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Treatment and Energy Valorisation of an Agro-Industrial Effluent in Upflow Anaerobic Sludge Reactor (UASB)

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Abstract. The accelerated growth of the population brings with it an increase in the generation of agro-industrial effluents. The inadequate discharge of these effluents significantly affects the quality of water resources. In this way, it becomes important to invest in treatment processes for agro-industrial effluents, particularly low-cost ones. In this context, the present study includes the design and construction of an UASB reactor and optimization of the anaerobic digestion treatment of the raw effluent from sweet chestnut production in the agro-industrial company Sortegel. The efficiency of the system was evaluated through the determination / monitoring of oxygen chemical oxygen demand (COD), biochemical oxygen demand (BOD5), total suspended solids (TSS), biogas production rate and quality (% methane). The reactor was fed for 25 weeks and operated under mesophilic conditions (temperature 30-40 °C). Different values were tested for the hydraulic retention time (HRT) and volumetric flow rate (VF): 0.66 days (VF=1509 L.m⁻³.d⁻¹); 1.33 days (VF=755 L.m⁻³.d⁻¹); 2.41 days (VF=415 L.m⁻³.d⁻¹). The average COD removal efficiency reached values of 69%, 82% and 75%, respectively, and simultaneously the associated BOD5 removal efficiency was 84%, 91% and 70%. As regards TSS, removal values were 78%, 94% and 63%. In addition, high methane production rates were obtained, between 2500 and 4800 L CH₄.kg⁻¹ COD removed d⁻¹. For all the hydraulic retention times tested, high concentrations of methane in the biogas were recorded: 66-75%, 70% and 75% for HRT of 0.66, 1.33 and 2.41 days, respectively.

1. Introduction

For many years the sweet chestnut has been used as a resource for human survival in Asia, Southern Europe and North Africa. At present, sweet chestnut production in Europe and West is no longer a source of subsistence, but continues to play an important role in food, wood harvesting and landscape
enhancement. Portugal was the third largest producer of sweet chestnut in Europe (EU 28), with an annual production of 24,700 tons, with the north of the country accounting for 84% of production [1].

The industrial production of sweet chestnut generates liquid effluents and its inadequate disposal may have as a consequence the reduction of drinking water quality for urban areas [2]. Thus, the intervention of governmental entities is required, either through regulations or national environmental policies and fines imposed for non-compliance with laws [3]. In this context, it is essential for the survival in the market, the implementation of effluent treatment systems, at minimum cost that, while ensuring the quality and commercialization of the product, combine the purification of the effluents with the production of renewable energy [4].

Anaerobic digestion has been implemented as a suitable, very efficient and low cost alternative [5]. This technology has been widely used in wastewater treatment processes where, under anaerobic and mesophilic conditions, anaerobic microorganisms convert organic matter into a more stabilized material, generating biogas as a co-product that can be used for the generation of electric and thermal energy [6]. This type of treatment can be carried out using an Upflow Anaerobic Sludge Blanket (UASB) reactor, which maintains a sludge bed with higher concentration of biomass at the bottom, improving the process efficiency [8].

In this project, the performance of the UASB reactor was evaluated in Sortegel agro industrial company wastewater treatment, for different values the hydraulic retention time (HRT) and volumetric flow rate (VF): 0.66 days (VF = 1509 L.m⁻³.d⁻¹); 1.33 days (VF = 755 L.m⁻³.d⁻¹); 2.41 days (VF = 415 L.m⁻³.d⁻¹). The following parameters were evaluated: biochemical oxygen demand after five days at 20°C (BOD₅) and total suspended solids (TSS), as well as the quality (% of methane) and production rate of biogas.

2. Materials and methods

The reactor used was of type "Y" UASB, constructed from tubes of PVC with diameter of 10 cm and a useful volume of 16 litres. Sludge from the anaerobic digester of the wastewater treatment plant (WWTP) of Bragança - Portugal, amounting 6.2 L (39% of the useful volume) was used as inoculum (biomass source).

As seen from Figure 1, the substrate feed takes place at point 1 through a peristaltic pump (WATSON 120S), in semi-continuous mode, and a timer controls the pump operating time. After the upward flow, the already stabilized liquid stream passes through the three-phase separator and exits through the inclined pipeline (point 5).

Figure 1. UASB reactor type "Y"
The generated biogas is collected at point 4, quantified in an "H"-shaped polyethylene device, which measures the volume produced through electrical impulses, the CH4 concentration being measured by a methane sensor (BlueSens, BCSCH4, biogás). A serpentine hose covers the reactor where hot water circulates to keep the temperature inside the reactor between 30 and 40°C. The internal temperature is monitored by means of two thermocouples (Multi mark) located in point 3 and between points 1 and 2.

The reactor was fed with agro-industrial effluent for 25 weeks and operated under mesophilic conditions (temperature 30-40°C). Different values were tested for the hydraulic retention time (HRT) and volumetric flow rate– (VF): 2.41 d (VF = 415 L.m⁻³reator.d⁻¹) - weeks 1 to 16; 1.33 d (VF = 755 L.m⁻³reator.d⁻¹) - weeks 17 to 23; 0.66 d (VF = 1509 L.m⁻³reator.d⁻¹) - weeks 24 to 25. The frequency of analyzes for the control of the physicochemical parameters and the analytical methods used for the determination of these parameters are shown in Table 1.

### Table 1. Monitored parameters and sampling frequency.

| Parameter                          | Frequency of Sampling | Analytical method |
|-----------------------------------|-----------------------|-------------------|
| pH                                | Daily                 | 4500 H2B [9]      |
| Temperature                        | Daily                 |                   |
| Total solids (TS)                  | Weekly                | 2540 B [9]        |
| Fixed solids (FS)                  | Weekly, Monthly       | 2540 E [9]        |
| Volatile solids (VS)               | Weekly, Monthly       | 2540 E [9]        |
| Total suspended solids (TSS)       | Weekly                | 2540 D [9]        |
| Alkalinity                         | Weekly, Weekly, Weekly| 2330 B [9]        |
| Biochemical oxygen demand (BODs)   | Weekly, Weekly        | 5210 B [9]        |
| Chemical oxygen demand (COD)       | Weekly, Weekly        | 5220 C [9]        |
| Volatile fatty acids (VFA)         | Weekly, Weekly, Weekly| [10]              |

### 3. Results and discussion

The optimum pH for microbial growth should be in the range of 6.5 to 8.2 [11]. Inside the UASB reactor the pH was between 5.6 to 8.0 during most of the operation time. The substrate pH ranged from 4.9 to 8.1, a common value for most industrial effluents [12]. Alkalinity directly interferes with pH because it provides a buffer effect when there is acid production in the anaerobic digestion, so it is necessary to maintain it between 2000 and 5000 mg.L⁻¹ CaCO₃ [13]. To compensate for the low alkalinity and acid pH of the substrate sodium bicarbonate was added to the feed.

Another parameter monitored was the concentration of volatile fatty acids (VFA) that can inhibit the biomass degradation potential when its value exceeds 1500 mg.L⁻¹ [14]. Values between 1200-9900 mg.L⁻¹ can partially inhibit the methanogenic phase and values above 5800 mg.L⁻¹ cause total inhibition [15]. The values analyzed at the inlet, outlet and inside the reactor did not exceed 300.0 mg.L⁻¹. In addition, the daily recorded values of temperature inside the reactor showed that the heating system allowed to maintain the temperature between 15 and 45°C, most of the time within the mesophilic range.

For the different HRT, 0.66 d (VF = 1509 L.m⁻³.d⁻¹), 1.33 d (VF = 755 L.m⁻³.d⁻¹) and 2.41 d VF = 415 L.m⁻³.d⁻¹), average removals of COD of 69% (range 50% - 70%), 82% (range 65% - 97%) and 75% (range 60% - 88%) were achieved respectively (Figure 2). These is a satisfactory result when compared with the work done by Khan et al. [16], in which COD removals between 50 and 90% were obtained from a study in several countries using the UASB reactor. In addition, COD values lower
than 150 mg.L\textsuperscript{-1} were recorded between the 5\textsuperscript{th} and 13\textsuperscript{th} week (with a VF of 416 L.m\textsuperscript{3}.d\textsuperscript{-1}), complying with Decree-Law no. 236/98 \cite{17} for discharge of industrial wastewater.

Furthermore, mean percentages of BOD\textsubscript{5} removal of 84\% (75 to 93\%), 91\% (76 to 96\%), and 70\% (55 to 97\%) were obtained for HRT values of 0.66 d, 1.33 d, 2.41 d, respectively (Figure 2). In the feed, the BOD\textsubscript{5} ranged from 60 to 1850 mg O\textsubscript{2}.L\textsuperscript{-1} and in the output between 6 to 832 mg O\textsubscript{2}.L\textsuperscript{-1}. Between the 5\textsuperscript{th} and 13\textsuperscript{th} week (VF = 415 L.m\textsuperscript{3}.d\textsuperscript{-1}) the recorded values were between 100 - 150 mg O\textsubscript{2}.L\textsuperscript{-1}. In this period, the BOD\textsubscript{5} and TSS concentration in the clarified effluent indicate the need for a post-treatment in order to meet the discharge limits.

![Figure 2](image.png)

**Figure 2.** Average removal of COD, BOD\textsubscript{5} and TSS in each week of the experimental study

According to Table 2, COD and BOD\textsubscript{5} removals were similar to values found in the literature. With respect to the TSS content, it was higher than values found in the literature for other types of effluents. In addition, the removals of TSS, COD and BOD\textsubscript{5} were higher for a volumetric organic charge (VOC) of 1.64 kg COD.m\textsuperscript{-3}.d\textsuperscript{-1} and a better performance of the reactor was observed for HRT = 1.33 d (VF = 755 Lm\textsuperscript{-3}.d\textsuperscript{-1}).

| Efluent                             | VOC (kg COD.m\textsuperscript{-3}.d\textsuperscript{-1}) | COD (% removal) | BOD\textsubscript{5} (% removal) | TSS (%removal) | Ref.  |
|-------------------------------------|----------------------------------------------------------|-----------------|-----------------------------------|---------------|-------|
| Domestic wastewater                 | 0.9                                                      | -               | 53                               | 46            | \cite{18} |
|                                     | 2.3                                                      | 57              | 64                               | 64            | \cite{19} |
|                                     | 7.8                                                      | 70              | -                                | 74            | \cite{20} |
|                                     | 10.4                                                     | 39              | -                                | 34            | \cite{20} |
| Domestic wastewater and dairy wastewater | 3.4                                                     | 69              | 79                               | 72            | \cite{21} |
| Synthetic wastewater                | 4.0                                                      | 90-92           | 94-96                            | -             | \cite{22} |
| Agroindustrial effluent (mean values) | 0.59                                                    | 75              | 73                               | 66            | In this study |
|                                     | 1.64                                                     | 80              | 90                               | 96            | study   |
|                                     | 7.85                                                     | 60              | 84                               | 78            |       |

**Table 2.** Results from the literature and obtained in this study for the removal of COD, BOD\textsubscript{5} and TSS.
Regarding TS content, minimum values of 316 mg.L$^{-1}$ and 719 mg.L$^{-1}$ were observed and maximum values were 3107 mg.L$^{-1}$ and 1738 mg.L$^{-1}$ in the feed and inside the reactor, respectively. In studies by Al-Jamal and Mahmound [23], when treating domestic wastewater in a UASB reactor, the samples had higher TS values (in the range of 53.9 to 66.9 g.L$^{-1}$).

The VS / TS ratio indirectly indicates the concentration of biomass in the reactor, being a minimum value of 0.67 considered adequate for a good system performance [23]. When studying the treatment of domestic effluent combined with the effluent from a dairy industry using a UASB reactor, Tawfik et al. [21] obtained VS/TS ratios of ≈0.66. From Figure 3, it can be observed that between the 3rd and 4th month the ratio is close to 0.67, which means a good performance of the reactor. This good performance is also confirmed by the average values of COD and BOD$_5$ removals, 82% and 91%, respectively, obtained in this period (Figure 2).

![Figure 3. Percentages of removal of COD, BOD$_5$ and TSS for each week analysed](image1)

From Figure 4, it can be observed that the biogas production reached around 4700 L.kg$^{-1}$ COD$_{substrate}.d^{-1}$, while the maximum methane production was around 2400 L.kg$^{-1}$ COD$_{substrate}.d^{-1}$ (values obtained at 19th week).

![Figure 4. Biogas and methane production](image2)
Figure 5 shows the accumulated production of biogas and methane. The average concentrations of methane in the biogas generated were as follows: 33%, 58% and 65% for HRT of 0.66, 1.33 and 2.41 d, respectively. However, during certain periods the percentage of CH$_4$ in the biogas reached values between 70 and 80%. For cogeneration systems, the methane content in the biogas cannot be less than 40 - 45%, since low levels impede the operation of the engines [24]. Due to the fact that the characteristics of the substrate are not constant over time, it is worth noting that there are some decreases in the percentage of methane, which could compromise the system.

![Figure 5. Cumulative production of biogas and methane](image)

Figure 6 shows the relationship between biogas and methane produced and the COD removed in the reactor. Throughout the study, the average daily yields were 0.47 and 0.33 L.kg$^{-1}$ of COD removed for biogas and methane, respectively. These values are relatively low but it is assumed that this may be due to the high variability of the composition of the industrial effluent (COD varied between 80 and 10000 mgO$_2$.L$^{-1}$). The maximum production of biogas and methane are respectively 59 and 30-35 L.d$^{-1}$ (for VF = 755 L.m$^{-3}$ reactor.d$^{-1}$), which correspond to 3.1 and 1.7 L.kg$^{-1}$ COD removed.

![Figure 6. Daily production of biogas and methane relative to COD removed](image)
4. Conclusions
The UASB reactor can be used in the primary treatment of an agro industrial sweet chestnut effluent, obtaining very satisfactory COD, BOD5 and TSS removal values for volumetric flow rates from 415 to 1509 L.m$^{-3}$.d$^{-1}$. In addition, there was energy recovery from the effluent, associated to methane production during long periods of operation, reaching around 35 L.d$^{-1}$.

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