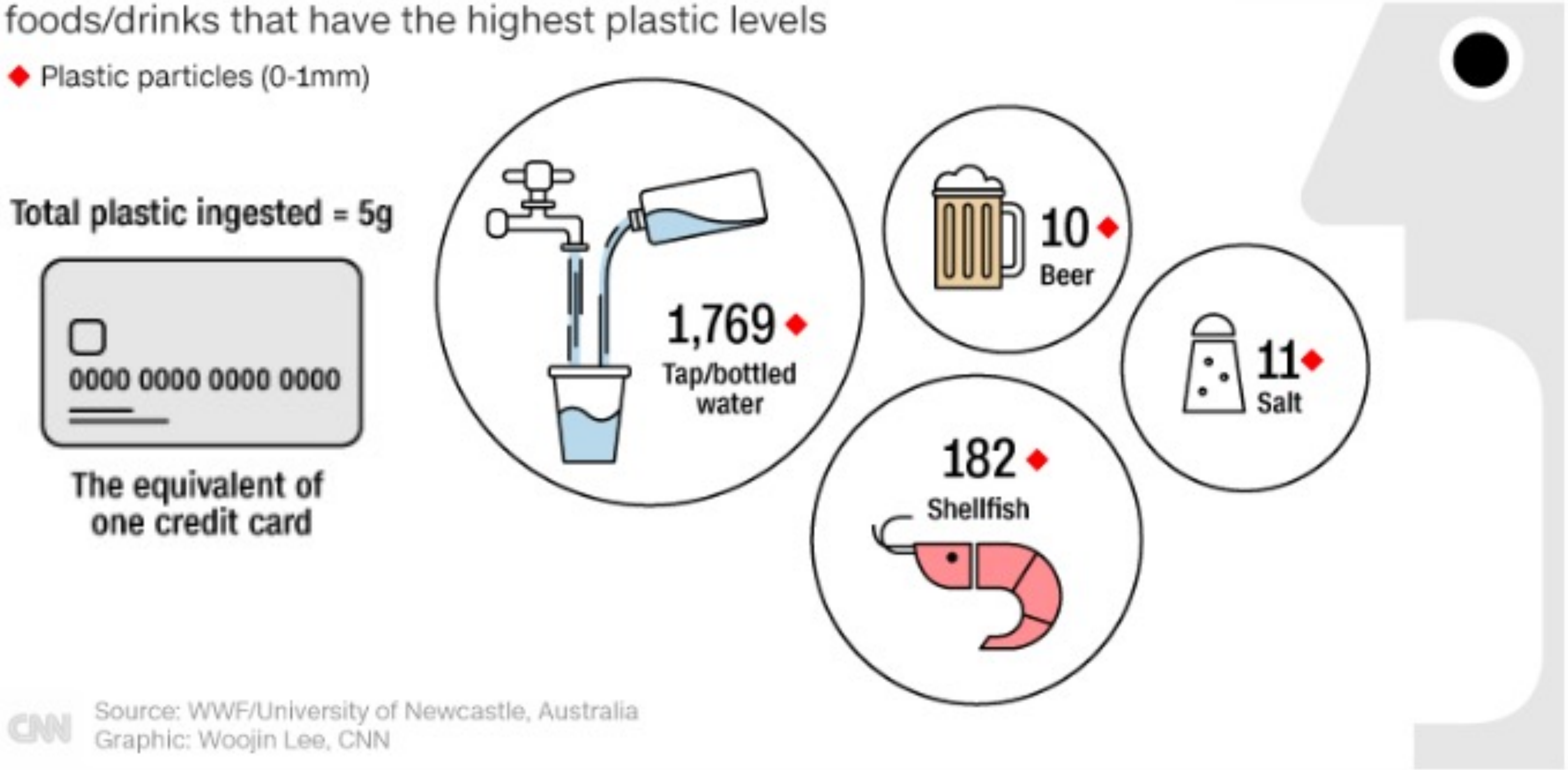
METHOD OPTIMIZATION FOR REDUCTION ANALYSIS OF MICROPLASTICS IN WASTEWATER

BY: SARAH E. ABNEY

Global average weekly plastic consumption

On average people swallow this number of plastic particles each week from the following foods/drinks that have the highest plastic levels



Source: WWF/University of Newcastle, Australia
Graphic: Woojin Lee, CNN

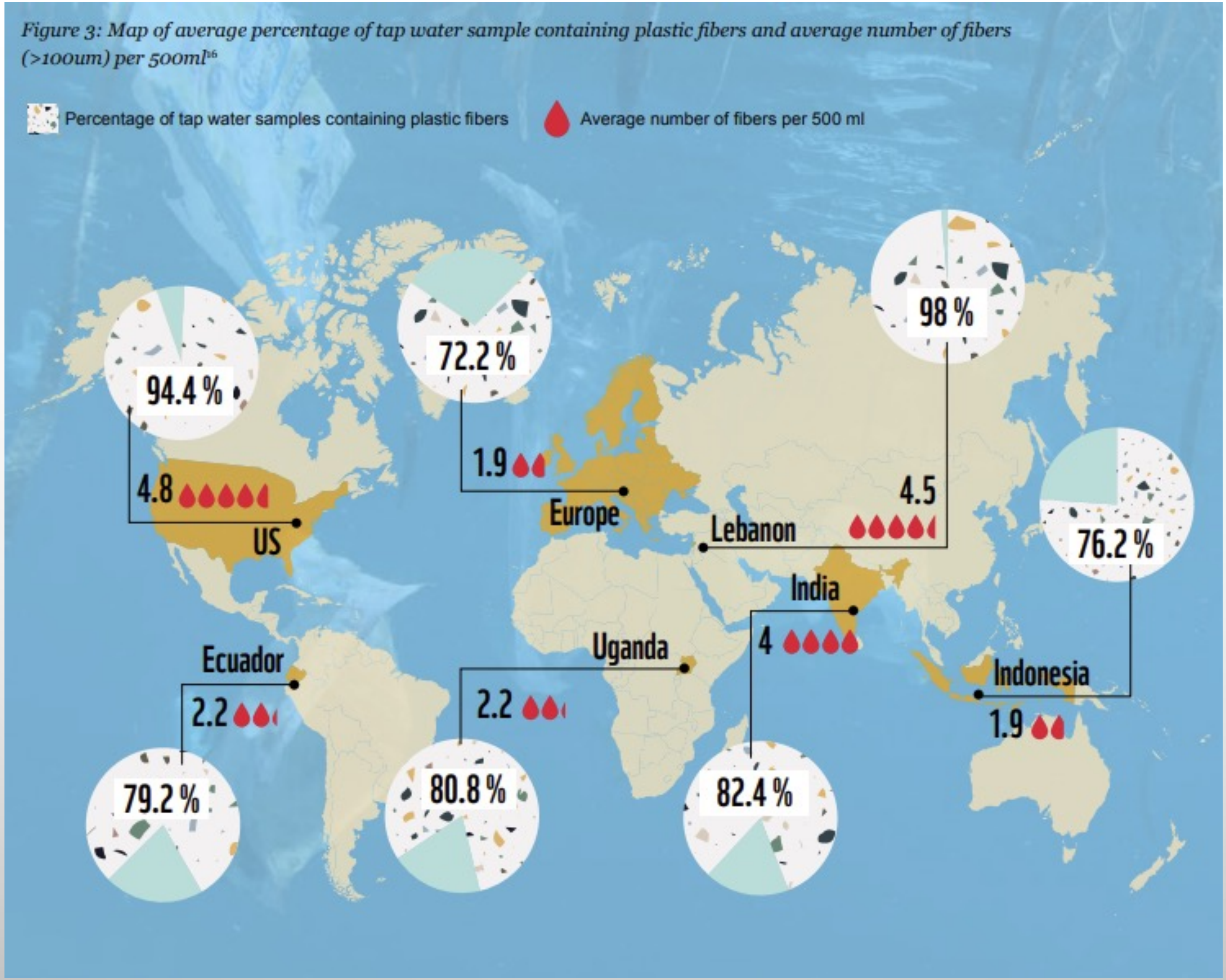
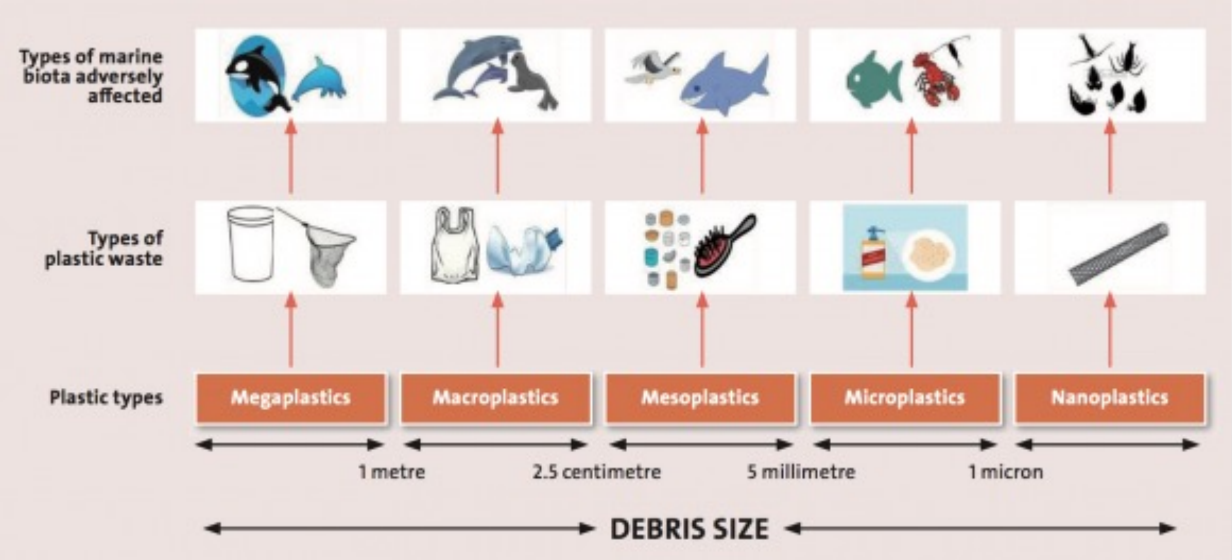
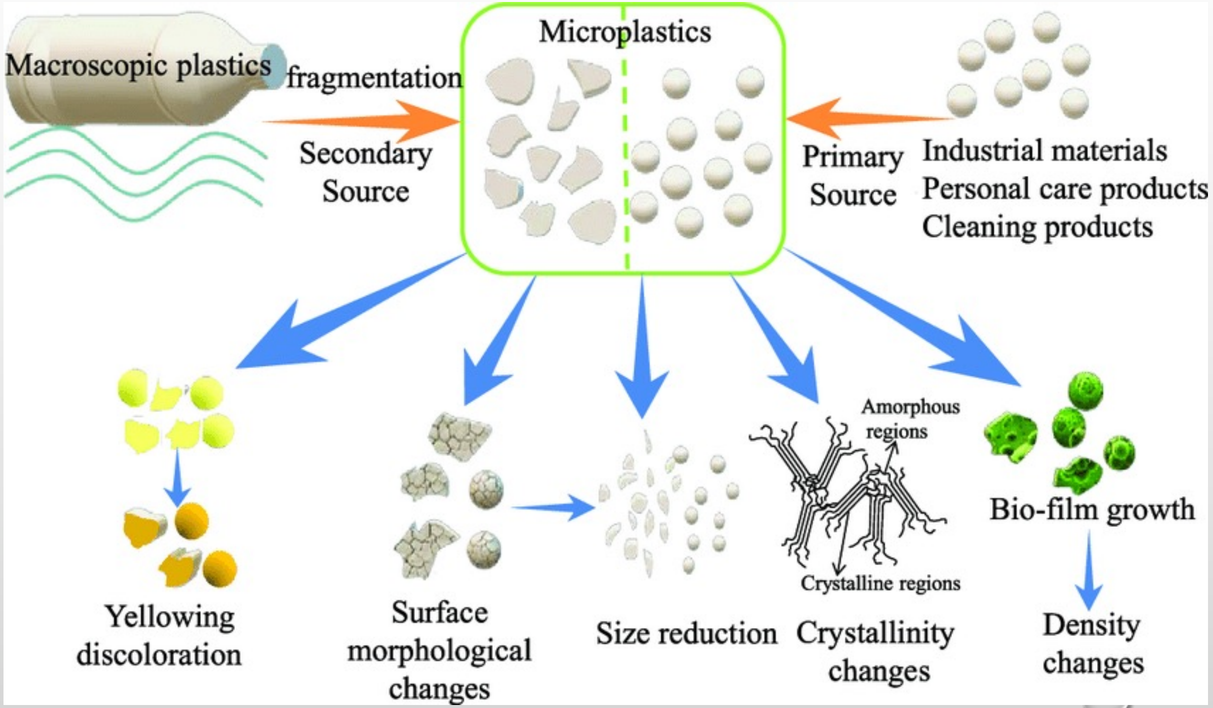
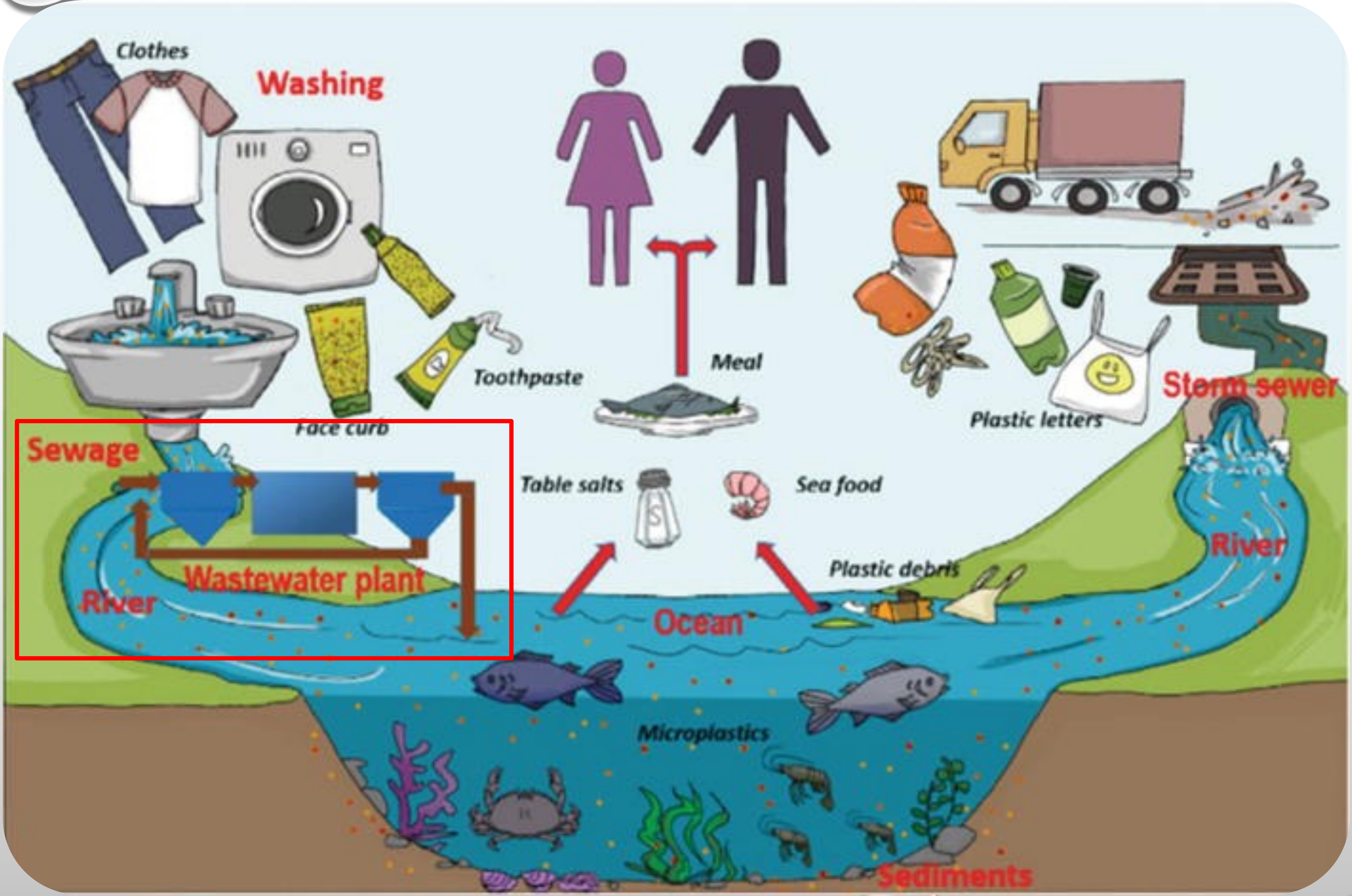


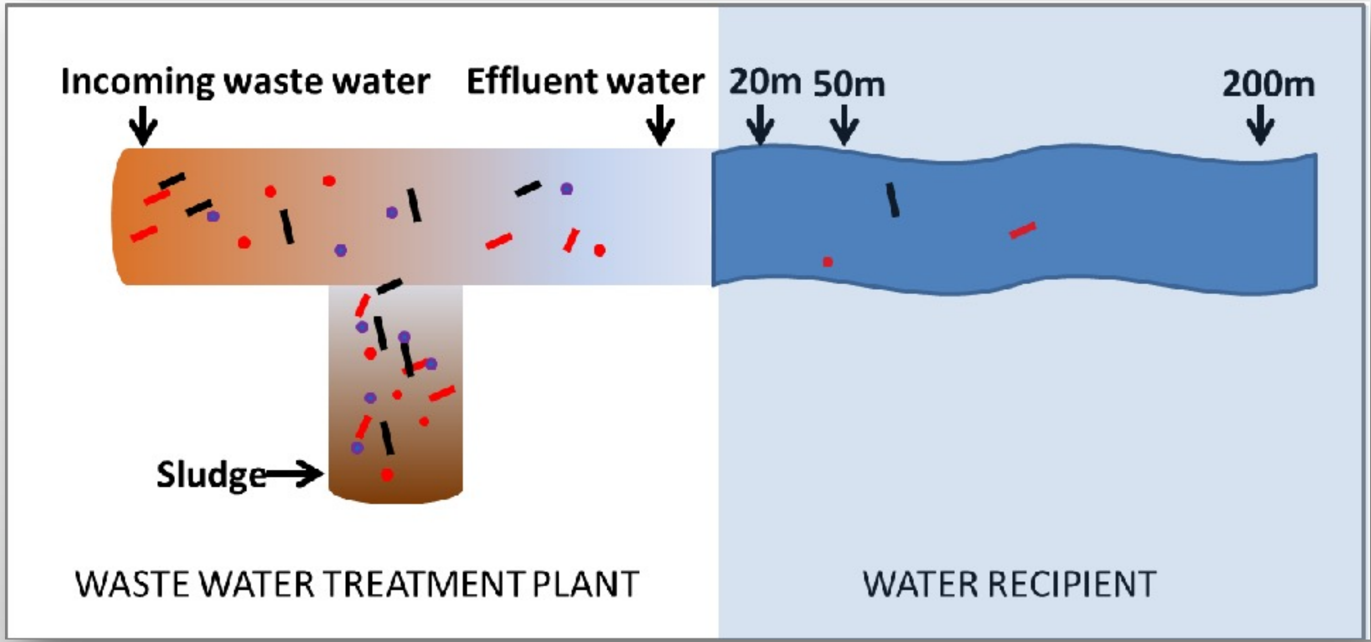
Figure 3: Map of average percentage of tap water sample containing plastic fibers and average number of fibers (>100um) per 500ml[16]
Percentage of tap water samples containing plastic fibers
Average number of fibers per 500 ml
94.4 %
4.8
US
72.2 %
1.9
Europe
98 %
4.5
Lebanon
76.2 %
Indonesia
1.9
India
4
82.4 %
Uganda
2.2
80.8 %
Ecuador
2.2
79.2 %

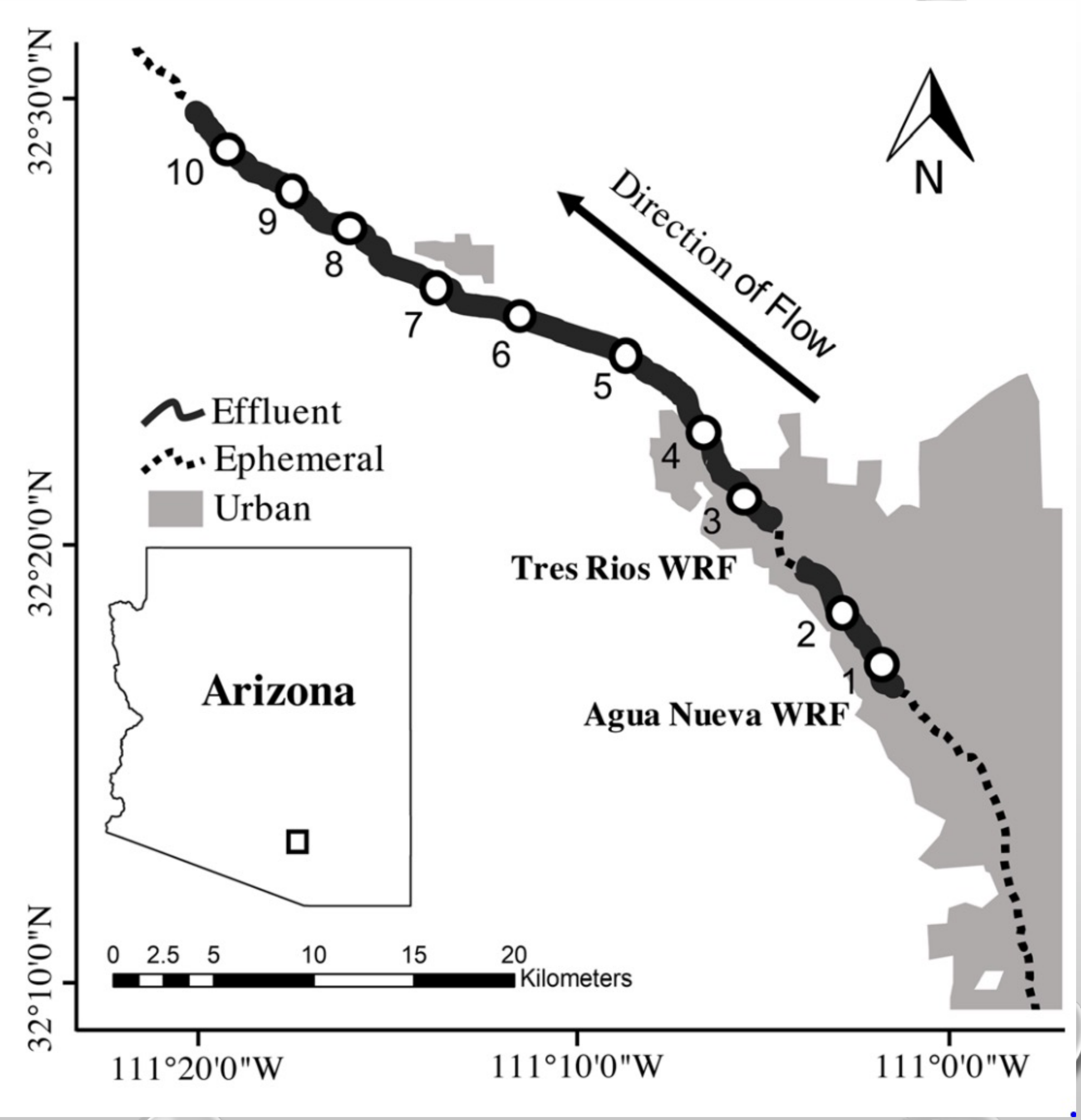

Types of marine biota adversely affected
Types of plastic waste
Plastic types
Megaplastics
Macroplastics
Mesoplastics
Microplastics
Nanoplastics
1 metre
2.5 centimetre
5 millimetre
1 micron
DEBRIS SIZE


Macroscopic plastics
fragmentation
Secondary Source
Microplastics
Primary Source
Industrial materials
Personal care products
Cleaning products
Amorphous regions
Crystalline regions
Bio-film growth
Yellowing discoloration
Surface morphological changes
Size reduction
Crystallinity changes
Density changes

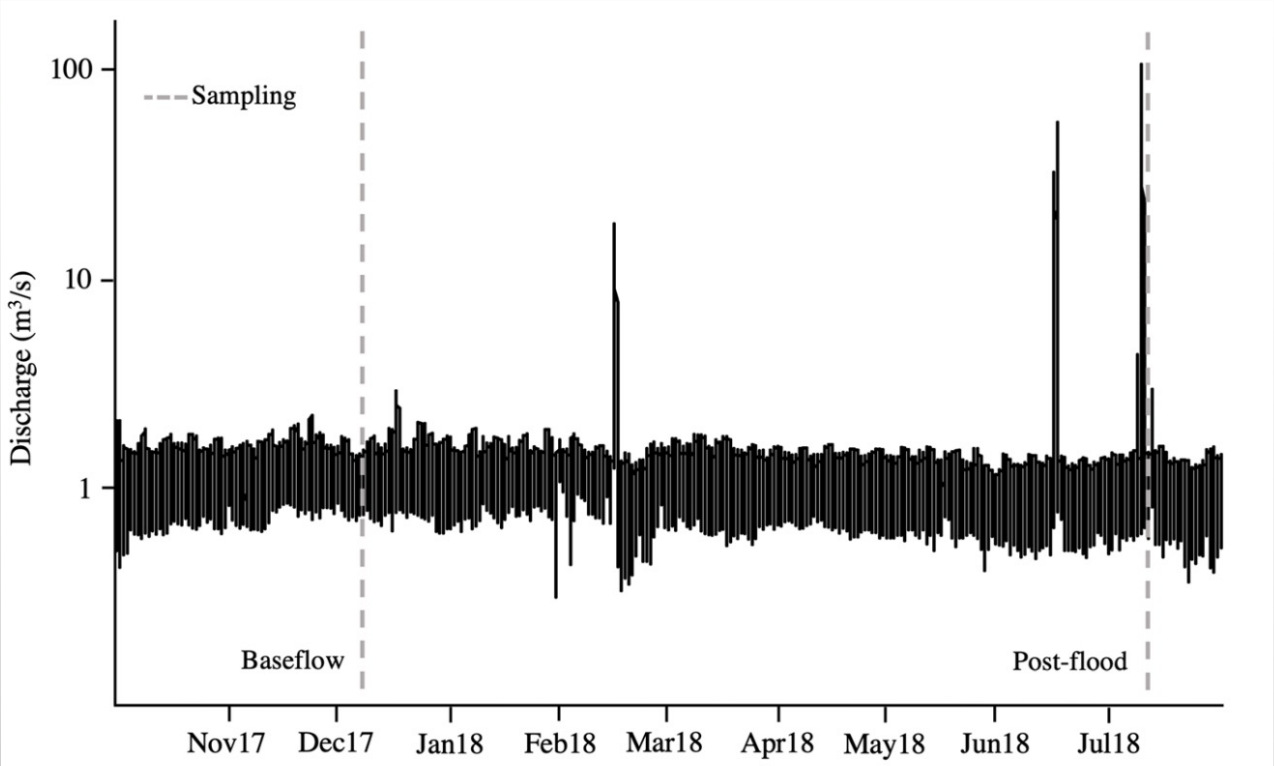


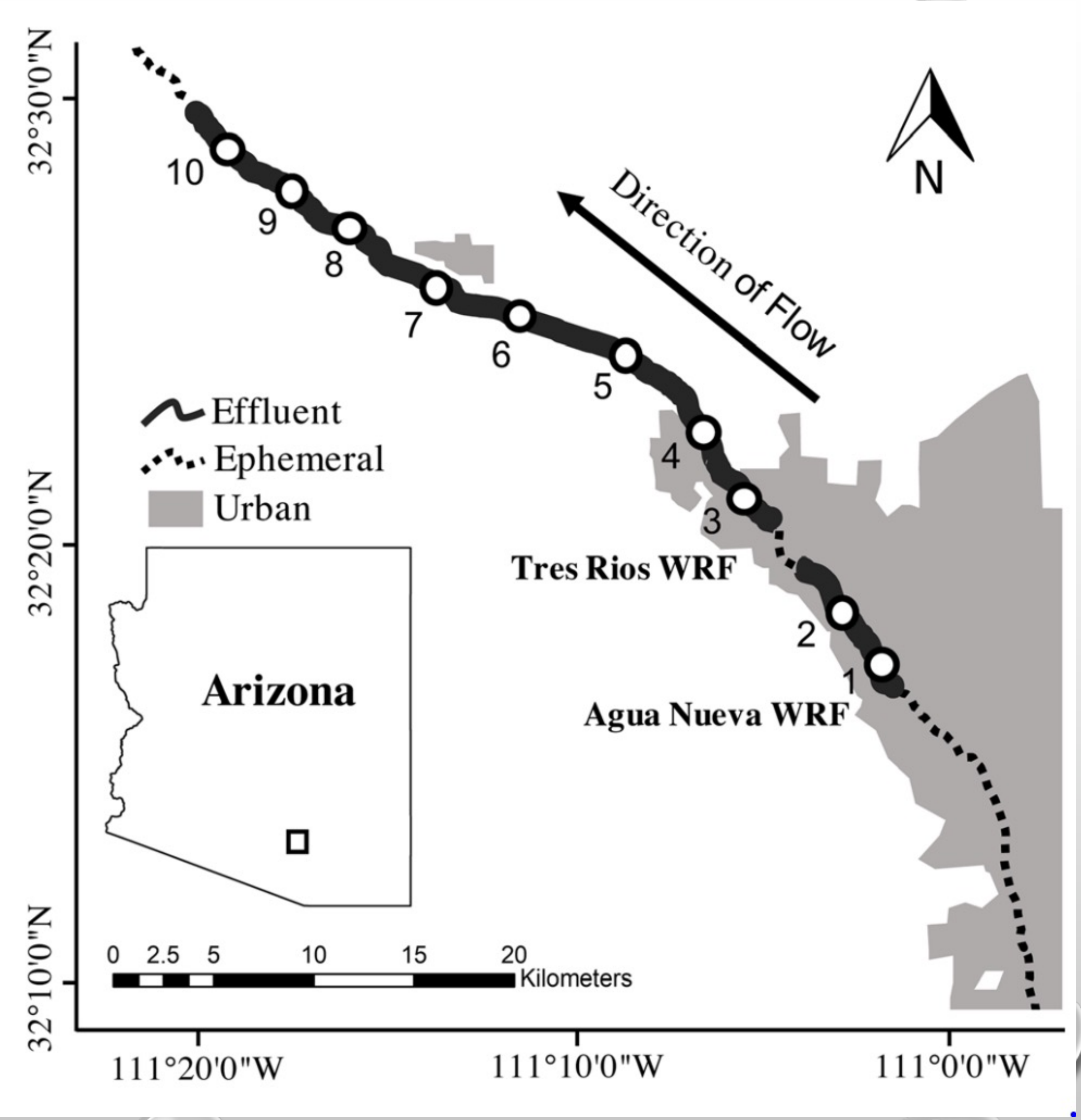
https://www.intechopen.com/chapters/57396





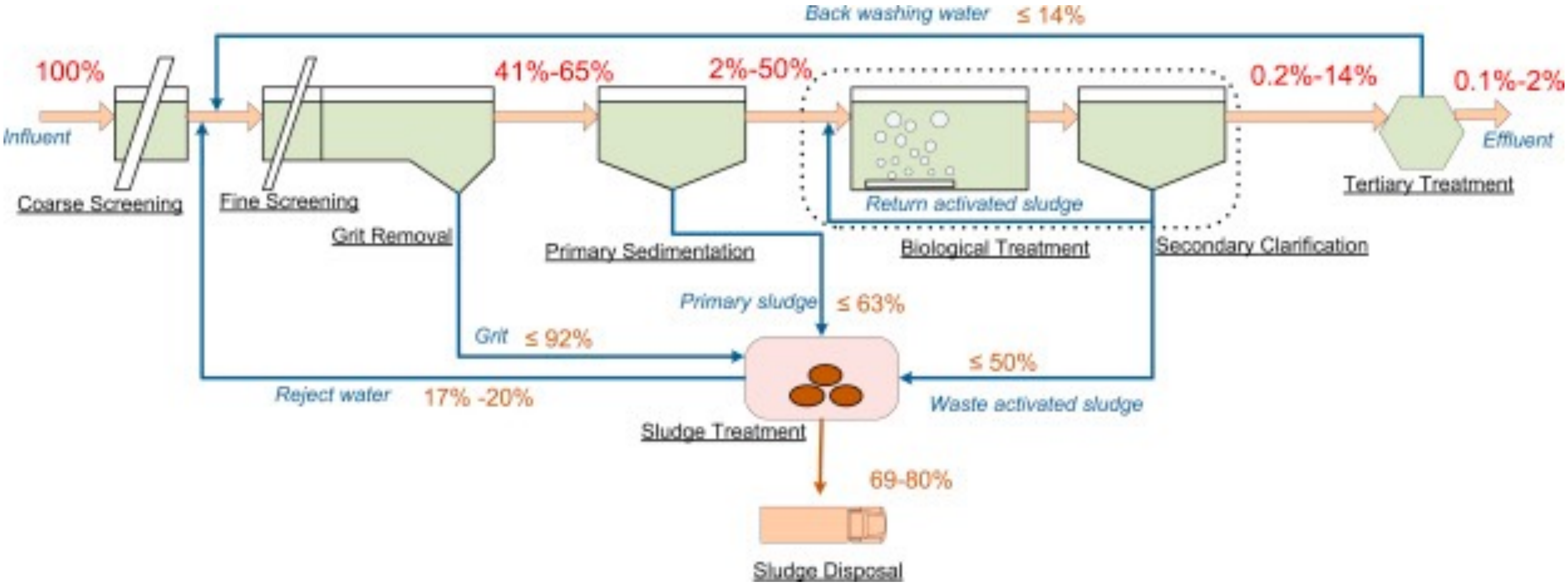
https://www.semanticscholar.org/paper/Screening-of-microplastic-particles-in-and-a-plant-Magnusson-Nor%C3%A9n/5a62de092403c29b6e2de1f87021ee5d30849ffe

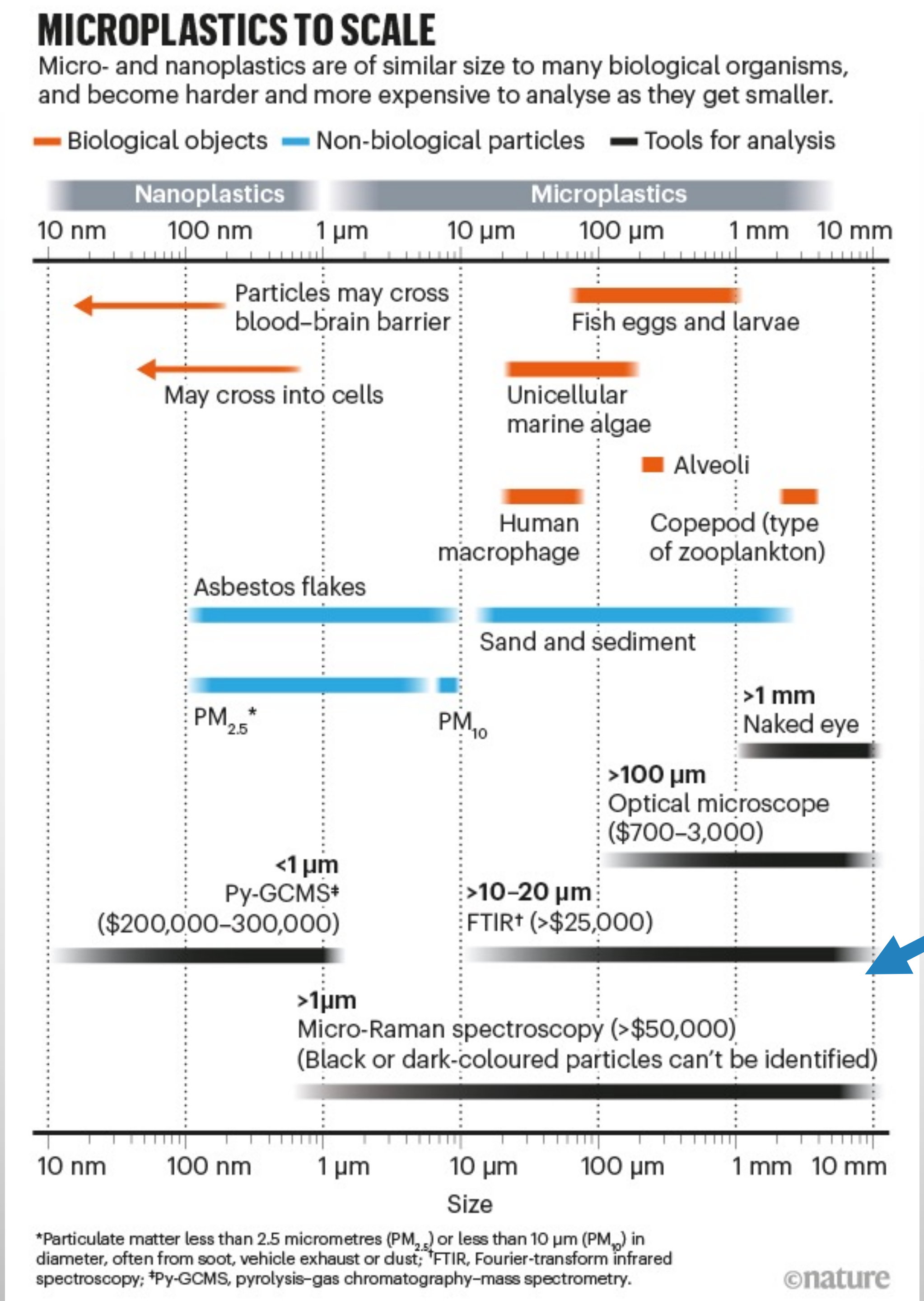




Impacts of baseflow and flooding on microplastic pollution in an effluent-dependent arid land river in the USA

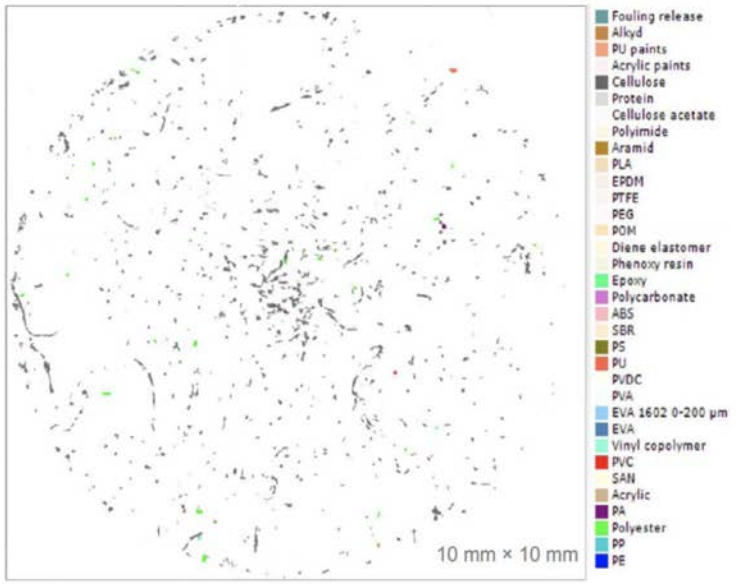
Typical Removal Rates of Wastewater Treatment Plants





HTTPS://WWW.NATURE.COM/ARTICLES/D41586-021-01143-3

FTIR VS LDIR: TIME IS $$



- Imaging FTIR acquires many spectra in parallel using an array detector.
- Example analysis of filtered sediment from a wet retention pond in Denmark[1]:
  - 16×16 mosaic using a 128×128-pixel array
  - A spectrum every 5.5 µm over 10×10 mm^2
  - 3 hours collection + 8 hours data processing (custom software)
  - 33 GB of data
  - 4.2 million spectra
- … for only 871 particles

Figure 4. How does LDIR compare to state-of-the-art FTIR?

Fourier transform infrared (FTIR) spectroscopy is the traditional choice for plastics analysis.
Con: The large incoherent light source can be difficult to focus onto a small microparticle.

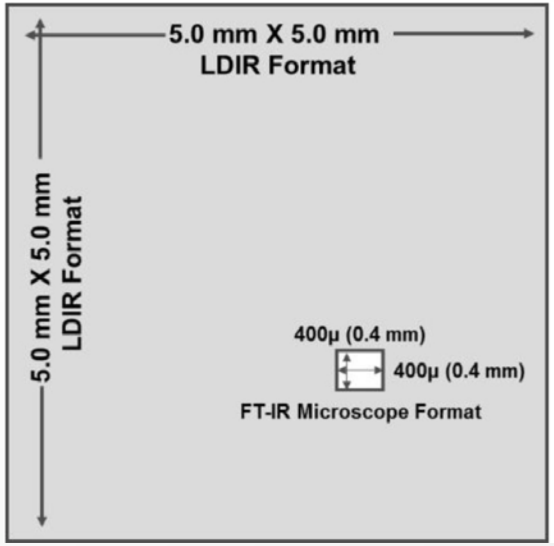
Laser Direct Infrared (LDIR) chemical imaging system features a bright infrared laser source with proprietary **Quantum Cascade Laser (QCL)** technology

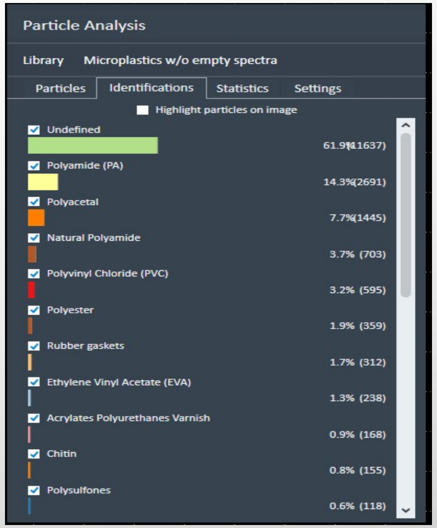
<u>Analysis time for a 33 GB data file:</u>

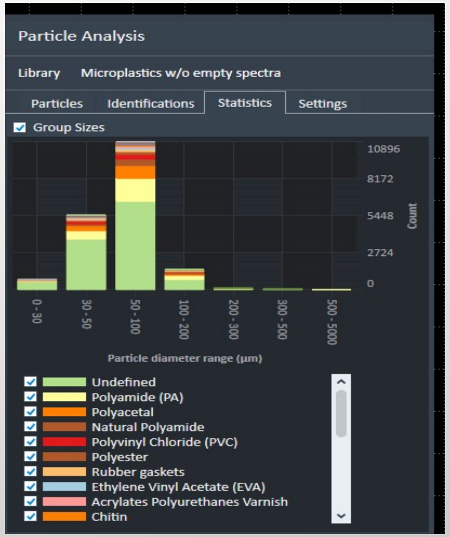
FTIR = 11 hours
LDIR = 2 hours

The key benefits claimed by Agilent

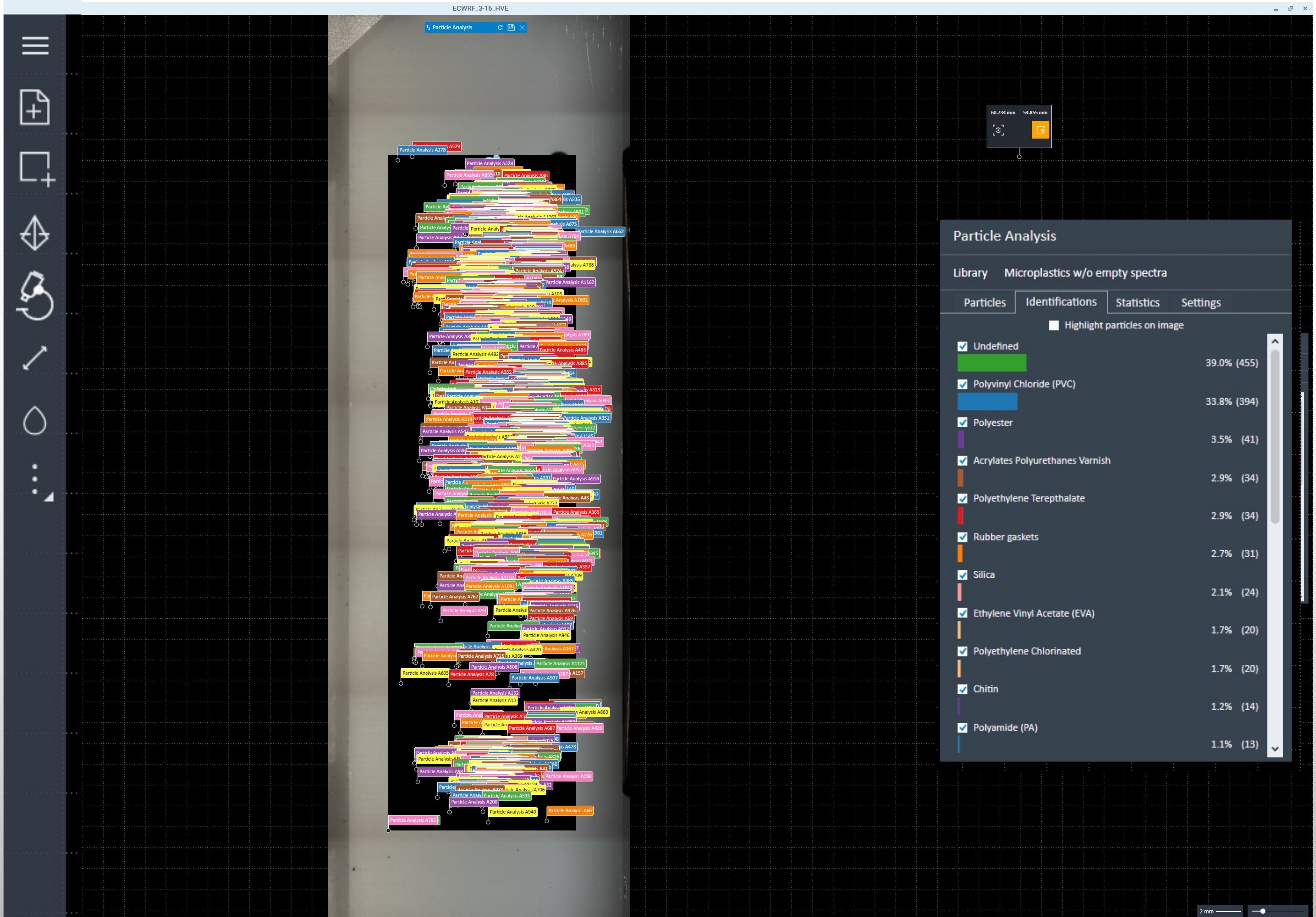
– **Automated** sample analysis.
– Ability to survey large sample areas and then explore smaller areas of interest in **more detail** without changing any optics.
– Full software control allows changing the field of view from **microns to centimeters** or the pixel size from 1 to 40 µm.
– Acquire ATR imaging data with pixel size as small as 0.1 µm for **unmatched image detail and spectral quality.**
– **Rapidly identify** unknowns using either commercial or custom libraries via ATR capabilities.
– Obtain relative quantitative information of sample constituents **without complex method development**.
– No requirement for liquid nitrogen **reduces operating costs** and simplifies maintenance.







HTTPS://WWF.PANDA.ORG/WWF_NEWS/PRESS_RELEASES/?348337/REVEALED-PLASTIC-INGESTION-BY-PEOPLE-COULD-BE-EQUATING-TO-A-CREDIT-CARD-A-WEEK


ECWRF_3-16_HVE
Particle Analysis
60.734 mm
54.855 mm
Particle Analysis
Library
Microplastics w/o empty spectra
Particles
Identifications
Statistics
Settings
Highlight particles on image
Undefined
39.0% (455)
Polyvinyl Chloride (PVC)
33.8% (394)
Polyester
3.5% (41)
Acrylates Polyurethanes Varnish
2.9% (34)
Polyethylene Terepthalate
2.9% (34)
Rubber gaskets
2.7% (31)
Silica
2.1% (24)
Ethylene Vinyl Acetate (EVA)
1.7% (20)
Polyethylene Chlorinated
1.7% (20)
Chitin
1.2% (14)
Polyamide (PA)
1.1% (13)
2 mm

Source	Method	Volume Processed	Analysis	Cost
Loder (2017)	Basic Enzymatic Purification Protocol (BEPP) w/ SSS	>10L	FPA-based FTIR analysis	**3214.65**
Long et al. (2019)	Filtration	<350L	micro-Raman spectroscopy	**759.3**
Ziajahromi et al. (2017)	Seiving	<200L	FT-IR analysis	**345.7**
Mason et al. (2016)	Seiving	500-21000L	visual-only identification	**1657**
Mintenig et al. (2017)	Pumping	390-1000L	ATR-FT-IR//Micro-FT-IR	**2601.94**
Talvitie et al. (2015)	Seiving	<285L	stereomicroscope Visual identification	**833.81**
Schymanski(2017)	Filtered	700 mL - 1500 mL	u-Raman spectroscopy	**2200**
Murphy (2016)	Filtering	1000	FT-IR analysis	**1455**
Carr (2016)	Filtering	2.8x10^6	Visual identification//FT-IR	**4194.5**
Magnussun (2014)	Plankton Sieves	cubic meters	FT-IR analysis	**4350**
Simon (2018)	Filtering	<81.5L	FT-IR analysis	**3548**
Uurasjärvi (2020)	Plankton Sieves	<468L	FT-IR analysis	**1590.1**

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

INTERNATIONAL

Designation: D8332 – 20

Standard Practice for Collection of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers[1]

This standard is issued under the fixed designation D8332; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

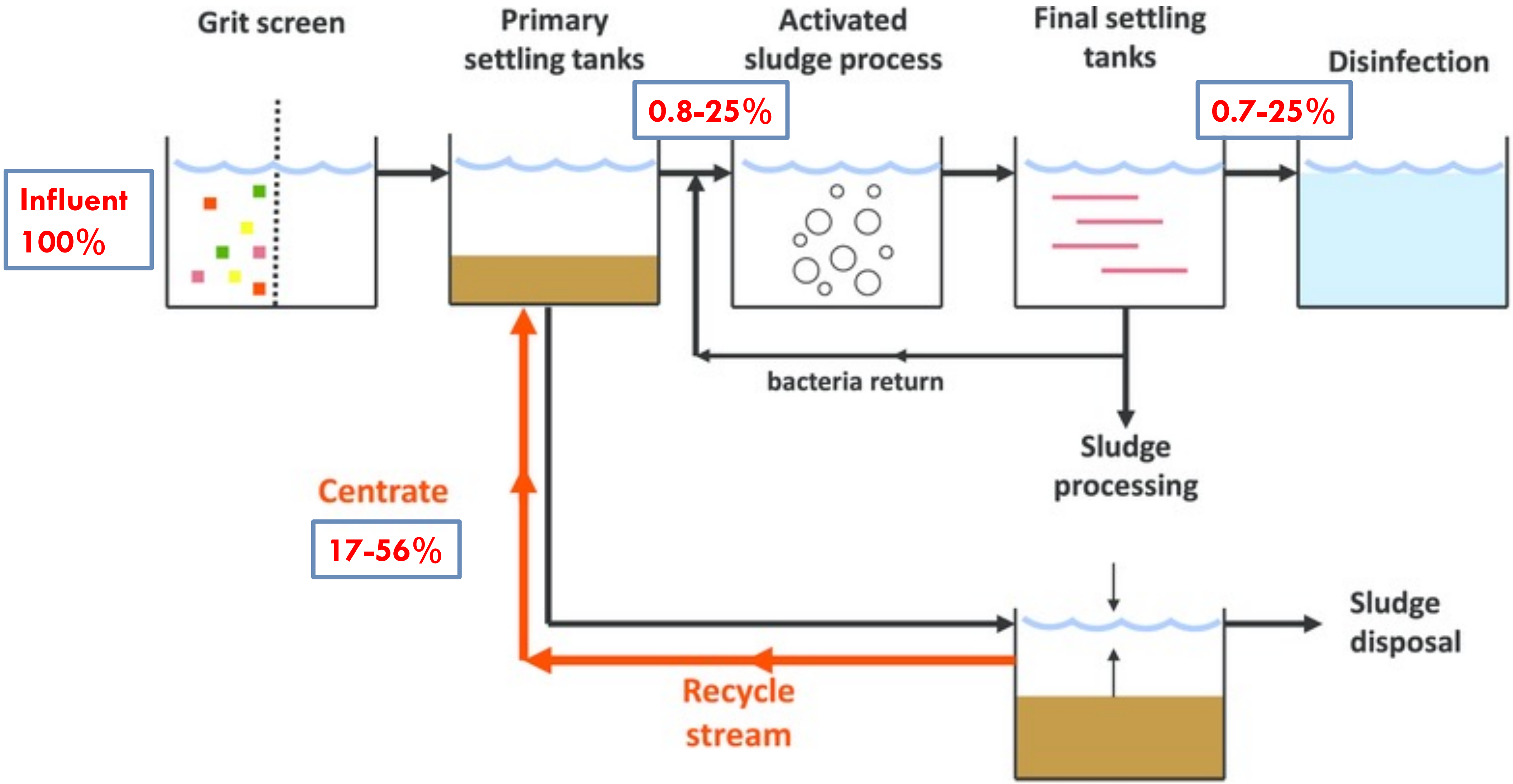
INTERNATIONAL

Designation: D8333 – 20

Standard Practice for Preparation of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers Using Raman Spectroscopy, IR Spectroscopy, or Pyrolysis-GC/MS[1]

This standard is issued under the fixed designation D8333; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

NEW METHOD FOR LARGE VOLUME COLLECTION: (SUBMITTING FOR ASTM STANDARD)


Grit screen
Primary settling tanks
Activated sludge process
Final settling tanks
Disinfection
Influent 100%
0.8-25%
0.7-25%
bacteria return
Sludge processing
Centrate
17-56%
Recycle stream
Sludge disposal

SUMMARY:

- Polymer plastics (PolyVinyl Chloride, Polystyrene, Polyethylene terephthalate, Polytetrafluoroethylene, Cellulose Acetate) along with some polyamides (protein), cellulosic, silica (sand) particles were observed in the samples
- Polyamides was the most prevalent particle identified which could be wool, silk and other textile product or the protein rich debris introduced from sample preparation
- Polyurethane, Synthetic wax, Alkyd, fatty acids, and acrylic polymers were grouped into one as these spectra has high similarity
- Manual analysis has now been replaced by automated particle analysis workflow in the software
- Example analysis based on automated particle analysis feature in the software was shown

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- **Water Resources Research Institute (WRRI) program 104(b) Student Research Grant**
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- Dr. Ian Pepper
- Fast (vacuum filtered)
- No need for separation with Agilent

Q & A