingly, a practical approach based on the combination of SLS with chemometric methods is proposed here for the batch analysis of size and concentration of microplastics in water. PCA is applied on the SLS signals of polystyrene (PS) samples to classify the size of the particles. Subsequently, an LDA model is trained using the PCA results of monodisperse samples. Next, the PCA-LDA model is used to classify the size of the particles in unknown mono- and polydisperse samples. Finally, the concentration of particles is determined using a simple linear fit.

2 Materials and methods

Monodisperse PS microspheres with different diameters were used as the target MPs in this work. Light scattering measurements were performed using a REFLET-180S goniophotometer (Light Tec) which can work either in transmittance or reflectance mode. As shown in Fig. 1, the optical design consists of two symmetric arms being the illumination and the detection parts which are focused at the same point on the sample. The collimated light from a 100 W Tungsten Halogen lamp with a beam diameter of 0.2 millimetre passes through the sample container at a fixed position and the photodetector detects the scattered light by scanning from θ = -90° to +90° with an angular

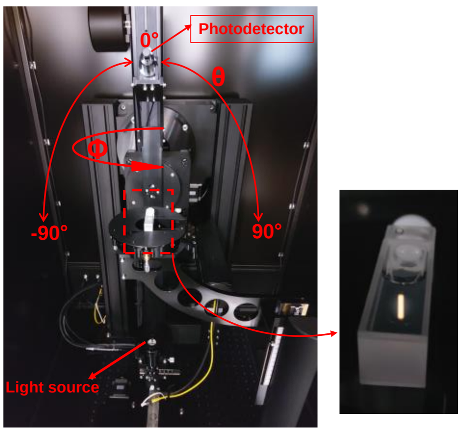

Fig. 1. Multi-angle static light scattering measurement setup.

resolution of 0.1°. The total duration of one measurement was 30 s. The format of the output file was a two columns matrix consisting of the Bidirectional Transmittance

* Corresponding author: Mehrdad.Lotfi.Choobbari@vub.be

© The Authors, published by EDP Sciences. This is an open access article distributed under the terms of the Creative Commons Attribution License 4.0 (http://creativecommons.org/licenses/by/4.0/).

Distribution Function (BTDF) versus the scattering angles. Further processing and analysis on the output data were performed using MATLAB and The Unscrambler (Aspen Technology).

3 Results and discussion

After measuring the BTDF signal of the MPs, the first derivative of the scattering intensity patterns was calculated using a second order, nine-point Savitsky-Golay filter as shown in Fig. 2. As a result, most of the information about the concentration of particles was removed, however, the difference between the shape of the scattering intensity patterns was significantly enhanced with respect to the size of the scatterers. Accordingly, this data was used as the input for the PCA analysis to discriminate the size of the particles.

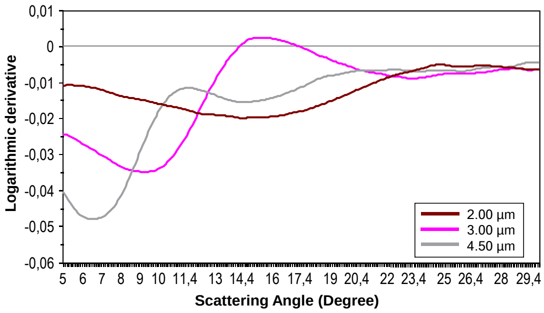

Fig. 2. Logarithmic derivative of the scattering intensity plots for different microplastic diameters.

PCA is known as an unsupervised chemometric method that can split between the groups of a dataset by identifying the existing patterns among them with no prior knowledge. Therefore, PCA was applied on the first derivative of the logarithmic scattering intensity patterns of the monodisperse samples to discriminate the size of the particles. As it is clear from Fig. 3, the first two PCs accounting for more than 80% of the total variance, were enough to discriminate between the size of the particles. Fig. 3 also demonstrates the projection of the PCA map of polydisperse samples (pink-diamond symbols). As seen, the PCA map of a polydisperse sample is located between the PCA maps of its constituent particles, i.e., the monodisperse samples. Thanks to this relationship, a PCA-LDA model was trained to classify the size of the particles in unknown mono- and polydisperse samples. The classification/prediction rate was 100% in both scenarios.

After specifying the size of the particles in a sample, their concentration can be determined using the scattering intensity at an arbitrary angle from the selected angular range. Fig. 4 represents the decimal logarithms of particle concentrations against the scattering intensity which was measured at 30° for all particle sizes. As seen, almost a linear relationship was obtained. Therefore, a simple linear fit was enough for measuring the concentration of MPs in a sample, provided that the size of the particles was known.

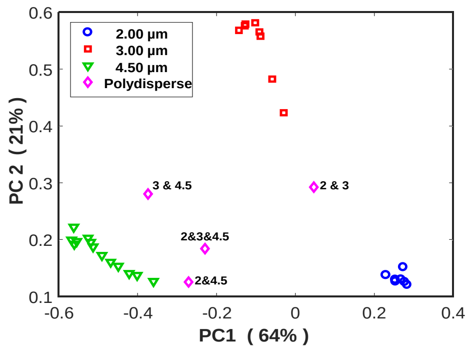

Fig. 3. PCA maps of mono- and polydisperse samples.

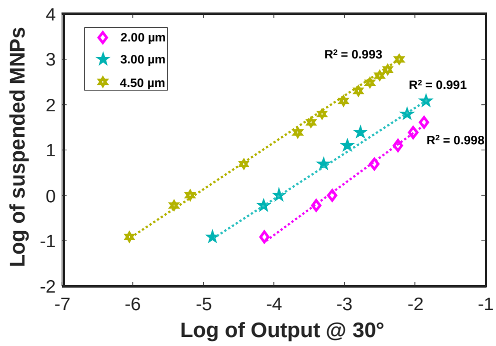

Fig. 4. The logarithm of MPs concentration in water versus the logarithm of scattering intensity at 30°.

4 Conclusion

Despite being a well-stablished technique for the characterization of small particles, light scattering received the past years less attention in comparison to other common optical techniques such as Raman spectroscopy and FTIR, for the analysis of microplastics. Here, it was demonstrated that the combination of light scattering with chemometric pattern recognition algorithms, such as PCA and LDA, can bring valuable advantages for the batch analysis of the size and concentration of microplastics in water. In a next step SLS will be combined with an optofluidic chip for in-situ monitoring of microplastics in water.

5 Acknowledgment

This work was supported by the “MONPLAS” European Union’s Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 860775.

References

[1] D. M. Mitrano and W. Wohlleben, *Nat. Commun.*, vol. 11, no. 1, pp. 1–12, 2020, doi: 10.1038/s41467-020-19069-1.

[2] A. Ragusa *et al.*, *Environ. Int.*, vol. 146, p. 106274, 2021, doi: 10.1016/j.envint.2020.106274.

[3] G. Balakrishnan, F. Lagarde, C. Chassenieux, and T. Nicolai, *Preprints*, vol. 2020030189, no. March, pp. 1–27, 2020, doi: 10.20944/preprints202003.0189.v1.