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Qualitative and quantitative analysis of microplastic and pigment particles in freshwater

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Motivation

- It was recently suggested to classify plastic waste as hazardous.[1]

- Microplastic (<5 mm) is constantly accumulating in aquatic environments; colorfulness of microplastic is gained by adding pigments. However, colorfulness is also a feature of particles of paint and coatings

Beach on the northern shore of Lake Garda [2]

- A reliable and standardized method to quantify the amount of (micro)plastic and pigment particles is required

- Raman microspectroscopy is a suitable tool for identification and quantification of (micro)plastic and pigment particles in sediments of aquatic ecosystems

Raman microspectroscopy (RM)
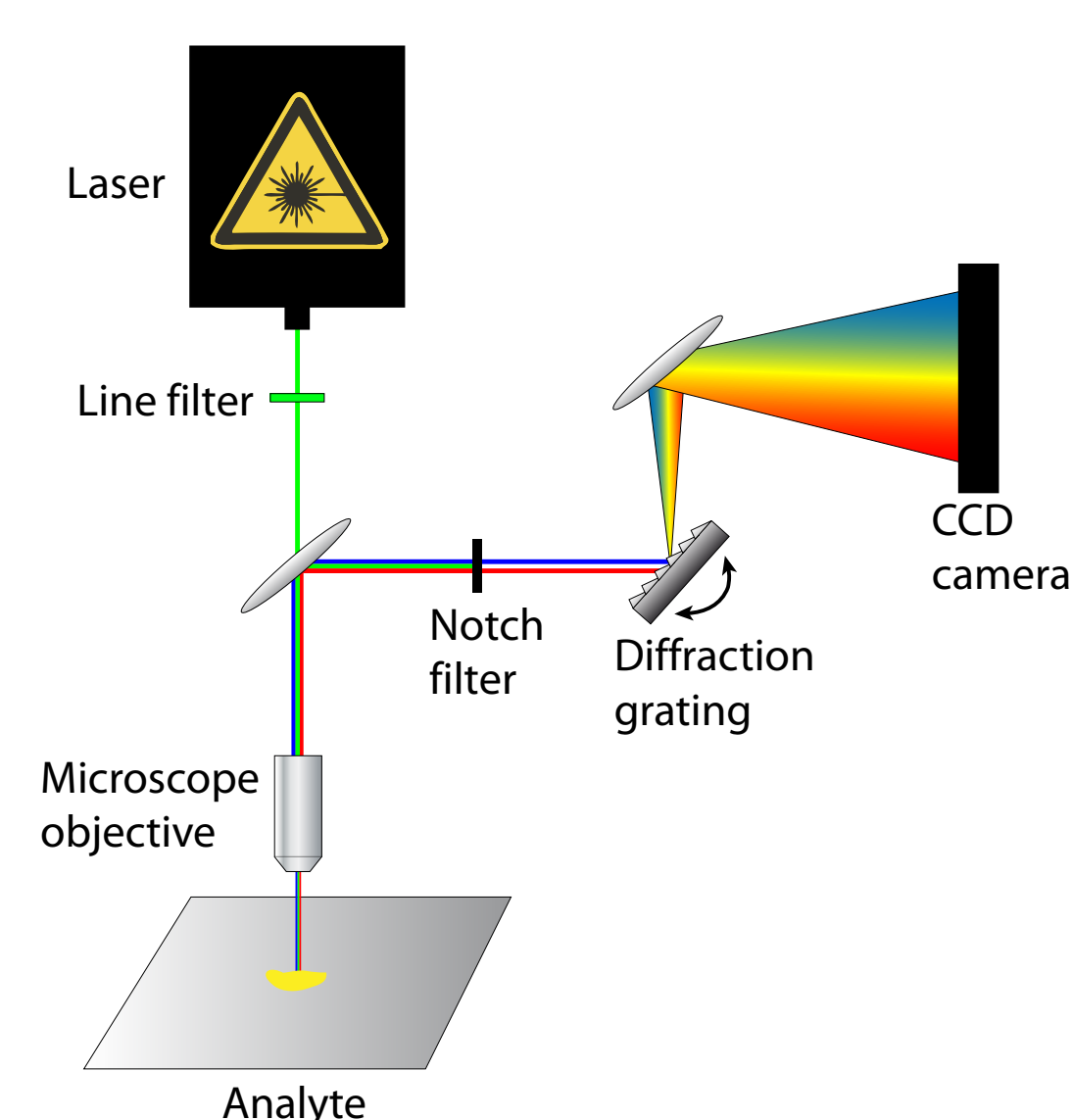
- Based on inelastic light scattering
- Provides molecular fingerprint spectra
- Noninvasive, nondestructive and water-insensitive
- Spatial resolution in the µm-range
- Very sensitive to polymer samples
- Identification of pigments possible

Schematic drawing of a Raman microscopic system

Experimental: plastic separation from sediment

- Sediment samples from Lake Garda
- Density fractionation with $ZnCl_2$ solution (density 1.6-1.7 kg/L)
- Application of the Munich Plastic Sediment Separator (MPSS)
- Collection on a quartz fibre filter
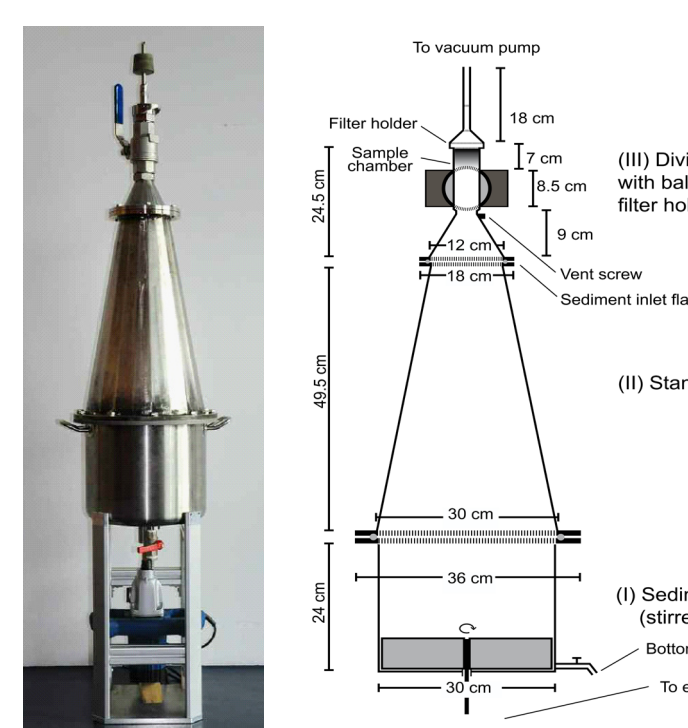- Microplastic size classes: [3]

Foto and scheme of MPSS [3]

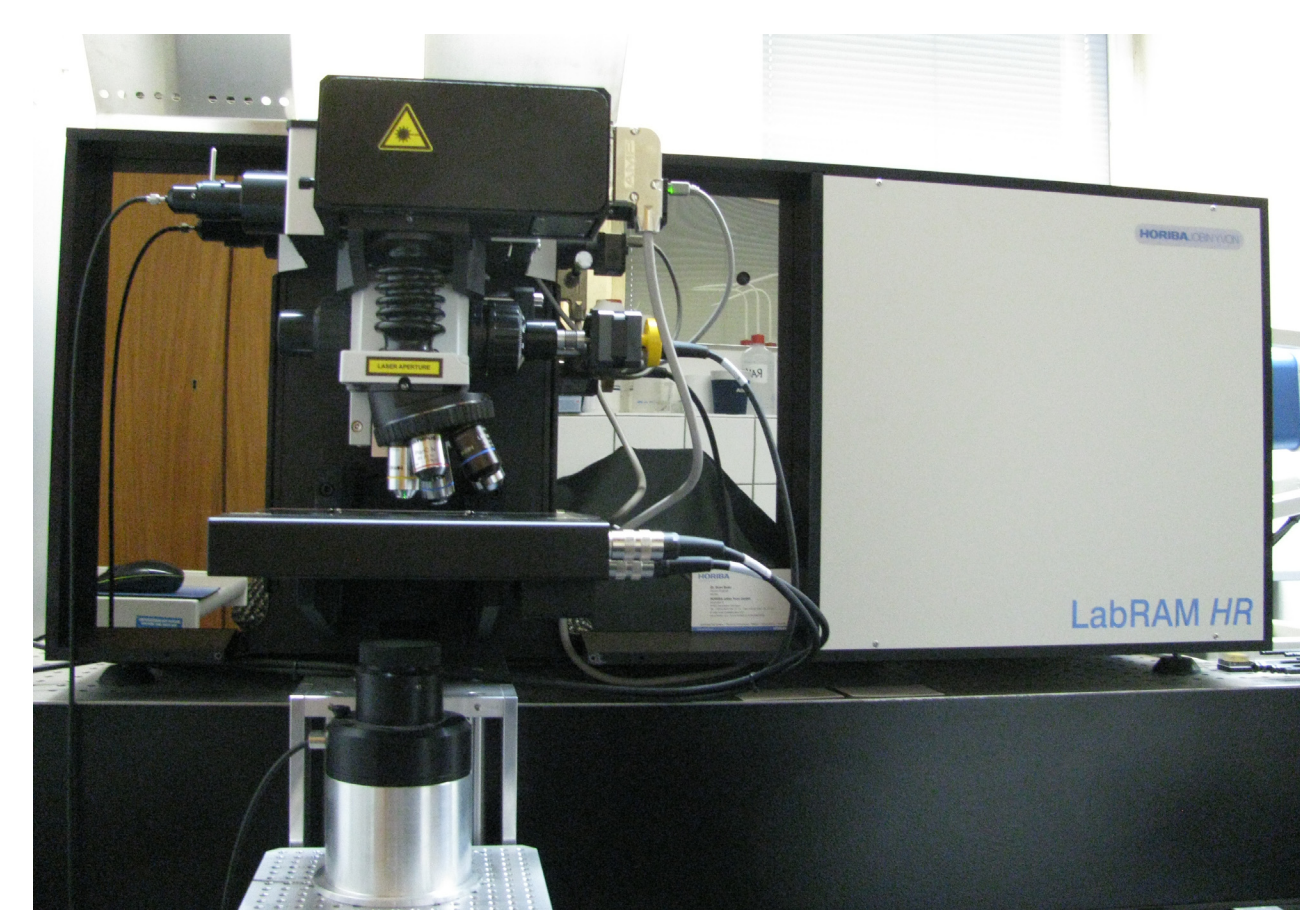|	
Large Microplastic (L-MPP):	500 µm - 5 mm
Small Microplastic (S-MPP):	50 µm - 500 µm
Very Small Microplastic (VS-MPP):	1 µm - 50 µm

Experimental: identification of plastic by RM

Photo of Raman microscope LabRAM HR
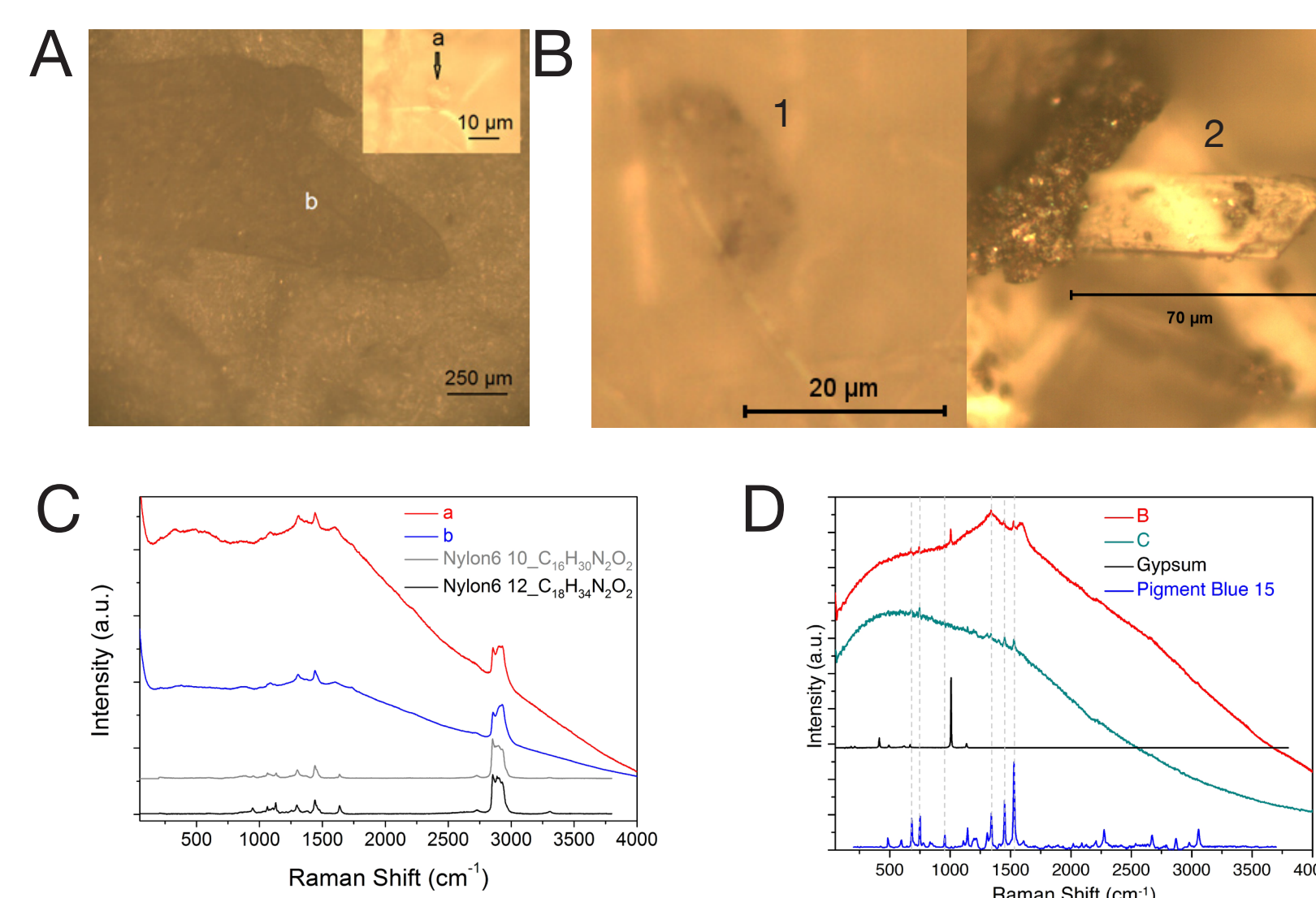
Raman microscope LabRAM HR (Horiba Scientific)

- 633-nm excitation laser (He-Ne)
- 3 objectives: 50× (NA=0.75), 50× long working distance (LWD) (NA=0.5) and 100× (NA=0.90)
- 1 – 100 s acquisition time

Results: Microplastic and -pigment particles

- Microsized plastic and pigment particles were found in all size classes
- Smallest microplastic particle found with 9 µm size

Optical microscope images of a found microplastic with magnification in the inlet (A) and two microsized pigmented particles (B) and their corresponding spectra with references (C and D) [4,5]

Results: Particles found in Lake Garda
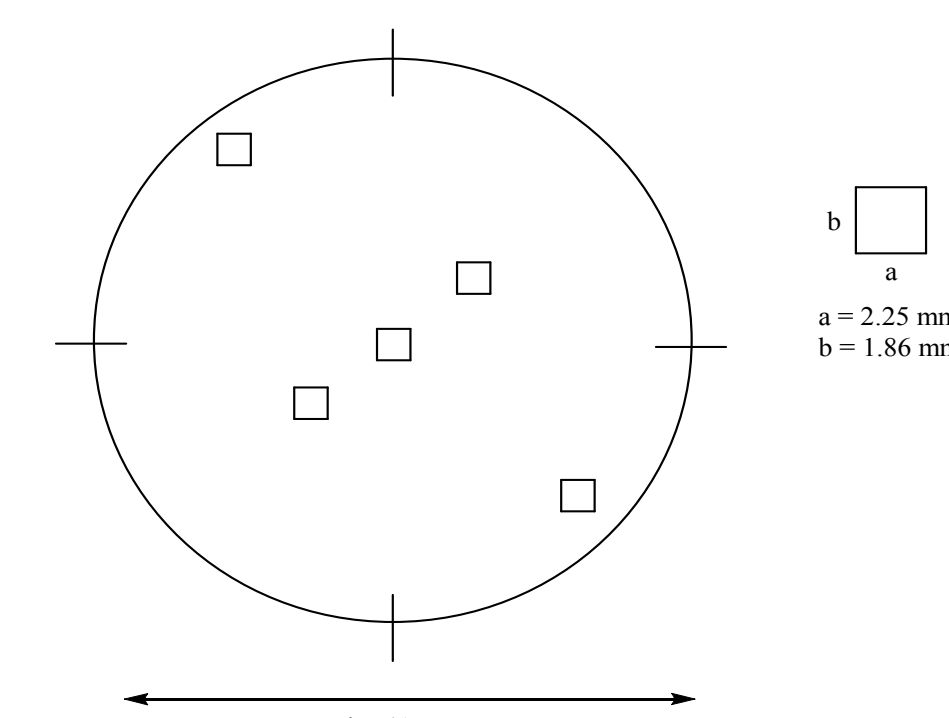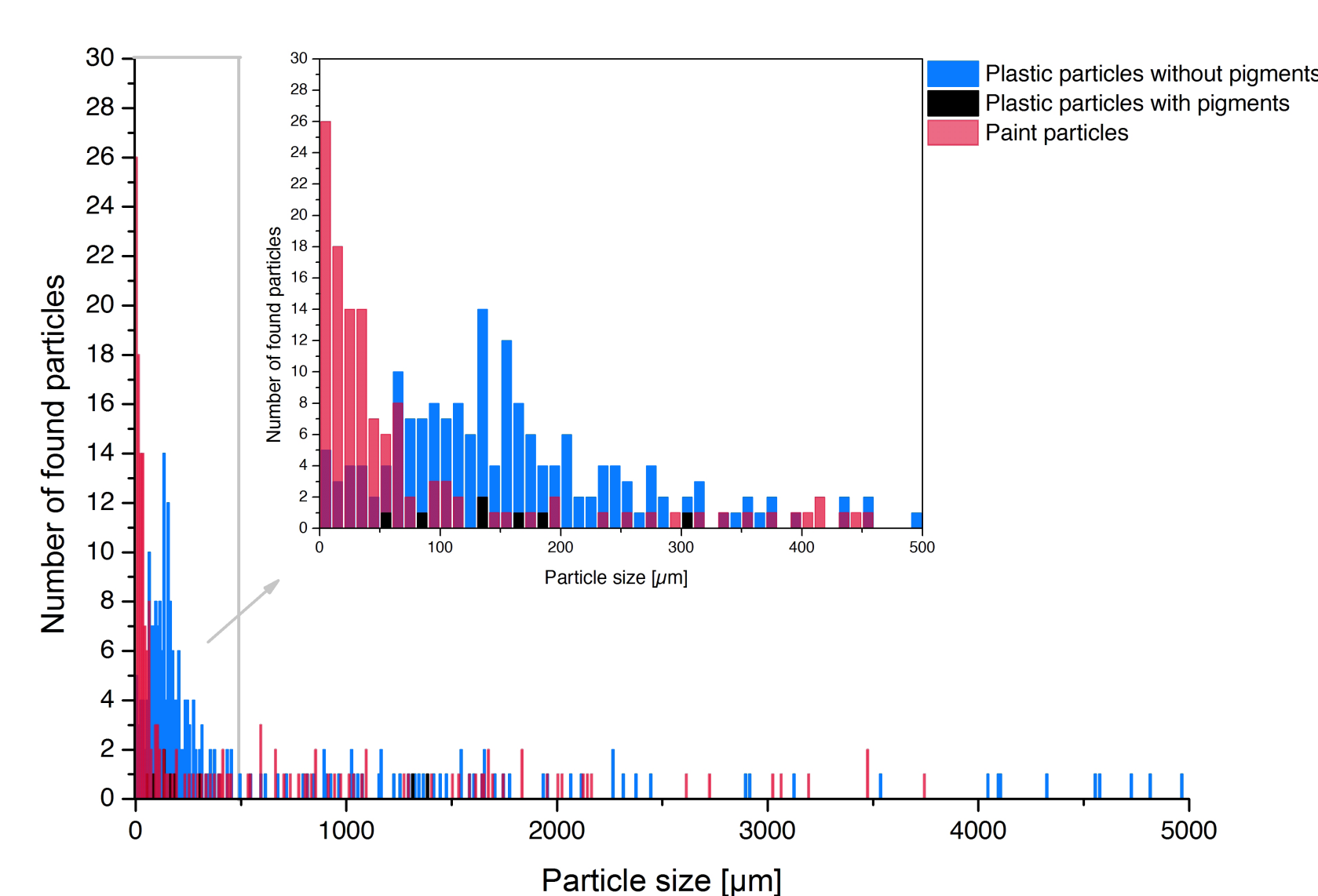
*Left: Overview of all identified particles with magnification inlett [5]*

Right: Schematic image of filter area analyzed for S-MPP and VS-MPP [5]

- More than 600 particles identified
- Pigments detected additionally to (colored) plastic particles
- Distribution of microplastic shows a maximum around 120 µm
- Number of pigments increases strongly below 50 µm
- Most prevalent plastic types: PE, PP, PS and PA
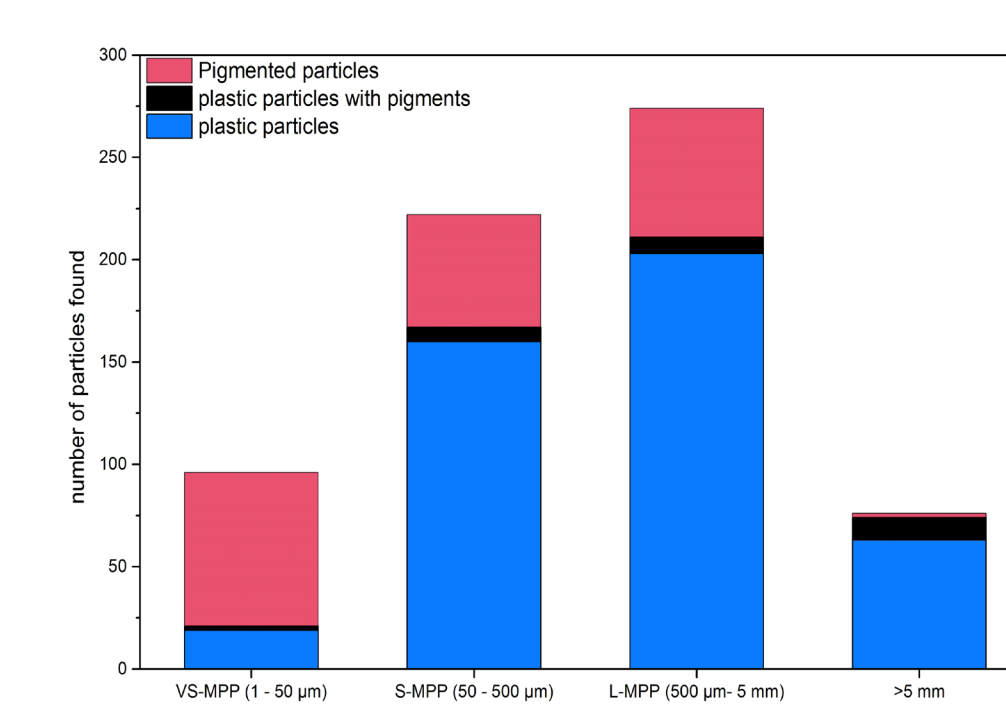
Top: Amount of plastic particles, colored plastic and pigment particles found in the different size classes (L-MPP, S-MPP, VS-MPP)

Bottom: Microsized pigment (A) and plastic (B) particles found
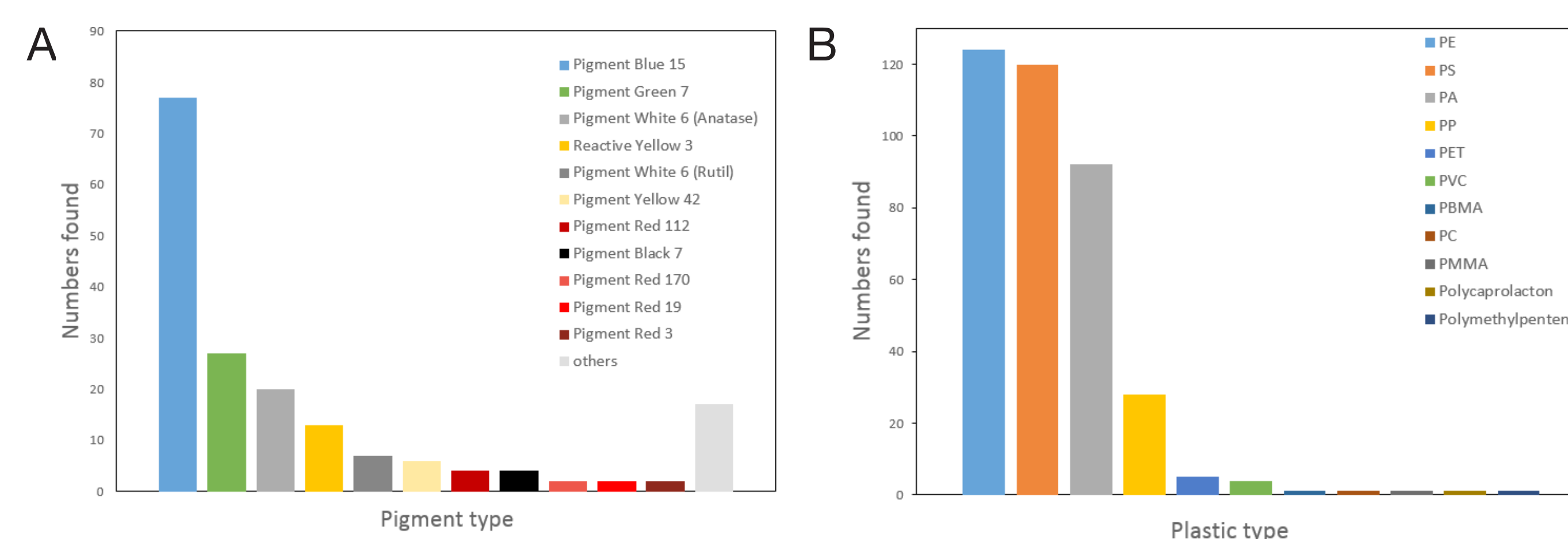
Summary

- RM is well suited for the identification and quantification of different types and size classes of plastic and pigment particles from sediments of aquatic ecosystems down to 1 µm
- Microplastic shows maximum around 120 µm, amount of pigments increases with decreasing size
- Highest number of pigments found in the region of very small microparticles (1 µm - 50 µm)

References

[1] C.M. Rochman, M.A. Browne, *Nature* **2016**, *494*, 169-171.
[2] N.P. Ivleva, R. Niessner, *Nachrichten aus der Chemie* **2015**, *63*, 46-50.
[3] H.K. Imhof, J. Schmid, R. Niessner, N.P. Ivleva, C. Laforsch, *Limnology and Oceanography* **2012**, *10*, 524-537.
[4] H.K. Imhof, J. Schmid, R. Niessner, N.P. Ivleva, C. Laforsch, *Current Biology*, **2013**, *23*, R867-R868.
[5] H. K. Imhof, C. Laforsch, A. C. Wiesheu, J. Schmid, P. M. Anger, R. Niessner, N. P. Ivleva *Water Research*, **2016**, *98*, 64-74.

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