Supplementary Material

Analytical Strategies for Micro- and Nanoplastics in Aqueous Matrices: Progress and the Separation Bottleneck.

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Tables

Table S1. Sample pretreatment methods for micro- and nanoplastics in spiked and environmental samples, based on data from 102 peer-reviewed articles (2019-2025). The articles were selected from two queries: (1) “microplastics” OR “nanoplastics” AND “pretreatment”; and (2) “separation” OR “extraction” AND “nanoplastics” AND (“aqueous” OR “water”). The literature search was conducted in May 2025 in the Scopus database.

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Solvent washing. Acid alkaline, and oxidative digestion	PET, PE, PP	-	-	[1]
Acid, alkaline, oxidative and enzyme digestion	PET, PE, PVC, PP, PA, PES, PMMA, PU, SBR	Up to 95%	-	[2]
Alkaline and oxidative digestion	PE, PVC, PP, PS, and PMMA	-	-	[3]
Oxidative digestion	PET, PE, PP, PA, PHB, PLA and PMMA	-	72.49 - 134.08%	[4]
Solvent washing. Polymer dissolution	PET	-	-	[5]
Oxidative digestion	PET, PE and PS	-	93.7% - 99.3%	[6]
Oxidative digestion	PE and PS	-	-	[7]
Sieving, filtration, oxidative digestion	PET, PE, PVC, PP, PS and PMMA	-	61.4 ± 13.5%	[8]
Alkaline and oxidative digestion, sonication and heating	PS	-	-	[9]
Solvent washing. Sonication. Oxidative digestion	PP and tires	-	-	[10]
Enzymatic digestion	PET, PE, PVC and PS	Up to 89%	71 - 110%	[11]
Oxidative and enzymatic digestion	PET, PE, PP, PS, PA, PMMA and POM	-	57 - 91%	[12]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Solvent washing, sonication acid digestion and oxidative digestion	PS	-	66.80 - 84.69%	[13]
Solvent washing. Oxidative digestion	PVC and PS	-	-	[14]
Solvent washing. Alkaline and oxidative digestion	PE, PS, EVA and PA	Up to 100%	> 95%	[15]
Solvent washing. Acid alkaline, and oxidative digestion	PET, PE, PVC, PP, PS, PA and PU	-	-	[16]
Solvent washing. Oxidative digestion. Sonication	PET, PE, PVC, PP, PS, PA, PC, PES, and PU	-	82.5 - 94%	[17]
Sieving, filtration	Acrylic, PA and PES	-	-	[18]
Polymer dissolution	PET, PE, PVC, PP, PS, PBAT, PHB and PLA	-	79.0 -129%	[19]
Alkaline and oxidative digestion	PVC, AC and PA	99%	89%	[20]
Oxidative digestion	PET, PE, PVC, PP, PS, PA, PC, PO, PU and PVDF	-	-	[21]
Oxidative digestion	PE, PVC, PP, PS, PA, PES and PTFE	-	90.80%	[22]
Oxidative and enzymatic digestion	PET, PE, PP, PS and PMMA	-	61 - 76%	[23]
Solvent washing. Oxidative digestion	PET, PE, PS, PA, PMMA and PTFE	-	-	[24]
Oxidative digestion, heating	PET, PE and PES	-	-	[25]
Oxidative digestion, sonication	PET, PE, PVC, PP, PS, ABS, PA, PC, PLA, PMMA, POM, PTFE and PU	Up to 83%	-	[26]
Oxidative digestion	PE, PP and PS	-	-	[27]
Oxidative and alkaline digestion, sonication	PET	-	-	[28]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Oxidative digestion	PET, PE, PVC, PP and PS	-	90%	[29]
Solvent washing. Oxidative and enzymatic digestion	PET, PE, PVC, PP, PS, ABS, PA and PMMA	-	75.0 ± 10.0%	[30]
Oxidative digestion	PET, PE, PVC, PP, PS, PA and PMMA	-	-	[31]
Oxidative digestion	PE and PA	Up to 96%	-	[32]
Oxidative and enzymatic digestion	Tire rubber	-	-	[33]
Alkaline digestion	PET, PE, PVC and PS	Up to 95%	93 - 99%	[34]
Solvent washing, Alkaline, Oxidative and enzymatic digestion	PET and PA	-	> 95%	[35]
Solvent washing, Oxidative digestion	PET, PE, PP. EVA, PB	-	-	[36]
Sieving, Solvent washing, Filtration, Alkaline and oxidative digestion	PE, PVC, PP and PA	-	64 - 90%	[37]
Sieving, filtration, Oxidative digestion	PET, PE, PVC, PP, PS and AC	Up to 98%	76 - 98%	[38]
Solvent washing	PE, PP, PS, and PA66	-	78 - 130%	[39]
Solvent washing, Alkaline and oxidative digestion	PET, PE, PP, PS and PA6	-	71 - 84%	[40]
Solvent washing and oxidative digestion	PE, PP, PP-PE copolymer, EN, P2PMA, PA, PARA, PEA, POM, PPE, PPA, PO, PVDF, and Si-polymer	-	~ 95%	[41]
Solvent washing and alkaline digestion	PET, PE, PVC, PP and PS	-	-	[42]
Acid, alkaline and oxidative digestion	PES	-	-	[43]
Solvent washing and oxidative digestion	PET, PE, PVC, PP and PS	-	> 94%	[44]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Oxidative and enzymatic digestion	Tire rubber	-	-	[45]
Acid, alkaline and oxidative digestion	PET, PE, PP and PS	-	-	[46]
Oxidative digestion	PET, PE, PP and PS	-	-	[47]
Solvent washing. Acid, alkaline and oxidative digestion	PET, PE, PP, PS, PA and PMMA	-	-	[48]
Solvent washing, oxidative digestion	PET, PE, PVC, PP and PS	-	-	[49]
Solvent washing, oxidative digestion	PET, PE, PP, PS, acrylic, CN, epoxy resin, EPM, PA, PES, and PVAC	-	-	[50]
Solvent washing, oxidative digestion	PE, PP, Aramid, BR, ECTFE, EPM, EVOH, FKM, Modacrylic, PA, PA12, PAA, PBA, PC, PEA, PEAA-Zn, PES, PFA, PO, PPA and PTFE	-	-	[51]
Solvent washing, oxidative digestion	PE, PP, PS, EEA, EMA, ETFE, EVOH, Fluoroelastomer, PA, PARA, PES, PMA PO, POM, PPA, PPE, PVDF and XT Polymer 375	-	-	[52]
Acid, alkaline, oxidative and enzymatic digestion	PET, AS, PC and PU	20 - 160%	86 - 100%	[53]
Freeze drying	PP	-	76.4 - 118%,	[54]
Acid, alkaline and oxidative digestion	PET, PA and PU	70 - 85%	70 - 90%	[55]
Solvent washing. Oxidative and enzymatic digestion	PET, PE, PP, PS, PA, PC, PMMA, PTFE, PU and tire rubber	54.21 ± 2.00%	~ 100%	[56]
Oxidative digestion	PE, PP, PS, AC, AKD, PES and PTFE	-	-	[57]
Solvent washing and oxidative digestion	PET, PE, PVC, PP, acrylic resin and PTFE	-	70 - 93%	[58]
Oxidative digestion	PET, PE, PVC, PP, PS, ABS, EEA, EPM, PA, PBD, PES, PU and tire rubber	-	-	[59]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Oxidative digestion	PET, PE and PA	90-97%	60 - 95%	[60]
Acid and oxidative digestion	PET, PE, PP, EEA, PA, PEA, PTFE and rayon	-	-	[61]
Oxidative digestion	PET, PE, PVC, PP, PS, ABS-PS, CA, PA, PE, PE-CA, PE-PP, PE-PVC, PP-PA, PU-PA, PVC, rayon and rubbers (Styrene-Isoprene, tall oil rosin	92.35 - 93.55%	-	[62]
Solvent washing, oxidative digestion	PET, PE, PVC, PP, PS and PA	-	100%	[63]
Oxidative digestion	PET, PE, PVC, PP, PS, ABS, alkyd, AS, epoxy, PA, PIP, PMMA, PPS, PU, PVAc, PVAl, PVME and SBR	-	94 - 100%	[64]
Solvent washing, oxidative digestion	PE, PP, PS, Acrylic polymer, ABS, EMA, EVA, EVOH, modacrylic, PA, PARA, PES, PO, POM, PPE, PPA, PTFE, PU, SBR, and VE	-	-	[65]
Sieving, filtration, Oxidative digestion	PE, PVC, PP, PS, PLA and rayon	76.05 - 98.3 %	-	[66]
Oxidative digestion	PP, Acrylic, PA, PES, and rayon	-	Up to 93%	[67]
Sieving, filtration, Alkaline digestion	PET, PE, PP, PS, PA, PC, PE-PB, PMMA, PNB, PNMAAm, Polyacrylate, POM, PVDF, and PVDF-PHFP	-	76.7 - 310%	[68]
Oxidative digestion	PET, PE, PVC, PP, PS, ABS, PA and PMMA	-	-	[69]
Acid, alkaline and oxidative digestion	Tire rubber	-	106 ± 3%	[70]
Alkaline and oxidative digestion	PET, PE, PVC, PP, PS, PA and PMMA	-	-	[71]
Oxidative digestion, heating	PE and PVC	-	~ 80%	[72]
Alkaline, oxidative and enzymatic digestion	PET, PE, PVC, PP, PS, ABS, epoxy resin, melamine, PA, PAN, PB, PBS, PC, PEEA, PES, PU, PVAE, silicone, and synthetic cellulose	-	70 - 100%	[73]
Acid and oxidative digestion	PE, PP and PS	95.6 - 99.6%	95.6 - 118.4%	[74]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Sonication, oxidative digestion	PE, PVC, PP, PS, EVA, PA, PAM, PES, PMMA, PMP, polyacetylene, polyoxides and PU	-	0 - 100%	[75]
Drying, oxidative digestion, heating	PET, PE, PVC, PP and PS	-	-	[76]
Acid and oxidative digestion	PE, PP and PS	-	-	[77]
Oxidative digestion, heating	PET, PE, PVC, PP, PS and PA	-	-	[78]
Oxidative and enzymatic digestion	PET, PE, PVC, PP, PS, PA and PBAT-PLA	-	-	[79]
Polymer dissolution	PE, PP, PS, and PMMA	-	61 - 113%	[80]
Oxidative digestion	PET, PE and PP	-	~ 90%	[81]
Oxidative digestion	PVC, PS, PA and PO	-	97.50 ± 0.58	[82]
Enzymatic digestion	PS	-	65 ± 10% - 122 ± 22%	[83]
Sonication	PET	-	91.5 - 109.3 %	[84]
Solvent washing	PE and PS	-	-	[85]
Solvent washing	PE, PP, PS, PI, and PDMS	-	~ 63%	[86]
Polymer dissolution	PET and PS	-	-	[87]
Sieving, filtration	PE, PVC PS, PMMA, PTFE	-	-	[88]
Oxidative and enzymatic digestion	PE and PVC	-	7 - 109%	[89]
Polymer dissolution	PE, PVC, PP, PS, PC and PMMA	-	60 - 140%	[90]

Sample pretreatment	Polymers	Organic matter removal	Recovery	Ref.
Oxidative digestion. Filtration	PET, PP, PS, PMMA, PA6, PA66 and PC	-	54.6 - 68.1%	[91]
Alkaline and oxidative digestion	PE, PP, PS and PC	-	-	[92]
Polymer dissolution	PET and PS	-	-	[93]
Alkaline digestion, solvent washing, polymer dissolution	PET, PE, PVC, PP, PS and PMMA	-	79.6 - 91.4%	[94]
Acid digestion	PS	-	-	[95]
Acid digestion	PS	-	72.5 - 87.2 %	[96]
Sieving, filtration	PE, PP, PS, PA, PBD, PDMS and PI	-	55 - 82%	[97]
Oxidative digestion	PET, PP, PS, PA, PC and PMMA	-	-	[98]
Filtration	PS and PMMA	-	72.1 - 98.9%	[99]
Filtration, acid digestion	PET, PE, PVC, PP, PS, and PMMA	-	72.9 - 92.8%	[100]
Sieving, filtration	PE, PP, PS, PBD, PI and Polysiloxanes	-	-	[101]
Solvent washing, acid digestion, polymer dissolution	PS	-	60 - 70%	[102]

Note: ABS = Acrylonitrile Butadiene Styrene; AKD = Alkyl resin; AS = Acrylonitrile Styrene; BR = Butadiene Rubber; CA = Cellulose Acetate; CN = Cellulose Nitrate; EEA = Ethylene Ethyl Acrylate; EMA = Ethylene Methyl Acrylate; EN = Epoxy Novolac; ECTFE = Ethylene Chlorotrifluoroethylene; EPM = Ethylene Propylene Monomer; EVA = Ethylene-Vinyl Acetate; EVOH = Ethylene-Vinyl Alcohol; FKM = Fluoroelastomer; P2PMA = Polybyphenil metacrylate; PA = Polyamide; PA12 = Polyamide 12; PA6 = Polyamide 6; PA66 = Polyamide 66; PAA = Polyacrylic Acid; PAM = Polyacrylamide; PARA = Polyarylamide; PB = Polybutylene; PBA = Polybutylacrylate; PBD = Polybutadiene; PBAT = Polybutylene Adipate Terephthalate; PBS = Polybutylene Succinate; PC = Polycarbonate; PDMS = Polydimethylsiloxane; PE = Polyethylene; PES = Polyester; PFA = Perfluoroalkoxy; PHB = Polyhydroxybutyrate; PHFP = Polyhexafluoropropylene; PI = Polyimide; PIP = Polyisoprene; PLA = Polylactic Acid; PMA = Polymethylacrylate; PMMA = Polymethyl Methacrylate; PMP = Polymethylpentene; PNB = Polynorbornene; PNMAAm = Poly(N-methyl acrylamide); PAcr = Polyacrylate; PO = Polyolefin; POM = Polyoxymethylene; PP = Polypropylene; PPA = Polyphthalamide; PPE = Polyphenylene Ether; PPS = Polyphenylene Sulfide; PS = Polystyrene; PTFE = Polytetrafluoroethylene; PU = Polyurethane; PVAE = Polyvinyl Acetate Ethylene; PVAc = Polyvinyl Acetate; PVAl = Polyvinyl Alcohol; PVDF = Polyvinylidene Fluoride; PVME = Polyvinyl Methyl Ether; SBR = Styrene-Butadiene Rubber; VE = Vinyl Ester.

Table S2. Separation methods for micro- and nanoplastics in spiked and environmental aqueous samples, based on data from 112 peer-reviewed articles (2019–2025). The articles were selected using the terms “separation” OR “extraction” AND “nanoplastics” AND (“aqueous” OR “water”). The search was conducted in March 2026 in Scopus database.

Separation method	Reference
Membrane Filtration/Ultrafiltration	[81,82,85,88,89,91–93,95,98,101,103–120]
Centrifugation/Ultracentrifugation	[100,121–130]
Field flow fractionation (FFF)/ Asymmetric flow field flow fractionation (AF4) Centrifugal field flow fractionation (CF3)	[102,123,125,131–136]
Density separation	[81,98]
Flotation	[137,138]
Acoustofluidic device	[139]
Magnetic adsorption	[84,140–154]
Adsorption on engineered or bio-based materials	[99,141,148,155–164]
Chromatographic methods	[86,87,97,165–168]
Biomass-mediated adsorption	[169–172]
Solid-phase microextraction	[114]
Surfactants/ Cloud Point Extraction (CPE)	[100,121,127,129,173–175]
Polar aprotic solvents	[90,93,94,176–178]
Deep eutectic solvents	[179–181]
Nonpolar organic solvents	[97]
Ionic liquids	[182]

Nonpolar natural solvents	[183]
Flocculation	[184–190]
Electrophoresis	[110,191–193]
Electrofloatation	[194]
Micro-electromembrane	[106]

Table S3. Representative studies of physical–mechanical separation methods for MPs and NPs, summarising their key advantages and limitations.

Extraction Method	Target size (particle or cut-off)	Polymer type	Matrix	Recovery rate	Challenges	Analytical techniques	Mechanism	Key innovation	Ref.
Membrane filtration	Glass fibre 450 nm (cut-off), $Al_2O_3$20-200 nm (cut-off), 58–255 nm (particle)	PVC, PS, PA, PO	Freshwater	97.5 ± 0.6% *(context-specific)*	Competitive precipitation, particle labelling	DLS, XPS, FTIR, AFM-IR, Py-GC/MS	Consecutive filtrations	High-sensitivity isolation and identification of 58–255 nm particles	[82]
Ultrafiltration	10–1000 nm (cut-off)	PET, PP, PS, PA, PC, PMMA	Freshwater, wastewater	54.6–68.1%	Matrix interferences	Py-GC/MS	Filtration + dissolution	Workflow to quantify NPs in environmental samples	[91]
Density separation	—	PET, PVC, PP, PE, PS, PA, PMMA	Freshwater, soils	—	Aged PE/ natural organic matter spectral overlap	STXM in combination with NEXAFS	Buoyancy in salt solutions	STXM identification	[98]
Flotation	100 nm	PS	Freshwater, wastewater	Average 92.8%	Matrix interferences	Zeta potential, SEM and FTIR	Bubble attachment (collector-assisted)	Chitosan greatly improved removal efficiency	[137]
Centrifugation	50–500 nm (particle)	PS, PLA, PMMA	Fresh/seawater	90–120%	Lower recoveries in seawater	SERS	Velocity sedimentation (Stokes)	Zr/tannic acid/rhodamine labelling	[122]
Ultracentrifugation	600–1000 nm (particle)	PS	Freshwater	57–103%	Lower recoveries with fragmented PS	UV–Vis, NTA, SEM, HSI	Sedimentation by g-force	First reported use of the technique for NP separation	
Field-Flow Fractionation (FFF)	76–216 nm (particle)	PE, PS	Ultrapure water	66–80%	Agglomeration, transfer steps	DLS, NTA, TRPS, MALS, Py-GC/MS, Raman	Flow-induced sedimentation	Multi-detector compatibility	[123]

Notes: AFM-IR = Atomic Force Microscopy–Infrared; DLS = Dynamic Light Scattering; FTIR = Fourier Transform Infrared Spectroscopy; HSI = Hyperspectral Imaging; MALS = Multi-Angle Light Scattering; NEXAFS = Near-Edge X-ray Absorption Fine Structure; NTA = Nanoparticle Tracking Analysis; Py-GC/MS = Pyrolysis–Gas Chromatography–Mass Spectrometry; SEM = Scanning Electron Microscopy; STXM = Scanning Transmission X-ray Microscopy; TRPS = Tunable Resistive Pulse Sensing; XPS = X-ray Photoelectron Spectroscopy.

Table S4. Representative studies of solvent-extraction techniques for MPs and NPs.

Solvent category	Solvent	Polymer Type	Matrix	Recovery (%)	Challenges	Analytical Techniques	Mechanism	Key innovation	Ref.
Polar aprotic organic solvent	Dichloromethane	PS, PE, PMMA	Air	95.8 ± 24.3% (NPs)	PE/PP identification hindered by hydrocarbon peak overlap.	Py-GC/MS	Polymer dissolution	Py-GC/MS multiplexed workflow	[195]
Non-polar organic solvent	Toluene	PE, PP, PS, PA, PBD, PDMS, PI	Freshwater	55–82%	Limited quantification, cross-study comparability	HPLC/HRMS	Ultrasonic-assisted extraction, dissolution	Early household tap-water MPs/NPs dataset	[97]
Natural organic solvent	Olive oil	PE, PS, PMMA	Seawater, soil, tea leaves, toothpaste	75–87%	Emulsion formation requiring demulsifiers	DLS, fluorescence, Raman	Hydrophobic interactions	Simple, sustainable, efficient	[183]
Surfactant (non-ionic surfactant)	Triton X-45	PS, PMMA	Environmental water	>90.7% (MPs), >93.0% (NPs)	Matrix complexity, surfactant residues	Py-GC/MS	Micelle encapsulation	Refined MP/NP separation protocol	[173]
Surfactant (cationic surfactant)	CTAB	PS	Biological matrices	>90% (NPs)	Surfactant interference, low-abundance detection	spICP-MS (Au labelling)	Surfactant-enhanced aggregation	High-sensitivity detection of PS via Au labelling	
Hydrophobic ES	Decanoic acid:menthol (1:1, 1:2)	PS, PET, PLA	Freshwater, saltwater	50–93% (MPs, NPs)	Inconsistent extraction in saltwater due to particle aggregation	DLS, SEM, contact angle	Hydrophobic interactions, H-bonding	Integrated molecular simulations to explain polymer–solvent affinity	[179]
Hydrophobic ES	Thymol:2,6-dimethoxyphenol (1:2), Menthol:2,6-dimethoxyphenol (1:1)	PS, PET	Aqueous	95–99% (MPs, NPs)	Instability of Men-Dmp (1:1), PS dissolution at prolonged contact (Thy-Dmp 1:2)	FTIR, DLS, contact angle, Py-GC/MS	Hydrophobic, H-bonding, π–π interactions	Sustainable lignin-based ES with interface-driven extraction mechanism	[180]
Hydrophobic ES	TBAB:decanoic acid (1:2)	PS	Freshwater, saline	97–100% (NPs)	Difficult polymer recovery from ES phase	H NMR, UV–Vis, fluorescence, ESEM	Hydrophobic interactions	High efficiency and extraction capacity in saline systems	[181]

Notes: FTIR = Fourier Transform Infrared; HPLC/HRMS = High-Performance Liquid Chromatography/High-Resolution MS; spICP-MS = Single-Particle Inductively Coupled Plasma MS; Py-GC/MS = Pyrolysis–Gas Chromatography–Mass Spectrometry; SEM = Scanning Electron Microscopy.

Table S5. Representative studies of adsorption-based extraction and separation techniques for MPs and NPs.

Extraction Method	Target size (particle)	Polymer type	Matrix	Recovery rate	Challenges	Analytical techniques	Mechanism	Key innovation	Ref.
Adsorption (cellulose hydrogel beads)	50 nm	PS	Freshwater, seawater, wastewater	Up to 99.8%	Only spiked samples, reduced number of polymer types and sizes	MADLS, FESEM, FTIR, BET, zeta potential	Electrostatic + H-bonding + π–π interactions + pore-filling mechanisms	Tailored hydrogel beads, high recoveries, excellent reusability	[158]
Solid-phase microextraction	500–3000 nm	PMMA	Freshwater, seawater, soil	75.6–98.9%	Soil samples challenging	GC/MS, TGA	Filtration + thermal decomposition	Solvent-free, robust to complex matrices	[114]
Adsorption (functionalised magnetic biochar)	50–100 nm	PS	Freshwater	up to ≈99.6% (baseline), lower with HPO_4^{2-}/CO_3^{2-} or humic acid	Ion and NOM interference	SEM, FTIR, XPS, zeta potential	Electrostatic + hydrophobic, magnetic retrieval	CTAB-modified magnetic rice-straw biochar, rapid magnetic separation	[171]
Adsorption (magnetic IONPs)	100–1000 nm, 2–5 mm	PS	Freshwater, saline	≈89–93% (100–1000 nm), ≈100% (mm)	not reported	FTIR, UV–Vis, contact angle, SEM	Hydrophobic functionalisation, magnetic retrieval	High efficiency across sizes, easy magnetic recovery	[145]
Flocculation (synthetic and bio-based)	≈100 nm (NPs, very low for MPs ~2000 nm)	PS, PMMA	Wastewater	65–90% for jellyfish mucin, ≤40% for $FeCl_3$/PAC, negligible for PAM	Synthetic: additive residues and charge inversion, Bio-based: matrix dependence	DLS, zeta potential, Cryo-SEM	Synthetic: charge neutralisation + bridging, Bio-based: adsorption-bridging + mesh filtration	Jellyfish mucin as a superior bio-flocculant	[187]
Chromatography (SEC/HPLC)	–	PE, PP, PS, PI, PDMS	Airborne, aqueous, biological	55–82%	Clogging, maintenance, small volumes	ICP-AES/MS, μ-FTIR, SEM, SEC-HRMS, HPLC/HRMS	Polymer–stationary-phase interactions	SEC fractionation (PLgel MIXED-D), APC/RP-HPLC profiling, single-cell NP detection	[86]

Notes: BET = Brunauer–Emmett–Teller; FESEM = Field-Emission Scanning Electron Microscopy; ICP-AES/MS = Inductively Coupled Plasma–Atomic Emission/Mass Spectrometry; SEC-HRMS = Size-Exclusion Chromatography–High-Resolution Mass Spectrometry; TGA = Thermogravimetric Analysis.

Table S6. Representative studies of electrokinetic and electro-assisted separation techniques for MPs and NPs, summarising their main advantages and limitations.

Extraction Method	Particle size	Polymer type	Matrix	Recovery rate	Challenges	Analytical techniques	Mechanism	Key innovation	Ref.
Electroflotation	≈176 nm	PS	Wastewater	91–95%	not reported	FESEM, DLS, TGA, SEM	Electrolysis-driven bubble attachment/coagulative flotation	Low energy, reduced sludge	[194]
Electrocoagulation	100–2000 nm	PS, PMMA	Wastewater	—	Electrode coagulants, local pH shifts	DLS, zeta potential	In situ coagulant generation, charge neutralisation	Tuneable aggregation without chemical dosing	[196]
Electrophoresis (deposition/foam coupling)	80–1000 nm	PS, PBMA, PMMA	Wastewater	> 90%	not reported	Zeta potential, UV–Vis	Electrophoretic deposition, hydrophobic interactions	Coupled foam formation for capture	[197]
Capillary electrophoresis (voltage-driven solvent-drop microextraction)	≈200 nm	PS	Beverages	47–60%	Low volumes, peak overlap	Capillary zone electrophoresis	Voltage-driven solvent-drop microextraction	Membrane-free support of immiscible solvent	[110]
Electromembrane processes (micro-scale)	100–2000 nm	PS, PMMA	Wastewater	65–90%	not reported	DLS, zeta potential, Cryo-SEM	Field-assisted transport across membranes	Electrical control of membrane selectivity	[106]

Note: Cryo-SEM = Cryogenic Scanning Electron Microscopy; FESEM = Field-Emission Scanning Electron Microscopy.

Table S7. UV–Vis absorbance (400 nm) and DLS PDI for PS at increasing concentrations.

Sample Concentration (µg/mL)	Absorbance at 400 nm	Polydispersity Index (PDI)
10	0.15	0.60
25	0.40	0.50
50	0.85	0.55
100	1.75	0.70

Table S8. SEM observations of PS NPs.

Sample Type	Average Particle Size (nm)	Observed Issues
Polystyrene, 50 µg	200–500	Aggregation, adhesion
Polystyrene, 100 µg	400–700	Surface roughness

Table S9. Number of suspected polystyrene particles detected using MP-VAT 2.0.

Sample	Column 1	Column 2	Column 3	Column 4	Total Particles
Procedural Blank	151	42	22	11	226
Treated water after ES separation	23	32	202	133	390

Table S10. Indicative quantitative performance of analytical techniques for MPs and NPs

Technique	Quantification metric	Indicative LOD/LOQ)*	Indicative RSD	Ref.
DSC	Thermal transitions	LOD: 0.05-0.76 mg/sample LOQ:0.13 – 1.84 mg/sample	Not reported**	[198]
TGA/TGA-FTIR	Mass loss / evolved gases	LOD: 0.041 - 0.061 mg/sample LOQ: 0.258 - 0.383 mg/sample	Not reported**	[199]
Py-GC/MS	Polymer-specific pyrolysates	LOD: 0.02-0.5 µg/sample LOQ: 0.06 – 1.5 µg/sample	<9.6%	[200]
TED-GC/MS	Volatile degradation products	LOD: 0.09 µg/sample LOQ: 0.28 µg/sample	8.7%	[201]
TD-PTR-MS	Real-time volatile fragments	LOD: 1.7-2 µg/sample LOQ: 5-6 µg/sample	Not reported**	[202]
MALDI-ToF-MS	Polymer mass distribution	LOD: 1.8-18.1 mg/sample LOQ: 9.0-18.1 ng/sample	0.8-7.5%	[203]
spICP-MS	Particle size and concentration	LOD: 3.6 x 10^7 NPs L^{-1} LOQ: 3.3 x 10^8 NPs L^{-1}	Not reported**	[204]

*Indicative LOD, LOQ, and RSD values were compiled from representative studies and reflect typical performance under controlled experimental conditions. These parameters are highly dependent on the method, polymer type, matrix composition, and analyst, and may vary significantly with sample pretreatment, instrumental configuration, method calibration, and data processing. Therefore, direct quantitative comparisons across techniques should be interpreted with caution.

**The corresponding metric was not explicitly provided in the cited reference.

Figures

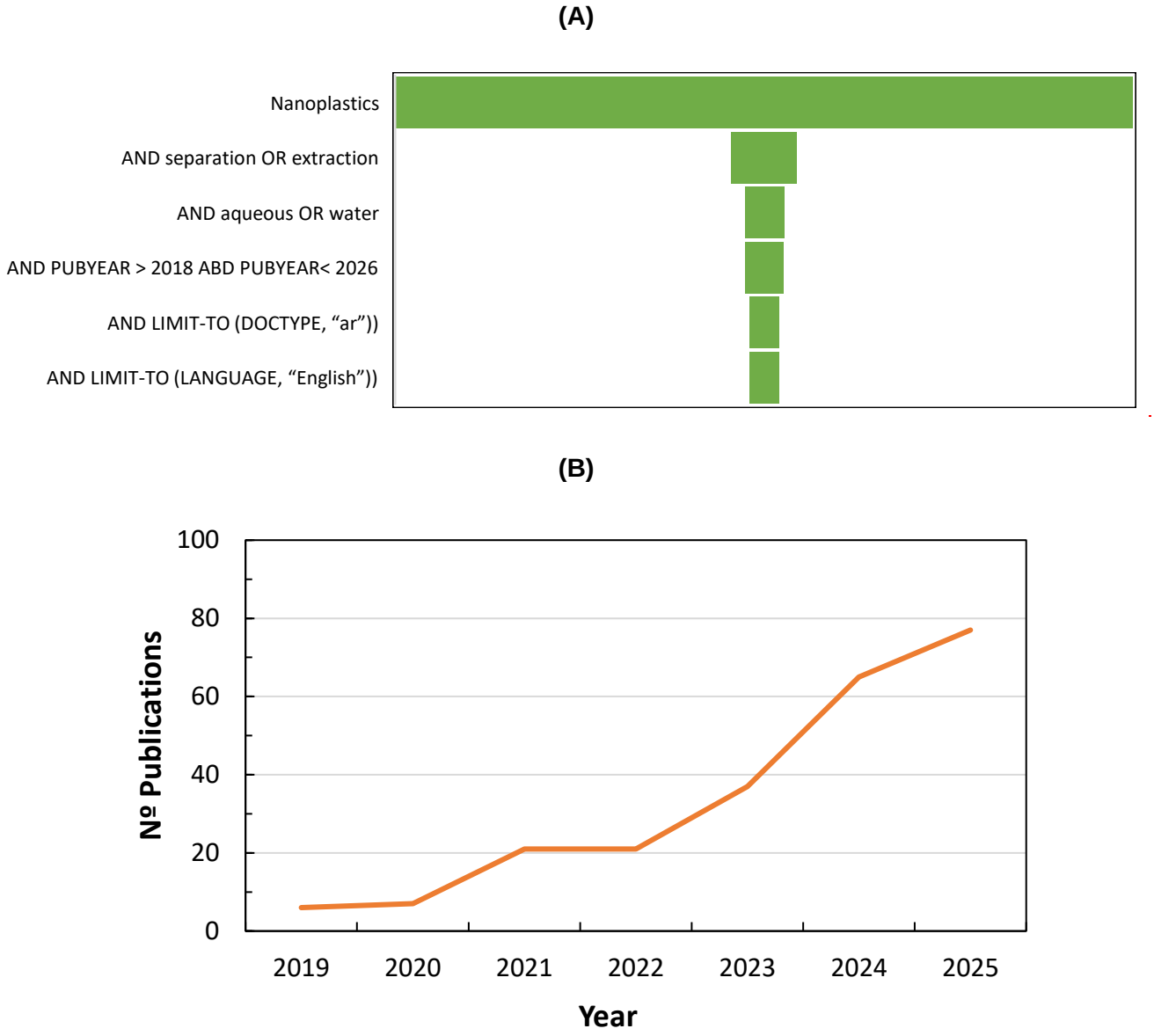

Figure S1. Literature screening for nanoplastic (NP) separation methods. (A) Keywords used in the Scopus search. (B) Number of publications per year (2019–2025)

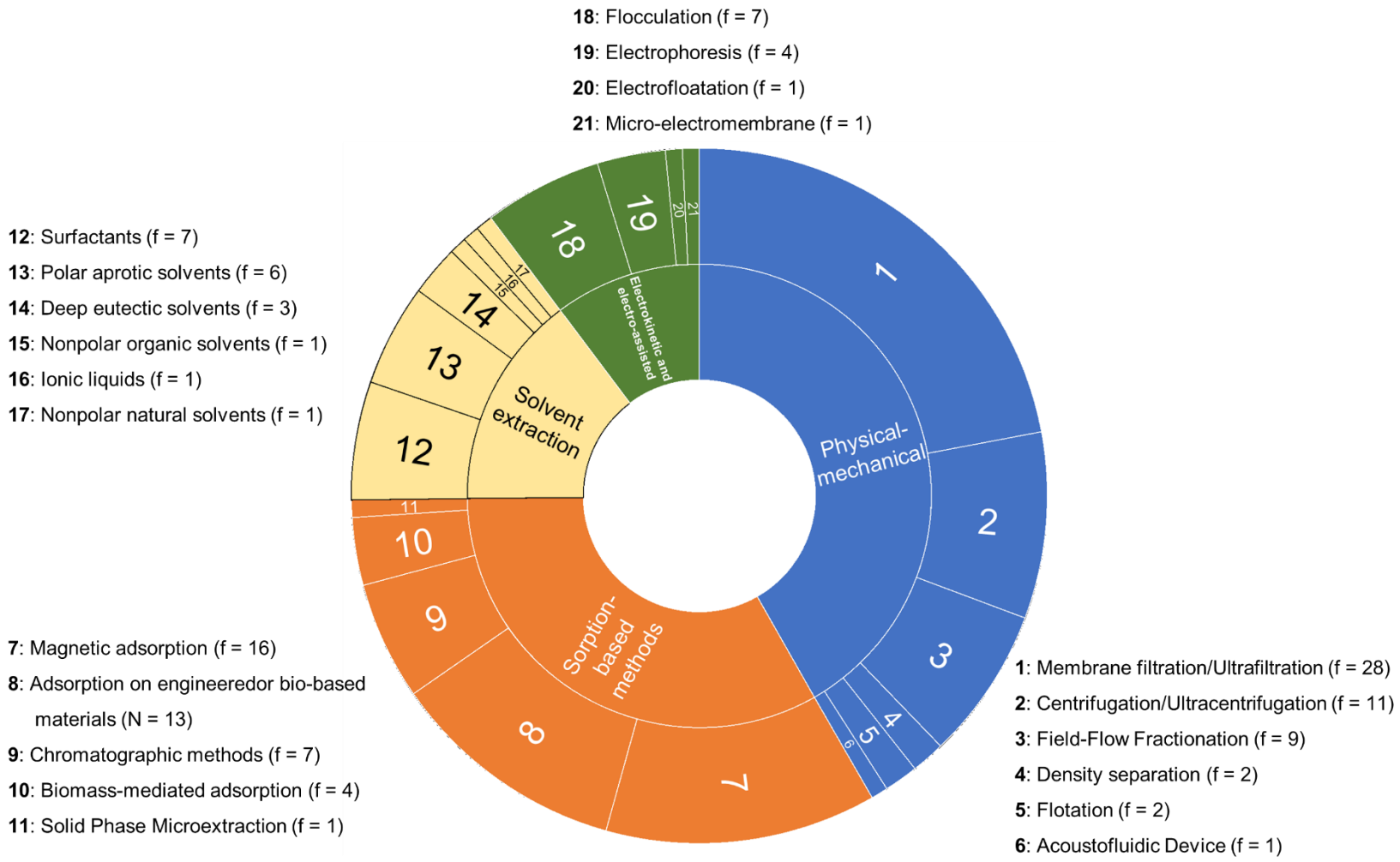

Figure S2. Distribution of separation methods identified across 112 studies (127 individual applications, 2019–2025) involving micro- and nanoplastics in spiked and environmental aqueous samples.

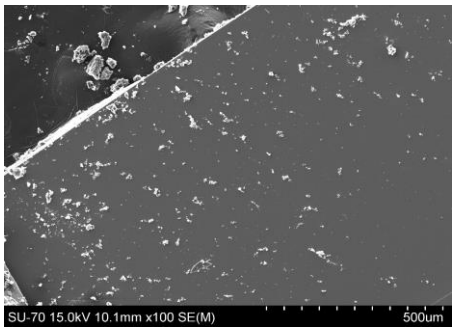

Figure S3. SEM image of polystyrene (PS, 20 kDa) particles acquired on a Hitachi SU-70 microscope at 100× magnification in secondary electron mode.

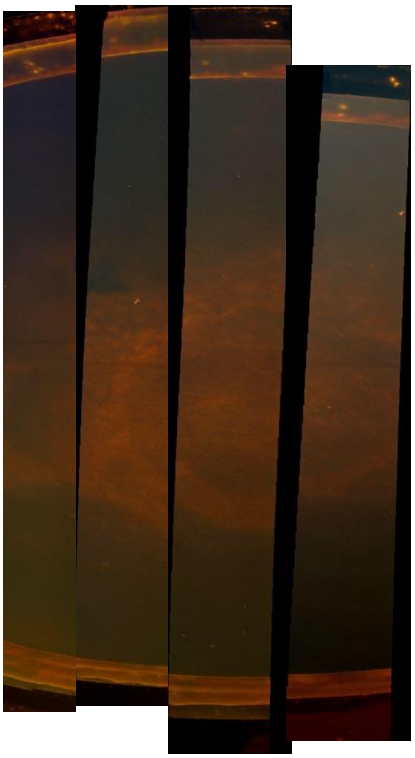

Figure S4. Composite images of the central sections of alumina filters used for the quantification of suspected polystyrene particles stained with Nile Red.

Text

Text S1. Methodology for hyperspectral imaging in hydrophobic ES extraction quantification.

The extraction process involved agitation in separatory funnels for 24 hours, followed by 2 hours of phase separation. The aqueous (bottom) phase was filtered through 0.1 µm Whatman® alumina filters, which were subsequently subjected to acetone digestion, rinsed with deionized water, stained with 1 % Nile Red, and air-dried. Imaging was performed under 470 nm illumination using an Olympus BX41 microscope equipped with a Canon 1200D camera.

The filters were divided into 3 mm columns for sequential photography. Composite images were assembled using Microsoft Image Composite Editor, and particle counting was carried out automatically with MP-VAT 2.0 (in-house ImageJ/Fiji macro, University of Aveiro, 2025). Composite images for each filter column were superimposed to enhance visualization (see Figure S4).

Automatic particle count data, summarized in Table S9, revealed variations between procedural blanks and treated samples, reflecting both the extraction efficiency and the limitations of the detection methods employed.

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