SINAYA
Philippine Journal for Senior High School Teachers and Students

"Recent Advances on Density Separation Techniques for Microplastic Recovery from Sediments"

Andrei Luigi T. Ang
De La Salle University Integrated School

Carlos Miguel D. Jose
De La Salle University Integrated School

Cara Isabel P. Del Rosario
De La Salle University Integrated School

Orrin Landon T. Uy
De La Salle University Integrated School

Dr. Joel Garcia
De La Salle University Integrated School

ABSTRACT: Microplastics are small plastic fragments commonly less than 5 mm in size (Khatmullina et al., 2016), making up about 92% of the total plastic pollution. Since these plastics are transparent due to their small size, they cause invisible plastic pollution. Due to their adsorptive properties, they can potentially harm marine and human life if ingested. Microplastic extraction is used to isolate microplastics from their original matrix using various methods and instruments to ease the process. Electrostatic separation, magnetization, and pressurized fluid extraction are among the established microplastic extraction methods, but these separation techniques require more effort and use a more advanced setup than density separation. Salt-assisted density separation of microplastics has been demonstrated to provide modest to excellent recovery rates. This review survey reported different salt solutions used in microplastic extraction via density separation in the last five years. Among the inorganic salts used in density separation, sodium dihydrogen phosphate (NaH_2PO_4) holds promise in separating microplastics because of its high extraction efficiency while being cost-effective and non-hazardous.

KEYWORDS: microplastics; microplastic quantification; density separation; salt solutions; sediment sampling

INTRODUCTION

Microplastics in marine environments date back to the 18th century (Carpenter & Smith, 1972). These small plastic fragments were later discovered to be common and present in most marine environments. The global release of primary microplastics into the ocean was estimated at 1.5 million tons per year (Mtons/year) (Boucher & Friot, 2017). The largest regional releases are in India and South Asia (18.3%) and North America (17.2%), Europe and Central Asia (15.9%), China (15.8%), and East Asia and Oceania (15.0%), South America (9.1%), and Africa and the Middle East (8.7%) (Boucher & Friot, 2017). Minimum estimates of the number of marine plastics in the world's oceans are currently placed at 5.25 trillion pieces, endangering marine life (Eriksen et al., 2014; Sutherland et al., 2010).

Microplastics in bodies of water pose a serious threat to marine biota because microplastics can absorb and spread toxins. Marine organisms commonly mistake microplastics for food, thus underlying the many deaths of marine life, ultimately affecting the food chain and web. While it is currently unclear if microplastic ingestion is harmful to health, it is established that toxins can adsorb onto microplastics. Ingestion of microplastics introduces toxins such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons, and petroleum hydrocarbons into organisms because of the high propensity of these plastics to adhere to organic pollutants (Avio et al., 2015; Teuten et al., 2009). It is critical to develop approaches to reduce the number of microplastics in bodies of water due to their harmful effects on the environment. The complexities and differences of ocean depths can make it difficult to properly and routinely sample and detect microplastics (Cole et al., 2011). To this end, several methods (Table 1), namely electrostatic separation, magnetic extraction, pressurized fluid extraction, and density separation, have been reported to separate microplastics from their original matrix (Felsing et al., 2018; Fuller & Gautam, 2016; Grbic et al., 2019; Prata et al., 2019).

Table 1

Comparison of the different methods used for microplastic extraction

Extraction Method	Advantages	Disadvantages	Author
Visual Sorting	Low cost/accessible equipment Simple No chemical hazards	Time consuming Results may vary widely for each person	Prata et al., 2018
Density Separation	Does not require specialized equipment Simple Easy	Can be time consuming Recovery rates will vary per flotation medium used	Prata et al., 2018
Electrostatic Separation	Mass reduction of almost 99% can be achieved Simple Smaller amounts of harmful solutions needed	Availability of specialized equipment needed Surface charge of the plastic may change Potential saturation of equipment used	Felsing et al., 2018
Magnetic Extraction	High recovery rate for large and small microplastics	Brittle microplastics will fragment Lower than average recovery rate for medium sized microplastics Effectiveness is reduced in less clean samples	Grbic et al., 2019
Pressurized Fluid Extraction	Simple Fast Fully automated Cost	Inability to measure size fractions of microplastics Physical characterization tests of microplastics are not possible Additional work is needed for identification especially if sample contains multiple microplastic types	Fuller & Gautam, 2016

SAMPLING AND MICROPLASTIC EXTRACTION

The quantity and quality of microplastics recovered are highly dependent on sampling location and depth and may compromise the consistency and reproducibility of the results (Prata et al., 2018). The samples differ when retrieved from *tidelines*, *intertidals*, and *supralittoral* environments. At beaches, particles can be best sampled with trowels, shovels, or spatulae at the high tide lines (Figure 1). The sampling procedure requires a *wrack* (Figure 2) wherein the microplastic-containing sediment samples will be collected along the beach. The area covered by the quadrat must be free from any debris. Collection of microplastics on beaches requires sieving, thus requiring a gallon bucket for use. Silt and clay sediments with fine grain sizes hinder visual analysis during extraction and quantification (Graca et al., 2017).

Figure 1

Diagram illustrating the different areas covered by Tide Lines from Florida Sea Grant (2016).

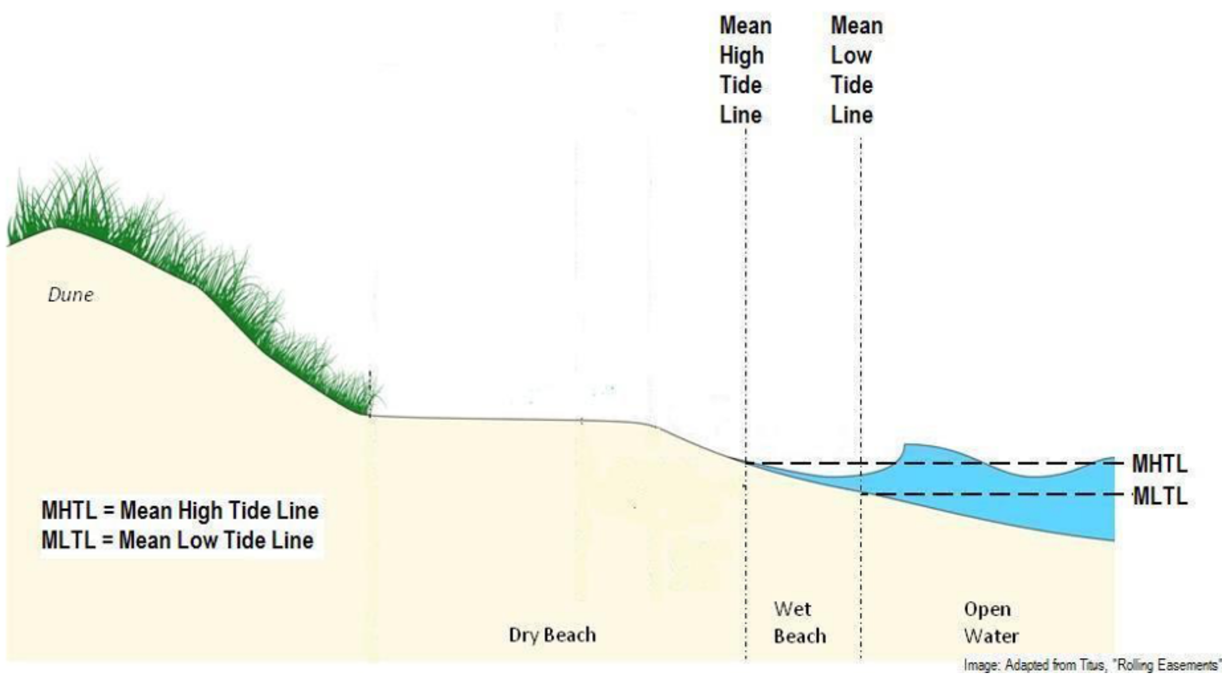

Figure 2

Quadrat Specifications via Microplastics Sampling and Processing Guidebook.

Cashman et al. (2020) noted that varying microplastic isolation and extraction techniques result in different microplastic recovery efficiencies. However, some microplastics may not be identified depending on the isolation and extraction method used in the study. This limitation could be attributed to the role of surface tension as it makes sediments adhere to a microplastic particle (Stolte et al., 2015). Finally, the lower extraction efficiency rates of microplastics from fine-grained sediments show the need for refinement in extraction techniques.

In density separation, saturated salt solutions are introduced to microplastic-containing samples; their concentration is modified accordingly to allow differential separation of microplastics by changing the density of the samples (Quinn et al., 2017). This technique allows suspended low-density microplastics to float while other high-density particles settle at the bottom during microplastic extraction (Li et al., 2018). Subsequently, the supernatant of the solution is commonly collected via a vacuum filter system, after which the supernatant is then filtered through a filter paper of mesh sizes ranging from 0.45 μm to 1.2 μm (Besley et al., 2017; de Carvalho & Neto, 2016; Horton et al., 2017; Quinn et al., 2017; Zhao et al., 2018). After separating the supernatant, portions of the sediment-containing sample with remaining particles are rinsed several times, and the washing solutions used are filtered using the same filter to minimize sample loss (Coppock et al., 2017; Nakajima et al., 2019; Zhao et al., 2018). After extraction, the microplastics are dried, often in an oven for several minutes at around 60-70ºC (Horton et al., 2017; Nakajima et al., 2019; Quinn et al., 2017; Zhao et al., 2018;). Once the microplastic has successfully been recovered, the recovery rate is then calculated by using either of the following formula:

$$\left(\frac{final\ weight\ of\ plastic}{initial\ weight\ of\ plastic}\right) \times 100\%$$

or

$$\left(\frac{final\ count\ of\ plastic}{initial\ count\ of\ plastic}\right) \times 100\%$$

In general, salt solutions with higher densities tend to have higher extraction efficiency rates when compared to those with lower densities (Table 2).

Table 2

Summary of microplastic quantification and extraction efficiency using different inorganic salts in density separation of microplastics

Floatation medium	Density (g/cm^3)	Mean extraction efficiency	Sediment sample	Identified polymers	Microplastic size range (μm)	Author
NaCl	1.17	<90.0%	Artificially spiked beach sediments	PP, LDPE, PE, HDPE, PS, nylon, PVC P, PVC UP, PET	200 - 400	Bayo et al. (2019); de Carvalho & Baptista-Neto (2016); Graca et al. (2017); Reed et al. (2018); Zhang et al. (2019); Quinn et al. (2017)
$ZnCl_2$	1.37	98.0%	Artificially spiked coarse sand sediments	Nylon	>100	Coppock et al. (2017); Maes et al. (2017); Horton et al. (2017)
		97.0%	Artificially spiked coarse sand sediments	PE	>100	

		85.0%	Artificially spiked fine silt sediments	Nylon	>100	
		88.0%	Artificially spiked fine silt sediments	PE	>100	
$CaCl_2$	1.46	69.0%	Artificially spiked beach sediments	EPS, PVC, ABS, PA, PES	40.1 - 20100	Crichton et al. (2017); Scheurer and Bigalke (2018)
NaH_2PO_4	1.4	>93.0%	Artificially spiked beach sediments	PE, PP PMMA, PS, PVC, POM, PET	500 - 5000	Zhang et al. (2020)
NaI	1.57	91% - 98%	Artificially spiked beach sediments	PP, LDPE, PE, HDPE, PS, nylon, PVC P, PVC UP, PET	200 - 400	Claessens et al. (2016); Pagter et al. (2018); Quinn et al. (2017); Espiritu et al. (2019)
$ZnBr_2$	1.71	99.00%	Artificially spiked beach sediments	PA, PE, PVC, PC HDPE, PET, PP	200 - 400	Sanchez-Nieva et al. (2017); Quinn et al. (2017)

Finally, microplastics are quantified by their composition, source, color, RIC, and density using visual identification, FTIR spectroscopy, and Raman spectroscopy.

SELF-ASSISTED DENSITY SEPARATION

New methods developed for separating plastic particles from sediments include electrostatic separation, magnetic separation, and pressurized fluid extraction (Cutroneo et al., 2021). However, these methods require heavy investment and a sophisticated setup. On the other hand, salt-assisted density separation is favorable for microplastic separation due to the accessibility of most salt solutions and a highly efficient separation process.

The choice of salt plays an important role in microplastic extraction via density separation. For example, sodium chloride (NaCl) is widely used in density separation because of its environmental and cost-friendly properties (Prata et al., 2019). This saline solution harbored a recovery rate of <90% for sediment samples containing mixed microplastics but has low recovery rates for some polyethylene terephthalate (PET)-based plastics, such as soft drink bottles (Quinn et al., 2017). The use of the saline solution is ineffective in PET as this material contains various additives or wetting agents that decrease its floatability, thus affecting its extraction efficiency (Wang et al., 2014). Moreover, density separation using NaCl is only effective for microplastics such as polyvinyl chloride (PVC) and PET, which are less dense than salt.

As NaCl may lead to an underestimation of microplastic abundance in sediments as its density is too low to allow flotation of certain microplastic particles (Nuelle et al., 2014), other inorganic salts such as zinc(II) bromide ($ZnBr_2$), NaCl, calcium chloride ($CaCl_2$), sodium iodide (NaI), and sodium dihydrogen phosphate (NaH_2PO_4) are used as alternatives for density separation of microplastics (Table 3). Differences in the density of inorganic salt solutions can be utilized to separate microplastics of density in the range of 0.8–1.6 g/cm^3 from sediments by carefully mixing the microplastic-contaminated sediment with saturated salt solutions and collecting the supernatant containing microplastics for further filtration (Rocha-Santos & Duarte, 2015).

Table 3

Advantages and disadvantages of the reported salts used in density separation

Salt Solution	Advantages	Disadvantages	Author
NaCl	Significantly cheaper than most options	Requires multiple washings of samples	Bayo et al. (2019); de Carvalho & Baptista-Neto (2016); Graca et al. (2017); Reed et al. (2018); Zhang et al. (2019); Quinn et al. (2017)

$ZnCl_2$	High Density Cost-effective	Highly hazardous Noxious to aquatic life	Coppock et al. (2017); Maes et al. (2017); Horton et al. (2017)
$CaCl_2$	Cost-effective	Ca^+ ions caused organic materials to gather	Crichton et al. (2017); Scheurer and Bigalke (2018)
NaH_2PO_4	Accessible and low cost Not toxic or hazardous Higher density than NaCl	Relies on temperature to increase solubility	Zhang et al. (2020)
NaI	Requires single washing of samples Reusable up to 10 times if cellulose fibers are absent Environmentally safe	Reacts with cellulose fibers causing samples to turn black Reported to be 70 times more expensive than NaCl Potentially hazardous	Claessens et al. (2016); Pagter et al. (2018); Quinn et al. (2017); Espiritu et al. (2019)
$ZnBr_2$	Requires single washing of samples Able to separate heavier polymers	More hazardous than other salt solutions due to toxicity Very expensive due to its high extraction rates	Sanchez-Nieva et al. (2017); Quinn et al. (2017)

As an alternative to NaCl, salts denser than NaCl were explored, including NaI, $ZnBr_2$, $ZnCl_2$, $CaCl_2$, and NaH_2PO_4. NaI and $ZnBr_2$ solutions can successfully isolate medium- to high-density polymers (Quinn et al., 2017). In one study that investigated the efficiency of salt-assisted density separation using $ZnBr_2$ and NaI salt solutions, artificially spiked beach sediments were prepared using different microplastics generated from different post-consumer products. Marine sediments were gathered from a beach and cleansed thrice using $ZnBr_2$ solution to eliminate floating debris and contamination traces. Following isolation of marine sediments from debris, microplastics were introduced using a 3:1 ratio of sediments and microplastics and mixed with NaI and $ZnBr_2$ salt solution. The microplastics on the solution's surface were recovered by vacuum suction and sieved with filter paper. While using NaI and $ZnBr_2$ salt solutions in microplastic separation resulted in good recoveries of 91–99% and 99%, respectively, these salts are expensive and hazardous to the environment. In addition, the hygroscopic nature and instability of NaI limit its utility for microplastic extraction (Kedzierski et al., 2017).

$ZnCl_2$ solution was also employed in the density separation of microplastics, likely because of this salt's high density and cost-effectiveness (Maes et al., 2017). In one study, microplastic samples from scraped consumer plastic items determined the recovery rate of microplastics using $ZnCl_2$. The microplastics were then seeded into multiple 1-g oven-dried marine sediments. The spiked sediments were suspended in 5-mL water and dyed with 50-μL Nile red in acetone with a shaker to render fluorescence, speeding up the identification and quantification process. Subsequently, the seeded sediments were gradually added to a 30-mL $ZnCl_2$ solution, mixed, then centrifuged. To visualize the stained microplastics, blue-green incident light via an orange mask was used to extract them from the solution's surface with glass Pasteur pipettes. Three extractions for each sample have been replicated in this process. Finally, the recovered plastic particles were combined, filtered, photographed, and counted. A recovery rate of 97–98% and 85–88% for coarse sand and fine slit were obtained, respectively. While the recovery rates were promising, there are hazards associated with using $ZnCl_2$. The substance is considered acutely toxic and very noxious to aquatic life, highlighting the need for careful operations in dealing with this substance (Frias et al., 2018).

$CaCl_2$ salt was also explored because it is safe and cost-effective (Crichton et al., 2017). In one study, microplastic-containing sediment was sieved, followed by adding $CaCl_2$ solution. The top surface was decanted into a filter funnel using a glass pipette with a polycarbonate membrane for vacuum filtration. Forceps were used to manually transfer the extracted polymers that failed to be filtered. A modest recovery rate of 69% was obtained, but Ca^{2+}-induced agglomeration of organic matter interferes with the identification of microplastics (Scheurer & Bigalke, 2018).

With the shortcomings of the mentioned salt solutions, it is still imperative to explore potential salt solutions for microplastic extraction. Several research groups investigated the use of NaH_2PO_4 in the density separation of microplastics. In their studies, the temperature was varied as density can be increased through heating. Artificially spiked beach sediments were collected, dried, and sieved to separate the attached debris. NaH_2PO_4 solution at 40 °C to achieve a density of 1.4 g/cm^3 was added to these sediment-containing samples. The upper solution was subsequently filtered with a sieve to extract the microplastics. Lastly, the extracted microplastics were rinsed, and the organic matter was separated using 35% hydrogen peroxide (H_2O_2). All microplastics involved in the experiment were extracted effectively using NaH_2PO_4. The salt solution demonstrated a recovery rate of >93% failing only to fully recover polymers of 0.5–1.0 mm. Despite its inability to recover smaller polymers, this salt may still possess a significant advantage in microplastic extraction as it is considered a high-density, low-cost, and non-toxic salt (Zhang et al., 2020).

In general, exploring the use of multiple complementary procedures is recommended to maximize the amount of extracted microplastics and recover all kinds of polymers from a sample. For example, performing density separation using a low-density salt followed by another round of density separation using a higher-density salt solution may present a higher recovery of microplastics. As chemical waste should be reduced as much as possible, investigating the recyclability of inorganic salts is recommended. In particular, the effect of repeated use of expensive and toxic $ZnCl_2$-containing solutions on the efficacy of this salt solution in separating microplastics warrants investigation. This study could determine the feasibility of recycling this salt solution in salt-assisted density separation of microplastics.

CONCLUSION AND RECOMMENDATION

The current strain of microplastic pollution is projected to persist in the future due to the forecasted increase in plastic manufacturing and a lack of effective waste management. Weathered plastics acquired from coastal habitats display substantial physical and chemical variations compared to unblemished plastics. Different strategies for recovering microplastics from sediments have been reported. Among these strategies, salt-assisted density separation holds promise for extracting microplastics because of this separation technique's simplicity and high recovery rates. The inorganic salts that facilitate microplastic density separation include NaCl, NaI, $ZnBr_2$, $ZnCl_2$, $CaCl_2$, and NaH_2PO_4. NaCl is non-hazardous and low-cost, but it fails to extract larger and denser microplastics. NaI, $ZnBr_2$, and $ZnCl_2$ demonstrated high recovery rates due to their high density; however, they are often expensive and harmful to the environment. On the other hand, $CaCl_2$ is not recommended as it tends to flocculate to organic substances despite its advantageous characteristics. A non-hazardous, economical, available, and high-density salt, such as NaH_2PO_4, is the most feasible to improve the separation efficiency.

Multi-step salt-assisted density separation is recommended to address some inherent limitations of each salt and improve recovery rates. A high-density salt is used first, followed by a low-density salt or vice versa in the density separation of microplastics. Given that there are different polymeric materials, it is recommended to identify the optimal low-density and high-density salt combinations for each type of polymer. Further research on the environment's interaction between the salts and microplastics is necessary to advance the knowledge of each salt's viability for separating microplastics with a wide range of properties. Also, promising salts such as NaH_2PO_4 may be studied further by combining with other inorganic salts to form a more effective hybrid salt solution in separating microplastics from sediments.

In the succeeding phase after microplastic extraction, the identification and detection of microplastics through microscopy and spectroscopy performs a vital function as this step distinguishes and classifies the isolated particles. The use of spectroscopic techniques such as FTIR and Raman spectroscopy is highly recommended as these modalities provide more accurate detail on the extracted particles and are more efficient and less prone to error than visual identification. Finally, with the enormous variety of techniques used in extracting microplastics, comparing the results obtained from multiple studies conducted worldwide is exceedingly challenging. Therefore, enforcing a standardized and validated protocol is necessary for a reliable comparison of numerous results.

REFERENCES

Avio, C. G., Gorbi, S., & Regoli, F. (2015). Experimental development of a new protocol for extraction and characterization of microplastics in fish tissues: first observations in commercial species from Adriatic Sea. *Marine Environmental Research*, *111*, 18-26. https://doi.org/10.1016/j.marenvres.2015.06.014

Bayo, J., Rojo, D., & Olmos, S. (2019). Abundance, morphology and chemical composition of microplastics in sand and sediments from a protected coastal area: The Mar Menor lagoon (SE Spain). *Environmental Pollution*, *252*, 1357-1366.

Besley, A., Vijver, M., Behrens, P., & Bosker, T. (2017). A standardized method for sampling and extraction methods for quantifying microplastics in beach sand. *Marine Pollution Bulletin*, *114*(1), 77-83. https://doi.org/10.1016/j.marpolbul.2016.08.055

Boucher, J., & Friot, D. (2017). Primary microplastics in the oceans: a global evaluation of sources. *IUCN*. https://doi.org/10.2305/IUCN.CH.2017.01.en

Brander, S., Renick, V., Foley, M., Steele, C., Woo, M., Lusher, A., Carr, S., Helm, P., Box, C., Cherniak, S., Andrews, R., & Rochman, C. (2020). Sampling and QA/QC: A guide for scientists investigating the occurrence of microplastics across matrices. *Applied Spectroscopy*, *74*(9), 1099-1125. https://doi.org/10.1177%2F0003702820945713

Brown, D. M., & Cheng, L. (1981). New net for sampling the ocean surface. *Marine Ecology Progress Series*, *5*, 225-227.

Carpenter, E. J., & Smith, K. L. (1972). Plastics on the Sargasso Sea surface. *Science*, *175*(4027), 1240-1241. https://doi.org/10.1126/science.175.4027.1240

de Carvalho, G., & Baptista-Neto, J. A. (2016). Microplastic pollution in the beaches of Guanabara Bay, Southeast Brazil. *Ocean and Coastal Management, 128*, 10-17. https://doi.org/10.1016/j.ocecoaman.2016.04.009

Cashman, M. A., Ho, K. T., Boving, T. B., Russo, S., Robinson, S., & Burgess, R. M. (2020). Comparison of microplastic isolation and extraction procedures from marine sediments. *Marine Pollution Bulletin*, *159*. https://doi.org/10.1016/j.marpolbul.2020.111507

Castro, R. O., Silva, M. L., Marques, M. R. C., & de Araújo, F. V. (2016). Evaluation of microplastics in Jurujuba Cove, Niterói, RJ, Brazil, an area of mussels farming. *Marine Pollution Bulletin*, *110*(1), 555-558. https://doi.org/10.1016/j.marpolbul.2016.05.037

Chubarenko, I., Bagaev, A., Zobkov, M., Esiukova, E. (2016). On some physical and dynamical properties of microplastic particles in marine environment. *Marine Pollution Bulletin*, *108*, 105-112. https://doi.org/10.1016/j.trac.2019.01.014

Claessens, M., Meester, S. D., Landuyt, L. V., Clerck, K. D., & Janssen, C. R. (2011). Occurrence and distribution of microplastics in marine sediments along the Belgian coast. *Marine Pollution Bulletin*, *62*(10), 2199-2204. https://doi.org/10.1016/j.marpolbul.2011.06.030

Cole, M., Lindeque, P., Halsband, C., & Galloway, T. S. (2011). Microplastics as contaminants in the marine environment: a review. *Marine Pollution Bulletin*, *62*(12), 2588-2597.

Common Law & Statutes - Florida Sea Grant. (2016). Florida Sea Grant. https://www.flseagrant.org/wateraccess/common-law-statutes/

Coppock, R. L., Cole, M., Lindeque, P. K., Queirós, A. M., & Galloway, T. S. (2017). A small- scale, portable method for extracting microplastics from marine sediments. *Environmental Pollution*, *230*, 829-837. https://doi.org/10.1016/j.envpol.2017.07.017

Covernton, G. A., Pearce, C. M., Gurney-Smith, H. J., Chastain, S. G., Ross, P. S., Dower, J. F., & Dudas, S. E. (2019). Size and shape matter: A preliminary analysis of microplastic sampling technique in seawater studies with implications for ecological risk assessment. *Science of The Total Environment*, *667*, 124-132. https://doi.org/10.1016/j.scitotenv.2019.02.346

Crichton, E. M., Noël, M., Gies, E. A., & Ross, P. S. (2017). A novel, density-independent and FTIR-compatible approach for the rapid extraction of microplastics from aquatic sediments. *Analytical Methods*, *9*(9), 1419-1428. https://doi.org/10.1039/c6ay02733d

Cutroneo, L., Reboa, A., Geneselli, I., & Capello, M. (2021). Considerations on salts used for density separation in the extraction of microplastics from sediments. *Marine Pollution Bulletin*, *166*. https://doi.org/10.1016/j.marpolbul.2021.112216

Dris, R., Gasperi, V., Rocher, V., Saad, M., Renault, N., Tassin, B. (2015). Microplastic contamination in an urban area: a case study in Greater Paris. *Environmental Chemostry, 12*(5), 592-599. https://doi.org/10.1071/EN14167

Eriksen, M., Lebreton, L. C., Carson, H. S., Thiel, M., Moore, C. J., Borerro, J. C., Galgani, F., Peter, R., Reisser, J. (2014). Plastic pollution in the world's oceans: more than 5 trillion plastic pieces weighing over 250,000 tons afloat at sea. *PloS ONE*, *9*(12). https://doi.org/10.1371/journal.pone.0111913

Erni-Cassola, G., Zadjelovic, V., Gibson, M. I., & Christie-Oleza, J. A. (2019). Distribution of plastic polymer types in the marine environment; A meta-analysis. *Journal of Hazardous Materials*, *369*, 691-698. https://doi.org/10.1016/j.jhazmat.2019.02.067

Espiritu, E. Q., Dayrit, S. A. S., Coronel, A. S. O., Paz, N. S. C., Ronquillo, P. I. L., Castillo, V. C. G., & Enriquez, E. P. (2019). Assessment of quantity and quality of microplastics in the sediments, waters, oysters, and selected fish species in key sites along the bombong estuary and the coastal waters of ticalan in San Juan, Batangas. *Philippine Journal of Science*, *148*(4), 789-801.

Felsing, S., Kochleus, C., Buchinger, S., Brennholt, N., Stock, F., & Reifferscheid, G. (2018). A new approach in separating microplastics from environmental samples based on their electrostatic behavior. *Environmental Pollution*, *234*, 20-28. https://doi.org/10.1016/j.envpol.2017.11.013

Frias, J., Pagter, E., Nash, R., O'Connor, I., Carretero, O., Filgueiras, A., Viñas, L., Gago, J., Antunes, J., Bessa, F., Sobral, P., Goruppi, A., Tirelli, V., Pedrotti, M.L., Suaria, G., Alliani, S., Lopes, C., Raimundo, J., Caetano, M., & Gerdts, G. (2018). Standardised protocol for monitoring microplastics in sediments. *Deliverable* 4.2. JPI-Oceans BASEMAN Project. http://dx.doi.org/10.25607/OBP-723

Fuller, S., & Gautam, A. (2016). A procedure for measuring microplastics using pressurized fluid extraction. *Environmental Science & Technology*, *50*(11), 5774-5780. https://doi.org/10.1021/acs.est.6b00816

Graca, B., Szewc, K., Zakrzewska, D., Dołęga, A., & Szczerbowska-Boruchowska, M. (2017). Sources and fate of microplastics in marine and beach sediments of the Southern Baltic Sea — a preliminary study. *Environmental Science and Pollution Research*, *24*(8), 7650-7661. https://doi.org/10.1007/s11356-017-8419-5

Grbic, J., Nguyen, B., Guo, E., You, J. B., Sinton, D., & Rochman, C. M. (2019). Magnetic extraction of microplastics from environmental samples. *Environmental Science & Technology Letters*, *6*(2), 68-72. https://doi.org/10.1021/acs.estlett.8b00671

Han, X., Lu, X., & Vogt, R. D. (2019). An optimized density-based approach for extracting microplastics from soil and sediment samples. *Environmental Pollution*, *254*. https://doi.org/10.1016/j.envpol.2019.113009

Horton, A., Svendsen, C., Williams, R., Spurjeon, D., & Lahive, E. (2017). Large microplastic particles in sediments of tributaries of the River Thames, UK – Abundance, sources and methods for effective quantification. *Marine Pollution Bulletin, 114*(1), 218-226. https://doi.org/10.1016/j.marpolbul.2016.09.004

Jahnke, A., Arp, H. P., Escher, B., Gewert, B., Gorokhova, E., Kühnel, D., Ogonowski, M., Potthoff, A., Rummel, C., Schmit-Jansen, M., Toorman, E., & MacLeod, M. (2017). Reducing uncertainty and confronting ignorance about the possible impacts of weathering plastic in the marine environment. *Environmental Science & Technology Letters*, *4*(3), 85-90. https://doi.org/10.1021/acs.estlett.7b00008

Kedzierski, M., Le Tilly, V., César, G., Sire, O., & Bruzaud, S. (2017). Efficient microplastics extraction from sand. A cost effective methodology based on sodium iodide recycling. *Marine Pollution Bulletin*, *115*(1-2), 120-129. https://doi.org/10.1016/j.marpolbul.2016.12.002

Khatmullina, L., Isachenko, I., Esiukova, E., Chibarenko, I. (2016). EXPERIMENTING ON SETTLING VELOCITIES OF NEGATIVELY BUOYANT MICROPLASTICS. 1-1. http://dx.doi.org/10.21610/conferencearticle_58b4315c8f7df

Kühn, S., Van Oyen, A., Booth, A. M., Meijboom, A., & Van Franeker, J. A. (2018). Marine microplastic: Preparation of relevant test materials for laboratory assessment of ecosystem impacts. *Chemosphere*, *213*, 103-113. https://doi.org/10.1016/j.chemosphere.2018.09.032

Lenz, R., & Labrenz, M. (2018). Small microplastic sampling in water: development of an encapsulated filtration device. *Water*, *10*(8). https://doi.org/10.3390/w10081055

Li, J., Liu, H., & Chen, J. P. (2018). Microplastics in freshwater systems: A review on occurrence, environmental effects, and methods for microplastics detection. *Water Research*, *137*, 362-374. https://doi.org/10.1016/j.watres.2017.12.056

Löder M. G. J., & Gerdts G. (2015). Methodology Used for the Detection and Identification of Microplastics—A Critical Appraisal. Marine Anthropogenic Litter. 201-227. https://doi.org/10.1007/978-3-319-16510-3_8

Maes, T., Jessop, R., Wellner, N., Haupt, K., & Mayes, A. G. (2017). A rapid-screening approach to detect and quantify microplastics based on fluorescent tagging with Nile Red. *Scientific Reports*, *7*(1), 1-10. https://doi.org/10.1038/srep44501

Masura, J., Baker, J., Foster, G., Arthur, C., & Herring, C. (2015). Laboratory Methods for the Analysis of Microplastics in the Marine Environment: Recommendations for Quantifying

Synthetic Particles in Waters and Sediments. *NOAA Marine Debris Division*. http://dx.doi.org/10.25607/OBP-604

Metz, T., Koch, M., & Lenz, P. (2020). Quantification of microplastics: Which parameters are essential for a reliable inter-study comparison? *Marine Pollution Bulletin*, *157.* https://doi.org/10.1016/j.marpolbul.2020.111330

Microplastics Sampling and Processing Guidebook. (n.d.). http://extension.msstate.edu/sites/default/files/publications/p3243.pdf

Nakajima, R., Tsuchiya, M., Lindsay, D. J., Kitahashi, T., Fujikura, K., & Fukushima, T. (2019). A new small device made of glass for separating microplastics from marine and freshwater sediments. *PeerJ*, *7*. https://doi.org/10.7717/peerj.7915

Ng, K. L., & Obbard, J. P. (2006). Prevalence of microplastics in Singapore's coastal marine environment. *Marine Pollution Bulletin, 52*(7), 761-767. https://doi.org/10.1016/j.marpolbul.2005.11.017

Nuelle, M. T., Dekiff, J. H., Remy, D., & Fries, E. (2014). A new analytical approach for monitoring microplastics in marine sediments. *Environmental Pollution*, *184*, 161-169. https://doi.org/10.1016/j.envpol.2013.07.027

Pagter, E., Frias, J., & Nash, R. (2018). Microplastics in Galway Bay: A comparison of sampling and separation methods. *Marine Pollution Bulletin*, *135*, 932-940. https://doi.org/10.1016/j.marpolbul.2018.08.013

Prata, J. C., da Costa, J. P., Duarte, A. C., & Rocha-Santos, T. (2018). Methods for sampling and detection of microplastics in water and sediment: A critical review. *Centre for Environmental and Marine Studies, 110,* 150-159. https://doi.org/10.1016/j.trac.2018.10.029

Quinn, B., Murphy, F., Ewins, C. (2017). Validation of density separation for the rapid recovery of microplastics from sediment. *Royal Society of Chemistry*, *9*, 1491-1498. https://doi.org/10.1039/C6AY02542K

Rocha-Santos, T., & Duarte, A. C. (2015). A critical overview of the analytical approaches to the occurrence, the fate and the behavior of microplastics in the environment. *TrAC Trends in Analytical Chemistry*, *65*, 47-53. https://doi.org/10.1016/j.trac.2014.10.011

Rodrigues, M. O., Gonçalves, A. M. M., Gonçalves, F. J. M., & Abrantes, N. (2020). Improving cost-efficiency for MPs density separation by zinc chloride reuse. *MethodsX*, *7*. https://doi.org/10.1016/j.mex.2020.100785

Sánchez-Nieva, J., Perales, J. A., González-Leal, J. M., & Rojo-Nieto, E. (2017). A new analytical technique for the extraction and quantification of microplastics in marine sediments focused on easy implementation and repeatability. *Analytical Methods*, *9*(45), 6371-6378. https://doi.org/10.1039/C7AY01800B

Scheurer, M., & Bigalke, M. (2018). Microplastics in Swiss floodplain soils. *Environmental Science & Technology*, *52*(6), 3591-3598. https://doi.org/10.1021/acs.est.7b06003

Serranti, S., Palmieri, R., Bonifazi, G., & Cózar, A. (2018). Characterization of microplastic litter from oceans by an innovative approach based on hyperspectral imaging. *Waste Management*, *76*, 117-125. https://doi.org/10.1016/j.wasman.2018.03.003

Song, Y. K., Hong, S. H., Jang, J., Kang, J. H., Kwon, O. Y., Han, G. M., & Shim, W. J. (2014). Large Accumulation of Micro-sized Synthetic Polymer Particles in the Sea Surface Microlayer. *Environmental Science Technology, 48*(16), 9014-9021. https://doi.org/10.1021/es501757s

Stock, F. Kochleus, C., Bänsch-Baltruschat, B., Brennholt, N., & Reifferscheid, G. (2019). Sampling techniques and preparation methods for microplastic analyses in the aquatic environments - A review. *Trends in Analytical Chemistry*, *113*, 84-92. https://doi.org/10.1016/j.trac.2019.01.014

Stolte, A., Forster, S., Gerdts, G., & Schubert, H. (2015). Microplastic concentrations in beach sediments along the German Baltic coast. *Marine Pollution Bulletin*, *99*(1-2), 216-229. https://doi.org/10.1016/j.marpolbul.2015.07.022

Teuten, E. L., Saquing, J. M., Knappe, D. R., Barlaz, M. A., Jonsson, S., Björn, A., Rowland, S., Thompson, R., Galloway, T., Yamashita, R., Ochi, D., Watanuki, Y., Moore, C., Hung Viet, P., Tana, T. S., Prudente, M., Boonyatumanond, R., Zakaria, M., Akkhavong, K., Ogata, Y., ... & Takada, H. (2009). Transport and release of chemicals from plastics to the environment and to wildlife. *Philosophical Transactions of the Royal Society B: Biological Sciences*, *364*(1526), 2027-2045. https://doi.org/10.1098/rstb.2008.0284

Wang, H., Wang, C. Q., Fu, J. G., & Gu, G. H. (2014). Flotability and flotation separation of polymer materials modulated by wetting agents. *Waste Management*, *34*(2), 309-315. https://doi.org/10.1016/j.wasman.2013.11.007

Zhang, B., Wu, D., Yang, X., Teng, J., Liu, Y., Zhang, C., Zhao, J., Yin, X., You, L., Liu, Y., & Wang, Q. (2019). Microplastic pollution in the surface sediments collected from Sishili Bay, North Yellow Sea, China. *Marine Pollution Bulletin*, *141*, 9-15. https://doi.org/10.1016/j.marpolbul.2019.02.021

Zhang, X., Yu, K., Zhang, H., Liu, Y., He, J., Liu, X., & Jiang, J., (2020). A novel heating-assisted density separation method for extracting microplastics from sediments. *Chemosphere*, *256.* https://doi.org/10.1016/j.chemosphere.2020.12703

Zhao, J., Ren, W., Teng, J., Liu, Y., Liu, H., Yin, X., Cao, R., & Wang, Q. (2018). Microplastic pollution in sediments from the Bohai Sea and the Yellow Sea, China. *Science of the Total Environment, 640-641,* 637-645. https://doi.org/10.1016/j.scitotenv.2018.05.346

Zhu, X., Nguyen, B., You, J. B., Karakolis, E., Sinton, D., & Rochman, C. (2019). Identification of Microfibers in the Environment using Multiple Lines of Evidence. *Environmental Science & Technology*. *53*(20), 11877-11887. https://doi.org/10.1021/acs.est.9b05262

ACKNOWLEDGMENTS

The completion of this manuscript could not have been possible without the supervision and support of Dr. Joel Garcia and Dr. Roger Tan of De La Salle University's Chemistry department.

ABOUT THE AUTHORS

Andrei Luigi T. Ang

Andrei Luigi T. Ang finished his middle school at St. Peter the Apostle School. Currently, he is studying at De La Salle University Integrated School and has chosen the academic field of Science, Technology, Engineering, and Mathematics. To date, he has already acquired two silver and one gold medal award.

Cara Isabel P. Del Rosario

Cara Isabel P. Del Rosario is a Science, Technology, Engineering and Mathematics strand student at De La Salle University Integrated School. She graduated from Pasig City Science High School with academic honors. Her research interests include environmental policies, climate adaptation, and ecological sustainability.

Carlos Miguel D. Jose

Carlos Miguel D. Jose was given multiple academic awards during junior high school and graduated with academic honors. He is currently a student in De La Salle University Integrated School under the Science, Technology, Engineering, and Mathematics strand; he likes technology and environmental research. He plans to pursue a computer studies degree.

Orrin Landon T. Uy

Orrin Landon T. Uy is currently a senior high school student at De La Salle University Integrated School in the Science, Technology, Engineering, and Mathematics Strand. He graduated junior high school at Sacred Heart School Tacloban City with academic honors.

Dr. Joel Garcia

Dr. Joel Garcia is a full professor of Chemistry from De La Salle University, Manila. He obtained his B.S. in Chemistry from the University of the Philippines, Diliman, and received his Ph.D. in Inorganic Chemistry from Wayne State University, USA where he investigated the physical properties of air-sensitive Eu2+ compounds for use as contrast agents in magnetic resonance imaging (MRI). His postdoctoral work in the Department of Biomedical Engineering, University of California, Davis, USA was focused on activatable MRI contrast agents using nanoparticles and small molecules. Currently, his research work is directed towards probe design and development using nanoparticles and small molecules, and utilization of NMR techniques for mechanistic studies and metabolic profiling of beverages.