



# Optimization of atmospheric carbonation in the integrated treatment immediate one-step lime precipitation and atmospheric carbonation. The case study of slaughterhouse effluents

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## ABSTRACT

Long carbonation time has been a common feature in the integrated process composed by immediate one-step lime precipitation and atmospheric carbonation. This work aims to understand how carbonation time can be influenced by reaction pH, as well as how reactor area/volume ratio affects carbonation time and ammonia removal, using slaughterhouse wastewater due to its variable characteristics. In the integrated immediate one-step lime precipitation and atmospheric carbonation process, the immediate one-step lime precipitation results showed that the reaction pH and the type of slaughterhouse wastewater influenced the removal, however, removals were the highest at reaction pH 12. In atmospheric carbonation process, the carbonation time required to reach pH 8 was independent of the reaction pH used. Additionally, at reaction pH 12, the reactor area/volume ratios applied (from 0 to 155.4 m<sup>2</sup>/m<sup>3</sup>) showed that higher reactor area/volume ratios caused lower carbonation time, but ammonia removal was not affected. For reactor area/volume ratios of 5 and 155.4 m<sup>2</sup>/m<sup>3</sup>, 15 and 1 day were spent to reduce the pH from 11.9 to 8.2, with removals of 71 and 82.6% for NH<sub>4</sub><sup>+</sup> and 10 and 79.1% for calcium, respectively. High removals of total Kjeldahl nitrogen (≥71%), biological oxygen demand (≥80%), ammonium nitrogen (≥52%), total phosphorus (98%), total suspended solids (≥52%), turbidity (≥62%), absorbance at 254 nm (≥87%), absorbance at 410 nm (≥83%) and oils & fats (≥47%) were obtained using immediate one-step lime precipitation and atmospheric carbonation integrated process to treatment slaughterhouse wastewater, indicating that the these process is an efficient pretreatment for slaughterhouse wastewaters.

## 1. Introduction

Although most municipal wastewater treatment plants are capable of removing biodegradable compounds (e.g. organic matter, phosphorus, and nitrogen), they are not designed to remove high concentrations of these compounds, so on-site pre-treatment is required [1,2]. The integrated process, immediate one-step lime precipitation (IOSLM) followed by atmospheric carbonation (AC), has been used as a pretreatment for industrial wastewaters, such as explosives [3], vinasse from sugarcane ethanol industry [4], winery [5], cheese whey [6], urban wastewaters [7], and landfill leachate [8]. This integrated solution is recognized for its low cost, simplicity, eco-friendly technology and high efficiency in removing contaminants, like organic matter and nitrogen (Madeira et al. [3]). However, this treatment process needs to be studied in detail, since

the ability of the atmospheric CO<sub>2</sub> to be transferred to the aqueous phase depends on factors, such as concentration of CO<sub>2</sub> in the atmosphere, area at gas-liquid interface, contact time and solution properties (including pH, temperature, concentration of dissolved salts, and others) (Viswanaathan et al. [9]). Lately, the capture of atmospheric CO<sub>2</sub> has deserved the attention of several researchers [10]. In AC process, the atmospheric CO<sub>2</sub> is transferred to aqueous phase reducing the pH, but long retention times are needed to reduce pH of the effluent [8]. Madeira et al. [3] and Prazeres et al. [4] observed that the AC process in the presence of sludge precipitate (resulting from the IOSLM process) needs a long time to reduce the pH from 12 to 8, but significant advantages in the removal of ammoniacal nitrogen was observed (Madeira et al. [3]). On the other hand, Correia et al. [7] observed that the injection of air (at 85 L/h) to urban effluent treated by IOSLM, in the presence of the sludge IOSLM precipitate, reduced the carbonation time from 220 to 100 h to reduce

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**Abbreviation**

Abs 254	Absorbance at 254 nm
Abs 410	Absorbance at 410 nm
AC	Atmospheric Carbonation
A/V	Area/Volume
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
Cond	Conductivity
DAF	Dissolved Air Flotation
EU	European Union
IOSLM	Immediate One-Step Lime Precipitation
MSTP	Municipal Sewage Treatment Plant
P. Alkalinity	Phenolphthalein Alkalinity

PCA	Principal Component Analysis
pH	Potential Hydrogen
SCOD	Soluble Chemical Oxygen Demand
Sludge V	Sludge Volume
S. Turbidity	Soluble Turbidity
SWPP	On-Site Pretreatment Plant
SWW	Slaughterhouse Wastewater
T. Alkalinity	Total Alkalinity
TKN	Total Kjeldahl Nitrogen
TN	Total Nitrogen
TP	Total Phosphorus
TSS	Total Suspended Solids
Turb	Turbidity
UV/Vis	Ultraviolet Visible Radiation

the pH from 11.5 to 8, but its effect on ammonia removal was not analyzed. Even so, aeration systems have been recognized as a significant energy cost in wastewater treatment plants [11,12]. In addition, since the transfer of the CO<sub>2</sub> to water must be maximised, the reactor area/volume (A/V) ratio in the AC process is very important. According to the literature, low reactor A/V ratios such as 3.78 m<sup>2</sup>/m<sup>3</sup> [3], 6.23 m<sup>2</sup>/m<sup>3</sup> [4], 4.63 m<sup>2</sup>/m<sup>3</sup> [6], 5.00 m<sup>2</sup>/m<sup>3</sup> [7], and 5.00–6.67 m<sup>2</sup>/m<sup>3</sup> [8] have been used in the AC process. However, it is not possible to establish any relationship between these reactors A/V ratios and the carbonation time, as the characteristics of effluent (e.g. pH, temperature, nitrogen and organic contents) and experimental conditions were different among the various authors. Therefore, the effect of high reactor A/V ratios on carbonation time of AC process and hence its effect on ammonia removal is unknown.

This work aims to fill these gaps in the integrated IOSLM and AC process, namely evaluate the effect of the reaction pH and reactor A/V ratio on the carbonation time and ammonium nitrogen removal. Slaughterhouse wastewater was used as a case study, given that current slaughterhouse coagulation pre-treatment systems use coagulants (such as ferric chloride or ferric sulfate) [13] that do not remove ammonium nitrogen [14]. Thus, the subsequent treatment processes, such as biological treatments (e.g. activated sludge) or constructed wetlands, need alternating aerobic and anoxic phases and consequently a longer hydraulic retention time, to effectively remove ammonium nitrogen and organic matter, as heterotrophic and nitrifying bacteria compete for available oxygen [15]. This effluent is recognized for the great variability in its composition (for organic matter, nitrogen and total phosphorus (TP), and total suspended solids (TSS)) due to the number and type of animal slaughtered, and water consumption [13,16,17]. Lime precipitation has been applied by several authors in the slaughterhouse wastewater treatment [18–21], but this treatment has never been integrated with the AC process, neither the impact of different slaughterhouse wastewater characteristics. In this way, this work also intends to know if the reaction pH can be a determining factor in the treatment of slaughterhouse wastewater with variable characteristics.

## 2. Materials and methods

### 2.1. Slaughterhouse wastewater sampling

Slaughterhouse wastewater (SWW) used in this study is a Portuguese on-site wastewater that resulted from the slaughtering process (cattle, pigs, goats, sheep, and ratite) and the cleaning facilities. The slaughterhouse wastewater is pretreated in an on-site pretreatment plant (SWPP), after which is discharged to the municipal sewage treatment plant (MSTP). Three slaughterhouse wastewaters coming from different SWPP stages were studied: SWW1 was collected in the aeration and homogenization tank, SWW2 was collected at the output of the rotary

drum screen filter, and SWW3 was collected at the output of the flocculator/dissolved air flotation (DAF) system. All samples were collected during the working hours and only once. If not immediately analyzed, the collected samples were stored at 4 °C until use. Table 1 shows the SWW1, SWW2 and SWW3 physicochemical characterization.

### 2.2. Experimental set-up

The experimental set-up used in this work is presented in Fig. 1, which includes the IOSLM followed by AC process.

#### 2.2.1. Immediate one-step lime precipitation (IOSLM)

IOSLM process is already described in Madeira et al. [3]. Various hydrated lime concentrations were added to pretreated wastewaters SWW1, SWW2 and SWW3, drop by drop, under vigorous agitation (magnetic stirrer at speed of 3 s<sup>-1</sup>) and agitation was maintained until precipitation pH reached, from 9.5 to 12 (called in this work reaction

**Table 1**

– Physicochemical characterization of SWW1, SWW2 and SWW3 effluents.

Parameters	Unit	SWW1	SWW2	SWW3
pH	Sorensen	7.2 ± 0.1	7.2 ± 0.2	7.5 ± 0.1
Conductivity	mS cm <sup>-1</sup>	3.1 ± 0.3	3.0 ± 0.1	3.0 ± 0.1
COD	mg O <sub>2</sub> L <sup>-1</sup>	5430 ± 1646	1810 ± 274	226 ± 41
SCOD	mg O <sub>2</sub> L <sup>-1</sup>	172 ± 38	450 ± 47	183 ± 2
BOD <sub>5</sub>	mg O <sub>2</sub> L <sup>-1</sup>	2813 ± 208	1013 ± 29	40 ± 0
BOD <sub>5</sub> /COD	–	0.52 ± 0.16	0.56 ± 0.09	0.18 ± 0.03
TKN	mg N-Kj. L <sup>-1</sup>	212 ± 2	97 ± 2	58 ± 0
COD/TKN	–	25.6	18.7	3.9
NH <sub>4</sub> <sup>+</sup>	mg N L <sup>-1</sup>	88 ± 1	69 ± 0	52 ± 0
Organic-N	mg N L <sup>-1</sup>	124 ± 2	28 ± 2	6 ± 0
TP	mg L <sup>-1</sup>	491.6 ± 89.6	255.6 ± 90.1	147.6 ± 12.0
TSS	mg L <sup>-1</sup>	3703 ± 10	848 ± 17	61 ± 7
Turbidity	NTU	1193 ± 84	469 ± 42	17 ± 6
STurbidity	NTU	24 ± 1	38 ± 1	11 ± 1
Abs. at 254 nm	cm <sup>-1</sup>	1.659 ± 0.298	0.884 ± 0.150	0.198 ± 0.040
Abs. at 410 nm	cm <sup>-1</sup>	2.072 ± 0.295	1.471 ± 0.296	0.314 ± 0.099
Oils & Fats	mg L <sup>-1</sup>	1736 ± 170	376 ± 96	86 ± 8
P. Alkalinity	mg CaCO <sub>3</sub> L <sup>-1</sup>	0 ± 0	0 ± 0	0 ± 0
T. Alkalinity	mg CaCO <sub>3</sub> L <sup>-1</sup>	560 ± 16	675 ± 16	583 ± 16
Calcium	mg L <sup>-1</sup>	101.2 ± 6.0	158.7 ± 6.9	190.5 ± 0.0
Magnesium	mg L <sup>-1</sup>	54.1 ± 3.6	74.6 ± 4.2	60.2 ± 4.2
Color	–	dark brown	dark brown	yellowish
Odor	–	unpleasant	unpleasant	slight smell

Note: COD - chemical oxygen demand; SCOD - soluble chemical oxygen demand total; BOD - biological oxygen demand; TKN - total Kjeldahl nitrogen; TP - total phosphorus; TSS - total suspended solids; STurbidity - soluble turbidity; P. Alkalinity - phenolphthalein alkalinity; T. Alkalinity - total alkalinity.

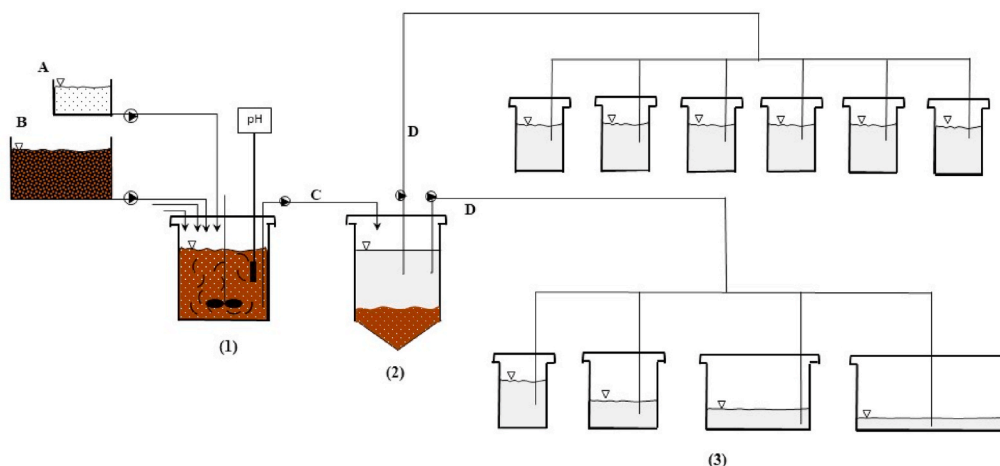


Fig. 1. Schematic diagram of the treatment processes used in this work, namely: (1) IOSLM process, (2) sedimentation process and (3) AC process. A – hydrated lime tank, B–SWW tank, C – IOSLM effluent, D – supernatant.

pH). The agitation was kept for 1 min and then stopped, and the effluent was transferred to sedimentation tank (without agitation) for 1 h. After that, the physicochemical characteristics of supernatant were analyzed (section 2.3) and the effluent passed to AC process. Calcium hydroxide ( $\geq 95\%$ , PanReac S.A) was used to prepare the hydrated lime solution at a concentration of  $200 \text{ g L}^{-1}$ .

#### 2.2.2. Atmospheric $\text{CO}_2$ carbonation (AC) tests

In all these experiments, 1–2 L of IOSLM supernatants from SWW1, SWW2 and SWW3 were tested and applied to AC reactors. Three AC tests were made. In the first set of experiments, the carbonation time was studied for the different IOSLM supernatants and pH (from 9.5 to 12), and were tested in AC reactors with  $9.5 \text{ m}^2/\text{m}^3$  of A/V ratio for SWW1 supernatant, and  $5 \text{ m}^2/\text{m}^3$  for SWW2 and SWW3 supernatants. In the second set, the supernatant pHs were maintained in 12 and the carbonation time was studied for different reactors area/volume (A/V) ratios, from 0 to  $155.4 \text{ m}^2/\text{m}^3$ . Finally, in the third experimental set, the best conditions were studied to maximize ammonium removal. In all experiments, the IOSLM supernatants were kept in contact with atmospheric air without any chemical and agitation. Process efficiency was monitored (section 2.3) over time, once a day, until the supernatant pH was close to 8 or kept constant over time. All AC tests were at room temperature  $23 \pm 2 \text{ }^\circ\text{C}$ .

Subsequently, to estimate the amount of atmospheric  $\text{CO}_2$  captured during the carbonation process, the IOSLM supernatant at pH 12 was neutralized with different volumes of pure  $\text{CO}_2$  from a  $\text{CO}_2$  cylinder (CEVIK brand), and the pH obtained was recorded.  $\text{CO}_2$  was quantified by Stevin's law and the ideal gas law, applying the sample in a U-Tube attached to a  $\text{CO}_2$  bottle and subsequent vigorous mixing between the added  $\text{CO}_2$  gas and the sample.

#### 2.3. Analytical methods

The parameters and methods used are shown in Table 2. Chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN), ammonium nitrogen ( $\text{NH}_4^+$ ), total phosphorus (TP), total suspended solids (TSS), oils and fats, biological oxygen demand (BOD), calcium, magnesium, phenolphthalein alkalinity, total alkalinity and turbidity were determined according to Standard Methods [22]. Absorbance at 410 nm indicates the presence of compounds responsible for color, while absorbance at 254 nm indicates the presence of high molecular weight organic compounds with high degree of aromaticity, high number of double and triple bonds, and phenolic groups [23].

#### 2.4. Statistical analysis

All samples and experimental treatments were analyzed in triplicate, being the results presented as means  $\pm$  standard deviation. For comparison between averages, the data were submitted to One-way ANOVA with Tukey's test at 95% confidence level, using IBM SPSS Statistics for Windows. Principal component analysis (PCA) and correlation analysis were performed using the XLSTAT 2021 statistical software. GraphPad Prism for Windows (version 5.0, GraphPad Software, La Jolla California USA) was used to draw the graphs.

In this work, the equipment used for parameter measurement were associated with the following accuracy values:  $\pm 0.01$  for WTW InoLab pH Level 1 apparatus; 0.5% for Jenway 4510 conductivity meter;  $\pm 1 \text{ nm}$  for UV/Vis spectrophotometer Pharmacia Biotech Ultrospec 2000;  $\pm 1\%$  of value for WTW OxiTop® IS 12 system; and  $\pm 2\%$  of readings plus 0.01 NTU from 0 to 1000 NTU for HACH 2100 N turbidimeter.

### 3. Results and discussion

#### 3.1. Physicochemical characterization of SWW

SWW physicochemical characteristics are shown in Table 1. Briefly, the COD concentrations vary according with the collected place at SWPP, being the highest COD concentrations for SWW1 ( $5430 \pm 1646 \text{ mg O}_2 \text{ L}^{-1}$ ) and the lowest for SWW3 ( $226 \pm 41 \text{ mg O}_2 \text{ L}^{-1}$ ). SWW1 and SWW2 organic matter is in insoluble form, while SWW3 organic matter is practically soluble. The biodegradability index ( $\text{BOD}_5/\text{COD}$  ratio) of SWW1 and SWW2 is around 0.5, similar to Al Smadi et al. [16]; which means that these effluents have moderate biodegradability. SWW3 has a low  $\text{BOD}_5/\text{COD}$  ratio (around 0.18), which according to Dinçer [24] it is not possible to treat using conventional biological treatments. For nitrogen, all SWW present considerable values of TKN ( $58\text{--}212 \text{ mg TKN L}^{-1}$ ), either in the form of ammonia ( $52\text{--}88 \text{ mg N-NH}_4^+ \text{ L}^{-1}$ ) or organic nitrogen ( $6\text{--}124 \text{ mg N L}^{-1}$ ), as well as are rich in phosphorus ( $148\text{--}492 \text{ mg L}^{-1}$ ). Turbidity values are high in SWW1 ( $1193 \pm 84 \text{ NTU}$ ) and SWW2 ( $469 \pm 42 \text{ NTU}$ ), and much lower in SWW3 ( $17 \pm 6 \text{ NTU}$ ), which is excepted since this wastewater was collected after flocculator/DAF processes. The presence of aromatic and unsaturated compounds given by absorbance at 254 nm (Table 1) is common in SWW due to the use of pharmaceutical drugs [25,26]. Other SWW characteristics can be seen in Table 1.

#### 3.2. Immediate one-step lime precipitation (IOSLM)

The supernatant quality and removals after IOSLM are presented in

**Table 2**  
– The parameters analyzed and analytical methods used.

Parameters	Method	Equipment
pH	Potentiometric method	WTW InoLab pH Level 1 apparatus and a pH electrode SenTix® 41
Conductivity	Electrometric method	Jenway 4510 conductivity meter and a conductivity sensor VWR phenomenal CO 11
COD and SCOD	Closed reflux colorimetric method	COD digester WPA Hydrocheck HC 6016, UV/Vis spectrophotometer Pharmacia Biotech Ultrospec 2000; 0.45-µm pore-size filter for soluble COD.
BOD <sub>5</sub>	Respirometric method	WTW OxiTop® IS 12 system
TKN	Kjeldahl method	Digester Bloc Digest 6 P-Selecta; distillation unit BUCHI B-316
NH <sub>4</sub> <sup>+</sup>	Distillation method	Distillation unit BUCHI B-316
TP	Vanadomolybdophosphoric acid colorimetric method	Muffle P SELECTA-HORN 186331, UV/Vis spectrophotometer Pharmacia Biotech Ultrospec 2000
TSS	Gravimetric method	Whatman® glass microfiber filters (Grade 934-AH®)
Turbidity and STurbidity	Nephelometric method	HACH 2100 N turbidimeter; 0.45-µm pore-size filter for soluble turbidity.
Abs. at 254 nm	Spectroscopic method	UV/Vis spectrophotometer Pharmacia Biotech Ultrospec 2000
Abs. at 410 nm	Spectroscopic method	UV/Vis spectrophotometer Pharmacia Biotech Ultrospec 2000
Oils & Fats	Gravimetric method	Soxhlet extractor with petroleum ether and Soxhlet heating mantles electrothermal EM O250/CE
P. Alkalinity	Neutralization titration	
T. Alkalinity	Neutralization titration	
Calcium	Volumetric complexation with EDTA	
Magnesium	Difference between total hardness and calcium hardness. Total hardness determined by Volumetric complexation with EDTA.	

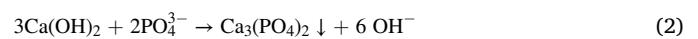
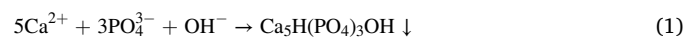
Note: P. Alkalinity - phenolphthalein alkalinity; T. Alkalinity - total alkalinity; SCOD – soluble COD; STurbidity – soluble turbidity.

Figs. 2 and 3. For TKN, NH<sub>4</sub><sup>+</sup>, organic-N and COD removals practically did not vary with reaction pH for all the studied waters. After the IOSLM, the supernatants quality was very similar between reaction pHs in each SWW but different between SWW, because the initial concentrations of these parameters were different between SWW (Fig. 2). Thus, for these parameters, the final supernatant quality is not dependent of reaction pH, and it seems that lower pH may be used in IOSLM process (pH of 9.5). This is very important in sludge production and consumption (see Fig. S1 in supplementary information).

The TKN removals were essentially due to the removal of organic nitrogen, since the ammonium nitrogen removals were insignificant ( $P < 0.05$ ). Although the studied pH range is greater than 9.3 (pKa of the reaction between NH<sub>3</sub> and NH<sub>4</sub><sup>+</sup> is 9.26, so at higher pH than 9.26 the ammoniacal nitrogen is in the non-ionized form (NH<sub>3</sub>) and susceptible to being volatilized [27]), the removal of ammonia was negligible since the

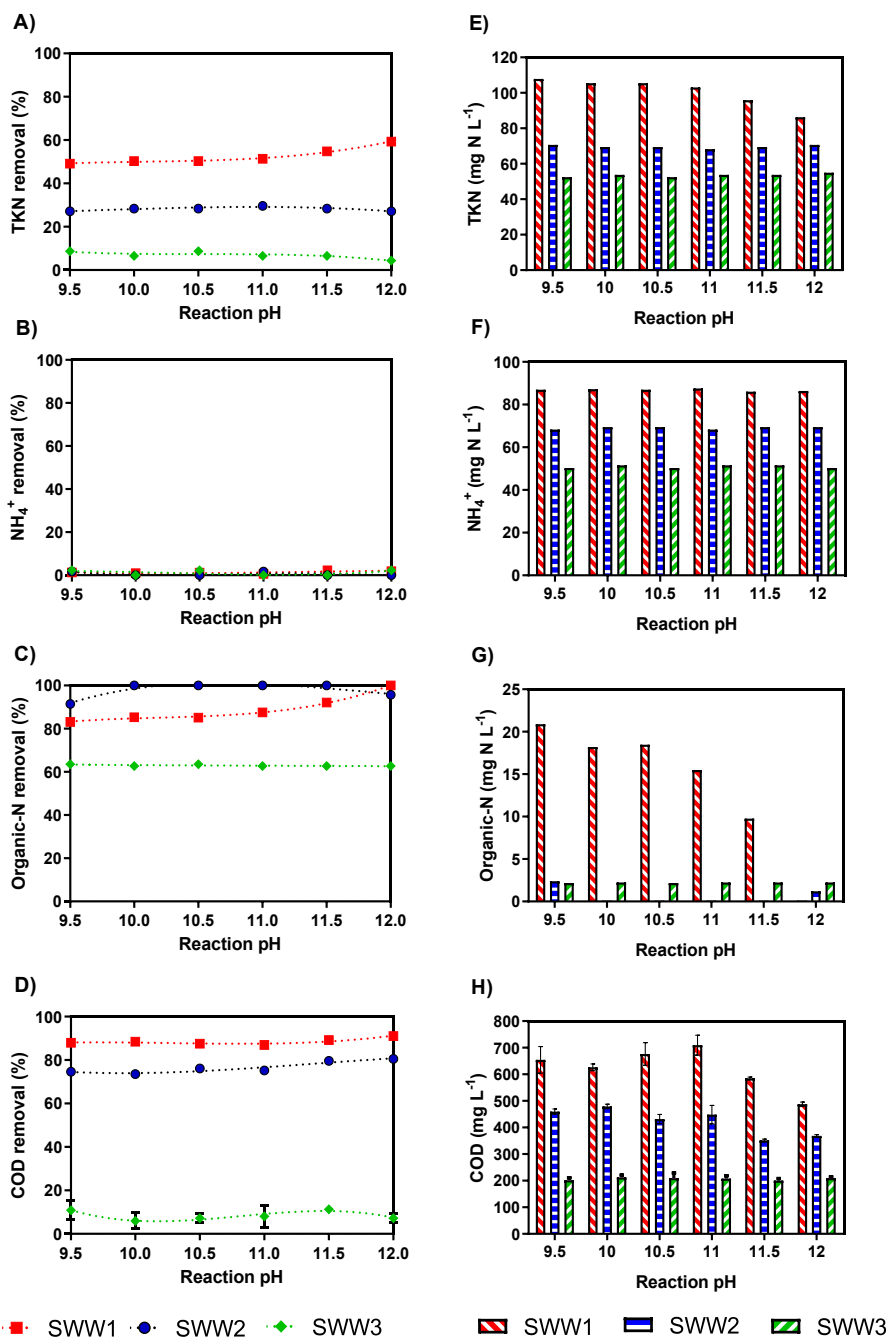
IOSLM process was quick. The IOSLM process was not very effective in removing COD for SWW3 (6.0–11.3%, Fig. 2d), which indicates that this effluent presents non-precipitable organic matter (in addition to being non-biodegradable). In this way, this effluent can only be treated by unconventional treatment processes (e.g., advanced oxidation processes or adsorption processes). Low COD removals in the lime precipitation process has also been found in the treatment of winery wastewaters [5] and landfill leachate [8]. On the other hand, high COD removals were obtained for SWW1 (86.9–91.0%) and SWW2 (73.5–80.6%), similar to those obtained by Tariq et al. [21]. These removals are justified by the addition of lime in excess to wastewater which results in the destabilization and aggregation of charged particles, as well as the formation of an intense and spontaneous precipitate [3]. This in addition to precipitate adsorptive capacity, contributes to a vigorous “sweeping” of suspended impurities and organic matters [3,28]. Moreover, the removal of some organic matter is also due to the sedimentation operation since these effluents present easily sedimentable organic matter, as observed by Satyanarayan et al. [20].

For absorbances at 254 and 410 nm, turbidity and TP, the removals increased with reaction pH and the supernatant quality was much better for pH 12 (Fig. 3) for all the studied waters. In fact, it is at high pH that larger precipitates of CaCO<sub>3</sub>, MgCO<sub>3</sub> and Mg(OH)<sub>2</sub> are formed, as a result of the reaction between lime and different chemical species (such as carbonic acid, calcium bicarbonate, magnesium bicarbonate, magnesium carbonate) present in SWW [3]. These precipitates have a high adsorption capacity for some organic compounds [29–31] and phosphorus [32]. According to Zhao et al. [33], the Mg(OH)<sub>2</sub> precipitate can neutralize charge, cause enmeshment of colloidal particles, beyond to its adsorptive capacity. In fact, the magnesium concentration was reduced in the entire reaction pH range and for all effluents (Fig. 4B). The observed decrease in turbidity appears to be related to the removal of absorbances at 254 nm and 410 nm, and COD, since strong and positive correlations were found between these parameters and turbidity, for all SWW types (Table S1). On the other hand, TP had a negative correlation with calcium, which varies between strong ( $r = -0.63$ , for SWW1 and  $r = -0.66$  for SWW2) and weak ( $r = -0.24$  for SWW3) correlation (Table S1). In fact, phosphorus removals involve also large amounts of soluble calcium and alkaline conditions (pH > 9) [34], being associated with the formation of hydroxyapatite precipitate (Eq. (1)), calcium phosphate (Eq. (2)) and octacalcium phosphate (Eq. (3)) [35].



Despite the reduction in contamination observed for the pH range studied, the calcium was not completely consumed in the IOSLM process, for all SWW (Fig. 4a), which may be important in the following AC process. On the contrary, the calcium that is present seems to be responsible for the conductivity since high and positive correlation were found, for all SWW (Table S1). As a result of the IOSLM process, only SWW1 (pH ≥ 11.5) showed a higher conductivity than it had initially (Fig. S1).

Finally, a PCA was applied for the IOSLM results (Fig. S2). The first principal component separates the three SWW treated at different reaction pH, showing greater contamination of SWW1, followed by SWW2 and then SWW3. For the three treated SWW, the increase in reaction pH decreases the concentration of several pollutants, although this decrease was more noticeable for treated SWW1, followed by SWW2 and finally SWW3. This means that the application of the same reaction pH did not guarantee the same effluent quality for the three SWW. Thus, the reaction pH is not a key parameter for slaughterhouse water treatment, which will depend on the characteristics of the SWW to be treated. However, at reaction pH 12 the quality of the three treated SWWs is better for the studied parameters than raw SWWs. SWW1 had higher



**Fig. 2.** Effect of the reaction pH in IOSLM tests on some parameters removal efficiency: a) TKN, b) ammonium nitrogen, c) organic-N and d) COD, and on the effluent quality: e) TKN concentration, f) ammonium nitrogen concentration, g) organic-N concentration and h) COD concentration, for SWW1, SWW2 and SWW3. Bars represent standard deviation of the mean.

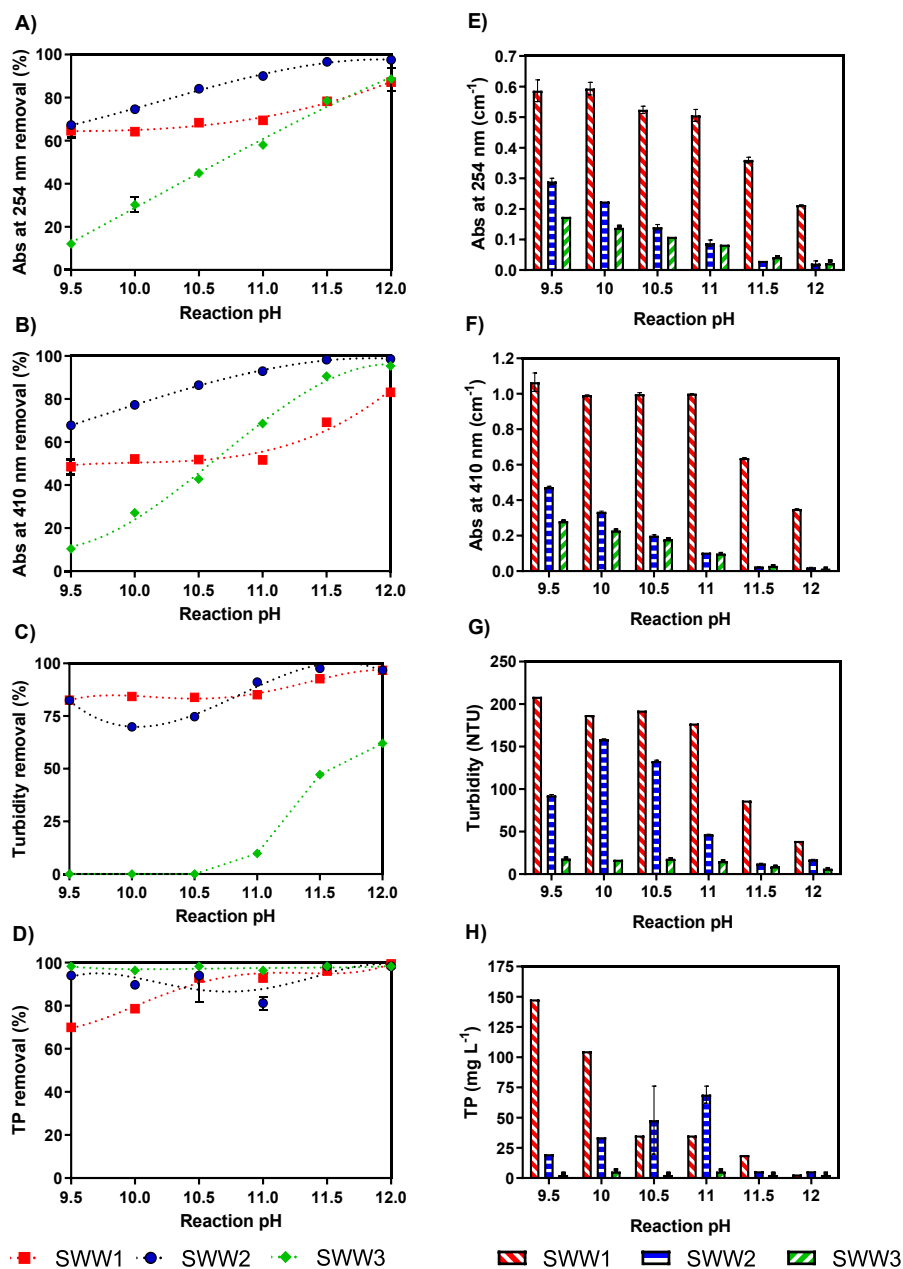
lime consumption and sludge production than SWW2 or SWW3, and SWW2 higher than SWW3, justified by the different initial contaminations (Fig. S1). This lime consumption (which ranged from 0.46 to 1.33 g L<sup>-1</sup>) was lower than that found in the treatment of explosive effluents (about 6–12 g L<sup>-1</sup>) [3], for the same range of reaction pH (9.5–12), and can be justified by the differences in ammonium nitrogen concentrations existing in these effluents (about 52–88 mg N L<sup>-1</sup> for SWW and 1554 mg N L<sup>-1</sup> for explosive wastewater). In general, the supernatant pH values were close to the reaction pH values, for the three SWW types (Fig. S1).

### 3.3. Atmospheric CO<sub>2</sub> carbonation (AC)

#### 3.3.1. Effect of the reaction pH

Fig. 5 shows the variation of the supernatant quality with carbonation time and reaction pH in the AC process, for the SWW1, SWW2 and SWW3. According to Fig. 5A, the supernatant pH decreased linearly with time for all reaction pH studied, excepted for pH 12. At this pH, the supernatant pH slightly decreased in the first seven days for all SWW, after which a sudden drop in supernatant pH was observed. This behavior could be related to the buffer capacity of the supernatant, that is, the ability of the supernatant to neutralize (through its alkalinity) the protons that were meanwhile released [36]. Similar behavior was observed by Luz et al. [5] when the authors applied AC process to





**Fig. 3.** Effect of the reaction pH in IOSLM tests on some parameters removal efficiency: a) absorbance at 254 nm, b) absorbance at 410 nm, c) turbidity, and d) TP), and on the effluent quality: e) absorbance at 254 nm, f) absorbance at 410 nm, g) turbidity, and h) TP concentration, for SWW1, SWW2 and SWW3. Bars represent standard deviation of the mean.

pretreated winery wastewaters at reaction pH 12. At 9th day (for SWW1), 10th day (for SWW2) and 12th day (for SWW3), all the reaction pH had reached the same supernatant pH. The decrease in supernatant pH over time was due to the reaction of the supernatant with atmospheric  $\text{CO}_2$  [3]. Very strong and significant ( $P < 0.05$ ) negative correlations were observed between supernatant pH and time, SWW1 ( $r \geq -0.95$ , Table S2), SWW2 ( $r \geq -0.92$ , Table S2) and SWW3 ( $r \geq -0.90$ , Table S3), at different reaction pH (9.5–12).

No significant variation in conductivity with time was observed for the studied reaction pH, being the final values of the conductivity very close to the initial ones before the integrated treatment processes by IOSLM + AC (Fig. 5B).

Calcium concentrations decreased in the first 10 days, after which remain practically unchanged, despite the concentration of the wastewaters after IOSLM process (Fig. 5C). Thus, the AC process is efficient in

removing calcium added in the IOSLM process. On the 10th day for SWW1 and 15th day for SWW2 and SWW3, the calcium concentrations were below the initial values (ca.  $101 \text{ mg L}^{-1}$  for SWW1,  $159 \text{ mg L}^{-1}$  for SWW2 and ca.  $191 \text{ mg L}^{-1}$  for SWW3, Table 1) before applying the IOSLM process. Despite the high calcium removal, the calcium was not a limiting factor in the AC process to drop pH to neutral values, as was observed by Ramalho et al. [8]. Ramalho et al. [8] observed that the calcium concentration was totally consumed during the AC process, leading to a decrease in pH from only 12.5 to 10.1, after 32 days of AC. The calcium removal is justified by the formation of calcium carbonate (Eq. (4)) since the carbonate ions are formed by dissolving atmospheric  $\text{CO}_2$  in water and reducing pH [6] (a strong positive correlation between calcium and supernatant pH,  $r \geq 0.46$ ,  $P < 0.05$ , for SWW1 (Table S2),  $r \geq 0.70$ ,  $P < 0.05$ , for SWW2 (Table S3) and  $r \geq 0.53$ ,  $P < 0.05$ , for SWW3 (Table S4)).

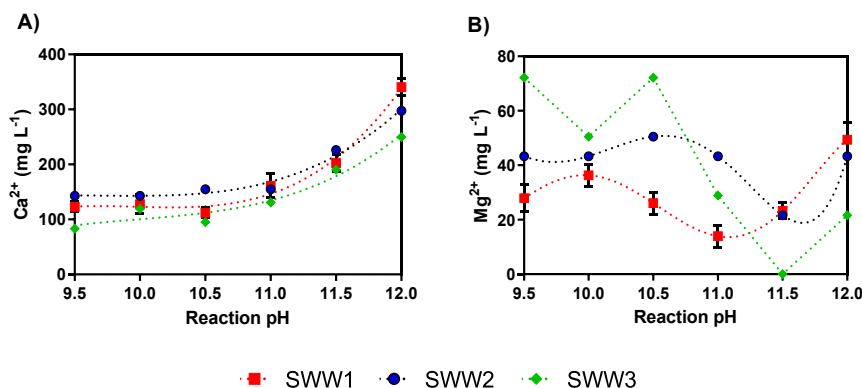


Fig. 4. Effect of the reaction pH in IOSLM tests on the: a) calcium concentration and b) magnesium concentration, for SWW1, SWW2 and SWW3. Bars represent one standard deviation of the mean.



Regarding the magnesium (Fig. 5D), its concentration varied considerably over time for all SWW, regardless of the reaction pH. In this way, there is no significant correlation ( $P < 0.05$ ) between magnesium and time, for reaction pHs and waters (Tables S2, S3 and S4). This variation may be due to the simultaneous occurrence of a set of reactions, namely the formation of magnesium carbonate (Eq. (5)), dissolution reactions of magnesium precipitates (Eq. (6)) and evaporation phenomena.



Overall, the AC process proved to be effective in removing aromatic and unsaturated compounds (Fig. 5E) and compounds responsible for color (Fig. 5F). It is expected that the sedimentability of small particles will still occur during the first days of the carbonation process. The decrease in absorbances (at 254 and 410 nm) may also be associated with the interaction of organic compounds with the precipitates formed during AC process, through adsorption processes since some positive correlations were found between absorbances and calcium or magnesium (Tables S2, S3 and S4).

A decrease in the concentration of nitrogen ammonium over time was observed, for all reaction pH and waters (Fig. 5G). In fact, very strong negative correlations were observed for SWW1 ( $-0.79 \leq r \leq -0.86$ , Table S2), SWW2 ( $-0.96 \leq r \leq -0.98$ , Table S3) and SWW3 ( $-0.96 \leq r \leq -0.98$ , Table S4). At the end of AC process, about 93–96% of ammonium nitrogen were removed in SWW1 for reaction pH 11.5 and 12, while 70.7 and 71.4%, respectively, in SWW2 and SWW3 at the reaction pH 12. It is observed that, over time, the decrease of ammonium nitrogen concentration is hampered due to the decrease in pH (Fig. 5A). In fact, since the supernatant pH decreases over time, ammonia tends to be converted to ammonium nitrogen, while the other part of the ammonia is available to be removed [37]. Ammonium nitrogen is not removed from the supernatant by volatilization [38]. In this way, higher reaction pHs provide greater removal of ammonia during the carbonation process, as can be seen in Fig. 5G. Ramalho et al. [8] obtained 100% ammonia removal during the carbonation process of a leachate effluent, as the pH decrease was slower over time due to calcium limitation, using an A/V ratio of 5–6.7  $\text{m}^2/\text{m}^3$ . These authors also observed the presence of nitrogen in the sludge resulting from the carbonation process, thus indicating that in addition to volatilization, it is also possible to have some adsorption of ammonia nitrogen during this process.

Finally, after 15 days, the best quality for the tested parameters was observed at reaction pH of 12 for all the studied supernatants and with

the final supernatant pH was 8 (Fig. 5). Therefore, independently of the water characteristics, it is possible to use AC process after the IOSLM to decrease IOSLM supernatants pH and remove ammonia.

### 3.3.2. Effect of the reactor A/V ratio

Fig. 6 shows the variation of supernatant pH with experimental time in AC process, for different reactor A/V ratios (0–155.4  $\text{m}^2/\text{m}^3$ ), for SWW1 (Fig. 6a), SWW2 (Fig. 6b) and SWW3 supernatants (Fig. 6c), at reaction pH 12. These results show that below a/v ratio (A/V ratio of 9.5  $\text{m}^2/\text{m}^3$  for SWW1, and A/V ratio of 3  $\text{m}^2/\text{m}^3$  for SWW2 and SWW3, Fig. 6), the AC process was not efficient to decrease the supernatant pH to values that allow its discharge or reuse, requiring more time (>7 days). However, for SWW2 and SWW3 at A/V ratio of 19  $\text{m}^2/\text{m}^3$ , 7 days were enough to put the supernatant pH at 8 and using high A/V ratio ( $\geq 52.4 \text{ m}^2/\text{m}^3$ ) 2 days were enough. For the highest A/V ratio 155.4  $\text{m}^2/\text{m}^3$  a more detail analysis was made (Fig. 6B) showing that the supernatant pH was 9.5 after 8 h and 8 after 24 h.

A decrease in the supernatant pH from 12 to 8 during AC process was obtained by Prazeres et al. [4] and Prazeres et al. [6]; using a reactor A/V ratio of 6.23  $\text{m}^2/\text{m}^3$  (for 10 days with vinasse effluent) and 4.63  $\text{m}^2/\text{m}^3$  (for 8 days with cheese whey wastewaters). However, longer carbonation times (about 13 days) were achieved in this work using similar A/V ratios (5  $\text{m}^2/\text{m}^3$ ). This means that the A/V ratio is not the only factor determining the pH drop. In fact, others factors (e.g., concentration of  $\text{CO}_2$  in the atmosphere during the experiments, as discussed below) can change the ability of the atmospheric  $\text{CO}_2$  to be transferred to the aqueous phase [9], and justify these differences in carbonation times. Results of SWW2 supernatant using A/V ratios of 5 and 155.4  $\text{m}^2/\text{m}^3$  to reach the pH 8 (13 days and 1 day for 5 and 155.4  $\text{m}^2/\text{m}^3$ , respectively) showed higher  $\text{NH}_4^+$  removals for the highest A/V ratio (82% for 155.4  $\text{m}^2/\text{m}^3$  compared 68% for 5  $\text{m}^2/\text{m}^3$ ) (Table 3). This means that despite the rapid decrease in pH,  $\text{NH}_4^+$  removal is not hampered, on the contrary, it allows greater volatilization due to its larger area of exposure. Thus, higher reactor A/V ratio did not decrease the  $\text{NH}_4^+$  removal of the system.

The impact of  $\text{CO}_2$  concentration was analyzed. The amount of  $\text{CO}_2$  that would be necessary to reduce the pH of 12 to 8 during AC process was estimated by adding  $\text{CO}_2$  and by reaction stoichiometry, for all waters studied. About 0.48 g of  $\text{CO}_2 \text{ L}^{-1}$  was necessary to reduce the pH from 12 to 8, for the three SWWs (Fig. 7), while lower quantities about 0.35, 0.24 and 0.22 g  $\text{CO}_2 \text{ L}^{-1}$  were estimated by reaction stoichiometry, for SWW1, SWW2 and SWW3, respectively. This same amount of  $\text{CO}_2$  injected for all SWW is justified by the similarity of the initial values of phenolphthalein alkalinity (about  $480 \pm 40$ ,  $549 \pm 40$  and  $556 \pm 35 \text{ mg L}^{-1} \text{ CaCO}_3$ , for SWW1, SWW2 and SWW3, respectively), total alkalinity (about  $709 \pm 79$ ,  $694 \pm 87$  and  $610 \pm 13 \text{ mg L}^{-1} \text{ CaCO}_3$ , for SWW1, SWW2 and SWW3, respectively), and pH. However, these  $\text{CO}_2$  quantities

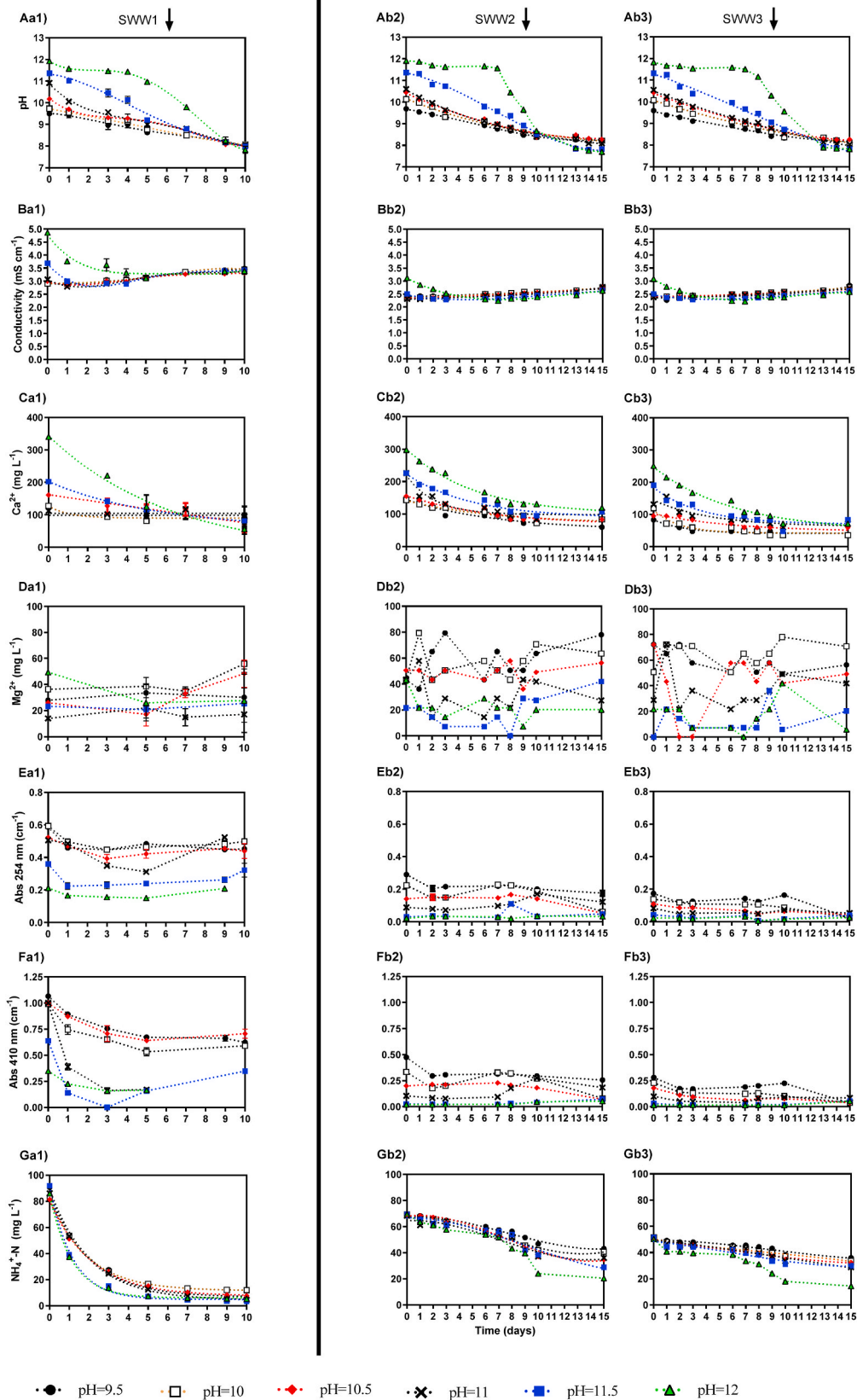


Fig. 5. Variation of: A) supernatant pH; B) electrical conductivity; C) calcium concentration; D) magnesium concentration; E) absorbance at 254 nm; F) absorbance at 410 nm, and G) ammonium nitrogen concentration, over experimental time at different reaction pH (9.5 and 12), in AC process. Numbers 1, 2 and 3 mean SWW1, SWW2 and SWW3, respectively. Lower case letters a and b mean the A/V ratio, for 9.5 m<sup>2</sup>/m<sup>3</sup> was used for SWW1 and b for 5 m<sup>2</sup>/m<sup>3</sup> was used for SWW2 and SWW3 pretreated by IOSLM. Bars represent one standard deviation of the mean.



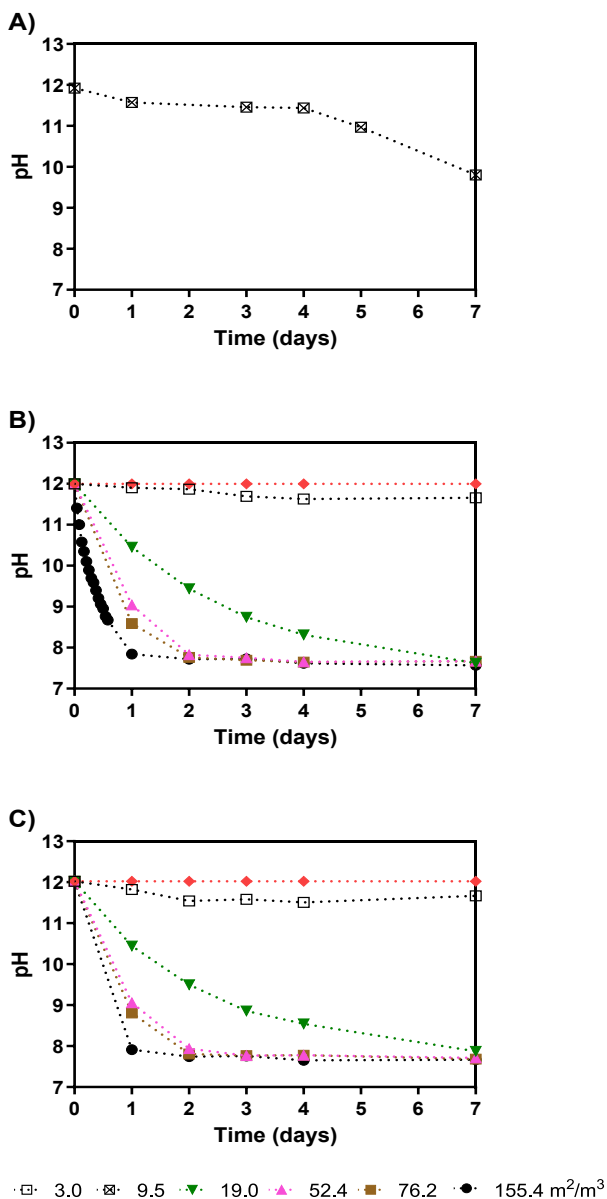
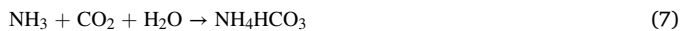


Fig. 6. Variation of the supernatant pH over time in AC process, using: a) SWW 1, b) SWW 2 and c) SWW 3 supernatants, all pretreated by IOSLM process (at pH 12), for different reactor A/V ratios (0–155.4 m<sup>2</sup>/m<sup>3</sup>). Bars represent one standard deviation of the mean.

are overestimated for the addition of injected CO<sub>2</sub> and underestimated for the reaction stoichiometry. According to Eq. (7), part of the injected CO<sub>2</sub> was consumed by the reaction with the ammonia present in the SWWs forming ammonium bicarbonate, since the immediate injection of CO<sub>2</sub> prevents the volatilization of ammonia.



On the other hand, for stoichiometric calculation the occurrence of evaporation during the AC process leads to higher concentrations of calcium or magnesium in the SWWs and the differences obtained in CO<sub>2</sub> calculated for SWW1, SWW2 and SWW3 reflect this. Calcium was the ion that most contributed (in about 81–89%) to CO<sub>2</sub> sequestration in this work compared to magnesium ion. Thus, the estimate of the value of CO<sub>2</sub> captured during the AC process must be considered by CO<sub>2</sub> injection. Quantifying the air-water flux of carbon dioxide is still a challenge [39].

Table 3

Results of the SWW2 supernatant in AC process, using A/V ratio of 5 m<sup>2</sup>/m<sup>3</sup> (after 1 day) and 155.4 m<sup>2</sup>/m<sup>3</sup> (after 13 days) to reach a pH of about 8.

Parameters	Unit	Initial	Final	
			A/V = 5 m <sup>2</sup> /m <sup>3</sup>	A/V = 155.4 m <sup>2</sup> /m <sup>3</sup>
pH	Sorensen	11.9 ± 0.2	7.9 ± 0.2	8.2 ± 0.1
Conductivity	mS cm <sup>-1</sup>	3.12 ± 0.02	2.46 ± 0.01	2.40 ± 0.01
NH <sub>4</sub> <sup>+</sup>	mg N L <sup>-1</sup>	69 ± 0	22 ± 1	12 ± 1
Abs. 254 nm	cm <sup>-1</sup>	0.022 ± 0.012	0.035 ± 0.006	0.025 ± 0.002
Abs. 410 nm	cm <sup>-1</sup>	0.020 ± 0.001	0.047 ± 0.001	0.012 ± 0.000
P. Alkalinity	mg CaCO <sub>3</sub> L <sup>-1</sup>	549 ± 40	–	0 ± 0
T. Alkalinity	mg CaCO <sub>3</sub> L <sup>-1</sup>	694 ± 87	–	234 ± 40
Calcium	mg L <sup>-1</sup>	297.6 ± 3.4	130.9 ± 8.4	62.2 ± 6.9
Magnesium	mg L <sup>-1</sup>	43.3 ± 2.1	20.2 ± 1.1	31.4 ± 7.2
Evaporation rate	%	–	6.0 ± 0.4	4.0 ± 0.6

Note: P. Alkalinity - phenolphthalein alkalinity; T. Alkalinity - total alkalinity.

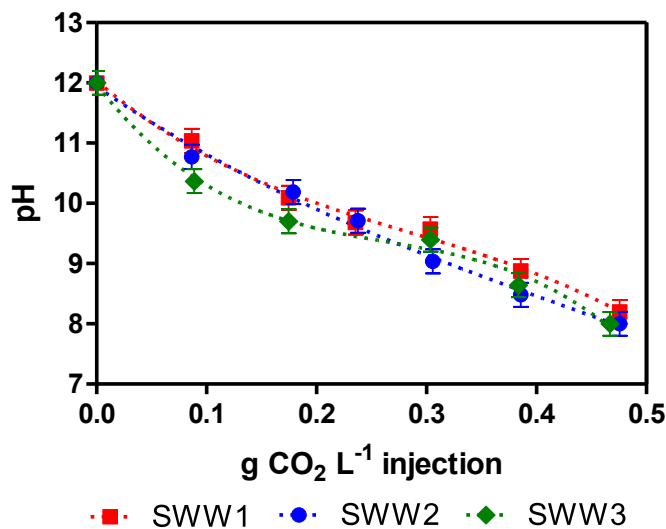


Fig. 7. Supernatant pH reached as a function of added CO<sub>2</sub>, for SWW1, SWW2 and SWW3 supernatants treated by IOSLM process at reaction pH 12. Bars represent one standard deviation of the mean.

### 3.4. IOSLM + AC integrated process

Table 4 shows the characteristics and removals obtained by the IOSLM + AC integrated process, using the optimized conditions (IOSLM process at pH 12 and AC process at A/V ratios of 9.5 m<sup>2</sup>/m<sup>3</sup> for SWW1 during 10 days and 5 m<sup>2</sup>/m<sup>3</sup> for SWW2 and SWW3 during 15 days). According to Table 4, the effluents treated by the IOSLM + AC integrated process had a pH around 7.8 and conductivity values close to the initial values. High TKN (97, 78 and 71%), BOD<sub>5</sub> (86, 82 and 80%), NH<sub>4</sub><sup>+</sup> (88, 69 and 52%), TP (99, 98 and 98%), TSS (99, 98 and 52%), turbidity (97, 96 and 62%), absorbance at 254 nm (87, 96, 87%), absorbance at 410 nm (92, 96 and 83%) and oils & fats (92, 71 and 47%) removals were obtained for SWW1, SWW2 and SWW3, respectively. COD removals were 91 and 80% for SWW1 and SWW2, respectively, and 7% for SWW3 due to their previous treatment on-site pretreatment plant. The quality of the treated effluents showed concentrations of COD (for all SWWs), BOD (for SWW1 and SWW2), TSS (for SWW1), TN (SWW2 and SWW3) and TP (for all SWWs) still above EU standards for discharge (COD =

**Table 4**

Characteristics and removals of three SWWs after IOSLM + AC integrated process. Removals are shown in parentheses (at IOSLM reaction pH = 12, and A/V ratios of 9.5 m<sup>2</sup>/m<sup>3</sup> for SWW1 during 10 days and 5 m<sup>2</sup>/m<sup>3</sup> for SWW2 and SWW3 during 15 days).

Parameters	Unit	SWW1	SWW2	SWW3
pH	Sorensen	7.8 ± 0.2	7.7 ± 0.1	7.8 ± 0.1
Conductivity	mS cm <sup>-1</sup>	3.4 ± 0.1	2.6 ± 0.1	2.6 ± 0.1
COD	mg O <sub>2</sub> L <sup>-1</sup>	488 ± 8 (91%)	368 ± 5 (80%)	210 ± 5 (7%)
TKN	mg N-Kj. L <sup>-1</sup>	6 ± 1 (97%)	22 ± 1 (78%)	17 ± 1 (71%)
BOD <sub>5</sub>	mg O <sub>2</sub> L <sup>-1</sup>	390 ± 14 (86%)	180 ± 0 (82%)	8 ± 4 (80%)
NH <sub>4</sub> <sup>+</sup>	mg N L <sup>-1</sup>	5 ± 1 (88%)	20 ± 1 (69%)	14 ± 1 (52%)
TP	mg L <sup>-1</sup>	3.0 ± 1.6 (99%)	5.6 ± 0.5 (98%)	2.5 ± 2.0 (98%)
TSS	mg L <sup>-1</sup>	47 ± 10 (99%)	21 ± 1 (98%)	29 ± 4 (52%)
Turbidity	NTU	39 ± 0 (97%)	17 ± 0 (96%)	6 ± 0 (62%)
Abs. at 254	cm <sup>-1</sup>	0.210 ± 0.015 (87%)	0.033 ± 0.005 (96%)	0.026 ± 0.001 (87%)
Abs. at 410	cm <sup>-1</sup>	0.164 ± 0.001 (92%)	0.054 ± 0.005 (96%)	0.053 ± 0.000 (83%)
Oils & Fats	mg L <sup>-1</sup>	131 ± 17 (92%)	110 ± 15 (71%)	46 ± 3 (47%)
Calcium	mg L <sup>-1</sup>	55.7 ± 15.7 (45%)	119.0 ± 7.5 (25%)	71.4 ± 3.7 (63%)
Magnesium	mg L <sup>-1</sup>	27.6 ± 24.3 (49%)	20.2 ± 1.1 (73%)	5.8 ± 1.8 (90%)

125 mg L<sup>-1</sup>; BOD = 25 mg L<sup>-1</sup>; TSS = 35 mg L<sup>-1</sup>; TN = 10 mg L<sup>-1</sup>; TP = 1 mg L<sup>-1</sup>). Therefore, additional treatments will be necessary for all SWWs. Biological treatments could be a solution for SWW1 and SWW2 since both presented a high biodegradability index values after IOSLM + AC integrated process. On the other hand, constructed wetlands could be a solution to treat low biodegradable effluents such as SWW3 after IOSLM + AC integrated process [15].

#### 4. Conclusion

This work investigated the AC process in immediate one-step lime precipitation and atmospheric carbonation integrated processes for slaughterhouse wastewaters treatment. Slaughterhouse wastewaters with different characteristics were evaluated.

IOSLM results showed that the reaction pH was not a key parameter for slaughterhouse wastewaters treatment, which will depend on the characteristics of the SWW to be treated. However, at reaction pH 12 the SWW characteristics were better for the studied parameters where higher removals were obtained.

The AC process, as solution for atmospheric mitigation of CO<sub>2</sub> emissions, proved to be efficient in reducing supernatant pH, conductivity, ammonium nitrogen and calcium concentrations over time. Longer carbonation times are required using higher reaction pH to achieve a supernatant pH of 9.5, however, if the objective is to achieve a supernatant pH of 8, the carbonation time may be the same regardless of the reaction pH applied. The reactor A/V ratio can be a significant factor in reducing supernatant pH, where high reactor A/V ratios mean less carbonation time. Ammonia removal was not affected by the reduction in carbonation time caused by the increase in the A/V ratio.

IOSLM + AC integrated process proved to be an efficient pre-treatment in the removal of contamination from the slaughterhouse wastewater for discharge into the municipal sewage treatment plant, however, an effluent tuning is still necessary if it is to be discharged into the water environment or to be reused in agriculture.

#### Credit author statement

Luís Madeira: Conceptualization, Methodology, Investigation,

Writing – original draft; Fátima Carvalho: Methodology, Writing – review & editing, Supervision, Funding acquisition; Adelaide Almeida: Writing – review & editing; Margarida Ribau Teixeira: Conceptualization, Writing – review & editing, Supervision.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

The authors are unable or have chosen not to specify which data has been used.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.rineng.2022.100807>.

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