

Agricultural reuse of cheese whey wastewater treated by NaOH precipitation for tomato production under several saline conditions and sludge management

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Abstract

NaOH precipitation applied to cheese whey wastewater (CWW) has been investigated in the pH range of 8.5-12.5. Optimum conditions were found at pH 11.0. High reductions of chemical oxygen demand–COD (40%), turbidity–T (91%), total suspended solids–TSS (69%), sulphates (93%), phosphorus (53%), total hardness (40%), calcium (50%), magnesium (27%), chlorides (12%), Kjeldahl nitrogen (23%), etc. were achieved. Treated CWW by the aforementioned process has been used for agricultural irrigation of two tomato cultivars (Roma and Rio Grande) after dilution with fresh water, which was used as control experiment (1.44 dS m^{-1}). Five different irrigation treatments, with salinity level in the range of $1.75\text{--}10.02 \text{ dS m}^{-1}$, were implemented with treated wastewater. Treatment did not show a significant effect on the total and marketable yield, production losses and tomato yield with physiological disorder of blossom-end rot. Nevertheless, the cultivar Rio Grande presented an increase up to 21% in the marketable yield, for salinity levels of $1.75\text{--}3.22 \text{ dS m}^{-1}$, owing to an increment of the fruit fresh weight. Furthermore, treatment significantly influenced the tomato yield with epidermis deformations by solar exposure, unit fruit fresh weight and tomato number per kilogram. Fruit epidermis deformations due to solar exposition were minimized in about 27–93% when using treated wastewater. Raw sludge generated in the NaOH precipitation presented an average content of organic matter, phosphorus and nitrogen of (g kg^{-1} on a dry basis) 512, 5.8 and 11.2, respectively. Additionally, this sludge was treated by means of several processes. Centrifugation was quite efficient in the sludge volume reduction. The integrated sequence: aerobic digestion + sedimentation + centrifugation constituted a suitable treatment line, achieving a sludge volume reduction of 80% and simultaneously producing an effluent with organic matter depletion around 40%. Infiltrated water from sand filters was more contaminated organic and inorganically than the effluent obtained in the sequence: aerobic digestion + sedimentation + centrifugation.

1. Introduction

The majority of the Mediterranean countries, including Portugal, Spain, Italy and Greece, has severe and frequent problems in the water availability for the diverse consuming sectors, with common and extensive periods of drought (Aiello et al., 2007), low rainfalls and hot summers (Prazeres et al., 2014). Agriculture is the largest water-consuming sector in the world, requiring about 70% of the total water consumption, compared with 20% and 10% for the industrial and domestic sectors, respectively (<http://www.worldometers.info/water/> (23.07.15)). Concerning Portugal, the water consumption is estimated at 7500 millions of $\text{m}^3 \text{ year}^{-1}$, where 87% is sought for the agriculture sector (<http://portaldaagua.inag.pt/PT/InfoUtilizador/UsoEficiente/Pages/ConsumoPortugal.aspx> (26.01.11)). As a consequence, the search for new water sources to agriculture has been increasing in several regions like North and South Africa, Southern Europe, Mexico and South America (Boyden and Rababah, 1996). The industrial wastewater reuse is one of the most important challenges in agricultural irrigation (Angelakis et al., 1999). Meanwhile, the reuse of reclaimed wastewater by farmers presents several advantages, such as the use of low-cost water resources, nutrient supplementation, minimizing of the application rate of commercial

fertilizers, improvement of the soil and crop productivity (Angelakis et al., 1999; Jiménez-Cisneros, 1995; Paranychianakis et al., 2006). Additionally, a decrease of the wastewater treatment costs can be achieved when using reclaimed wastewater in agricultural irrigation.

The growing attractiveness of fruits and vegetables is mainly due to the nutritional and health benefits linked to their consumption (Klaiber et al., 2005). At present, the demand and preference of consumers for fruits and vegetables are becoming more diverse, for example, consumers are looking for sweet tomatoes on the market (Sato et al., 2006). In this sense, the improvement of this property can be achieved when salinity conditions are applied in the tomato production. Numerous studies have often described increments in the tomato sweetness, flavor, umami, epidermis firmness, acidity, titratable acidity, soluble solids, chloride, ascorbic acid, glucose, fructose, sucrose, organic acids, free amino acids and lycopene (Petersen et al., 1998; Sato et al., 2006; Wu and Kubota, 2008; Zushi and Matsuzoe, 1998, 2009) when salinity conditions have been used. As well, in previous works, we have studied the production of two tomato cultivars (Roma and Rio Grande), obtaining increases in the total soluble solids content (Brix grade) and epidermis firmness of fruits when using pretreated CWW as a source of water and nutrients at different salinity levels (Prazeres et al., 2013a,b, 2014).

Raw CWW is characterized as a relatively high contamination source of chemical and biochemical oxygen demand (COD and BOD), turbidity, oils and fats, suspended solids, phosphorus, nitrogen and anoxic conditions, etc. Additionally, wastewater coming from the cheese manufacture, usually, presents high salinity, monitored by electrical conductivity values in the range 11-14 dS m⁻¹. The salinity level results of the salt addition during the manufacturing process, so the concentrations of chloride and sodium are fairly elevated (2.1-3.8 g L⁻¹ and 0.9-1.7 g L⁻¹, respectively).

The direct use of cheese effluents on the soil is a common and long-lasting practice (Jones et al., 1993; Lehrsch et al., 2008; Prazeres et al., 2012; Robbins and Lehrsch, 1998), but a previous treatment is typically required so as to prevent several emerging public health risk and environmental damages. Nowadays, the treatment of CWW is carried out by the application of physicochemical and biological processes, the latter being extensively used, under anaerobic (Gavala et al., 1999; Gutiérrez et al., 1991; Kalyuzhnyi et al., 1997) and aerobic (Fang, 1991; Martins and Quinta-Ferreira, 2010; Rivas et al., 2010, 2011) conditions. Bio-process efficiencies can attain values above 80%; nonetheless, high hydraulic retention times are normally mandatory. What is more, biotreated wastewater does not ordinarily comply with the European Environmental Legislation, exceeding the COD and BOD limits allowed for the direct discharge. The levels of phosphorus and nitrogen can also lead to the eutrophication phenomena.

Physicochemical treatments partially eliminate the organic matter and nitrogen compounds (Rivas et al., 2010, 2011). Moreover, these processes are also very efficient in the removal of total coliforms, TSS, fats and phosphorus. Although treated wastewater exceeds the limits imposed by legislation, this effluent is a rich source of biodegradable organic matter and nutrients, for example, nitrogen, potassium, phosphorus, calcium, magnesium, chloride, etc., which can be recycled as growth factors in agriculture. In this context, physicochemical processes as coagulation-flocculation and basic precipitation are sustainable options to reuse the obtained effluent or apply a biological post-treatment. However, these physicochemical technologies are influenced by some variables as the reagent dose, temperature, operation pH, etc. Accordingly, the present work aimed at studying the NaOH precipitation applied to CWW under different operating conditions (pH 8.5-12.5), characterizing the supernatant and sludge obtained. The supernatant obtained from the NaOH precipitation, under optimal conditions, was reused for the production of two tomato cultivars, determining the effect of the treated CWW on the total and marketable yield, production losses, tomato yield with physiological disorders (blossom-end rot and deformations by solar exposure), unit fruit fresh weight and number of tomatoes per kilogram. Additionally, wet sludge generated in the NaOH precipitation was treated by centrifugation, compaction, filtration through sand beds, aerobic digestion + sedimentation, and aerobic digestion + sedimentation + centrifugation.

2. Materials and methods

2.1. Raw cheese whey wastewater

CWW was taken from a cheese factory located in the "Serpa Cheese" region of Alentejo (Portugal). Table 1 shows the physicochemical characterization of this raw effluent. As inferred from COD and BOD₅ maximum values (18.5 and 12.9 g L⁻¹, respectively), CWW presents a high organic matter content. The elevated biodegradability (BOD₅/COD^{0.7}) indicates that the effluent can be treated by biological processes. However, when this effluent was treated by aerobic digestion, a hydraulic retention time of approximately 8 days was required to reduce the COD content from 9525 mg L⁻¹ to 258 mg L⁻¹ (Rivas et al., 2010). Thus, slow biodegradation and bulking process formation, especially for high microorganisms concentration (6.5 g L⁻¹), constitute the principal limiting factors of the biological processes.

The low pH and high electrical conductivity values of raw CWW are the result of the manufactured whey

type, anaerobic conditions of the storage lagoons and NaCl incorporation in the cheese production. The effluent presents a greenish-whitish color with high values of sodium (0.9-1.7 g L⁻¹), chloride (2.1-3.8 g L⁻¹), total solids (5.7-15.1 g L⁻¹) and turbidity (405-1386 NTU). CWW also presents a disagreeable odor of butyric acid that causes discomfort and attracts insects. The hardness of CWW is mainly due to calcium, which can be found with a concentration about 2 times higher than the magnesium concentration.

2.2. Analytical procedures for wastewater and sludge characterization

pH and redox potential measurements were performed in a WTW InoLab apparatus (sludge/water ratio = 1:2.5). Electrical conductivity (sludge/water ratio = 1:2.5) and turbidity were evaluated in a Jenway 4510 meter and WTW Turb550 turbidimeter, respectively. COD, solids, ammonium and Kjeldahl nitrogen were quantified by spectrophotometric, gravimetric and Kjeldahl standard methods (APHA, 1998). BOD and chloride were determined by respirometric and Mohr methods, respectively. Phosphorus was assessed by absorbance measurement after calcination (600 °C), dry digestion and reaction of orthophosphates with vanadate-molybdate solution (APHA, 1998). Oil and fats were measured gravimetrically after Soxhlet extraction (Sawyer et al., 1994). Sodium and potassium were evaluated in a CORNING 410 flame photometer. Sulphates determination was made by ionic chromatography using a Metrohm 761 Compact Ion Chromatography Analyzer equipped with a Metrosep A Supp 5–150/4.0 column. For

Table 1
Physicochemical characterization of raw CWW.

Parameters	Average	Interval
Chemical oxygen demand—COD (mg L ⁻¹)	12563 ± 5147	9264–18493
Biochemical oxygen demand—BOD ₅ (mg L ⁻¹)	9150 ± 3269	6900–12900
BOD ₂₀ (mg L ⁻¹)	12200 ± 3978	8500–17000
BOD ₅ /COD	0.74 ± 0.04	0.70–0.77
Turbidity—T (NTU)	782.3 ± 528.0	404.8–1385.6
pH	4.17 ± 0.78	3.28–4.78
Temperature (°C)	24.0 ± 2.5	21.9–26.8
Electrical conductivity (dS m ⁻¹)	9.30 ± 1.95	7.13–10.91
Redox potential (mV)	195.6 ± 68.9	121.6–257.5
Total solids—TS (mg L ⁻¹)	9021 ± 5313	5678–15148
Total volatile solids—TVS (mg L ⁻¹)	4234 ± 4317	1418–9204
Total suspended solids—TSS (mg L ⁻¹)	1573 ± 228	1400–1832
Volatile suspended solids—VSS (mg L ⁻¹)	1184 ± 260	884–1352
Sulphates (mg L ⁻¹)	78.1 ± 8.4	72.1–84.0
Na (mg L ⁻¹)	1345.1 ± 417.9	898.5–1726.6
K (mg L ⁻¹)	228.2 ± 139.9	113.3–384.1
P (mg L ⁻¹)	103.0 ± 14.3	90.4–118.5
Cl (mg L ⁻¹)	2706.4 ± 929.3	2065.7–3772.2
N-Kjeldahl (mg L ⁻¹ N)	314.7 ± 64.1	255.1–382.5
Calcium hardness—CH (mg L ⁻¹ CaCO ₃)	293.0 ± 15.4	277.6–308.5
Ca (mg L ⁻¹)	117.2 ± 6.2	111.0–123.4
Magnesium hardness—MH (mg L ⁻¹ CaCO ₃)	201.8 ± 105.9	115.7–320.0
Mg (mg L ⁻¹)	49.2 ± 25.8	28.2–78.1
Total hardness—TH (mg L ⁻¹ CaCO ₃)	494.8 ± 91.1	424.1–597.6
Ca/Mg	2.9 ± 1.5	1.4–4.4
CH/MH	1.8 ± 0.9	0.9–2.7

Results obtained after 3 different collections of cheese whey wastewater.

this purpose, the samples were previously filtrated with cellulose acetate membrane filters of 0.2 μ m. Bicarbonates were measured by volumetric method using an HCl solution and methyl orange indicator. Calcium and magnesium determinations were made by volumetric complexation with EDTA using eriochrome black T (calcium + magnesium) and calcon (calcium) indicators. Dry matter (or total solids) and organic matter (or volatile solids) were determined by gravimetric method after drying at 105 °C and calcination at 550 °C, respectively. Analytical procedures for the studied absorbances can be found elsewhere (Rivas et al., 2010; Prazeres et al., 2013c).

2.3. Reagents and procedure

2.3.1. Cheese whey wastewater treatment by NaOH precipitation

Precipitation tests were conducted by using 400 mL of raw CWW in vessels of the Qlabo ISCO jar test apparatus at 20.0 °C. Initially, pH of raw CWW was adjusted under rapid agitation until the desired pH was attained. Thereafter, the agitation was turned off. After sludge sedimentation, the supernatants were analyzed to determine the main contaminant indicators. Optimal conditions were selected and the supernatant was characterized and reused in the tomato crop fertirrigation.

2.3.2. Reuse of treated CWW for tomato production

Treated CWW by NaOH precipitation under optimal conditions (pH 11.00) was used in the irrigation of two tomato cultivars *Lycopersicon esculentum* Mill. (Roma and Rio Grande). Irrigation experiments were implemented at the Experimental Center of Escola Superior Agrária de Beja (Portugal). The minimum and maximum average temperatures during the irrigation experiments were 13.92 ± 3.30 and 31.32 ± 5.06 , respectively. The trial had an area of 36 m^2 , plots of 1 m^2 and 5 fruit producing plants for each repetition in a soil of medium texture. The used soil presented the following properties: pH 8.3; electrical conductivity = 0.455 dS m^{-1} ; organic matter = 1.28%; P (as P_2O_5) = 135 mg kg^{-1} ; K (as K_2O) > 200 mg kg^{-1} ; Mg > 125 mg kg^{-1} ; N = 0.101%; Na = 77 mg kg^{-1} and Cl = 266.3 mg kg^{-1} . The soil detailed characterization can be found elsewhere (Prazeres et al., 2013b, 2014). Young plants were transferred with a distance in line and between lines of 0.20 m and 1 m, respectively.

The irrigation by gravity was done in furrows of 1 m of length and was performed three times per week during 72 days, based on the soil matric potential measured through “watermark” sen-

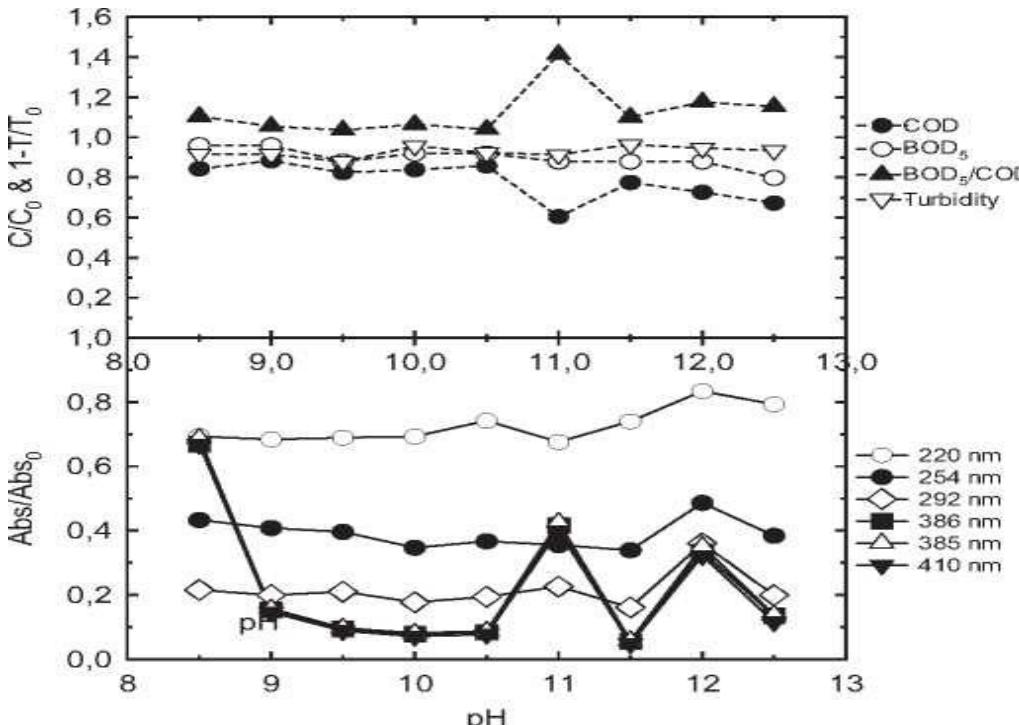


Fig. 1. Treatment of raw cheese whey wastewater by NaOH precipitation. Experimental conditions: pH 3.29, temperature = 20.5°C , COD = 12775 mg L^{-1} , BOD₅ = 12400 mg L^{-1} , BOD₅/COD = 0.97, turbidity (T) = 869.8 NTU, absorbance at 220 nm (1:50 dilution) = 0.589, absorbance at 254 nm (1:50 dilution) = 0.245, absorbance at 292 nm (1:50 dilution) = 0.186, absorbance at 386 nm (1:50 dilution) = 0.096, absorbance at 385 nm (1:50 dilution) = 0.094, absorbance at 410 nm (1:50 dilution) = 0.090.

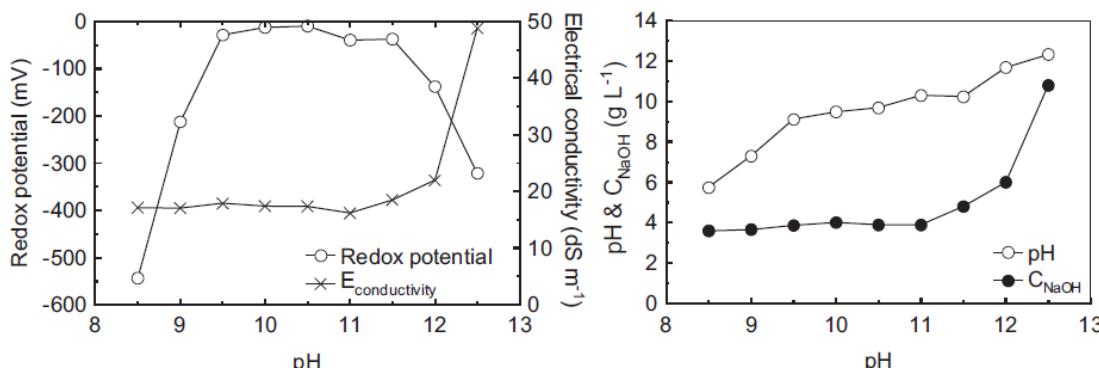


Fig. 2. Effect of the NaOH precipitation on the redox potential, electrical conductivity, pH and NaOH concentrations (C_{NaOH}). Experimental conditions: temperature = 20.5°C , redox potential = 229.7 mV, electrical conductivity = 14.25 dS m^{-1} , pH 3.29.

sors at depths of 20 and 35 cm (Prazeres et al., 2014) in order to minimize the water stress on plants. Up to six classes of irrigation waters were developed (treatment levels T₀-T₅). The treatment levels T₁-T₅ were obtained by dilution of treated CWW with fresh water (1:50; 1:22; 1:10; 1:5 and 1:2). The mean salinity levels of the treatments T₀, T₁, T₂, T₃, T₄ and T₅ corresponded to 1.44, 1.75, 2.22, 3.22, 5.02 and 10.02 dS m^{-1} . Irrigation water salinity levels of 1.7; 2.3; 3.4 and 5.0 dS m^{-1} provide the maximum tolerances to obtain potential

yield of 100; 90; 75 and 50%, respectively, for tomato crops (Fipps, 2003). Fresh water was used as control trial (T0), which has low organic matter concentration ($\text{COD} = 30 \text{ mg L}^{-1}$) and biodegradability index (BOD_5/COD) of about 0.1. Beyond that, the average salinity level (1.44 dS m^{-1}) and total dissolved solids (TDS) content (688 mg L^{-1}) make the fresh water an irrigation water of class 3 (permissible) (Fipps, 2003). It is worthwhile to remark that the treatments T1-T5 presented high nutrients content, such as phosphorus, nitrogen and potassium, compared with T0. Additionally, the irrigation waters T1-T5 had a lower calcium hardness than the treatment T0 and similar magnesium content. The detailed characterization of several irrigation waters can be found elsewhere (Prazeres et al., 2013a,b, 2014). The experimental design constituted a bifactorial in blocks with four replications per treatment (two repetitions for each cultivar). The following factors were studied: cultivar, treatment and interaction of cultivar x treatment.

The beginning of fruits ripening was recorded in the last week of July, corresponding to the test day 34 after the start of the irrigation. The fruits were harvested weekly. All production without deprecative quality defects (rot; blossom-end rot; lack of firmness; epidermis deformations; healed cracks; visible green parts; attack of parasites, etc.) was considered as marketable production.

2.3.3. Sludge treatment

Experiments of aerobic biodegradation were carried out at a volume of 2.0 L in batch mode under magnetic agitation and neutral conditions ($\text{pH } 7.05$). The magnetically stirred biological reactor was continuously oxygenated by an air flow stream (40 L h^{-1}). Microorganisms coming from an activated sludge process of a municipal wastewater treatment plant were used after 4 days of acclimatization to the sludge. Microorganisms were added to the biological reactor at a concentration of 3.8 g L^{-1} . Sludge sedimentation and compaction were performed in a normalized glass graduated cylinder (1 L) with 34 cm height. Sludge centrifugation was carried out at 3500 rpm for 10 min in a Jouan CR 1000.S5 centrifuge.

Sludge filtration tests were completed in a 42 cm height column, with external and internal diameters of 6.8 and 6.1 cm, respectively. Sand filters were composed of false fund completed with coarse gravel at a height of 2.1 cm. The drainage and collection of infiltrated (drained) water were performed from this false fund. The filtration medium consisted of gravel (16 cm, whose diameter decreased from 4 in 4 cm); coarse sand (2 cm), intermediate sand (4 cm) and fine sand (2 cm). The experiments of the sand filters were performed in triplicate. In the filtration experiments, 500 mL of the sludge (sludge height \times 17.1 cm) were placed in each column. The drained water from the filters was analyzed (BOD , COD , turbidity, pH , temperature, electrical conductivity, redox potential, absorbance at 220, 254, 292, 386, 385 and 410 nm) at different time intervals. When the water draining process was finished, the sludge was removed from the column and was placed in open dishes, starting the process of evaporation at a temperature of $27 \pm 1^\circ\text{C}$. The dry and organic matters of the sludge were determined periodically in the evaporation process, until the sludge showed the desired humidity.

2.4. Statistical analysis

The results were submitted to analysis of variance applying the statistical program MSTAT-C and for the distinction between averages was used the Least Significant Difference (LSD) test with a 95% confidence range.

3. Results and discussion

3.1. Cheese whey wastewater treatment by NaOH precipitation

NaOH addition to raw CWW was used to increase the initial pH to values in the range 8.5-12.5. High pH led to the formation of agglomerated particles that precipitated by gravity after a short period of time. A series of precipitation experiments was conducted to determine the best pH to be used in the tomato irrigation. The results of these experiments are illustrated in Fig. 1. COD elimination presented a slight increasing trend when the pH was raised from 8.5 to 12.5. Nevertheless, optimum conditions at $\text{pH } 11.0$ were experienced, achieving a COD removal near 40%. Additionally, the obtained supernatant presented a turbidity depletion of 91% and a high biodegradability value. Absorbance reductions between 32 and 77% for optimum pH were also found, depending on the studied wavelength.

Fig. 2 (left) depicts the evolution of the redox potential and electrical conductivity of the supernatant after NaOH precipitation. As concluded from this figure, the redox potential rapidly increased from -550 mV to values close to 0.0 mV by increasing the pH from 8.5 to 11.5. A further pH rise led to a new decrease of this parameter. NaOH addition induces the precipitation of magnesium and calcium hydroxides through the following reactions:



Table 2

Physicochemical characterization of the supernatant coming from NaOH precipitation of raw CWW.

Parameters	Average	Interval
Chemical oxygen demand—COD (mg L ⁻¹)	8553 ± 468	8064–8997
Biochemical oxygen demand—BOD ₅ (mg L ⁻¹)	8075 ± 2111	6100–10900
Turbidity—T (NTU)	48.9 ± 36.4	8.3–78.8
pH	11.74 ± 0.30	11.50–12.07
Temperature (°C)	24.0 ± 4.2	19.1–26.6
Electrical conductivity (dS m ⁻¹)	15.24 ± 1.36	13.87–16.58
Redox potential (mV)	-110.7 ± 38.5	-144.3–(-68.6)
Total solids—TS (mg L ⁻¹)	12066 ± 415	11758–12538
Total volatile solids—TVS (mg L ⁻¹)	4314 ± 309	4088–4666
Total suspended solids—TSS (mg L ⁻¹)	488 ± 481	148–828
Volatile suspended solids—VSS (mg L ⁻¹)	413 ± 342	44–720
Sulphates (mg L ⁻¹)	5.8 ± 1.0	5.0–6.9
Na (mg L ⁻¹)	3017.2 ± 332.7	2646.7–3290.4
K (mg L ⁻¹)	205.2 ± 2.9	201.9–206.9
P (mg L ⁻¹)	48.1 ± 7.7	39.2–52.8
Cl (mg L ⁻¹)	2371.1 ± 142.6	2263.3–2532.8
N-Kjeldahl (mg L ⁻¹ N)	242.4 ± 16.0	224.9–256.2
Calcium hardness—CH (mg L ⁻¹ CaCO ₃)	149.1 ± 8.9	138.8–154.2
Ca (mg L ⁻¹)	59.6 ± 3.6	55.5–61.7
Magnesium hardness—MH (mg L ⁻¹ CaCO ₃)	146.5 ± 13.4	131.1–154.2
Mg (mg L ⁻¹)	35.7 ± 3.3	32.0–37.6
Total hardness—TH (mg L ⁻¹ CaCO ₃)	295.6 ± 22.3	269.9–308.5
Ca/Mg	1.7 ± 0.1	1.6–1.7
CH/MH	1.0 ± 0.0	1.0–1.1

Results obtained after 3 different collections of cheese whey wastewater.



Additionally, reactions can also be developed in the medium that allow the precipitation of calcium and magnesium carbonates:



According to the solubility constants (K_s), Ca(OH)₂ precipitation occurs after CaCO₃ precipitation and MgCO₃ precipitation takes place after Mg(OH)₂ precipitation, once the required amount of calcium and carbonate or magnesium and hydroxide to precipitate CaCO₃ and Mg(OH)₂ is lower than the amount of calcium and hydroxide or magnesium and carbonate to precipitate Ca(OH)₂ and MgCO₃—K_s(CaCO₃) < K_s(Ca(OH)₂) and K_s(Mg(OH)₂) < K_s(MgCO₃).

The stabilizing role played by the calcium ions (50% reduction compared with raw CWW) in casein micelles is reduced leading to the appearance of the observed precipitate. For high precipitation pH (>9), the lowest electrical conductivity value (16.17 dS m⁻¹) was obtained at pH 11.0, coinciding with the maximum COD removal. Runs conducted at pH above 11.0 did not involve a higher precipitation efficacy. However, the excess of NaOH (Fig. 2) led to an increase in the supernatant electrical conductivity. Similar results were obtained by Renou et al. (2008) when studying lime precipitation applied to stabilized landfill leachate treatment. In such work, an increase of the precipitant dose, and consequently a precipitation pH rise, did not cause the improvement in the organic matter removal but an increase of the supernatant pH and electrical conductivity. The effects observed in the present study can be explained by the reactions that happen when NaOH is added to the raw wastewater containing organic matter, ions (Ca²⁺ and Mg²⁺), bicarbonates, suspended and colloidal particles, etc. Existing cations react with OH⁻ to form calcium and magnesium hydroxides. Additionally, calcium and magnesium carbonates can be formed in the medium. These insoluble species entrap and drag the suspended and colloidal particles, precipitating together with the reduction of organic matter, turbidity, suspended solids, calcium, magnesium, etc. Thus, the electrical conductivity reaches the minimum value and the removal of COD presents the maximum value. From this point, the rise of the precipitation pH (excess precipitant concentration) leads to an increase of the electrical conductivity due to the Na⁺ and OH⁻ enhanced concentrations in the solution, affecting the elimination of organic matter, turbidity, absorbances, solids, etc.

Table 2 shows the main properties of the supernatant after basic precipitation conducted at pH 11.0. This Table indicates TSS and turbidity significant reductions if compared with raw CWW. The supernatant is almost completely transparent, without whitish appearance. Sulphates, phosphorus, total hardness and Kjeldahl

nitrogen suffered 93%, 53%, 40% and 23% depletion. Permanent hardness was scarcely affected, since the chloride elimination registered values of only 12.4%. Additionally, the treated CWW is still a strong organic effluent with about 8.1 g L⁻¹ of BOD and 8.6 g L⁻¹ of COD. As inferred from Table 2, the obtained supernatant is a rich effluent in biodegradable organic matter and nutrients (Ca, Mg, P, N, K, Na and Cl) that can be recycled as plant growth factors.

3.2. Reuse of treated CWW for tomato production

When plants are exposed to a nutrient disequilibrium, they suffer disturbances in their metabolism (Prazeres et al., 2013b). The tomato crop response to adverse conditions such as salinity, depends on several factors, comprising environmental conditions, plant development stage, salinity level and exposure time (Caines and Shennan, 1999; Munns, 2002; Maggio et al., 2004; Perez-Alfocea et al., 1993; Prazeres et al., 2014). The key drawback of the treated CWW is the salinity level. For that reason, the treated CWW by NaOH precipitation was diluted with fresh water before being used for the tomato plants irrigation (Table 3). Table 4 describes the influence of different factors (cultivar, treatment and cultivar x treatment) on the production and development of industrial tomatoes. As observed from the Table, total yield, marketable yield and yield with physiological disorder of blossom-end rot were significantly influenced ($p < 0.05$) by the cultivar used (Roma and Rio Grande). Thus, the cultivar Rio Grande presented a marketable production 16% higher than the cultivar Roma. Additionally, the cultivar Roma showed a two-fold increase in the tomato yield with physiological disorder of blossom-end rot, compared with cultivar Rio Grande.

On the other hand, the tomato cultivars responded differently to the treatment (cultivar x treatment). Cultivar Roma presented a

Table 3
Characterization of different waters used in the agricultural irrigation of the tomato cultivars.

Parameters	T ₀	T ₁	T ₂	T ₃	T ₄	T ₅
Chemical oxygen demand—COD (mg L ⁻¹)	30 ± 19	172 ± 57	404 ± 107	895 ± 278	1883 ± 470	5014 ± 1481
Biological oxygen demand—BOD ₅ (mg L ⁻¹)	3 ± 1	140 ± 49	305 ± 100	738 ± 149	1675 ± 574	4450 ± 1652
BOD ₅ /COD (adimensional)	0.11 ± 0.02	0.81 ± 0.03	0.75 ± 0.05	0.84 ± 0.08	0.88 ± 0.08	0.88 ± 0.06
Turbidity (NTU)	1.9 ± 1.2	12.2 ± 2.9	19.0 ± 2.0	27.4 ± 5.9	33.8 ± 15.1	69.6 ± 40.7
pH	7.38 ± 0.16	7.51 ± 0.27	7.30 ± 0.29	7.32 ± 0.24	7.36 ± 0.28	7.43 ± 0.47
Electrical conductivity (dS m ⁻¹)	1.44 ± 0.05	1.75 ± 0.03	2.22 ± 0.11	3.22 ± 0.21	5.02 ± 0.36	10.02 ± 0.96
Redox potential (mV)	212.1 ± 32.8	110.8 ± 72.5	26.7 ± 155.4	-96.2 ± 181.2	-122.2 ± 172.1	-186.9 ± 79.0
Total suspended solids—TSS (mg L ⁻¹)	118 ± 48	82 ± 12	100 ± 58	193 ± 34	265 ± 27	241 ± 47
Total dissolved solids—TDS (mg L ⁻¹)	688 ± 51	796 ± 44	1043 ± 31	1602 ± 198	2899 ± 501	6653 ± 1183
Volatile suspended solids—VSS (mg L ⁻¹)	22 ± 8	107 ± 14	106 ± 70	170 ± 25	233 ± 19	219 ± 50
Volatile dissolved solids—VDS (mg L ⁻¹)	152 ± 6	163 ± 16	150 ± 54	314 ± 171	623 ± 296	1759 ± 742
Bicarbonates (mg L ⁻¹)	187.2 ± 0.0	205.3 ± 0.0	229.5 ± 10.5	257.7 ± 7.0	348.2 ± 15.2	628.1 ± 12.1
Sulphates (mg L ⁻¹)	48.6 ± 1.6	125.8 ± 0.2	240.6 ± 0.7	504.2 ± 1.0	947.7 ± 1.3	2134.9 ± 2.2
Na (mg L ⁻¹)	97.3 ± 13.0	147.1 ± 9.6	217.8 ± 13.2	419.1 ± 64.5	734.5 ± 137.6	1638.6 ± 329.0
K (mg L ⁻¹)	10.3 ± 1.4	14.6 ± 2.3	24.1 ± 9.6	41.4 ± 16.1	73.9 ± 22.8	141.6 ± 59.9
P (mg L ⁻¹)	<LOD	<LOD	<LOD	<LOD	<LOD	13.2 ± 6.6
Cl (mg L ⁻¹)	237.1 ± 0.0	265.9 ± 11.4	309.0 ± 11.7	421.2 ± 43.5	643.1 ± 106.5	1257.4 ± 302.4
N-Kjeldahl (mg L ⁻¹)	3.2 ± 2.8	7.7 ± 2.6	18.6 ± 8.8	35.2 ± 8.5	60.1 ± 20.6	136.4 ± 33.7
Ca (mg L ⁻¹)	74.0 ± 0.0	52.4 ± 3.6	51.7 ± 4.6	48.6 ± 1.5	75.6 ± 20.4	41.6 ± 5.9
Mg (mg L ⁻¹)	41.9 ± 3.3	45.6 ± 3.9	34.3 ± 2.8	48.0 ± 8.9	38.6 ± 9.8	49.9 ± 12.4
Sodium adsorption ratio—SAR (adimensional)	2.2	3.6	5.7	10.2	17.1	40.3

Some parameters are from Prazeres et al. (2013b) and Prazeres et al. (2014). Results obtained after 4 different collections of raw wastewater. T₀ symbolizes the fresh water (control run). T₁, T₂, T₃, T₄ and T₅ symbolize the pretreated CWW after dilution with fresh water at different ratios (1:50; 1:22; 1:10; 1:5 and 1:2). LOD—limit of detection.

drop in the marketable yield (6.7-19.3%) when tomato plants were exposed to salinity levels between 1.75 and 10.02 dS m⁻¹. Several research studies under osmotic stress conditions have reported a yield decrease (Fisher and Nel, 1990; Gianquinto et al., 1990; Latef and Chaoxing, 2011). Katerji et al. (1998) obtained a yield reduction of about 60% in tomato variety ELKO 190 for salinity level of 3.6 dS m⁻¹, compared to fresh water (0.9 dS m⁻¹). Similar results were reported by Santa-Cruz et al. (2002), where a fruit yield reduction in the range 36-46% was experienced when moderate salinity conditions (50 mM NaCl) were applied to tomato plants. Contrarily, the marketable yield of the cultivar Rio Grande increased in the interval of 14-21% for salinity levels of 1.75-3.22 dS m⁻¹. This effect can be due to the greater calcium and potassium absorption and accumulation capacity in the tissues of the cultivar Rio Grande (Prazeres et al., 2013b). Cuartero and Fernández-Munoz (1999) referred that when tomato plants absorb more calcium and potassium than sodium of a saline environment, they will have a nutritional balance closer to the plants growing in a no-saline

Table 4

Effect of cultivar, treatment and cultivar \times treatment interaction on the production and development of industrial tomato *Lycopersicum esculentum* Mill. irrigated with CWW treated by NaOH precipitation.

Study factors	TY (ton ha ⁻¹)	MY (ton ha ⁻¹)	\sum_{PL} (ton ha ⁻¹)	Y _{BER} (ton ha ⁻¹)	Y _{SE} (ton ha ⁻¹)	FW (g)	NT kg ⁻¹
Cultivar	*	*	n.s.	*	n.s.	**	**
Roma	95.7 \pm 8.0	73.4 \pm 6.1	22.3 \pm 2.6	9.8 \pm 3.6	3.4 \pm 2.8	47.6 \pm 9.0	21.7 \pm 3.6
Rio Grande	109.6 \pm 17.3	87.4 \pm 14.3	22.2 \pm 3.5	5.0 \pm 4.2	5.1 \pm 2.1	52.4 \pm 10.3	19.8 \pm 4.2
Treatment	n.s.	n.s.	n.s.	n.s.	*	***	***
T ₀	105.3 \pm 3.3	80.5 \pm 2.4	24.9 \pm 0.9	3.8 \pm 1.9	8.5 \pm 0.0a	57.3 \pm 7.4a	18 \pm 2d
T ₁	113.3 \pm 15.7	88.1 \pm 16.1	25.2 \pm 0.4	3.4 \pm 1.7	4.8 \pm 1.9b	60.4 \pm 7.9a	17 \pm 2d
T ₂	104.8 \pm 26.6	83.0 \pm 23.6	21.8 \pm 3.0	8.9 \pm 4.4	2.3 \pm 0.9b	51.0 \pm 9.1b	20 \pm 4c
T ₃	102.6 \pm 14.7	81.6 \pm 14.5	21.0 \pm 0.2	8.8 \pm 3.2	1.8 \pm 0.8b	49.3 \pm 12.4b	21 \pm 5c
T ₄	84.1 \pm 8.5	65.1 \pm 3.5	19.0 \pm 5.0	10.2 \pm 7.3	3.5 \pm 0.9b	42.9 \pm 0.5c	23 \pm 0b
T ₅	105.9 \pm 13.7	84.2 \pm 11.2	21.7 \pm 2.5	9.3 \pm 3.3	4.7 \pm 1.4b	39.4 \pm 1.9c	25 \pm 1a
Cultivar \times treatment	n.s.	n.s.	n.s.	n.s.	n.s.	***	***
Roma							
T ₀	107.7 \pm 15.2	82.2 \pm 8.6	25.5 \pm 6.6	6.5 \pm 0.2	8.5 \pm 2.8	62.5 \pm 9.4ab	16 \pm 2de
T ₁	102.1 \pm 10.8	76.7 \pm 13.0	25.4 \pm 2.3	6.8 \pm 1.1	3.5 \pm 0.6	54.8 \pm 5.5c	18 \pm 2cd
T ₂	86.0 \pm 7.5	66.3 \pm 3.5	19.7 \pm 11.0	12.0 \pm 6.7	0.6 \pm 0.1	44.6 \pm 7.1de	23 \pm 4b
T ₃	92.2 \pm 18.7	71.3 \pm 13.0	20.9 \pm 5.8	11.0 \pm 6.0	1.2 \pm 0.4	40.6 \pm 1.0e	25 \pm 1ab
T ₄	90.2 \pm 25.4	67.6 \pm 27.6	22.6 \pm 2.2	15.4 \pm 4.7	2.9 \pm 2.7	42.6 \pm 1.0e	24 \pm 1b
T ₅	96.2 \pm 19.5	76.3 \pm 10.2	19.9 \pm 9.3	7.0 \pm 0.5	3.6 \pm 1.3	40.7 \pm 0.5e	25 \pm 0ab
Rio Grande							
T ₀	103.0 \pm 5.7	78.8 \pm 5.4	24.2 \pm 0.3	1.2 \pm 1.1	8.5 \pm 1.4	52.0 \pm 6.8 cd	19 \pm 3c
T ₁	124.4 \pm 15.3	99.5 \pm 14.1	24.9 \pm 1.2	0.0	6.2 \pm 0.6	66.0 \pm 4.9a	15 \pm 1e
T ₂	123.6 \pm 24.1	99.7 \pm 9.6	23.9 \pm 14.6	5.7 \pm 4.9	3.9 \pm 1.7	57.4 \pm 6.5bc	18 \pm 2cde
T ₃	113.0 \pm 14.4	91.9 \pm 20.4	21.2 \pm 6.0	6.5 \pm 5.6	2.3 \pm 1.9	58.0 \pm 3.8bc	17 \pm 1cde
T ₄	78.1 \pm 8.4	62.6 \pm 14.8	15.4 \pm 6.5	5.0 \pm 0.7	4.2 \pm 3.5	43.2 \pm 5.6e	23 \pm 3b
T ₅	115.6 \pm 0.3	92.1 \pm 8.8	23.5 \pm 8.5	11.6 \pm 6.3	5.7 \pm 2.1	38.0 \pm 1.6e	26 \pm 1a
C.V. (%)	14.83	17.53	33.44	60.25	50.03	6.81	6.50

TY—total yield; MY—marketable yield; \sum_{PL} —production losses; Y_{BER}—yield with physiological disorder of blossom-end rot; Y_{SE}—yield with epidermis deformations by solar exposure; FW—unit fruit fresh weight; NT kg⁻¹—number of tomatoes per kilogram. T₀ represents the control experiment (fresh water). T₁, T₂, T₃, T₄ and T₅ represent the treated CWW (by NaOH precipitation) diluted with fresh water in the following ratios 1:50; 1:22; 1:10; 1:5 and 1:2, respectively. *, **, *** significant for $p \leq 0.05$, $p \leq 0.01$ and $p \leq 0.001$, respectively; n.s.—not significant. Different lowercase letters indicate significant differences with $p \leq 0.05$, according to LSD test. C.V.—coefficient of variation.

medium. Consequently, these plants show more tolerance to salinity. On the other hand, the leaf concentration of sodium increased more in the cultivar Roma than in the cultivar Rio Grande when the plants were exposed to salinity conditions of 10.02 dS m⁻¹, compared with a salinity level of 1.44 dS m⁻¹ (Prazeres et al., 2013b). Thus, the effect of sodium toxicity appears more significant in the cultivar Roma. Additionally, the tomato yield of the treatment T₅ was higher than that of the treatment T₄. This effect can be explained by the high activity of potassium and nitrogen in the plant tissues and competition phenomena between sodium- potassium and chloride-nitrogen (Prazeres et al., 2013b).

Although there are no significant statistical differences (Table 4), the water salinity level (treatment) severely affects the incidence of the production losses due to the blossom-end rot. Salinity level $>$ 2.22 dS m⁻¹ led to an increase in the tomato yield with physiological disorder of blossom-end rot. Therefore, it can be mentioned a rise of the yield with physiological disorder of blossom-end rot around 58 and 90% for the cultivar Roma and Rio Grande in the electrical conductivity levels of 5.02 dS m⁻¹ (T₄) and 10.02 dS m⁻¹ (T₅), respectively. Nevertheless, it was not observed such physiological disorder in the treatment T₁ for the cultivar Rio Grande. The physiological disorder of blossom-end rot can be attributed to irradiation, temperature, low water availability, soil composition, soil cationic balance, air humidity, salinity, NH₄⁺ toxicity, etc. (Barickman et al., 2014; Hayashi et al., 2014; Ho et al., 1993; Morales et al., 2015; Prazeres et al., 2014; Rubio et al., 2009; Saure, 2001). However, the increased incidence of yield with physiological disorder of blossom-end rot is one of the key drawbacks when salinity conditions are used (Martinez et al., 1987), resulting from the reduced uptake of Ca²⁺ (Adams and Ho, 1992; Dorais et al., 2001). As a consequence, the local deficiency of Ca²⁺ in the distal placental tissue of the fruit results in symptoms of minor dimpling in this tissue, invading gradually the pericarp (Cuartero and Fernández- Muñoz, 1999). The lower calcium content of the irrigation waters T₁-T₅ is explained by the calcium elimination around 50% in the NaOH precipitation of the raw CWW.

As observed from Table 4, the treatment affected ($p < 0.05$) the tomato yield with epidermis deformations by solar exposure, with statistical distinction of two groups: one constituted by T₀ (control run) and another represented by T₁-T₅. Accordingly, the salinity conditions reduced the yield with epidermis deformations by solar exposure in about 93 and 73% for the cultivar Roma (T₂) and Rio Grande (T₃), respectively. This effect can be explicated by the improvement of the epidermis resistance within the range of 30-40% when salinity conditions with pretreated CWW were applied to tomato plants (Prazeres et al., 2014).

The used cultivar had a very significant effect ($p < 0.01$) on the unit fresh weight of the fruit and number of fruits per kilogram, while the treatment and interaction of cultivar \times treatment had a highly significant influence ($p < 0.001$) (see Table 4). The cultivar Roma showed a linear reduction in the unit fruit fresh weight ($R^2 = 0.84$)

and a linear increase in the number of fruits per kilogram ($R^2 = 0.88$) for the treatments T0-T3. As a consequence, a reduction of about 12-35% in the unit fruit fresh weight was experienced for the cultivar Roma, compared with control plants. Fruit weight decline is consistently reported as a result of the tomato plants exposure to salinity conditions (Bolarin et al., 2001; Gianquinto et al., 1990; Katerji et al., 1998; Latef and Chaoxing, 2011; Sato et al., 2006; Zushi and Matsuzoe, 2009). In this approach, Santa-Cruz et al. (2002) and Zushi et al. (2009) described reductions in the fruit weight of 27-44% for salinity level of 50 mM NaCl and 40% for salinity level of 100 mM NaCl, respectively. The fruits of plants exposed to salinity conditions have a smaller development period, halting the growth in the turning stage, compared with fruits of plants that were not subjected to stress conditions (Zushi et al., 2009). Some researchers have observed that the fruit formation is disfavored at

Table 5

Sludge characterization coming from NaOH precipitation of raw CWW.

Parameters	Average ^a	Interval ^a
Sludge volume (mL of sludge L ⁻¹ of wastewater)	63 ± 0	63–63
pH	9.58 ± 0.03	9.56–9.60
Temperature (°C)	14 ± 0	14–14
Electrical conductivity (dS m ⁻¹)	5.16 ± 0.01	5.15–5.16
Dry matter (%)	3.15 ± 0.48 ^b	2.59–3.50 ^b
Organic content (dry basis) (%)	51.2 ± 10.0 ^b	38.7–58.6 ^b
Organic content (wet basis) (%)	1.66 ± 0.54 ^b	1.00–2.05 ^b
Phosphorus (dry basis) (g kg ⁻¹)	5.78 ± 3.95	2.98–8.57
Phosphorus (wet basis) (g kg ⁻¹)	0.06 ± 0.05	0.03–0.10
Kjedahl nitrogen (wet basis) (g kg ⁻¹)	0.352 ± 0.152	0.244–0.459
Kjedahl nitrogen (dry basis) (g kg ⁻¹)	11.17 ± 4.83	7.75–14.57
Ammonium nitrogen (wet basis) (g N-NH ₃ kg ⁻¹)	0.025 ± 0.004	0.022–0.028
Ammonium nitrogen (dry basis) (g N-NH ₃ kg ⁻¹)	0.79 ± 0.13	0.70–0.89
Total suspended solids—TSS (mg L ⁻¹)	11116 ± 2251	9524–12708
Volatile suspended solids—VSS (mg L ⁻¹)	5115 ± 765	4574–5656
Sedimentation type	Zone	

^a Results after 2 replicates.

^b Results after 5 replicates.

the expense of the plant growth and development under salinity conditions or water deficit (Fisher and Nel, 1990; Gianquinto et al., 1990; Katerji et al., 1998). Unlike the cultivar Roma, the unit fruit fresh weight of the cultivar Rio Grande increased within the range of 9.4-21.2% for the treatments T1-T3. The tomato fresh weight followed the same trend of the marketable yield. Accordingly, salinity effect on the yield must be related to the fruit development. The lowest number of fruits per kilogram observed for the cultivar Rio Grande (T1-T3) was associated to the production of large dimension fruits. In addition, it was observed, for the cultivar Rio Grande, a linear reduction in the unit fruit fresh weight ($R^2 = 0.82$) and a linear increase in the number of fruits per kilogram ($R^2 = 0.86$) for the treatments ranging between T₁ and T₅.

3.3. Sludge characterization

NaOH precipitation originated a white precipitate, which presented a density of roughly 1017 kg m⁻³. Table 5 summarizes the main properties of the generated sludge. The high pH value (pH 9.6) is undoubtedly due to the basic conditions used in the precipitation process. This pH value permits the use of the generated sludge as soil corrective. Additionally, electrical conductivity around 5.2 dS m⁻¹ seems to be an indicator of an elevated salt content and hydrosoluble ionic compounds (Acosta et al., 1998; Carvalho et al., 2012). The sludge volume was significantly lower than those obtained when using other precipitating agents (Rivas et al., 2010). The TSS content presented a value of approximately 11 g L⁻¹ (roughly 2 times higher than the volatile suspended solids—VSS content).

The organic matter, total phosphorus and nitrogen fractions suggest the hypothesis of using the obtained sludge as fertilizer. The phosphorus concentration was about 2 times higher than the critical limit reported by Singh and Jones (1976) for agricultural purposes.

3.4. Sludge sedimentation and settling tank design

In this study, the sedimentation properties and the settling tank design of the generated sludge after applying the NaOH precipitation was compared with the sludge obtained by lime precipitation (pH 11.5) and Fe(III) coagulation-flocculation (250 mg L^{-1}) processes. Fig. 3 depicts the sludge sedimentation curves as a function of time under triplicated experiments executed for the CWW treatment (lime precipitation, NaOH precipitation and Fe(III) coagulation-flocculation processes). Thus, the column height-time curves (resulting from the previous profiles) provide the average

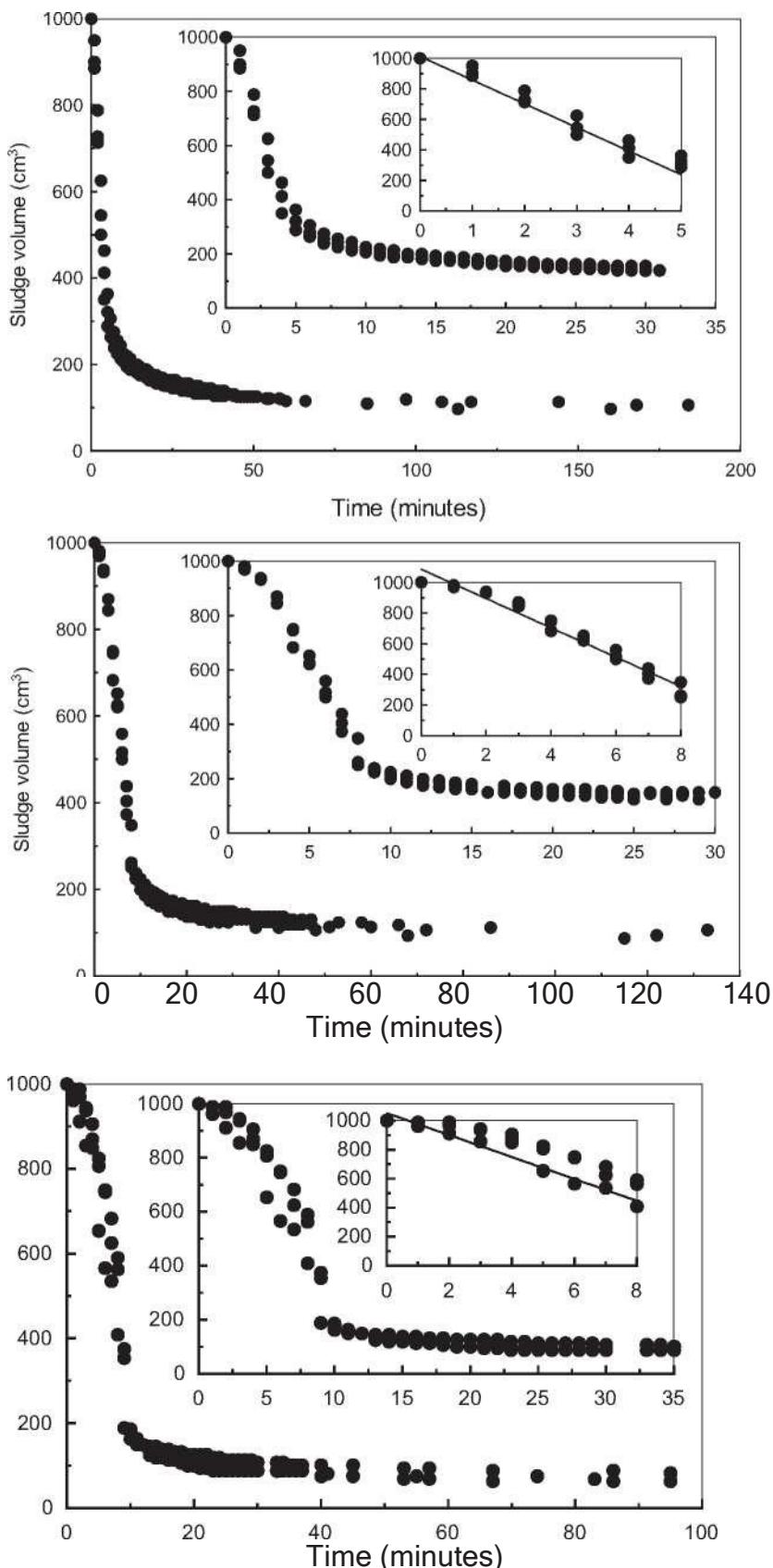


Fig. 3. Settling characteristics of the sludge obtained in the lime precipitation, NaOH precipitation and Fe(III) coagulation-flocculation processes applied to raw cheese whey wastewater. Three replicates: top—lime precipitation; middle—NaOH precipitation; bottom—Fe(III) coagulation-flocculation.

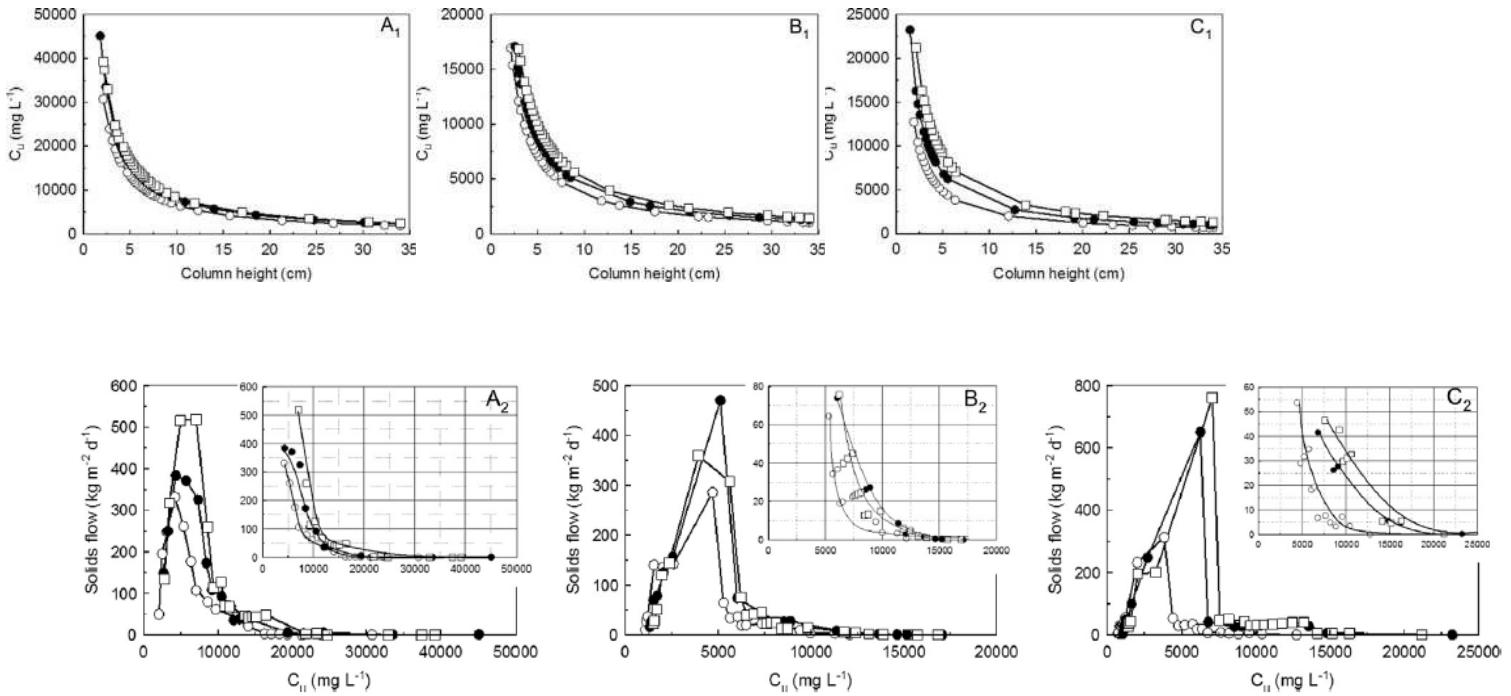


Fig. 4. Concentration and flow of solids obtained in the lime precipitation, NaOH precipitation and Fe(III) coagulation-flocculation processes applied to raw cheese whey wastewater. (A) Lime precipitation: O $C_0 = 1937.5 \text{ mg L}^{-1}$, • $C_0 = 2347.4 \text{ mg L}^{-1}$, □ $C_0 = 2470.4 \text{ mg L}^{-1}$. (B) NaOH precipitation: O $C_0 = 1051.8 \text{ mg L}^{-1}$, • $C_0 = 1281.5 \text{ mg L}^{-1}$, □ $C_0 = 1466.5 \text{ mg L}^{-1}$. (C) Fe(III) coagulation-flocculation: O $C_0 = 711.9 \text{ mg L}^{-1}$, • $C_0 = 1017.0 \text{ mg L}^{-1}$, □ $C_0 = 1333.4 \text{ mg L}^{-1}$. (1) Sludge solids concentration (C_u) as a function of the column height. (2) Solids flow (GB) as a function of the sludge solids concentration (C_u).

settling velocity (VS) of the sludge through the slope of the tangent to the sedimentation curve in the clarification zone (Metcalf and Eddy, 2003; Ramalho, 1996) for lime precipitation (5.23 cm min⁻¹ with $R^2 = 0.99$), NaOH precipitation (3.19 cm min⁻¹ with $R^2 = 0.98$) and Fe(III) coagulation-flocculation (2.09 cm min⁻¹ with $R^2 = 0.95$).

In addition, sedimentation experiments were performed by varying the initial solids concentration (C_0) for the different studied processes. Following this procedure, the solids concentration at different column heights C_u (H_i) was calculated from Kynch's theory (Ramalho, 1996):

$$C_u(H_i) = C_0 \frac{H_0}{H_i} \quad (5)$$

where H_0 is the initial column height (34 cm). These results are represented in Fig. 4 (graphics 1). At the same time, the total solids flow (GT) was determined using the graphical method proposed by Yoshioka and Dick (Ramalho, 1996). In a first approach, the solids flow in discontinuous operation due to the gravity (GB) (represented in Fig. 4, graphics 2) was deduced by the following expression:

$$C^B = C^n V_i \quad (6)$$

where C_u is the solids concentration in the sludge and V_i is the settling velocity in the zone of concentration C_u . The procedure for determining G_T was also based on the final concentration of solids desired in the sludge (C_{ud}), and consequently on the compacting index ($T = C_{ud}/C_0$). The next step consisted of plotting the tangent to the curve GB versus C_i containing the point $(C_{ud}, 0)$ until intercept the Y axis. This point provided the value $G_T = GB + GU$ ($GU =$ solids flow that coming out of the settling tank). The minimum area needed for the sludge thickening was given by $AT = Q_0 C_0 / G_T$ (with $Q_0 =$ inflow volumetric rate). In a following step, minimum clarification area of the settling tank (AC) was obtained from the expression (Metcalf and Eddy, 2003; Ramalho, 1996):

$$A_C = \frac{Q_e}{V_S} \quad (7)$$

where Q_e is the outflow volumetric rate, which depends on the inflow volumetric rate (Q_0), initial solids concentration (C_0), final solids concentration desired in the sludge (C_{ud}) and solids concen-

Table 6

Design parameters of the settling tanks for lime precipitation, NaOH precipitation and Fe(III) coagulation–flocculation processes.

Parameters	Units	Lime	NaOH	Fe(III)
Q_0	$\text{m}^3 \text{d}^{-1}$	1000	1000	1000
C_0	mg L^{-1}	1937.5	1466.5	711.9
C_e	mg L^{-1}	25.19	29.33	50.54
C_{ud}	mg L^{-1}	12500	9500	7500
Υ	–	6.45	6.48	10.54
Q_e	$\text{m}^3 \text{d}^{-1}$	846.71	848.25	911.22
Q_u	$\text{m}^3 \text{d}^{-1}$	153.29	151.75	88.78
V_s	m d^{-1}	67.27	46.54	25.60
A_C	m^2	12.59	18.23	35.60
G_T	$\text{kg m}^{-2} \text{d}^{-1}$	215.79	161.91	116.25
G_B	$\text{kg m}^{-2} \text{d}^{-1}$	68.42	32.38	33.02
C_i	mg L^{-1}	8787.88	7580.65	5357.14
G_U	$\text{kg m}^{-2} \text{d}^{-1}$	147.37	129.53	83.23
A_T	m^2	8.98	9.06	6.12
D	m	4.00	4.82	6.73
HRT	h	1	1	2
V	m^3	41.67	41.67	83.33
H	m	3.31	2.29	2.34

V—volume.

tration in the effluent (C_e). For this purpose, Q_e was determined using:

$$Q_e = Q_0 \frac{C_{ud} - C_0}{C_{ud} - C_e} \quad (8)$$

Fig. 5 illustrates the minimum clarification area as a function of the initial (C_0) and sludge (C_u) solids concentration for the lime precipitation, NaOH precipitation and Fe(III) coagulation–flocculation processes. The sludge solids concentration influences significantly the minimum clarification area. The minimum clarification area exceeded the minimum area needed for the sludge thickening. Therefore, the first was considered to determine the dimensions of the settling tank: diameter (D) and height (H). **Table 6** resumes the values considered (Q_0 , C_0 , C_{ud} , HRT—hydraulic retention time) and calculated in the design of the settling tank for the different processes. From this table, it can be concluded that the minimum clarification area obtained for the NaOH precipitation process is

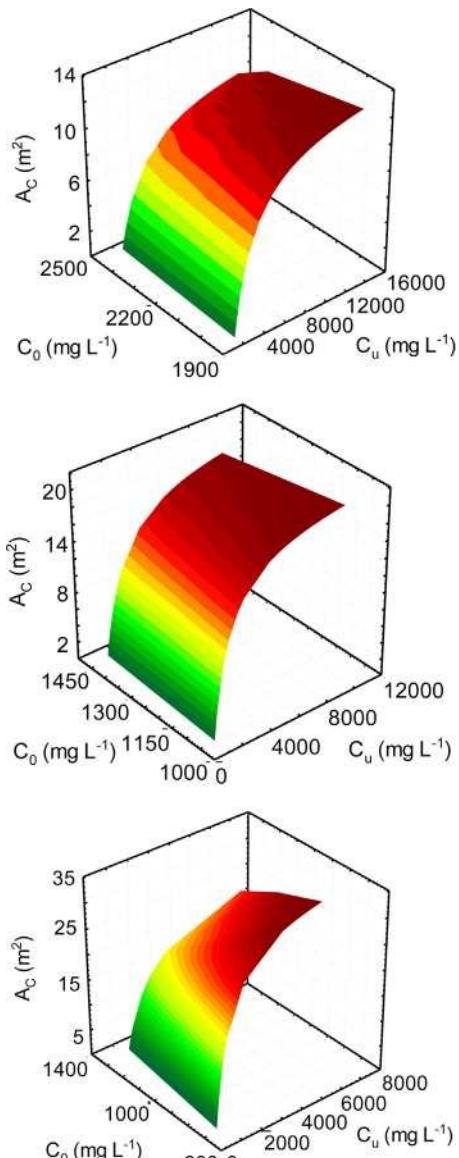


Fig. 5. Minimum clarification area as a function of the initial (C_0) and sludge (C_u) solids concentration in the lime precipitation, NaOH precipitation and Fe(III) coagulation-flocculation processes applied to raw cheese whey wastewater. Top—lime precipitation; middle—NaOH precipitation; bottom—Fe(III) coagulation-flocculation.

higher than the value achieved for the lime precipitation process. However, a similar value of minimum area needed for the sludge thickening was obtained for both processes (lime and NaOH precipitation).

3.5. Sludge management

In order to dispose/manage the generated sludge, this solid was subjected to the following processes: centrifugation, compaction, filtration + evaporation (sand filters), aerobic digestion + sedimentation and aerobic digestion + sedimentation + centrifugation. As previously mentioned, the sludge aerobic digestion was carried out at neutral pH. The obtained results are represented in Fig. 6. Aerobic digestion was effective in the volume reduction of sludge in about 62%. It was observed an increase of the microorganisms growth until a process time of 27 h (VSS 13 g L^{-1}) was attained, and then a decrease up to 48 h (40% of VSS depletion). Similar results were obtained for the TSS content. Aerobic digestion of the sludge

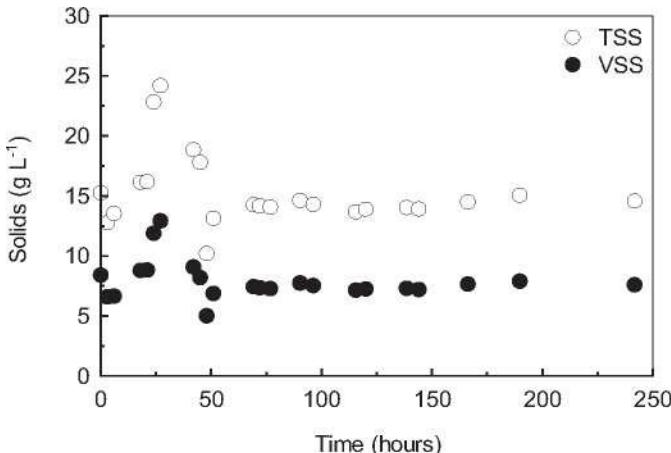
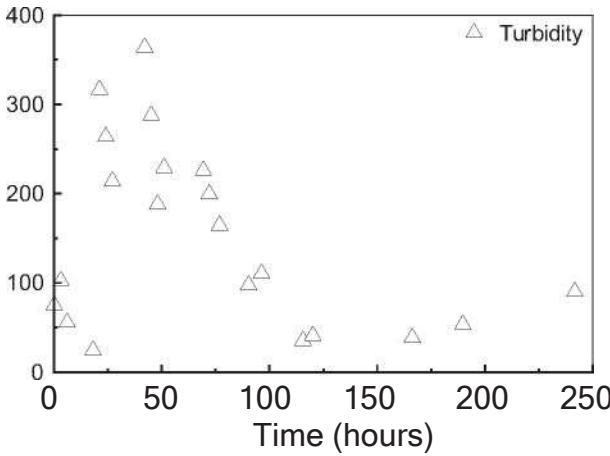
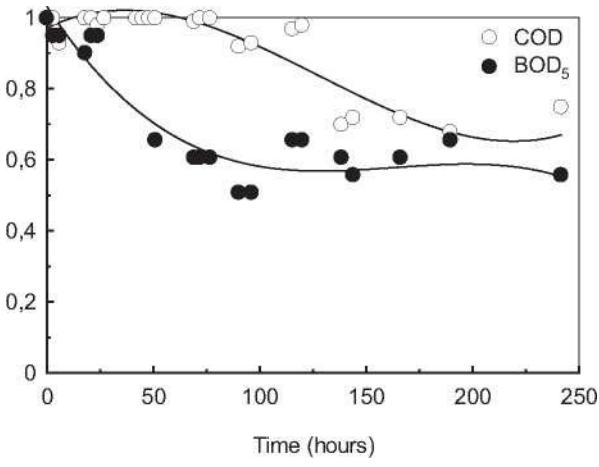


Fig. 6. Aerobic digestion of the sludge obtained in the NaOH precipitation. Experimental conditions: pH 7.77, temperature = 26.3 °C, potential redox = -184.0, electrical conductivity = 18.46 dS m⁻¹, COD = 6713 mg L⁻¹, BOD₅ = 10200 mg L⁻¹, turbidity (T) = 75.0 NTU, absorbance at 220 nm (1:50 dilution) = 0.568, absorbance at 254 nm (1:50 dilution) = 0.290, absorbance at 292 nm (1:50 dilution) = 0.215, absorbance at 386 nm (1:50 dilution) = 0.144, absorbance at 385 nm (1:50 dilution) = 0.145, absorbance at 410 nm (1:50 dilution) = 0.133, TSS = 15.284 g L⁻¹, VSS = 8.404 g L⁻¹.

revealed to be effective in the COD and BOD reduction (32-39%) of the obtained supernatant. However, this supernatant still constitutes a strong pollutant, presenting a COD value of 4.6 g L⁻¹, consequently it needs a post-treatment.

Table 7

Characterization of the effluent obtained in the sludge treatment: filtration and aerobic digestion + sedimentation + centrifugation.

Parameters	Filtration ^a	Aerobic digestion + sedimentation + centrifugation
Process time (h)	72	166–190
pH	9.71 ± 0.11 (2.87%)	8.43 (−8.5%)
Redox potential (mV)	−32.4 ± 4.7 (70.8%)	−118.3 (35.7%)
Electrical conductivity (dS m ^{−1})	27.57 ± 2.30 (−88.9%)	19.35 (−4.8%)
Chemical oxygen demand—COD (mg L ^{−1})	6358 ± 1180 (6%)	4575 (31.8%)
Biochemical oxygen demand—BOD (mg L ^{−1})	2533 ± 289 (66%)	6200 (39.2%)
Turbidity—T (NTU)	217 ± 20 (−758%)	38.8 (48.3%)
Absorbance at 220 nm ^b	1.175 ± 0.032 (−129%)	0.541 (52.4%)
Absorbance at 254 nm ^b	0.461 ± 0.024 (−232%)	0.151 (74.0%)
Absorbance at 292 nm ^b	0.297 ± 0.026 (−316%)	0.083 (80.7%)
Absorbance at 386 nm ^b	0.148 ± 0.067 (−208%)	0.008 (97.2%)
Absorbance at 385 nm ^b	0.147 ± 0.074 (−203%)	0.008 (97.2%)
Absorbance at 410 nm ^b	0.125 ± 0.068 (−197%)	0.005 (98.1%)

Values in parentheses indicate the removal in percentage relative to the initial time.

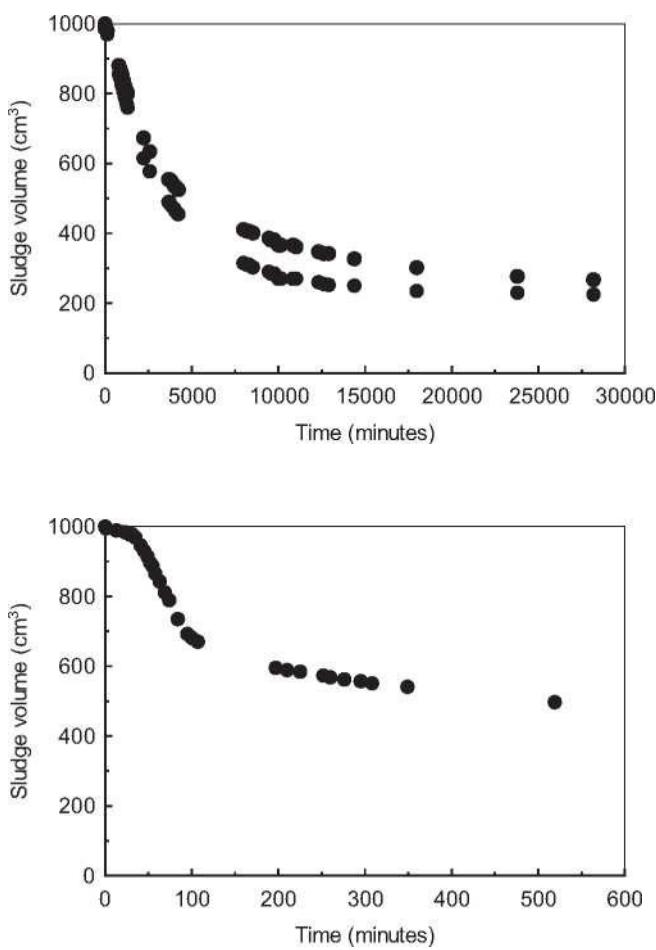
^a Results after 3 replicates.^b 1:25 dilution.

Fig. 7. Sludge compaction coming from NaOH precipitation and sludge sedimentation after aerobic digestion. Top—sludge compaction; bottom—sludge sedimentation.

The sludge compaction coming from the NaOH precipitation and sedimentation profiles after aerobic digestion are shown in Fig. 7. The obtained sludge exhibited zone sedimentation. The calculated compaction and sedimentation velocities were 0.0806 m d^{-1} ($R^2 = 0.999$) and 1.71 m d^{-1} ($R^2 = 0.932$), respectively. Additionally, a sedimentation time of 8.7 h after aerobic digestion was required to find a biosludge volume reduction of about 50%. The compaction time increased for 2.8 days to obtain a similar sludge volume.

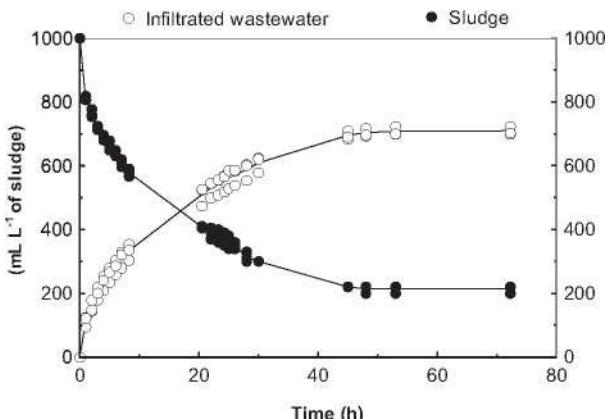


Fig. 8. Sludge filtration through sand beds: infiltrated water volume and sludge volume that remained on the filter.

Fig. 8 illustrates the sludge filtration process through sand filters. This figure indicates a sludge volume reduction of 79% when sand filters were used. The rate of sludge volume reduction in the sand filters was 0.153 m d^{-1} ($R^2 = 0.923$). Infiltrated water was collected at the bottom of the column at a rate of 0.139 m d^{-1} ($R^2 = 0.943$), i.e., similar to the sludge volume reduction rate. The time required for the maximum reduction of the sludge volume using sand filters depends on the sludge water content, filter fouling, filter washing type, etc. In this work, approximately 2 days were appropriate to eliminate the sludge maximum water content by infiltration. Infiltrated water characterization during the sludge filtration through sand beds is presented in Fig. 9. The final infiltrated water constitutes a saline and turbid effluent with COD around 6.4 g L^{-1} . Moreover, the effluent biodegradability decreased. The redox potential reduction during the filtration process can be due to the oxygen consumption in the BOD elimination. This fact suggests that the biofilm formation may occur in the filter composition.

Table 7 shows the effluent characterization obtained in the filtration and aerobic digestion + sedimentation + centrifugation of the sludge. The aerobic digestion + sedimentation + centrifugation allowed an acceptable removal of organic load (32-39%), turbidity (48%) and studied absorbances (52-98%), indicating the suitability of this sequence to manage the sludge generated in the NaOH precipitation.

The volume, dry matter and organic content of the sludge after the implementation of different physicochemical and biological processes are represented in Table 8. The centrifugation is an effective process, since it allowed the sludge

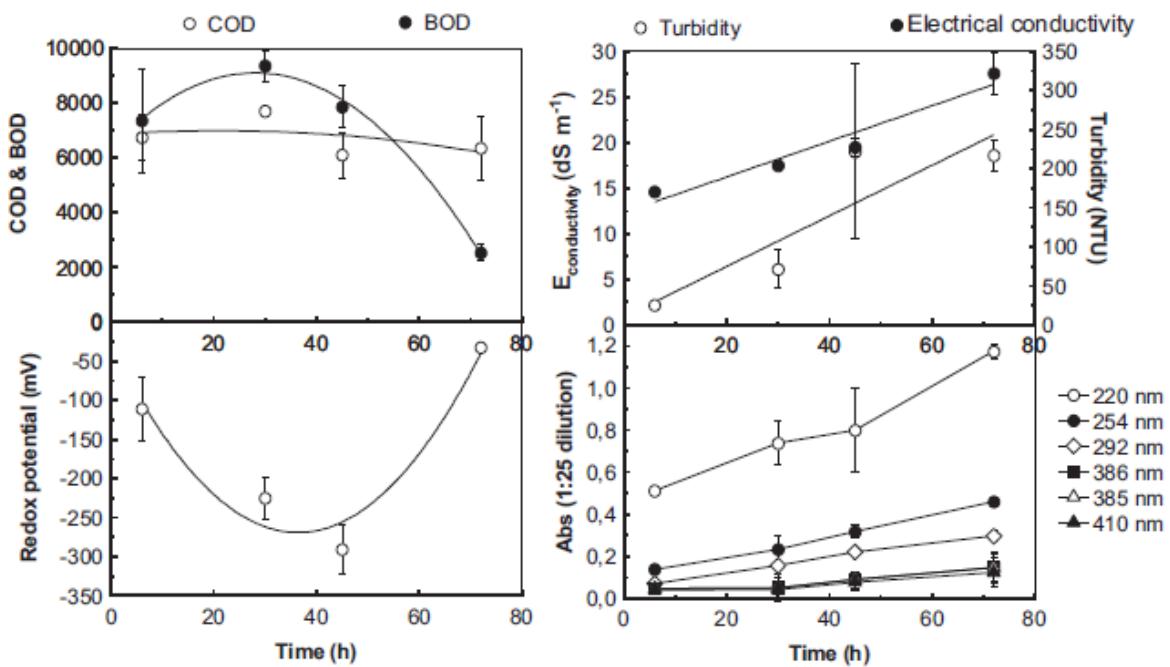


Fig. 9. Infiltrated water characterization during the sludge filtration through sand beds.

Table 8
Sludge characterization after application of different physicochemical and biological processes.

Process type	Sludge volume (mL L^{-1})	Dry matter (%)	Organic content(dry basis) (%)	Organic content(wet basis) (%)
Centrifugation	150 ± 10 (85%)	9.8 ± 0.7	64.3 ± 0.4	6.3 ± 0.4
Compaction	246 ± 30 (75.4%)	3.15 ± 0.48	51.2 ± 10.0	1.66 ± 0.54
Filtration	208 ± 17 (79.2%)	13.2 ± 0.8	24.4 ± 2.4	3.2 ± 0.1
Filtration + evaporation	-	94.8 ± 0.4	25.0 ± 3.1	23.7 ± 2.9
Aerobic digestion + sedimentation	378 (62.2%)	-	-	-
Aerobic digestion + sedimentation + centrifugation	200 (80%)	14.5 (5.1%) ^a	7.7 (8.9%) ^b	-

Values in parentheses indicate the sludge volume reduction or the removal in percentage relative to the initial time.

^a As total suspended solids—TSS (g L^{-1}).

^b As volatile suspended solids—VSS (g L^{-1}).

volume reduction in about 85% with an organic matter content of 643 g kg^{-1} . The sludge volume achieved in the aerobic digestion + sedimentation sequence was approximately 2.5 times the volume obtained in the centrifugation. However, aerobic digestion + sedimentation + centrifugation proved to be the most appropriate sequence, permitting the sludge volume reduction around 80%. Additionally, the obtained supernatant presented a lower organic load.

4. Conclusions

NaOH precipitation is a viable physicochemical process to pretreat CWW. Under optimal conditions, NaOH precipitation produces a clarified effluent, decreasing the organic load in approximately half. High biodegradability of the pretreated CWW indicates that the biological processes can be used as post-treatments. The saline effluent coming from the NaOH precipitation of raw CWW can constitute an option for agricultural irrigation of industrial tomato *Lycopersicon esculentum* Mill., since the treated CWW presents plant growth factors. Several studies have shown a lower production of tomato fruits under salinity conditions, as observed in this work for the cultivar Roma. However, the cultivar Rio Grande presented an increase of the marketable yield and fruit fresh weight up to 21% when irrigated with treated wastewater. This suggests that the cultivar Rio Grande has a higher salt-tolerance than the cultivar Roma and the use of treated CWW can lead to advantages for producers (tomato productivity and quality). The major draw-back of the saline irrigation with treated CWW was related with the production loss due to the physiological disorder of blossom-end rot. However, the production loss owing to the epidermis deformations by solar exposure was minimized for both culti-vars (Roma and Rio Grande). The wet sludge volume obtained in the CWW treatment by NaOH precipitation represented only 6.3% in relation to the wastewater initial volume. The sludge is rich in organic matter, phosphorus and nitrogen, which can be used as a fertilizer in agriculture. Additionally, the generated sludge can be treated by means of centrifugation, compaction, filtration + evaporation (sand filters), aerobic digestion + sedimentation, aerobic digestion + sedimentation + centrifugation, with sludge volume reduction above 60%.

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