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https://doi.org/10.3390/app15052630

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First Appraisal of Effective Microplastics Removal from the Textile Manufacturing Processes

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Abstract: The textile industry consumes large volumes of freshwater, producing enormous wastewater containing chemicals from dyeing and bathing, but also microplastics concentrations that have not been deeply studied. Liquid wastes from the synthetic and natural textile manufacturers were treated with a new disruptive technology (Adiabatic Sonic Evaporation and Crystallization, ASEC), which completely removed contaminants from water, providing distilled water and crystallized solids. The current study presents the characterization of the industrial residues and the obtained by-products: microplastics and organic matter contained in the solid residue were analyzed and characterized through chromatography. The results of the analyses displayed that compounds such as benzene, benzoic acid and 2,4-dymethyl-1-heptene were found in the synthetic industry water samples as degraded compounds of polyester and polypropylene. Meanwhile, the natural industry water also contained polyester, nylon and PMM polymer. After the depuration of samples, microplastics were completely retained in the solid phase, together with the organic matter (sulfate and surfactants) resulting on clean water. This is the first study focused on the study of microplastics generated by the textile industry and their prevention by removing them as solid waste.

Keywords: crystallized solids; microplastics; Adiabatic Sonic Evaporation and Crystallization; effective water depuration; plastics in water; synthetic fibers; natural fibers

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Received: 13 January 2025
Revised: 12 February 2025
Accepted: 26 February 2025
Published: 28 February 2025

Citation: Bonnail, E.; Vera, S.; Blasco, J.; DelValls, T.Á. First Appraisal of Effective Microplastics Removal from the Textile Manufacturing Processes. *Appl. Sci.* **2025**, *15*, 2630. https://doi.org/10.3390/app15052630

1. Introduction

Awareness is increasing about the presence of plastics and microplastics (MPs) in the environment, especially in aquatic environments (rivers, lagoons and oceans) and their impact on health is being more and more frequently studied [1–3]. The presence of MPs results from the degradation of larger pieces of plastic, or from being released directly into the environment. In the case of the fashion industry, MPs are provided by both sources: the manufacturing process, and the wearing and washing of textiles made from synthetic (plastic) fiber fragments. Around 66% of the textile fiber market is produced from synthetic fibers (mainly nylon and polyester), followed by cotton, cellulosic materials and wool [4]. The production processes of synthetic fibers (Sf), yarns, fabrics and products may be responsible for the increased release of microfibers [5]. In particular, the application of abrasive friction during production is an important factor in MP formation [6].

Periyasamy and Tehrani-Bangha [5] estimated that of global fiber production will reach 145 million metric tons in 2030, due to garments made from synthetic fibers (34.8%). Previous studies [7,8] have determined an annual estimation of MPs release into the ocean of between 0.2 and 0.5 million from synthetic textiles. MPs from aquatic environments can enter organisms through various pathways, commonly ingestion (bioaccumulation), but they are also biologically transported through the food chain [9,10] and enriched at higher trophic levels (biomagnification), ultimately affecting human health [11].

The release of MPs into the water environment is being regulated in Europe through the Council decision 2021/764 (May 2021) [12], which requires water pollution control (including MPs) to ensure that water bodies achieve a good ecological status. Therefore, controlling the potential plastic emission sources, such as the textile industry, would allow the aquatic ecosystem quality to improve. In terms of the water from textile manufacturing processes (wet processing: dyeing, pre-treatment, finishing), it is supposed that around 86% of consumed water [13] is usually dumped after water waste treatment in Europe. However, it is crucial to monitor process parameters to reduce waste, costs and environmental impacts [14].

MPs (and nanoplastics) removal during water treatment includes chemical removal (fourth flotation, agglomeration, coagulation), chemical degradation (hydrolysis, cavitation, advanced oxidation, photocatalysis) [15] and physical removal (such as micro-/nano-/ultra-filtration, reverse osmosis or dynamic membrane). Biological removal employs aerobic and anaerobic digestion, lagoons and septic tanks, but these are inefficient and might cause secondary contamination [16]. A recent study focused on the thermal pyrolysis process into fuel oil [17]. Ozonation can dissolve 90% of MPs into functional groups with O_2, but harmful intermediate chemicals might be generated [18]. UV oxidation makes MPs into smaller sizes or nanoplastics [18]. Therefore, most of these techniques are not completely efficient, and are extremely costly, with a high consumption of energy or other consumables (membranes, reagents) or are not scalable, among others. The search for a cost-effective technology with a high level of effectiveness for MPs removal to treat significant volumes of wastewater is still a challenge.

Zero liquid discharge (ZLD) technologies might represent a transformative solution because despite their high implementation costs, due to the use of renewable resources as energy supply, and their 100% efficiency in water recovery, they could be a cost-effective choice to significantly remove MPs from wastewater [19] and other environmental challenges. These ZLD technologies are reshaping wastewater management practices in the textile sector [20]. The utilization of water in the textile industry, the avoidance of chemicals and reduced waste disposal are some of their main advantages. Nevertheless, once these technologies are implemented in industry, the destiny of MPs is not clear. A previous study [21] characterized the removal of contaminants (metals and metalloids) from textile industry fluids after an innovative ZLD treatment. However, this study solely focuses on the removal of MPs from textile industry water using this technology, with the aim of checking its effectiveness.

2. Materials and Methods

2.1. Liquid Residues

The industrial processes for both synthetic (Sf) and natural (Nf) fiber manufacturing are composed of different steps, which are partially cyclical. Water at different temperatures is processed through baths with chemicals and additives (colorants, surfactants, glues, detergents, dyes, etc.) that appear in the solution of the output effluents. These are collected and treated in a local wastewater treatment plant (WWTP). In the case of Sf, the daily total average volume is 150 m^3, and a volume of 950 m^3/day is employed for Nf.

For the experiment, different aqueous residues were collected at different phases of the Sf textile processing line (Figure 1). Sample S1 was collected during the preparation phases from the autoclaves. Sample S2 corresponded to the staining and dyeing phases, and is called jiggers, a mixture with the fluid from the previous treatment. Sample S3 was taken before the WWTP. A fourth sample from the Nf manufacturer was collected as a homolog of S3 (after all the treatments plus the domestic wastes, and before the WWTP).

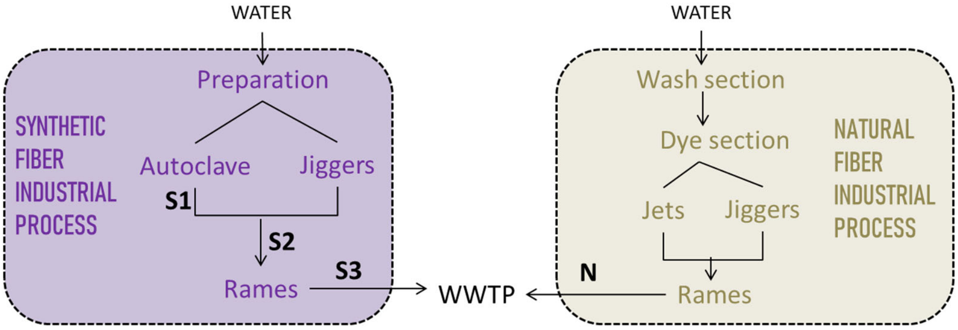


Figure 1. Scheme of the manufacture textile processes for synthetic (**left**) and natural (**right**) fibers and the sampling locations (S1, S2, S3, N).

2.2. Adiabatic Sonic Evaporation and Crystallization (ASEC) Technology

The ASEC system (EP:3135635) is a ZLD technology designed for contaminated fluid purification using the distillation–separation of dissolved solids and liquids based on physical changes, resulting in clean water and dried crystallized solid salts (moisture < 1%). The technology requires an energy supply between 3 and 5 kWh/m^3 of treated fluid (it can be replaced by renewable energies or residual heat from industrial processes) [21–23]. Briefly, the ASEC system uses a physical process combining evaporation and crystallization in one step to remove all the solutes and solids in the solution as salts and/or dried solids, without the use of reactants or fungible pieces. The fluid to be treated passes through a solid removal stage using self-cleaning filters. Then, it flows through two preheating exchangers to reach the required process temperature, before being directed to the nozzles. The fluid is injected into the evaporation chamber, impacting the transfer surfaces, and onto hot plates, forming a falling layer. The vapor is then routed to the interior of the plates, where it transfers heat to the falling layer of the fluid, subsequently condensing the pure water (distilled water). The water is then directed to the condensate tank existing in the evaporation chamber. Meanwhile, the concentrated fluid inside the crystallizer loses moisture until it reaches the target level. At this stage, the solid product falls into a screw conveyor that transports it out of the plant, where it is recovered.

2.3. Experiment

To test the efficiency of the ASEC technology, water samples (50 L) were collected from a different phase of the Sf (S1, S2, S3), and a new sample from the Nf textile manufacturer (Figure 1), as detailed in Bonnail et al. [21]. The same volume (50 L) of distilled water was obtained after depuration for all samples, together with the crystallized solids (950 g for $S1_{out;}$ 260 g for $S2_{out}$; 350 g for $S3_{out}$; and 200 g for N).

2.4. Sample Collection and Analyses

Liquid industrial waste samples (Sf: S1, S2, S3; Nf: N) were directly collected from industry (also called as $_{\text{-in}}$ samples) and injected into the ASEC system. Treated samples (also called as $_{\text{-out}}$ samples) were collected, together with their respective solid dried residues (humidity < 1%) as outlined in [21].

2.4.1. MPs Characterization

The extraction and analysis of MPs was carried out for 12 samples (8 liquid and 4 solid). For the MPs extraction in the liquid samples, a volume of 200 mL and 1 g was taken for liquid and solid samples, respectively. The first step was the removal of organic matter by adding an oxidizing agent. It was kept under contact stirring at a temperature of 70 °C for 3 h. Next, NaCl was added until saturation, and it was left to settle for 12 h to favor the separation by the density of the plastic materials. Finally, the supernatant was filtered with quartz fiber filters of 0.3 µm pore size (previously pre-treated at 500 °C for 2 h). Analyses were carried out by certified laboratories.

A gas chromatograph equipped with a pyrolyzer and a mass spectrometer (Pyr-GC/MS) were used for the identification of MPs. The pyrolysis was performed by sharp heating to 600 °C in an inert atmosphere (He), and the released products were analyzed. The chromatographic system employed Ultra Alloy UA+-5 capillary columns. The identification of the polymers was carried out thanks to the pyrolyzed compounds by comparing the mass spectra obtained and the spectral libraries (NIST and F-Search MPs).

2.4.2. Determination of Surfactants and Sulfates

The MBAS/CTA titration was applied for the surfactant's determination (MBAS measurement range: 0.1–5.0 mg/L; CTAB measuring range: 1–20 mg/L). The determination of SO_4^{2-} was made by using the turbidity measurement with barium sulfate and a specific kit (Visocolor® ECO, Macherey-Nagel GmbH & Co. KG, Düren, Germany) (measuring range: 25–250 mg/L).

3. Results

3.1. Mass Balance

Volumes of 50 L were supplied to the ASEC equipment, and about 50 L of each sample were recovered after the treatment (Figure 1) with complete transparency (~10 mg/L of suspended solids). The pH ranged between 6.87 and 7.01, with an electrical conductivity of 16 µS/cm [21]. So, the complete recuperation of the liquid volume was achieved after the treatment of all the samples. On the other hand, the dried solids and crystallized salts were collected from the output. Effluents from both industries before the WWTP have a dried conglomerate production (humidity < 1%) of 7 g per liter for Sf and 4 g per liter for Nf (Table 1).

Table 1. Salt formation collected in the ASEC, the MP mass in the liquids (Aq) and solids (Salt) at the entrance ($_{\text{-in}}$) and exit ($_{\text{-out}}$) of the equipment and the ratio of MPs retained per gram of salt formation.

Samples	**Salt Formation (g/L)**	**MP Mass (mg)**			**Retained Ratio (mg MP/Solid)**	**MPs Removal (%)**
		$\mathbf{Aq_{in}}$	**$\mathbf{Aq_{out}}$**	**$\mathbf{Solid_{out}}$**		
S1	19	44 ± 1	0.3 ± 0.1	95 ± 1.0	0.005	99.97
S2	5.8	182 ± 3	0.1 ± 0.1	0.27 ± 2.0	4.65×10^{-5}	99.99
S3	7	55 ± 2	0.1	101 ± 2.0	0.014	99.99
N	4	69 ± 3	0.25 ± 0.1	146 ± 2.0	0.0365	99.99

The samples with the highest MPs particle load (in mg) retained in the filter were all from the raw samples: $S2_{\text{-in}}$ (182) > $N_{\text{-in}}$ (69) > $S3_{\text{-in}}$ (55) > $S1_{\text{-in}}$ (44). The MP mass in the depurated water samples ($_{\text{-out}}$) was below 0.3 mg for the samples studied.

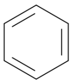
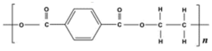
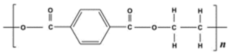
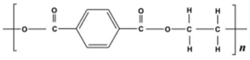
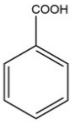
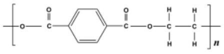
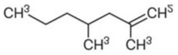
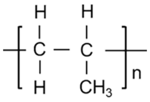
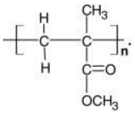
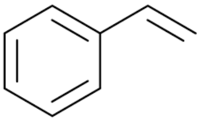
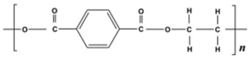
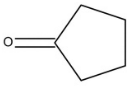
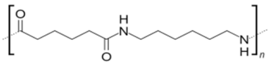
The number of MPs found in dried solids were 146 mg in N, 101 mg in S3, 95 mg in S1 and 0.27 mg in S2. The highest amount of MPs retained was in the N sample with a ratio of 0.014; this meant that most of the mass of the salt formed was composed of MPs.

3.2. By-Product Characterization

3.2.1. Water Intake Characterization

Regarding incoming samples (Table 2), in sample $S1_{in}$, the presence of polyester was detected in degraded compounds including benzoic acid and their structure. For sample $S2_{in}$, it was not possible to identify the type of polymer. 2,4-dymethy-1-heptene being found evidences the presence of polypropylene (low match). In addition, a compound containing benzene in its structure might suggest the presence of polyester (styrene, low match). The presence of any polymer is not completely guaranteed for $S3_{in}$, despite benzene and styrene being present (low match). In the case of the N_{in} sample, methyl methacrylate also appears, indicating the presence of polymethyl methacrylate (low match). Cyclopentanone also appears, which may be due to nylon-6,6. In addition, other compounds with the ester functional group appear due to polymethyl methacrylate or polyester.

Table 2. Compounds detected in the liquid samples from the input of the Sf and Nf samples.

Compound Input Water	$\mathbf{S1_{in}}$	$\mathbf{S2_{in}}$	$\mathbf{S3_{in}}$	$\mathbf{N_{in}}$
Benzene 	Polyester 	Polyester 	Low match	Polyester 
Benzoic acid 	Polyester 			
2,4-dymethyl-1-heptene 		Polypropylene 		PMMA polymer 
Styrene/ 		Polyester  (Low match)	Low match	Presence
Cyclopentanone 				Nylon-6,6 

3.2.2. Water Output Characterization

The collected water after the ASEC treatment was transparent for all the samples [13]. None of the compounds confirmed in the input samples were significantly detected in the $S1_{out}$ samples. For the output water of sample 2 ($S2_{out}$), the presence of any polymer was not guaranteed. Polyester may be present due to the presence of benzene, styrene and cyclohexane, 1,3,5- triphenyl (low match). For the sample $S3_{out}$, there was no significant detection of any of the compounds in Table 1. For the distilled water collected from the natural fiber sample (N_{out}), none of the compounds shown in Table 3 were significantly detected.

Table 3. MPs characterization in solid samples collected after the ASEC depuration of the textile effluents.

Solid $S1_{out}$

Compound	CAS	Characteristic ions m/z	Match
Benzene	71-43-2	50, 51, 77, 78	790
Cyclopentanone	120-92-3	41, 55, 84	714
2,4-Dimethyl-1-heptene	19549-87-2	43, 55, 70, 83	887
Styrene	100-42-5	51, 78, 104	888
Cyclohexane, 1,3,5-triphenyl-	117-81-7	149, 167	666
Bis(2-ethylhexyl) phthalate	117-81-7	149, 167	665

Solids $S2_{out}$

Compound	CAS	Characteristic ions m/z	Match
Benzene	71-43-2	50, 51, 77, 78	805
Styrene	100-42-5	51, 78, 104	834
Bis(2-ethylhexyl) phthalate	117-81-7	149, 167	820
2,4-Dimethyl-1-heptene	19549-87-2	43, 55, 70, 83	895
Toluene	108-88-3	91,92	708

Solids $S3_{out}$

Compound	CAS	Characteristic ions m/z	Match
Benzene	71-43-2	50, 51, 77, 78	886
Toluene	108-88-3	91, 92	773
2,4-Dimethyl-1-heptene	19549-87-2	43, 55, 70, 83	842
Styrene	100-42-5	51, 78, 104	843
1-Eicosene	01-07-3452	43, 55, 69, 71, 83, 97	830
Bis(2-ethylhexyl) phthalate	117-81-7	149, 167	676

N_{out}

Compound	CAS	Characteristic ions m/z	Match
Benzene	71-43-2	50, 51, 77, 78	798
2,4-Dimethyl-1-heptene	19549-87-2	43, 55, 70, 83	919
Styrene	100-42-5	51, 78, 104	709
Benzene, 1-ethyl-3-methyl-	620-14-4	120, 105	703
Bis(2-ethylhexyl) phthalate	117-81-7	149, 167	913

3.2.3. Solid Characterization

When analyzing the different dried solid samples (Table 3), different compounds were detected, confirming the presence of MPs. In the dried solids from S1, 2,4-dimethyl-1-heptene was detected, so polypropylene may be present. Polyester may be present due to the presence of styrene, benzene and cyclohexane, 1,3,5-triphenyl. However, this cannot be confirmed as no further pyrolyzed compounds appear to confirm the presence of polyester. Also, cyclohexane, 1,3,5-triphenyl- had a low match. Cyclopentanone also appeared, which may be due to nylon-6,6 (low match).

Solids from sample S2 displayed polypropylene, due to the presence of 2,4-dimethyl-1-heptene. Although benzene and styrene appeared, it is not certain that polyester was present as cyclohexane, 1,3,5-triphenyl and other pyrolyzed compounds did not appear.

The solids derived from treatment of S3 contained 2,4-dimethyl-1-heptene, so polypropylene might be present. In addition, peaks corresponding to alkenes were observed as 1-eicosene, although the typical profile of polyethylene was not observed. Therefore, the presence of polyethylene in the sample was not guaranteed.

Solids obtained from the treatment of the N sample showed 2,4-dimethyl-1-heptene in the chromatogram, indicating the presence of polypropylene. Polyester may be present due to the presence of styrene, benzene and 1-ethyl-3-methyl. However, this cannot be confirmed due to no further pyrolyzed compounds appearing to confirm the presence of polyester, and the matches were low.

The sulfate content and the concentrations of surfactants are summarized in Table 4. All the original samples contained a significant number of sulfates (above 200 mg/L). However, the ASEC treatment removed the sulfate ions (<25mg/L). Detergents were present in all the input samples, but the output liquid samples did not present with surfactant concentrations.

Table 4. Sulfate ions and concentration of surfactant results.

Sample	Sulfate Ions (mg/L)	Concentration of Surfactants (mg/L)	
		MBAS Anionic	CTAB Anionic
$S1_{in}$	8000	<0.1	1.0
S_{-in}	200	<0.1	1.0
S_{-in}	400	<0.1	1.0
N_{in}	200	2.0	3.0
$S1_{out}$	<25	<0.1	<0.1
$S2_{out}$	<25	<0.1	<0.1
$S3_{out}$	<25	<0.1	<0.1
N_{out}	<25	<0.1	<0.1

4. Discussion

There are almost 2 thousand chemicals involved in fabric production, and 165 of them are classified by the European Union as hazardous to the environment [13]. Among emerging contaminants, MPs involved in the fashion industry are detected in different environmental compartments and biological systems [24]. The release of Sf into the environment leads to widespread contamination, and has ecological consequences [25]. MPs are already found in the respiratory tract, blood, placenta, feces and other human body compartments; also, MPs might induce cancer, diseases and other immunotoxicity reactions [26]. On the other hand, fashion industry processes also consume huge volumes of water (79 billion m^3 [13]). Therefore, there is a dual problem associated with textile production: water consumption and contamination. Nowadays, in the search for new procedures, the use of alternative, friendly chemicals and innovative technology might help to solve environmental/health problems.

In the current study, the ASEC process has been demonstrated to be effective in eliminating MPs and other organic compounds (sulfates and surfactants) from depurated textile industry effluent water samples (Tables 2 and 4). Despite the surfactant lubricate [27], fiber fragment emissions occur from the use of detergents containing zeolite that promotes friction (powder) and the hydroxylation of surfaces such as PET (alkali-based detergents) [5]. The identification of MPs was carried out using a Pyr-GC/MS. This technique pyrolyzes the samples and analyzes the vapors. So, the identification of the polymers was based on pyrolyzed compounds. Sf samples showed polyester and polypropylene present (PP). Meanwhile, despite the natural origin of the fibers, Nf samples contained PP, PMMA, nylon and polyester. Once the samples were treated with ASEC technology, it was not possible to detect the presence of polymers. The generated dried solids derived from the treatment of the contaminated samples were also analyzed (Table 3). These displayed the presence of PP and nylon. Some limitations require further study or observation, such as the transformation of MPs into other potentially hazardous materials. The thermal processes that occur both in purification and those carried out in the analytical determination could lead to the formation of new compounds with a toxic potential greater than that of microplastics, such as phthalates. The treatment of new samples and greater volumes, or even the choice of other characterization methods, would help to clear up doubts. Meanwhile, the temperature of the depuration process might be adjusted to obtain better results with the same efficiency.

Previous studies [25,28] support that many Sf, such as nylon, polyester and acrylics, were found to shed in clothing and discharge from the stream wastewater into aquatic ecosystems. Vdovchenko and Remini [29] found significant environmental contamination originating from the textile industry, a notion supported by the prevalence of polyester, nylon, polyamid and polyurethane (textile materials), which are among the most frequently encountered polymer types in food and human samples.

Other industries releasing MPs into water sources include the pharmaceutical industry [30,31], food processing and agriculture [32] and the cosmetic industry [33], among others. By coupling ZLD into their wastewater cycles, they would save a huge volume of water, and more importantly, prevent the MPs being released into water sources and, consequently, the environment.

The study's main environmental implications include water recovery and recycling, and the minimization of wastewater discharge. The high-quality water byproduct free of MPs can be reinjected into the industry for a new use (Figure 1). The second by-product of dried solids requires further research for its potential value, but the main purpose—the immobilization of MPs—is achieved. There are some other environmental implications, such as potential hazards or toxicity derived from the solid phase, that require tackling. This residue is transformed into an easier and low-volume conglomerate of compounds. By using renewable energy sources (or even heat from industrial processes), it is possible to reduce the carbon footprint. Other economic implications are linked to saving costs in chemical and membrane usage. The implementation of ZLD technologies in the textile industry is subject to significant costs, but these are justified by the benefits obtained in terms of sustainability and environmental protection. This would allow the fulfillment of the European Green Deal, boosting the efficient use of resources, reducing GHG emissions and controlling pollution release (in these cases, from MPs and other chemicals from the textile industry).

5. Conclusions

The textile industry generates a significant number of MPs in the manufacturing process. These reach the aquatic systems, promoting environmental pollution and, consequently, environmental health, due to the incorporation of these emerging contaminants into organisms. Traditional depuration methods do not consider the removal of these types of contaminants. However, in the current study, the ASEC technology depurated textile effluents (synthetic and natural fibers), resulting in the complete removal of MPs from the liquid phase and achieving dried solids that captured the transformed MPs compounds. Due to MPs being immobilized in dried solids, further investigations are proposed to determine potential hazards and the commercial value of the solids. ZLD technologies are recommended to be developed and commercially implemented in the textile industries to fulfill the sustainable goals of the United Nations and the European Green Deal objectives.

Author Contributions: E.B. and T.Á.D. were involved in all aspects of the manuscript such as conceptualization, methodology, software, validation, formal analysis, investigation, resources, data curation, writing, reviewing and editing, visualization, supervision, project administration and funding acquisition. S.V. was involved in conceptualization, methodology, software, validation, formal analysis, investigation, resources, writing, reviewing and editing, visualization, supervision, project administration and funding acquisition. J.B. and T.Á.D. were involved in validation, investigation, writing, reviewing and editing, visualization and supervision. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by ASEC Grant #190180588 through the EU EIC/Accelerator Horizon Europe 2022 call.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are unavailable due to privacy or ethical restrictions.

Acknowledgments: The authors thank the Water Challenge S.L. technical team for all the activities conducted during the study in the ASEC plant, and the technical and laboratory staff from the ICMAN (CSIC) and AIMPLAS.

Conflicts of Interest: Sebastian Vera was employed by the company Water Challenge S.L. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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