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SÃO PAULO STATE UNIVERSITY - UNESP

School of Engineering- Bauru Campus

FERNANDA FILIPAKI

SEPARATION OF MICROALGAE AND MANAGEMENT OF MICROPLASTICS THROUGH INTEGRATED APPROACHES

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Thesis presented to the São Paulo State University (UNESP), School of Engineering, Bauru Campus, to obtain the title of doctor in Civil and Environmental Engineering.

Area of Concentration: Pollution and Control

Advisor: Prof. Dr. Gustavo Henrique Ribeiro da Silva

Co-advisor: Prof. Dr. Rodrigo Braga Moruzzi

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ABSTRACT

Microplastic pollution constitutes one of the major contemporary environmental challenges, directly affecting the quality of water resources and aquatic ecosystems. These emerging contaminants, characterized by their persistence, have been detected in a wide range of environments, which often act as diffuse sources of microplastics (MPs) in the environment. In parallel, the use of effluents for the cultivation and recovery of microalgae presents high potential as a sustainable strategy for tertiary treatment and biomass valorization. In this context, this thesis aimed to develop and evaluate integrated approaches for MPs monitoring and microalgae recovery. In Chapter 1, an offline ultrafiltration system coupled to a remotely operated vehicle (ROV) was developed and applied for in situ concentration of water samples. The concentration efficiency was evaluated using membranes with different filtration areas, and MPs were identified by Differential Scanning Calorimetry (DSC). In Chapter 2, microalgae were cultivated in photobioreactors with and without the addition of MPs, and the effluent was treated by coagulation–flocculation–sedimentation using natural coagulants (*Moringa oleifera* seeds and Tanfloc) and a chemical coagulant (aluminum sulfate). The coagulants were characterized by FTIR spectroscopy and the floc sedimentation dynamics analyzed using Particle Image Velocimetry. The results showed that the ROV system is a promising tool for environmental sampling in hard-to-reach areas, although DSC detection presented limitations at low concentrations. In the microalgae recovery stage, natural coagulants exhibited performance comparable to aluminum sulfate, promoting the formation of larger, denser flocs with higher sedimentation velocities. The integration of the two chapters demonstrates a synergistic approach: the first develops tools for environmental monitoring of MPs, while the second proposes solutions for their removal and the simultaneous recovery of biomass. Thus, this thesis contributes relevant methodological and technological advances toward addressing the challenges associated with water pollution and the sustainable reuse of resources in the twenty-first century.

Keywords: coagulants, microplastics, sedimentation analysis, infrared spectroscopy

RESUMO

A poluição por microplásticos constitui um dos principais desafios ambientais contemporâneos, afetando diretamente a qualidade dos recursos hídricos e dos ecossistemas aquáticos. Esses contaminantes emergentes, caracterizados por sua persistência, têm sido detectados em uma ampla variedade de ambientes, que frequentemente atuam como fontes difusas de microplásticos (MPs) no ambiente. Paralelamente, o uso de efluentes para o cultivo e a recuperação de microalgas apresenta alto potencial como estratégia sustentável para o tratamento terciário e a valorização da biomassa. Nesse contexto, esta tese teve como objetivo desenvolver e avaliar abordagens integradas para o monitoramento de MPs e a recuperação de microalgas. No Capítulo 1, foi desenvolvido um sistema de ultrafiltração offline acoplado a um veículo operado remotamente (VOR), aplicado à concentração in situ de amostras de água. A eficiência de concentração foi avaliada utilizando membranas com diferentes áreas de filtração, e os MPs foram identificados por Calorimetria Exploratória Diferencial (CED). No Capítulo 2, microalgas foram cultivadas em fotobiorreatores com e sem a adição de MPs, e o efluente foi tratado por coagulação–floculação–sedimentação utilizando coagulantes naturais (*Moringa oleifera* e Tanfloc) e um coagulante químico (sulfato de alumínio). Os coagulantes foram caracterizados por espectroscopia FTIR, e a dinâmica de sedimentação dos flocos foi analisada por Velocimetria por Imagem de Partículas. Os resultados mostraram que o sistema VOR é uma ferramenta promissora para amostragem ambiental em locais de difícil acesso, embora a detecção por CED apresente limitações em baixas concentrações. Na etapa de recuperação de microalgas, os coagulantes naturais exibiram desempenho comparável ao sulfato de alumínio, promovendo a formação de flocos maiores, mais densos e com maiores velocidades de sedimentação. A integração dos dois capítulos demonstra uma abordagem sinérgica: o primeiro desenvolve ferramentas para o monitoramento ambiental de MPs, enquanto o segundo propõe soluções para sua remoção e a recuperação simultânea de biomassa. Assim, esta tese contribui com avanços metodológicos e tecnológicos relevantes para o enfrentamento dos desafios associados à poluição hídrica e ao reaproveitamento sustentável de recursos no século XXI.

Keywords: coagulantes, microplásticos, análise de sedimentação, espectroscopia de infravermelho

SUMMARY

SCOPE OF THE RESEARCH

The pervasive presence of microplastics in the global water cycle represents a critical environmental challenge of the 21st century. This challenge manifests in two interconnected domains: their widespread dispersion in natural water bodies and their concerning accumulation within wastewater treatment systems. This thesis addresses both ends of this continuum through two complementary investigations. The first chapter focuses on the source and dispersion of microplastics, developing advanced methodologies for their detection in aquatic environments. The second chapter addresses their fate and removal, investigating innovative strategies for microalgae harvesting from contaminated wastewater. This sequential approach—from environmental monitoring to treatment optimization—provides a comprehensive framework for tackling microplastic pollution across different segments of the water cycle.

The first chapter of this doctoral research addresses the growing environmental and scientific concern surrounding microplastics (MPs). Defined as solid, water-insoluble plastic particles with dimensions ≤5 mm, microplastics have been detected across various environmental matrices, including freshwater, marine systems, sediments, and even biological tissues (Bergmann, Gutow and Klages, 2015). These small plastic particles, typically ranging from 100 nanometers to 5 millimeters in size, enter aquatic environments through both primary and secondary sources. Primary MPs are intentionally manufactured in small dimensions for use in industrial applications, while secondary MPs result from the physical, chemical, or biological degradation of larger plastic debris in the environment (Tirkey and Upadhyay, 2021).

Despite increasing global attention, there remains a methodological gap in standardized protocols for accurate sampling, detection, and characterization of microplastics, particularly in complex biological and aqueous matrices (Rocha-Santos and Duarte, 2015). Existing approaches often vary in terms of equipment, filtration pore size, sample treatment, and analytical techniques, leading to inconsistencies and difficulty in comparing results across studies.

In response to these challenges, this chapter explores and compares multiple strategies for sampling and concentrating microplastics from aquatic environments. The research initially tested an online ultrafiltration system designed to be integrated with an underwater vehicle for mobile water sampling. However, due to technical limitations and inefficiencies, the project pivoted toward evaluating an offline ultrafiltration setup, allowing the drone to operate independently from the filtration equipment. In this revised approach, the ultrafiltration unit was deployed either at a fixed shoreline location or mounted on a floating support structure that moved in tandem with an autonomous underwater vehicle.

By mapping the logistical and technical constraints involved in microplastic sampling across these methods, this chapter contributes to the development of more robust, scalable, and standardized protocols for the detection and quantification of microplastics in natural water bodies.

The second chapter of this research investigates integrated strategies for microalgae biomass harvesting and microplastic analysis in treated domestic sewage, with a special focus on the performance of natural coagulants. The study was divided into interconnected stages, each designed to address specific knowledge gaps and technological limitations in the field.

Initially, a comprehensive bibliometric and bibliographic review was conducted, covering publications from the last ten years, to identify the primary technologies used for microalgae harvesting and the main bioproducts derived from this biomass. This review analyzed contributions from different authors, countries, journals, and leading institutions to map current scientific and technological trends. The rationale for this review was to guide the experimental phase by identifying the most relevant and widely adopted methods for biomass harvesting. One key insight from the literature was the increasing use of combined harvesting technologies, which, although promising, raise a critical challenge: how can microalgal biomass be efficiently recovered for subsequent bioproduct development while avoiding contamination from chemical agents?

Chemical coagulants, although commonly used, present considerable drawbacks, such as toxicity and the risk of residual metal contamination in the biomass (Vandamme, Foubert and Muylaert, 2013). As a result, natural coagulants have garnered growing attention as safer and more sustainable alternatives (Abdul Hamid et al., 2016; Villaseñor-Basulto et al., 2018). Among these, Moringa oleifera (MO) seeds have shown great potential due to their low toxicity, biodegradability, and minimal interference with the biomass composition (De Moraes et al., 2021; Marinho et al., 2025; Silva et al., 2021; Yang, Hou and Miao, 2021). Similarly, tannin-based coagulants (e.g., Tanfloc S.G.) have demonstrated efficiency in removing pollutants from wastewater, including color and turbidity (Arismendi et al., 2018), and in recovering microalgae across various pH levels, outperforming aluminum sulfate in several contexts (Fuad et al., 2018; Mezzari et al., 2014; Niemia and Gentili, 2021; Roselet, Burkert and Abreu, 2016).

Building on this, the research explored the role of functional groups in coagulant performance through Fourier-transform infrared spectroscopy (FTIR), assessing their potential influence on microalgae harvesting mechanisms. This was followed by a modeling study aimed at identifying the optimal dosage for each coagulant. Using a factorial experimental design, the study evaluated different coagulant concentrations and the effect of rapid mixing gradients (RMG) on floc formation.

With the foundational gaps addressed, the core research focused on biomass production under conditions simulating microplastic contamination. The exponential growth of plastic production has resulted in widespread microplastic (MP) pollution in aquatic ecosystems. Wastewater treatment plants (WWTPs), while efficient in removing many pollutants, have been identified as significant sources of MPs—particularly polyester (PES), polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC) (Dronjak et al., 2023). These MPs pose significant challenges to microalgae cultivation and separation processes, potentially affecting the efficiency of biomass harvesting (Fu et al., 2019; Parsai et al., 2022; Xiang et al., 2022; Zahmatkesh Anbarani et al., 2023).

To simulate these conditions, microalgae were cultivated in two lab-scale flat-panel photobioreactors (PBRs): one containing only microalgae and another containing microalgae with added PVC particles. The effluent from both systems was then used for biomass harvesting via a lab-scale coagulation–flocculation–sedimentation (CFS) system. During this stage, a sedimentation column was adapted to allow the in-depth investigation of floc formation and characterization. Through image analysis, detailed information was obtained regarding floc attributes — such as size, shape, and structure — and their influence on sedimentation efficiency and microalgae harvesting.

This innovative approach enabled a deeper understanding of the gravitational settling behavior of flocs under the influence of different coagulants and operating conditions. Non-intrusive image-based techniques were employed to provide accurate particle size distributions and reveal the settling dynamics of heterodisperse aggregates (Dyer et al., 1996; Rijn, 2007).

The two chapters of this doctoral research are intrinsically connected through their shared focus on water quality and the challenges posed by emerging pollutants. While the first chapter develops methodologies to detect and quantify microplastics in natural water bodies, the second chapter addresses how these same contaminants interfere with wastewater treatment processes and biomass recovery. Together, they offer complementary perspectives: the first deepens our understanding of how microplastics are introduced and dispersed in the environment, while the second investigates their impact on treatment optimization and resource harvesting. This integrated approach not only highlights the complexity of working with contaminated water systems but also reinforces the need for technological innovation in both monitoring strategies and treatment solutions.

CHAPTER 01

1 INTRODUCTION

In recent years, there is increasing evidence of the harmful environmental impacts from improperly disposed plastics, which has greatly increased public concern over related hazards and risks they pose also to human health (Koelmans et al., 2019). Attention to nano- and microplastics (NMP) has also increased and regulatory measures already limit the use of different types of microplastics. Potential human health hazards NMPs can be associated with adverse effects of the polymeric particles as such, as well as with hazards of plastic-associated chemicals. To assess risks of the particles themselves, exposure levels need to be quantified first, which concerns the analysis of NMPs in environmental media, drinking water and food. However, there is still no consensus about the analytical methods which should be applied to quantify and qualify such particles (Mintenig et al., 2018).

Environmental NMPs represent highly diverse materials due to a myriad of morphologies and multiple polymer-additive- pollutant combinations. The microplastic size continuum ranges from 1 to 5000 µm, and most analytical studies address only particles bigger than 10 µm (practical size detection limit for optical microscopy). This diversity and very small particle size poses the greatest challenge to NMP detection and management strategies and mitigation actions. Hence the current knowledge on the actual levels of exposure to NMP in the environment or in food is limited and there is no data for the sub-micrometer size plastics.

The presence of such particles in the water cycle, with potential for human exposure, is an understudied yet urgent area of research. Hence, identification and monitoring of NMPs and their toxicological effects is crucial. Apart from the techniques used in analytical labs to characterize such particles, sampling them is also considered a big challenge., as they are extremely small and present in very small concentrations (Mintenig et al., 2018). The use of (beyond) state of the art technologies that can facilitate sampling and characterization of environmental NMPs is therefore highly demanding.

Driven by recent mechatronics and electro technical open-source internet of things developments, several novel technologies have appeared in the market that can contribute to more robust environmental monitoring systems. More and more, innovations such as autonomous or remotely operated vessels / aquatic drones (ROV; ASV), real-time sensor networks are being deployed in the field for smart and autonomous environmental data collection. These tools allow new opportunities to monitor biodiversity, or collect 3D water quality data, which is key to calibrating and validating different types of water quality, ecological or hydrodynamic models.

The Introduction is the initial section of the paper that presents the research objective, the rationale, and its importance to the field, the problem, and its limitations. It should be written succinctly, clearly, and objectively.

5 CONCLUSION

This thesis demonstrated, in an integrated manner, the feasibility and importance of innovative approaches to two of the main current challenges in water resources management: microplastic pollution and the pursuit of more sustainable effluent treatment systems. Through a strategic division into two investigative axes, it was possible to make significant advances in both environmental detection methodologies and technologies for biomass removal and valorization.

In the first axis, the combination of an ROV with an ultrafiltration concentration system proved to be a promising tool for microplastic sampling, overcoming several limitations of conventional methods. The system enabled more strategic sampling in terms of location and depth, with a drastic reduction in the volume of water to be transported, optimizing field logistics. However, the results obtained with the DSC technique highlighted the method's sensitivity to low concentrations and cross-contamination, indicating the need for more rigorous membrane cleaning protocols and, potentially, the use of more sensitive complementary analytical techniques for definitive polymer confirmation. The detection of Polypropylene (PP) in the environmental samples, however, reinforces the relevance of continuous monitoring of these materials.

In the second axis, the studies on microalgae harvesting confirmed coagulation-flocculation-sedimentation as a highly efficient process, with natural coagulants performing equivalently or superiorly to aluminum sulfate. The detailed morphological analysis of the flocs via PIV revealed that sedimentation efficiency is intrinsically linked to the physical characteristics of the formed aggregates. Larger and more compact flocs, such as those generated by Moringa oleifera, settled at significantly higher velocities. The presence of PVC microplastics in the effluent did not drastically compromise floc formation, although it required slight adjustments to the optimal coagulant dosages, demonstrating the robustness of the process and the ability of natural coagulants to agglomerate particles of different natures. Spectroscopic analysis (FTIR) provided valuable insights into the functional groups present in the coagulants, elucidating part of the chemical mechanisms involved in particle destabilization.

In summary, this thesis contributes to the body of scientific knowledge by validating an advanced methodology for microplastic sampling and by developing and optimizing a microalgae harvesting process using natural coagulants, even in the presence of emerging contaminants. The results emphasize the potential of accessible and sustainable technologies, such as the use of drones for monitoring and biocoagulants for treatment, paving the way for future research aimed at real-scale implementation. The integration between accurate environmental monitoring and the development of effective treatment processes represents a crucial step towards the holistic management of water quality and the promotion of a circular economy in environmental sanitation.

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RECOMMENDATIONS FOR FUTURE RESEARCHS

Considering the promising results achieved in this research, it is recommended that future studies continue to explore integrated approaches that combine physical and chemical methods for the treatment and recovery of complex aqueous matrices. The incorporation of more robust analytical frameworks and the adoption of in situ monitoring technologies are also encouraged to improve the accuracy, reproducibility, and scalability of the developed methodologies, thus enabling their application in real operational conditions.

For the subsequent stage of this research, it is recommended that the study continues to employ the drone-based sampling platform but implements a parallel membrane configuration or membranes with a larger surface area. This modification would enhance sample concentration rates and significantly optimize the total processing time. Furthermore, to address the issue of clogging and ensure a consistent flow rate, the installation of a pre-filtration unit at the sample intake is strongly advised. The development of more effective fouling control and cleaning protocols is also suggested, as this is crucial for preventing contamination and extending the operational lifespan of the membranes.

In the present research, the challenge of managing the sampling hose and ensuring a stable drone hover was noted; therefore, it is recommended to investigate the feasibility of a floating sampling system. Such a system could move synchronously with the drone, which would eliminate the complications associated with an extended hose connection. Finally, the pursuit of full system automation is recommended to enable precise control of the intake volume and facilitate fully autonomous sampling missions, thereby increasing the system's reproducibility and ease of use.

For the second stage, it is recommended that the study be continued using the same principles for obtaining the effluents but applying the flotation method as an alternative to sedimentation. This modification would allow for a more comprehensive comparison between treatment techniques and further elucidate the effectiveness of the coagulation process in removing microalgae and microplastics. In the present research, some flocs were observed to float due to differences in density, suggesting that flotation could be an effective complementary method for contaminant removal. Moreover, as the current results were obtained under specific experimental and hydrodynamic conditions—particularly during a period of extreme heat—it is suggested that future analyses use effluents generated on the same day, which may facilitate a more accurate interpretation of the phenomena involved. It is also recommended to evaluate dosage calculations based solely on the purity of the Moringa oleifera powder, to determine whether the presence of oil interferes with floc sedimentation. Regarding FTIR analysis, future work should investigate the application of the coagulant directly to the effluent to obtain a clearer understanding of the chemical interactions and structural modifications promoted by each coagulant on microalgae and microplastics.

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