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Reuse of Pretreated Agro-Industrial Wastewaters for Hydroponic Production of Lettuce

Alexandra Afonso [1,2,*], Mariana Regato [3], Mariana Patanita [4], Silvana Luz [1,5], Maria João Carvalho [1], Annabel Fernandes [2], Ana Lopes [2], Adelaide Almeida [1,2], Idália Costa [3] and Fátima Carvalho [1,2]

[1] Department of Applied Sciences and Technologies, Escola Superior Agrária de Beja, Instituto Politécnico de Beja, 7800-309 Beja, Portugal
[2] Fiber Materials and Environmental Technologies (FibEnTech-UBI), Universidade da Beira Interior, R. Marquês de Ávila e Bolama, 6201-001 Covilhã, Portugal
[3] Department of Bioscience, Escola Superior Agrária de Beja, Instituto Politécnico de Beja, 7800-295 Beja, Portugal
[4] MED—Mediterranean Institute for Agriculture, Environment and Development & CHANGE—Global Change and Sustainability Institute, Institute for Advanced Studies and Research, Universidade de Évora, Pólo da Mitra, Ap. 94, 7006-554 Évora, Portugal
[5] Departamento de Ingeniería Química y Química Física, Instituto Universitario de Investigación del Agua, Cambio Climático y Sostenibilidad (IACYS), Universidad de Extremadura, 06006 Badajoz, Spain
* Correspondence: alexandra.afonso@ipbeja.pt

Abstract: The utilization of agro-industrial wastewaters (AIWWs), pretreated by immediate one-step lime precipitation + natural carbonation, as a nutritive solution for the hydroponic production of lettuce was evaluated. The AIWWs studied were olive mill wastewater (OMW), winery wastewater (WW), and cheese whey wastewater (CWW). Lettuces (*Lactuca sativa* L. var. crispa) were grown in a closed nutrient film technique hydroponic system, using the pretreated AIWWs (OMW-T, WW-T, and CWW-T) and a control nutrient solution (CNS). The growth and sensory analysis of lettuces and the environmental parameters of effluents after hydroponics were evaluated. The average number of lettuce leaves obtained with nutrient solutions prepared with AIWW-T was lower than that from CNS, but the highest lettuce chlorophyll content was attained with CWW-T, which also presented the best grow results. In general, sensory analysis did not show significant differences from the lettuces grown in the different pretreated AIWWs and CNS. As for the environmental parameters of the effluents from hydroponics, according to the Portuguese legislation, only the chemical oxygen demand of the OMW-T and WW-T presented slightly higher values than that of the environmental limit values for discharge in surface waters, showing the feasibility of using pretreated agro-industrial effluents in hydroponic lettuce cultivation, while obtaining a treated effluent, in a circular economy perspective.

Keywords: agro-industrial wastewaters; hydroponics; lettuce crop; sensory analysis; circular economy

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Citation: Afonso, A.; Regato, M.; Patanita, M.; Luz, S.; Carvalho, M.J.; Fernandes, A.; Lopes, A.; Almeida, A.; Costa, I.; Carvalho, F. Reuse of Pretreated Agro-Industrial Wastewaters for Hydroponic Production of Lettuce. *Water* **2023**, *15*, 1856. https://doi.org/10.3390/w15101856

Academic Editor: Ranka Junge

Received: 12 April 2023
Revised: 9 May 2023
Accepted: 11 May 2023
Published: 13 May 2023

1. Introduction

The agro-industry sector, which includes the production, processing, industrialization, and commercialization of agricultural, animal, forestry, and fishery products, is the third biggest water consumer in the world and, inevitably, generates large volumes of wastewaters [1]. Although agro-industrial wastewaters (AIWWs) are seen as readily degradable and non-toxic, and are, therefore, often discharged untreated into water courses, depending on their characteristics, AIWWs can cause severe adverse effects on the environment, requiring proper treatment before disposal [2].

AIWW composition and characteristics depend, among other things, on the type of feedstock, the desired final product, on the industrial process design, which may present horizontal or vertical diversification, and on the type of technology used (manual or mechanical processing), etc., [2]. The industrial processes and the products' seasonality are other important factors that contribute to the characteristics of the AIWWs, as in the case of the wastewaters from the olive mill (OMW), winery (WW), and cheese whey (CWW) industries.

When considering AIWW treatment options, two determining factors must be considered: the AIWW composition and characteristics and the intended quality of the treated effluent. Several different treatment technologies have been applied to AIWWs, such as physicochemical and biological processes, constructed wetlands, advanced oxidation processes, including electrochemical processes, and membrane technologies [1,3–5]. Among the different processes described in the literature for AIWW treatment, immediate one-step lime precipitation (IOSLP) is presented as a simple and economic process that can be used to obtain, in a fast and efficient way, a clarified and disinfected effluent, rich in nutrients and with very low content in heavy metals, apparently capable of being reutilized for agricultural purposes [6]. Furthermore, IOSLP has been proven to be effectively reduce phenolic content when dealing with AIWWs with high phenolic content, such as OMW [7]. According to these authors, this process, consisting of adding lime to the wastewater, also contributes to the reduction in greenhouse gases through posterior spontaneous carbonation reactions with atmospheric CO_2.

The reuse of treated AIWWs in agriculture irrigation has been addressed by different authors, since AIWW can be a valuable source of nitrogen, phosphorous, potassium, organic matter, and meso- and micro-nutrients for plant growth [8]. Cultivation in hydroponic systems fed by treated wastewaters, as a nutrient solution, has being pointed out as a promising strategy to reduce fresh-water consumption by reusing the treated wastewater, thus preventing soil and groundwater contamination [9].

Hydroponic systems allow the cultivation of a wide variety of cultivars without soil use and can be classified as "passive type" when the nutrient solution is fed to the plant by capillarity, or "active type", when the nutrient solution circulates within the system with the aid of a pump, also allowing its oxygenation [10]. The "passive type" system can be further designated as "opened" if the nutrient solution passes through the culture only once, or "closed" if it is recirculated until it runs out of nutrients. Cultivation in a "closed" hydroponic system leads to higher quality products and productivity, although crops can suffer from autotoxicity due to the accumulation of root exudates in the nutrient solution; the continuous monitoring of pH and electrical conductivity (EC) is necessary [11]. The most used "closed" hydroponic system is the nutrient film technique (NFT), through which the plants are fed by a nutrient solution that continuously flows as a thin film over their roots [12], decreasing the stagnant boundary layer surrounding each root and allowing an enhanced mass transfer of nutrients to the root surface [12].

The quality of the water to be used in a hydroponic system must fulfill some requirements, including [13]: (i) Adequate conductivity, since soluble salt in excess increases the osmotic pressure of the liquids and plant roots cannot absorb water; the relationship between hazard to the plants and EC (in mS cm^{-1}) is expressed as (None, <0.75), (Some, 0.75–1.5), (Moderate, 1.5–3), and (Severe, 3–7.5); (ii) The recommended suitable sodium adsorption ratio (SAR, Equation (1)), an indicator of the suitability of water for use in agricultural irrigation, is SAR < 10 $(\text{mmol L}^{-1})^{0.5}$, since higher values may promote severe damage to crops; (iii) Residual sodium carbonate (RSC, Equation (2)) not exceeding 1 meq L^{-1}; the ideal value for irrigation purposes is lower than 0.5 meq L^{-1}; (iv) Low concentration of elements that may cause an ionic imbalance to plants or even plant toxicity.

$$\text{SAR} = \frac{[\text{Na}^+]}{\sqrt{\frac{1}{2}\left\{[\text{Ca}^{2+}] + [\text{Mg}^{2+}]\right\}}} \text{ (with concentrations in meq L}^{-1}\text{)} \quad (1)$$

$$\text{RSC} = [\text{HCO}_3^{\,-}] + [\text{CO}_3^{\,2-}] - [\text{Ca}^{2+}] - [\text{Mg}^{2+}] \text{ (with concentration in meq L}^{-1}\text{)} \quad (2)$$

Lettuce cultivation in hydroponic systems fed by wastewaters has already been described in the literature. In the study conducted by Adler et al. [12], an NFT hydroponic system was evaluated as a method of removing phosphorus from an aquaculture effluent. The low content of organic matter and nutrients allowed the direct use of the aquaculture effluent in the hydroponic system, which requires effluent supplementation with some nutrients. Conversely, in the studies reported by Santos et al. [14] and Jung and Kim [15], where highly polluted vinasse and fishery processing wastewater were used, respectively, a pretreatment of the AIWW and its dilution were required to obtain an adequate nutrient solution that led to a good plant growth and high crop yield. When using a model mixture of petroleum, textile, pharmaceutical, domestic, and agricultural runoff wastewater, Egbuikwem et al. [16] experienced even more complex difficulties. Even after treating the mixed wastewater, which significantly reduced its pollutant load, the lettuce development and growth was poor, which was attributed to the presence of residual recalcitrant compounds that inhibited the development of the plant roots at the early stage and, consequently, hindered the plants' access to nutrients.

Following a previous study [6], where winery wastewater was treated by IOSLP + carbonation and an effluent supposedly capable of being used in agriculture was obtained, the objective of this work was to verify this potential use of WW, CWW, and OMW pretreated by IOSLP + carbonation in hydroponics lettuce growth. The system's performance was evaluated in terms of: (1) Efficiency of the hydroponic system in the removal of organic load and nutrients; (2) Possibility of discharging the final effluent in natural water resources; (3) Production and quality of the grown lettuces.

2. Materials and Methods

2.1. Agro-Industrial Wastewater Sampling and Pretreatment

OMW was collected from the storage lagoon located next to the oil mill in the town of Ferreira do Alentejo, Portugal. WW was gathered from a storage tank at the wine factory in the village of Vidigueira, Portugal. CWW, resulting from the production of sheep and goat cheese, was collected at the wastewater treatment plant entrance from the cheese factory located in the village of Serpa, Portugal. All the AIWW samples were collected in September 2021, and stored in PVC deposits with a capacity of 1000 L.

The raw AIWW samples were pretreated by IOSLP, followed by a carbonation (CB) step through atmospheric CO_2. Winery wastewater pretreatment followed the procedure described by Luz et al. [6]. OMW and CWW were pretreated by IOSLP up to a pH of 12.5, using a 200 g L^{-1} hydrated lime solution, prepared with groundwater and commercial hydrated lime ($Ca(OH)_2$, $\geq$95%, Spectrum® chemical, Brunswick, NJ, USA). To form a precipitate that allowed the removal of organic matter and other contaminants, 38 and 25 mL of the hydrated lime solution per liter of OMW or CWW were utilized, respectively [6,17]. The obtained suspensions were then left to rest for 48 h, and the supernatant was separated from the sludge, using a pump to remove the supernatant, which was stored in an open 1000 L tank, allowing carbonation by atmospheric CO_2 for natural pH neutralization. The initial CB process took around 90 days, and it was extended throughout the hydroponic reuse of the pretreated AIWWs.

2.2. Nutritive Solutions Characterization

2.2.1. Analytical Methods

pH, EC, redox potential (ORP), temperature, dissolved oxygen (DO), total dissolved solids (TDS), and salinity were measured using a Multiparameter Water Quality Meter—HI98194.

Nitrate, nitrite, chloride, fluoride, and sulfate ion concentrations were determined by ionic chromatography, following the procedure described by Ramalho et al. [18].

Chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), ammonia nitrogen (AN), total alkalinity and alkalinity to phenolphthalein, total phosphorus (P_{total}), total calcium and total magnesium hardness, and turbidity were determined according to the standard methods [19].

COD was determined by the spectrophotometric method, utilizing a WPA HC 6016 digester, purchased from ILC (Lisbon, Portugal), and a UV-visible spectrophotometer, purchased from Izasa Scientific (Carnaxide, Portugal). BOD_5 was determined by the respirometric method, using a WTW's Oxitop® gauge system, purchased from VWR International (Amadora, Portugal). Ammonia nitrogen was determined by distillation followed by titration, using a Tashiro indicator, boric acid as the absorbent in distillation, and sulfuric acid as the titrant. Total alkalinity and alkalinity to phenolphthalein were determined by the volumetric method. For the total phosphorus determination, samples were digested in acid medium, and the color development by vanadomolibdophosphoric acid colorimetry was monitored on a UV-visible spectrophotometer, at a wavelength of 470 nm, purchased from Izasa Scientific (Carnaxide, Portugal). Total hardness and calcium and magnesium hardness determinations followed the EDTA titrimetric method, as described by Ramalho et al. [18]. Turbidity was determined through a WTW Turb550 turbidimeter, purchased from VWR International (Amadora, Portugal), using a formazine suspension as a calibration standard. Potassium and sodium ions were analyzed by flame photometry, using a Model 410 Corning Flame Photometer, purchased from Dias de Sousa S.A. (Alcochete, Portugal).

2.2.2. Samples Characterization

Considering the high EC values presented by the AIWW samples pretreated by IOSLP + CB (OMW 7.51; WW 6.35; CWW 8.94 mS cm^{-1}), their dilution with groundwater became necessary at a ratio of 1:2 (AIWW:groundwater), so that they could be used as nutrient solutions in the hydroponic system. The characterization of the diluted pretreated AIWW samples, utilized as nutrient solutions in the hydroponic system, the groundwater, and the nutrient solution (CNS), is presented in Table 1. The three different hydroponic solutions, composed of the pretreated AIWWs diluted with groundwater, were named OMW-T, WW-T, and CWW-T. The parameter values presented for the OMW-T, WW-T, and CWW-T correspond to the mean values of the different characterizations performed along the experiments, since, as the CB process continued throughout the hydroponic study duration, some of the initial values varied over time. The CNS, formulated according to Almeida et al. [20], was prepared with groundwater and the following commercial fertilizers: 510 mg L^{-1} $Ca(NO_3)_2$, 480 mg L^{-1} KNO_3, 300 mg L^{-1} KH_2PO_4, 80 mg L^{-1} $MgSO_4$, and 1.5 mL L^{-1} of NPK 3-8-11 fertilizer solution (Tecnifol). The Larson–Skold index (LSI) was determined according to Equation (3).

$$LSI = \frac{[Cl^-] + [SO_4^{2-}]}{[HCO_3^-] + [CO_3^{2-}]} \text{ (with concentrations in meq L}^{-1}\text{)} \quad (3)$$

Table 1. Physicochemical characterization of the groundwater, the nutrient solutions containing the pretreated agro-industrial wastewaters, and the control nutrient solution.

Parameter	Groundwater	OMW-T	WW-T	CWW-T	CNS
pH	7.80 ± 0.05	9.2 ± 0.3	8.1 ± 0.4	8.0 ± 0.3	7.3 ± 0.4
EC, mS cm^{-1}	0.5 ± 0.2	2.1 ± 0.1	1.2 ± 0.9	1.8 ± 0.1	2.0 ± 0.2
ORP, V	0.19 ± 0.01	0.19 ± 0.01	0.23 ± 0.01	0.23 ± 0.02	0.26 ± 0.02
TDS, g L^{-1}	0.2627 ± 0.0003	0.10 ± 0.06	0.60 ± 0.06	0.88 ± 0.06	1.0 ± 0.1
BOD_5, mg L^{-1}	11 ± 1	19 ± 12	33 ± 9	77 ± 38	ND
COD, g L^{-1}	<0.005	0.6 ± 0.1	0.8 ± 0.2	0.51 ± 0.04	0.030 ± 0.003
DO, mg L^{-1}	ND	5 ± 2	6 ± 2	3 ± 1	5 ± 2
Turbidity, NTU	ND	76 ± 7	52 ± 6	7 ± 2	17 ± 3
P_{total}, mg L^{-1}	4 ± 1	0.6 ± 0.1	1.0 ± 0.4	0.9 ± 0.6	259.1 ± 0.1

Table 1. *Cont.*

Parameter	Groundwater	OMW-T	WW-T	CWW-T	CNS
AN, mg L^{-1}	< 0.1	0.6 ± 0.1	0.31 ± 0.07	7 ± 2	17 ± 4
NO_3^-, mg L^{-1}	86.5 ± 0.3	47 ± 9	21 ± 8	24 ± 9	837 ± 9
NO_2^-, mg L^{-1}	ND	4.2 ± 0.4	2.8 ± 0.9	0.4 ± 0.3	0.200 ± 0.007
SO_4^{2-}, mg L^{-1}	34.4 ± 0.1	38 ± 9	50 ± 9	58 ± 9	48 ± 3
Cl^-, g L^{-1}	0.0230 ± 0.0001	0.19 ± 0.03	0.09 ± 0.03	0.38 ± 0.03	0.034 ± 0.004
Ca^{2+}, g L^{-1}	0.075 ± 0.005	0.03 ± 0.01	0.21 ± 0.05	0.19 ± 0.06	0.5 ± 0.1
Mg^{2+}, mg L^{-1}	32 ± 2	15 ± 7	24 ± 9	12 ± 5	82 ± 9
K^+, mg L^{-1}	ND	559 ± 9	29 ± 3	22 ± 1	$3.6 \times 10^2 \pm 6 \times 10$
Na^+, mg L^{-1}	ND	89 ± 5	47 ± 5	137 ± 6	14 ± 4
CO_3^{2-}, g L^{-1}	ND	0.27 ± 0.07	0	0	0
HCO_3^-, g L^{-1}	ND	0.6 ± 0.1	0.6 ± 0.1	0.50 ± 0.09	0
SAR, (meq $L^{-1})^{0.5}$	ND	19 ± 7	4.3 ± 0.4	14 ± 3	0.8 ± 0.4
RSC, meq L^{-1}	ND	16 ± 3	<0	<0	<0
LSI, meq L^{-1}	ND	0.32 ± 0.07	0.4 ± 0.1	1.5 ± 0.4	-

Notes: OMW-T—pretreated olive mill wastewater; WW-T—pretreated winery wastewater; CWW-T—pretreated cheese whey wastewater; CNS—control nutrient solution; ND—not determined.

Although the microbial characterization of the diluted pretreated samples was not performed, it is known from previous studies that lime precipitation leads to the complete elimination of coliforms, and, thus, the treated wastewaters pose a low risk of pathogen contamination [6,21].

2.3. Hydroponic System

Lettuces (*Lactuca sativa* L. var. crispa) with 6 to 7 leaves per plant were obtained from commercial points and planted in the hydroponic system, and their growth was carried out for 72 days in a closed NFT hydroponic system, containing 4 lines with 5 plants each, a line for each of the nutrient solutions studied: OMW-T, WW-T, CWW-T, and CNS. Bench-scale hydroponics was performed using cylindrical white PVC tubes with 1 m length, 90 mm diameter, and with a 0.3% slope. The pots, containing perlite as support for the plant, were placed in holes with a diameter of 5.5 cm and 16 cm apart from each other. The height reached by the blade ranged between 4 and 6 cm. The hydroponic system worked on an intermittent recirculation system of 5 L of nutrient solutions, regulated by an electric pump, at a flow rate of 3 L min^{-1}. The nutrient solutions were replaced at the 15th, 31st, and 49th days.

During the hydroponic experiments, the different nutrient solutions were monitored daily for pH, EC, temperature, DO, and TDS. Lettuce growth was monitored weekly (diameter of lettuce and number of leaves). Whenever the nutrient solutions were replaced, the physicochemical characteristics of the withdrawn solutions and the solutions that replaced them were analyzed (according to Section 2.2.2).

2.4. Plant Analysis

Throughout the study, the diameter of the plant was measured and the number of lettuce leaves was counted. The harvest of the plants was carried out after 72 days in the hydroponic system. The vegetative parts (aerial part) and the roots of the lettuces were weighed separately. The chlorophyll content was determined using a SPAD 502 chlorophyl meter. Three representative leaves from each lettuce grown in each line were reserved, and the leaf area was determined using LI 3100C AREA METER, which allows measurement by a direct method. Then, the same representative leaves or roots were used to determine the dry weight of the lettuce by placing it at a temperature of 65−70 °C for 48 h [22]; the water content of the leaves was determined by the difference between the initial mass and the mass after drying. The remaining leaves were used for sensory analysis.

A hedonic sensory analysis was carried out with 30 volunteer tasters and panelists, aged between 19 and 60, in a sensory laboratory equipped with individual booths [23]. A lettuce whole leaf of one of the lettuces grown in one of the lines with the different nutrient solutions, or approximately 20 g of randomly selected cut lettuce, at room temperature, was presented to the assessors in a closed odorless plastic container labeled with three-digit random numbers. The panelists were also provided with water to clean their palate between samples. Sensory profile attributes were established based on previous studies [24,25]. The intensity of the sensory attributes was scored on a mixed structured hedonic scale of four points (4 = very present; 3 = present; 2 = not very present; 1 = absent) for each of the samples. The descriptors were: (a) Visual appearance (color intensity, uniformity of color, and withering of leaves), (b) Smell (fresh vegetable or unpleasant); (c) Flavor (sweet, bitter, or unpleasant); and (d) Texture (crunchy, tender, or succulent). Evaluations were performed under artificial daylight illumination, temperature control (between 22 and 24 °C) and using an air circulation ventilation system. In the sensory evaluation form, in addition to the evaluation, the tasters were asked to indicate gender, age, lettuce consumption behavior, and purchase intention.

The results of the examined parameters were subjected to a variance analysis (ANOVA), and their means were compared using the Tukey HDS test at a significance level of 0.05, using the STATISTICA 12.0 software program. In the variance analysis (ANOVA), it was shown that there was a statistically significant difference between the treatments whenever the value of F was higher than the critical value of F. Every time F was lower than the critical F, there was no statistically significant difference.

3. Results and Discussion

3.1. Hydroponic System Monitoring

In Table 1, it can be observed that all the nutrient solutions prepared with pretreated AIWWs present higher BOD_5 and COD than that of CNS, since no organic compounds were added in this control solution. The content of phosphorous, ammonia nitrogen, and nitrate in the diluted pretreated AIWWs is much lower than that in the control solution, which can be a hindrance for the lettuces' growth since these species play an important role as nutrients. Furthermore, the nutrient solutions from the AIWWs present very high SAR, in particular OMW-T and CWW-T, which may damage crops. It should be noted that the high standard deviations presented by some of the parameters that characterize the nutrient solutions from AIWWs are due to variations in time suffered by the solutions during the hydroponic study since the natural CB process continued since the solutions were stored in an open tank.

The hydroponic assays, assembled and ran as described in Section 2.3, were monitored daily for the pH, EC, temperature, DO, and TDS of the nutrient solutions that were being recirculated to the system. Figure 1 presents the variation of pH, EC, and DO observed in the different nutrient solutions during the 72-day assays. The vertical dot lines mark the replacement of the nutrient solutions by fresh ones. Temperature and ORP variations are presented as Supplementary Material (Figure S1).

The pH values of OMW-T, WW-T, and CWW-T solutions (Figure 1a) varied slightly, from 8.8 to 9.6, 7.6 to 8.7, and 7.6 to 8.4, respectively; these oscillations were consistent with those verified for the CNS (6.85 to 8.28). High values of pH may change nutrient solubility, precipitating as calcium and phosphorus salts, for instance, and preventing their absorption by lettuce. Despite the pH variation and the values above the recommended (6.0–7.0), according to Sharma et al. [26], the plants' development was not inhibited, specifically in the lettuces produced by CWW-T and OMW-T. According to Zaman et al. [13], for crops grown in soil, the pH is not so important since the soil has a buffering effect. Although, in the present study, lettuces were not grown in soil, a similar buffering effect is observed for the different diluted pretreated AIWWs, but at different pH values. This buffering effect may be due to carbonates, which are related to the Ca^{2+}, K^{+}, CO_3^{2-}, and HCO_3^{-} content, which is similar for WW-T and CWW-T and different for OMW-T. Similar pH variations during lettuce culture have already been observed by Sapkota et al. [27] and Majid et al. [28]. Sudden changes in pH were observed after the substitution of the nutrient solutions for the diluted pretreated AIWWs and for the control solution. These sudden changes may be explained by the fact that some ions are more rapidly utilized by plants than others, as is the case with NH_4^+ and NO_3^- [29].

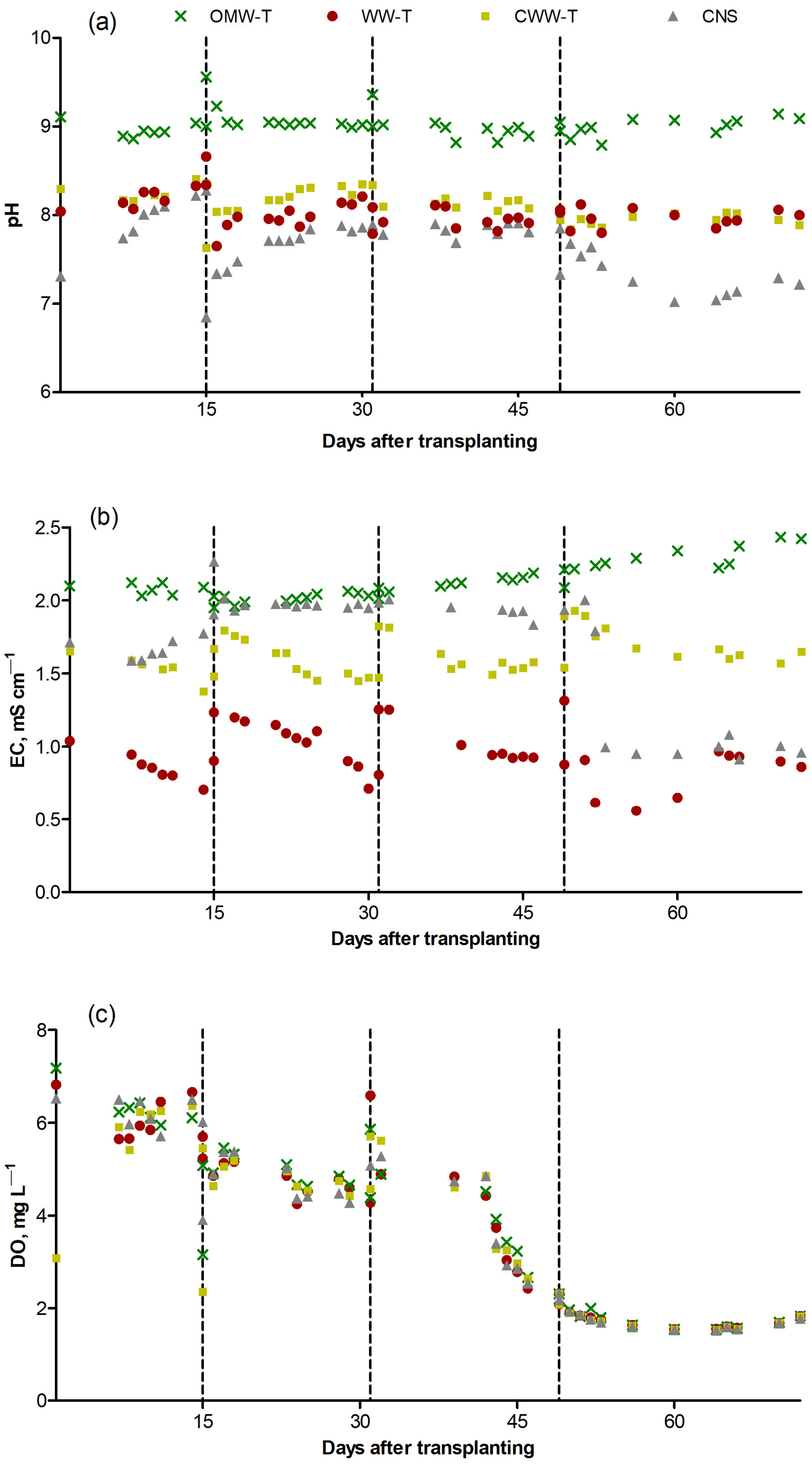

Figure 1. Variation of (**a**) pH, (**b**) electrical conductivity, and (**c**) dissolved oxygen in the different nutrient solutions, OMW-T, WW-T, CWW-T, and CNS, during the experimental period of the lettuce growth in the hydroponic system.

Regarding the electrical conductivity of the solutions fed to the hydroponic system (Table 1), and according to Zaman et al. [13], OMW-T, CWW-T, and CNS presented a moderate risk to crops, and only WW-T presented some risk. EC is related to the concentration of nutrients present in the solution, and it varies throughout the experiments due to the nutrient absorption by the lettuces [28]. The appropriate EC range for most hydroponic cultures is 1.5–2.5 mS cm^{-1}, although for lettuce it should be kept within 1.2 to 1.8 mS cm^{-1} [26]. In this study, the EC values of OMW-T, WW-T, and CWW-T (Figure 1b) presented variations between the average values of 1.96 and 2.43, 0.53 and 1.35, and 1.38 and 1.93 mS cm^{-1}, respectively, showing fluctuations that are consistent with that observed for CNS (0.91 and 2.27 mS cm^{-1}). For all diluted pretreated AIWWs except OMW-T, a decrease in the EC values along each phase was observed. This must be related to the quick absorption of the nutrients by the plants that had been in contact with a solution whose nutrients were almost depleted. The WW-T nutrient solution presented the lowest EC values, which is consistent with the fact that the lettuces produced were the smallest ones. This means that this type of nutrient solution has a low content of nutrients necessary for the development of the crop. In fact, it presented the lowest ammonium and nitrate contents. In addition to the low nutrient content, WW-T presented the highest COD and chloride concentration, as it is the most "corrosive" medium with the highest LSI.

DO varied between 1.5 and 7.1 mg L^{-1} (Figure 1c), presenting lower values in the final stage of development, which coincided with the increase in temperature and the consequent decrease in oxygen solubility (Figure S1a). However, this decrease did not cause the wilting of the lettuce nor did it weaken the root growth since the plant was in the final stage of growth.

Temperature is of utmost importance since it affects the other parameters and allows the evaluation of the absorption of nutrients and water by the plant, its growth, and chemical composition [30]. The oxygen consumed by the plants varies depending on the temperature of the nutrient solution, and an increase in temperature leads to lower DO values, causing plant wilting and poor root growth [31]. According to the literature, the ideal temperature range for lettuce growing is 15−25 °C since higher temperatures also favor water rather than nutrient absorption [29]. In this study, due to climatic conditions, the temperature varied between 13 and 23 °C, but most of the time the values remained within the ideal range.

3.2. Hydroponic System Performance for AIWW Polishing

Table 2 presents the COD, BOD_5, and biodegradability index (BI), given as the BOD_5/COD ratio, for the nutrient solutions prepared with pretreated AIWWs before and after being utilized in the hydroponic system at each of the four cycles. The hydroponic system allowed organic compounds to be removed during all the experiments, when compared to the composition of the nutrient solutions at the entrance of the system. In fact, the organic matter presented significant decrease, with COD removals ranging between 38% to 78% and BOD_5 removals of up to 92%.

For OMW-T, the highest COD removal was attained during Phase I of the experiment (1 to 15th day), i.e., in the first 15 days after transplanting the plants to the hydroponic system, when larger amounts of nutrients were required for their development, which coincides with the EC decrease in the solution. The biodegradability index also presented the highest increase during Phase I, smaller increases in Phases II and III, and a decrease in the final stage, when the temperature and DO were lower.

Regarding WW-T, the highest COD removal was observed during the final stage of the development; the lowest COD removal was attained in Phase I. Biodegradability increased during most of the phases, showing that non-biodegradable compounds are being oxidized.

For the CWW-T nutrient solution, an increase in COD and BOD_5 removals was observed from Phase I to Phase II, and then to Phase III, but, in the final stage, the removals decreased. This different behavior in the last phase is probably due to the low consumption of nutrients since this crop presented a faster growth.

Table 2. Variation between phases of COD, BOD_5, and biodegradability index (BI) of the different nutrient solutions used in the hydroponic system.

		OMW-T			WW-T			CWW-T		
		COD *	**BOD_5 ***	**BI**	**COD***	**BOD_5 ***	**BI**	**COD ***	**BOD_5 ***	**BI**
Phase I	Initial	0.67 ± 0.03	30 ± 0	0.04	0.56 ± 0.01	60 ± 0	0.11	0.47 ± 0.02	100 ± 0	0.21
	Final	0.21 ± 0.05	15 ± 7	0.07	0.33 ± 0.02	30 ± 0	0.09	0.14 ± 0.03	15 ± 7	0.11
	% removal	68	50		41	50		71	85	
Phase II	Initial	0.61 ± 0.06	30 ± 0	0.05	0.87 ± 0.01	30 ± 0	0.03	0.49 ± 0.01	115 ± 7	0.24
	Final	0.216 ± 0.005	10 ± 0	0.05	0.39 ± 0.01	29 ± 1	0.07	0.123 ± 0.005	15 ± 7	0.12
	% removal	64	67		55	3		75	85	
Phase III	Initial	0.50 ± 0.01	10 ± 0	0.02	0.95 ± 0.01	25 ± 7	0.03	0.553 ± 0.005	60 ± 9	0.10
	Final	0.204 ± 0.002	8 ± 4	0.04	0.47 ± 0.01	25 ± 0	0.05	0.119 ± 0.009	5 ± 0	0.04
	% removal	59	25		50	0		78	92	
Phase IV	Initial	0.43 ± 0.01	8 ± 4	0.02	0.96 ± 0.01	15 ± 0	0.02	0.536 ± 0.009	33 ± 4	0.06
	Final	0.266 ± 0.005	3 ± 2	0.01	0.41 ± 0.03	13 ± 4	0.03	0.173 ± 0.009	18 ± 0	0.10
	% removal	38	67		57	17		68	45	

Note: * COD in g L^{-1}; BOD_5 in mg L^{-1}.

In general, among the different nutrient solutions prepared with pretreated AIWWs, CWW-T presented the highest COD and BOD_5 removals, indicating a higher absorption of organic matter/nutrients by the lettuce roots. In fact, the lettuces produced with this nutrient solution showed a gradual evolution and better development than that presented by the control lettuce. Additionally, they presented the highest biodegradability index, ranging from 0.04 to 0.12, although, when compared to their initial value (0.21), a decrease was observed, probably because readily biodegradable organic matter had been utilized by the crops.

The nutrient removal in the hydroponic system, fed with the diluted pretreated AIWW, allowed the treatment of these effluents, while producing the lettuces. Table 3 presents the average values of the characterization parameters of the effluents from the hydroponic system collected after the four consecutive phases, for each AIWW. The removal values are also presented in Table 3 in percentages; the comparisons of the average final values with the initial values are presented in Table 1.

Table 3. Physicochemical characterization of the different nutrient solutions after utilization in the hydroponic system, presented as medium values of the effluents from the four cycles.

Parameter	OMW-T		WW-T		CWW-T		ELV *
	Final	**% Removal**	**Final**	**% Removal**	**Final**	**% Removal**	
COD, g L^{-1}	0.22 ± 0.03	59	0.40 ± 0.06	52	0.14 ± 0.02	73	0.150
BOD_5, mg L^{-1}	9 ± 5	55	24 ± 8	26	13 ± 5	83	40
Turbidity, NTU	2 ± 1	97	13 ± 5	74	5 ± 2	30	-
P_{total}, mg L^{-1}	0.28 ± 0.01	53	0.64 ± 0.08	34	0.60 ± 0.09	38	10
AN, mg L^{-1}	0.1 ± 0.3	78	0.2 ± 0.2	57	4 ± 3	49	10
K^+, mg L^{-1}	578 ± 9	−3	29 ± 2	0.4	15 ± 4	32	-
Na^+, mg L^{-1}	317 ± 6	−255	37 ± 6	23	141 ± 9	−4	-
NO_3^-, mg L^{-1}	28 ± 2	41	13 ± 2	38	13.6 ± 0.9	43	50
Total hardness, g $CaCO_3$ L^{-1}	0.10 ± 0.03	18	0.29 ± 0.04	53	0.24 ± 0.02	43	-
Hardness Ca, g $CaCO_3$ L^{-1}	0.04 ± 0.01	38	0.23 ± 0.04	56	0.17 ± 0.03	63	-

Notes: * ELV—environmental limit values for discharge in surface waters according to Portuguese legislation (Decree-Law No. 236/98, 1998 [32]).

In general, there was a decrease in turbidity in all the studied nutrient solutions, which is higher for the WW-T, which presented a middle initial value among the different nutrient solutions prepared with pretreated AIWWs. The ammonia nitrogen and nitrate levels in the different solutions decreased, as the different systems were more efficient in removing nitrogen in the form of ammonia than nitrate, which can be explained by the fact that ammonium ions are absorbed more quickly when compared to nitrate [29].

It was found that the highest P_{total} removals were in the WW-T solution. Potassium removal was only effective in the CWW-T. Sodium absorption was also very low in WW-T, and negative for the other AIWWs, which, in fact, presented unsuitable SAR, i.e., higher than 10 (Table 1). Regarding calcium removal, the highest value was observed in the CWW-T solution, followed by WW-T, and finally, by OMW-T. In general, with the exception of turbidity and ammonia nitrogen, CWW-T attained the highest removals for the different parameters. Table 3 also presents the environmental limit values (ELV) for effluent discharge in surface waters, according to the Portuguese legislation (Decree-Law No. 236/98, 1998) [32]. It can be observed that for the parameters under evaluation, only OMW-T and WW-T leave the hydroponic system with COD values slightly higher than the ELV; all the other parameters for all the nutrient solutions were prepared with pretreated AIWWs below the ELV.

3.3. Lettuce Agronomic Characteristics

Table 4 presents the growth parameters and chlorophyll of the lettuces produced in the hydroponic system fed with the different nutrient solutions evaluated. The results show that the lettuces produced with OMW-T, CWW-T, and WW-T present lower numbers of lettuce leaves per plant, lettuce diameter, and leaf area than those produced with CNS. A possible explanation for this fact is the deficiency of essential elements in the diluted pretreated AIWWs (Table 1) when compared to the CNS. Still, these results are better than those presented by Santos et al. [14], who utilized a vinasse solution, which obtained 8.5 leaves per plant. As for the average fresh shoot mass of the lettuces produced by OMW-T and CWW-T solutions, their values are similar, much higher than the lettuces produced by WW-T, and much lower than the lettuces produced by the control solution. However, the fresh roots of the lettuces produced in the WW-T experiment show the highest mass, those from the control solution being the smallest ones. The results obtained by the WW-T and CNS solutions show statistically significant differences between the fresh mass of the shoot and the fresh mass of the root. These differences are probably due to the WW-T solution having low content of P and N and high total hardness (mg $CaCO_3$ L^{-1}), which can hinder nutrient absorption by the lettuces, contributing to the poor development of the plant and, consequently, less fresh mass. Regarding the dry mass of the shoot, there are no significant differences between the lettuces produced by the different solutions; the obtained values are higher than those presented by Egbuikwem et al. [16], who evaluated the growth of lettuce in mixed wastewater, and attained, per plant, six to eight leaves, 0.03 to 0.20 g of fresh mass, and 0.01 to 0.03 g of dry mass. Other authors [27], who evaluated the effect of different nutrient concentrations of N, K, and Ca on lettuce production in hydroponic culture, obtained average values of 10 to 21 leaves and 41.9 to 115.3 g fresh weight per plant.

Table 4. Mean growth parameters and chlorophyll of the lettuces produced in the hydroponic system fed with the different nutrient solutions.

Parameter	OMW-T	WW-T	CWW-T	CNS
Leaf number	18 ± 3^{b}	16.0 ± 0.6^{b}	19 ± 2^{b}	25 ± 4^{a}
Lettuce diameter, cm	18 ± 1^{b}	14 ± 1^{c}	19 ± 2^{b}	30 ± 4^{a}
Fresh shoot mass weight, g	$12 \pm 2^{a,b}$	4.4 ± 0.9^{b}	$13 \pm 3^{a,b}$	25 ± 9^{a}
Dry shoot mass weight, g	0.42 ± 0.06^{a}	0.21 ± 0.04^{a}	0.45 ± 0.08^{a}	0.7 ± 0.4^{a}
Fresh root mass weight, g	$0.5 \pm 0.3^{b,c}$	$1.6 \pm 0.6^{a,c}$	1.2 ± 0.5^{a}	$0.5 \pm 0.4^{b,c}$
Dry root mass weight, g	$0.19 \pm 0.07^{b,c}$	$0.37 \pm 0.05^{a,c}$	0.31 ± 0.08^{a}	$0.2 \pm 0.1^{b,c}$
Leaf area, dm^2	$0.59 \pm 0.03^{a,b}$	0.31 ± 0.05^{b}	$0.58 \pm 0.09^{a,b}$	$1.2 \pm 0.7^{a,b}$
Water content, %	96.3 ± 0.8^{a}	94.7 ± 0.7^{b}	96.5 ± 0.5^{a}	97.5 ± 0.5^{a}
Shoot: Root mass weight	$2.4 \pm 0.7^{a,c}$	$0.6 \pm 0.2^{b,c}$	1.6 ± 0.6^{c}	4 ± 2^{a}
Chlorophyll, SPAD units	8 ± 1^{b}	15 ± 2^{a}	19 ± 3^{a}	15 ± 4^{a}

Note: ^{a,b,c} Different letters in different columns mean statistically significant differences ($p < 0.05$).

For the shoot:root ratio, there were only significant differences between the CNS solution and the CWW-T and WW-T, with lettuces grown in the CNS presenting the highest values. A high ratio means that the shoot has higher growth compared to the root of the lettuce; that is, the lettuces produced by the control solution present higher dimensions of the shoot when compared to the dimensions of the roots [33]. These results are similar to those obtained by Frasetya et al. [34], who evaluated five nutritional formulas with similar EC for the hydroponic culture of lettuce (*Lactuca sativa* Var. Arista), and obtained shoot:root ratio values between 0.57 and 1.43. Regarding leaf area, the lowest value was presented by lettuces grown in WW-T, and the highest value was from the CNS. However, these values are lower than those obtained by Frasetya et al. [34], where leaf area varied between 0.676 and 2.461 dm^2. The lettuces' water content was higher than 90% for all the nutritive solutions studied.

The highest average value for chlorophyll, expressed in SPAD units, was presented by lettuces produced with CWW-T, although only lettuces grown in OMW-T presented significant differences from the others, having the lowest chlorophyll content. This parameter is usually related to the nitrogen concentration in the nutrient solution, which is a main macronutrient necessary for the vegetative development of lettuce [35]. Although the chlorophyll value was lower in the lettuces produced by the OMW-T solution, it did not affect photosynthesis or the production of carbohydrates, since the plants produced through this solution did not differ from that of CNS solution in relation to the fresh mass of the shoot and dry mass of the roots and shoots. These results are in good agreement with those obtained by Liu et al. [36], who studied the growth of hydroponically cultivated lettuce with biogas slurry, supplemented with nutrients, and obtained chlorophyll values between 8.2 and 18.1 SPAD units.

From the parameters evaluated and the results obtained, it can be inferred that the lettuces produced by CWW-T presented the best characteristics among those grown in nutritional solutions prepared from AIWWs.

3.4. Lettuce Sensory Analysis

Post-harvest lettuce quality is largely governed by sensory factors, such as appearance, flavor, aroma, and texture. Its shelf-life reduction is usually first perceived as a decline of visual appearance, due to leaf tearing, yellowing, and browning, followed by a deterioration of the flavor and aroma profiles, caused by the development of fermentative metabolites of microbial contaminants and by a loss of leaf firmness, succulence, and juiciness [37]. The results of the sensory analysis of the lettuces leaves produced in the hydroponic system fed with the different nutrient solutions are presented in Table 5.

In the visual appearance descriptor, CWW-T led to the highest score in color intensity, although only lettuce leaves grown in the OMW-T presented significant differences compared to the other samples. The freshness of vegetables may significantly affect the evaluation on all the sensory attributes, including smell, taste, and appearance. The withered leaf descriptor had the highest score in the control sample, indicating that the lettuces from the AIWW nutrient solutions appeared fresher than the control, although there were no significant differences between them. Considering the overall results attained in the visual appearance descriptor, it can be said that the best scored lettuces, in terms of visual appearance, were those grown in the CWW-T nutrient solution, having an even higher score than the control lettuces.

Concerning flavor-related attributes, the sweetness of lettuce, largely determined by its glucose content, was found to be the main factor influencing consumer liking [38]. The study conducted by Chadwick et al. [38] supports the hypothesis that consumers can detect the sweet and the bitter compounds in lettuce, as well as the hypothesis that most consumers prefer sweeter and less bitter genotypes. The study suggests that the bitter and sweet components act to counterbalance each other, and the perceived sweetness scores are correlated with total sugar content, the primary factor being the glucose content. In the present study, lettuces from CNS and CWW-T attained the highest score in sweetness and the lowest in bitterness and unpleasant flavor. As for the sensory parameter of smell, no statistically significant differences were observed for the lettuces produced with the different nutrient solutions.

Table 5. Sensory characteristics of the lettuces produced in the hydroponic system fed with the different nutrient solutions: 4 = very present; 3 = present; 2 = not very present; 1 = absent.

Parameter		OMW-T	WW-T	CWW-T	CNS
Visual appearance	Color intensity	2.4 ± 0.9 [c]	2.9 ± 0.8 [b]	3.6 ± 0.6 [a]	3.4 ± 0.8 [a,b]
	Color Uniformity	2.9 ± 0.9 [a]	2.9 ± 0.7 [a]	3.3 ± 0.7 [a]	3.2 ± 0.7 [a]
	Withered leaves	1.6 ± 0.9 [b]	1.7 ± 0.9 [b]	1.7 ± 0.9 [a,b]	2.2 ± 0.9 [a]
Flavor	Sweet	2.0 ± 0.9 [a]	1.9 ± 0.9 [a]	2.1 ± 0.9 [a]	2.4 ± 0.9 [a]
	Bitter	2.3 ± 0.9 [a]	2.4 ± 0.9 [a]	2.3 ± 0.9 [a]	2.0 ± 0.9 [a]
	Unpleasant	1.5 ± 0.9 [a]	1.6 ± 0.9 [a]	1.4 ± 0.8 [a]	1.3 ± 0.6 [a]
Smell	Fresh vegetable	2.6 ± 0.9 [a]	2.9 ± 0.9 [a]	2.9 ± 0.8 [a]	2.9 ± 0.8 [a]
	Unpleasant	1.4 ± 0.9 [a]	1.3 ± 0.8 [a]	1.3 ± 0.8 [a]	1.2 ± 0.6 [a]
Texture	Crunchy	2.6 ± 0.9 [a]	2.6 ± 0.9 [a]	2.9 ± 0.9 [a]	2.3 ± 0.9 [a]
	Tender	2.7 ± 0.9 [a]	2.8 ± 0.8 [a]	2.7 ± 0.8 [a]	3.3 ± 0.8 [a]
	Succulent	2.7 ± 0.9 [a]	2.5 ± 0.9 [a]	2.8 ± 0.9 [a]	2.9 ± 0.9 [a]

Note: [a,b,c] Different letters in different columns means statistically significant differences ($p < 0.05$).

Leaf texture is another important sensory attribute for determining post-harvest quality and consumer acceptance. According to some authors, crispness is one of the most popular sensory parameters in lettuce [39]. In the present work, lettuce from CNS had the highest score in juiciness and tenderness and the lowest in crunchiness, but there were no significant differences among all samples.

From the lettuces grown in nutrient solutions prepared with pretreated AIWWs, and considering the overall results of the sensory analysis, lettuces from CWW-T attained the most favorable sensory evaluation. Nonetheless, there were no significant differences between the different samples and between them and the CNS lettuce, which validates the use of the diluted pretreated AIWW in the hydroponic culture of lettuce.

4. Conclusions

The use of IOSLP + CB pretreated agro-industrial wastewaters, namely WW-T, OMW-T, and CWW-T, for lettuce hydroponic culture is a feasible option to promote the circular economy of these types of wastewaters, leading to a marketable product while polishing the AIWWs, allowing their safe discharge in water courses. The hydroponic culture of lettuce, through an NFT system, using diluted solutions (1:2 dilution ratio) of AIWWs pretreated with IOSLP + CB, resulted in the reduction in the organic load, ammonia, nitrate nitrogen, turbidity, and hardness of the used AIWW solutions. The nutrient solution prepared from CWW presented the largest reductions in pollutant load, considering that all the parameters were evaluated in compliance with the Portuguese legal discharge limits in water courses. Regarding the quality of the lettuces produced, according to the physic-chemical parameters evaluated, no significant differences were found between the lettuces grown in the different AIWW solutions, although those from the CWW-T solution presented the best scores. Still, lettuces produced with AIWW solutions were smaller than the control ones, indicating that the supplementation of the AIWW solutions with some default nutrients might be required. On the other hand, the sensory analysis revealed no significant differences between the lettuces produced with AIWW solutions and those from control, although, once more, lettuces from the CWW-T solution attained the best results.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w15101856/s1, Figure S1: Variation of (a) temperature, (b) ORP, (c) salinity, and (d) TDS in the different nutrient solutions, OMW-T, WW-T, CWW-T, and CNS, during the experimental period of the lettuce growth in the hydroponic system.

Author Contributions: Conceptualization, F.C.; methodology, M.J.C., M.R. and A.A. (Adelaide Almeida); validation, A.A. (Alexandra Afonso), M.P. and M.J.C.; formal analysis, A.F. and A.L.; investigation, A.A. (Alexandra Afonso), S.L., I.C. and M.P.; data curation, M.J.C., M.R., F.C. and A.L.; writing—original draft preparation, A.A. (Alexandra Afonso), M.J.C. and A.F.; writing—review and editing, M.R., A.L. and F.C.; visualization, A.L. and F.C.; supervision, A.L., A.A. (Adelaide Almeida) and F.C.; project administration, A.A. (Adelaide Almeida) and F.C.; funding acquisition, F.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by NETA project, New Strategies in Wastewater Treatment (POCI-01-0247-FEDER-046959), through PORTUGAL2020, by Carbo2Soil project (PRR-C05-i03-I-000030), through Plano de Recuperação e Resiliência (PRR), and by Fundação para a Ciência e a Tecnologia, FCT, project UIDB/00195/2020, Ph.D. grant 2020.04822.BD awarded to A. Afonso, and contract awarded to A. Fernandes.

Data Availability Statement: Data sharing is not applicable to this article.

Acknowledgments: The authors are very grateful for the support from research unit Fiber Materials and Environmental Technologies (FibEnTech-UBI), project reference UIDB/00195/2020, funded by the Fundação para a Ciência e a Tecnologia, IP/MCTES through national funds (PIDDAC).

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

Abbreviations

AIWW: agro-industrial wastewaters; OMW: olive mill wastewater; WW: winery wastewater; CWW: cheese whey wastewater; OMW-T: pretreated olive mill wastewater; WW-T: pretreated winery wastewater; CWW-T: pretreated cheese whey wastewater; CNS: control nutrient solution; IOSLP: immediate one-step lime precipitation; NFT: nutrient film technique; SAR: sodium adsorption ratio; RSC: residual sodium carbonate; LSI: Larson–Skold index; ELV: environmental limit value; NTU: nephelometric turbidity unit; ND: not determined; PVC: polyvinyl chloride; CB: carbonation; EC: electrical conductivity; ORP: redox potential; DO: dissolved oxygen; TDS: total dissolved solids; COD: chemical oxygen demand; BOD_5: biochemical oxygen demand; AN: ammonia nitrogen; P_{total}: total phosphorus; EDTA: ethylenediamine tetraacetic acid; NPK: nitrogen, phosphorus and potassium ratio; BI: biodegradability index.

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