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Enhancing Sustainability in Wine Production: Evaluating Winery Wastewater Treatment using Sequencing Batch Reactors †

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Abstract: Wine production generates a high volume of wastewater with a significant fraction of biodegradable organic matter that must be removed before safe release into surface waters. Aerobic sequencing batch reactors (SBR) has been successfully applied in the treatment of a wide range of wastewaters. However, only few studies have described the use of SBR process for the treatment of winery wastewater (WW). The effectiveness of using an aerobic SBR process was investigated for the treatment of WW using two activated sludge concentration (i.e., 2 and 4 g_{VSS} L^{-1}) and nutrient-supplemented conditions. In nutrient-deficient conditions, COD removal efficiencies varied between 70% to 97% depending on the organic loading rate (OLR). In nutrient-supplemented assays, COD removal efficiencies remained above 91% in all conditions tested. However, the effluent quality decreased due to the increase in the total suspended solids concentration. Furthermore, the COD concentration of the treated effluent was unable to meet legal requirements (< 0.150 g L^{-1}) for safe wastewater discharge. Therefore, longer aeration periods and settling phases may be required in order to improve the effluent quality under high organic loadings. Overall, these findings demonstrate the potential of SBR as a biological WW treatment process.

Keywords: biological treatment; aerobic process; organic loading rate; agro-industrial effluents

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1. Introduction

From an economic standpoint, wine production plays a fundamental role in many countries. However, the viticulture and winemaking sectors have long overlooked the environmental concerns associated with wine production. In Europe, several wineries still face challenges in wastewater management and fail to meet the legal limits requirements for water discharge or reuse due to the lack of adequate treatment practices [1].

For every litre of wine produced, between 0.5 L to 14 L of wastewater can be generated from several processes, including grape rinsing and de-stemming, pressing grapes into must, cleaning installations, fermentation barrels, and even wine losses into the waste stream [2]. These waste stream are rich in biodegradable organic matter, with typical chemical oxygen demand (COD) concentration varying between 0.3 – 49 g L^{-1} [3]. Nutrients such as nitrogen and phosphorus can also be present in concentrations ranging from 10 – 415 mg L^{-1} and 2.1– 280 mg L^{-1}, respectively [3]. However, the characteristic of winery wastewater (WW) strongly depends on the winemaking stage and the technology applied. Although the oscillatory characteristics of WW streams emphasizes the complexity and challenges associated to its treatment, preventing the discharge of untreated or partially treated WW is crucial to mitigate the risks of nutrient leaching to groundwater and eutrophication of surface waters.

The choice of a specific treatment process depends on several factors, such as the size and location of the wineries, the volume of wastewater generated and its organic content, as well as the capital investment and operating costs [4,5]. Several treatment processes, including physical, chemical and biological methods have been studied to treat WW [6]. Advanced oxidation processes (AOP) has emerged as a promising treatment process to successfully achieve efficient WW treatment [7,8]. However, the high cost of reagents and energy associated to AOP hinders their widespread application [5]. Biological treatment is widely recognized as a cost-effective and environmental-friendly approach to treat WW [9]. Aerobic biological processes, such as aerated storage tanks and conventional activated sludge systems are commonly employed for the treatment of these waste streams due to their simplicity and high efficiency [9]. Sequencing batch reactor (SBR) has been successfully used to treat several industrial wastewaters, due to low infrastructure and energy requirements [10]. Furthermore, SBR processes offers significant advantages due to its simple automation, flexible operation and low operating cost when compared to conventional activated sludge systems [6,9,11]. Despite these advantages, only a few studies have reported the use of SBR for WW treatment [12–14]. Therefore, the present study aims to determine the viability of treating winery effluents in SBR.

2. Materials and methods

2.1. Winery wastewater

The WW was obtained from a winery located in the Douro region, North of Portugal. The WW was characterized in terms of chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS), volatile suspended solids (VSS), total nitrogen (TN) and total phosphorus (TP), in accordance with the standard methods [15] (Table 1). Five distinct $COD_{influent}$ concentrations were tested to assess the treatment process efficiency using a SBR system. In order to obtain $COD_{influent}$ concentrations from 3.0 g L^{-1} to 18.5 g L^{-1}, the WW was diluted with tap water and then neutralized (pH 7) with sodium hydroxide.

Table 1. Composition of the winery wastewater.

	COD ($g L^{-1}$)	**BOD ($g L^{-1}$)**	**TSS ($g L^{-1}$)**	**VSS ($g L^{-1}$)**	**TN ($g L^{-1}$)**	**TP ($g L^{-1}$)**	**COD:N:P**
Winery Wastewater	68	55	14.7	12.8	0.663	0.258	100:1:0.4

2.2. Sequencing batch reactors set-up and experimental conditions

A total of 15 identical sequencing batch reactors (SBRs), each with a working volume of 4 L, were used in this work. The SBRs were operated in cycles of 23 h, including an aeration phase (21 h) and a settling phase (2 h). The feeding and effluent withdrawal phases were fast and had a negligible impact on the overall cycle time. During the aeration phase, air was introduced at the bottom of the reactors with a variable airflow rate, aiming to achieve a minimum dissolved oxygen concentration of 2 mg L^{-1}. All reactors were operated for at least 35 cycles to achieve steady-state, before concluding each experiment.

All reactors were inoculated with activated sludge from a local municipal wastewater treatment plant (Vila Real, Portugal). Two sludge concentration were evaluated, namely 2 g_{VSS} L^{-1} (X_2) and 4 g_{VSS} L^{-1} (X_4). Specifically, reactors R1 – R4 and R9 – R11 were inoculated with 2 g_{VSS} L^{-1} of activated sludge, while reactors R5 – R8 and R12 – R15 were inoculated 4 g_{VSS} L^{-1}. Additionally, due to imbalanced COD:N:P ratio in the WW, the effect of nutrient supplementation on the treatment process was also evaluated. Hence, to ensure suitable nutrients for cellular synthesis, nitrogen (15.4 mg_N g_{COD}^{-1}) and phosphorus (2.0 mg_P g_{COD}^{-1}) were added to the feed of R9 – R11 (X_{2+N}) and R12 – R15 (X_{4+N}). The volume exchange ratio (VER) in R1 – R8 and R9 – R15 was 50% and 25%, respectively. A summary detailing the conditions applied to each reactor is presented in Table 2.

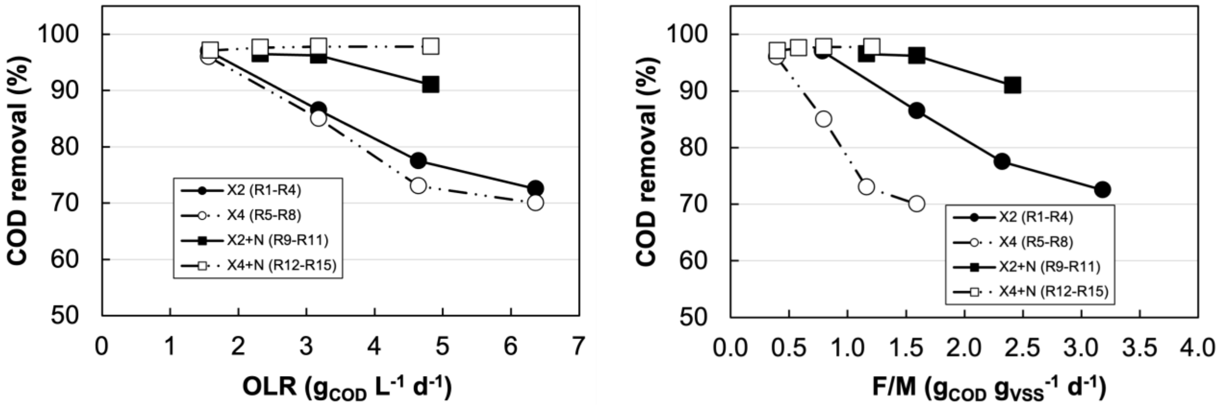
Table 2. Composition of the winery wastewater.

Reactors	**COD$_{\text{influent}}$ (g L^{-1})**	**OLR (g L^{-1} d^{-1})**	**Sludge concentration (g L^{-1})**	**VER %**	**HRT (d^{-1})**	**Nutrient supplementation**
R1	3.0	1.6	2	50	1.9	No
R2	6.1	3.2				
R3	8.9	4.6				
R4	12.2	6.4				
R5	3.0	1.6	4	50	1.9	No
R6	6.1	3.2				
R7	8.9	4.6				
R8	12.2	6.4				
R9	8.9	2.3	2	25	3.8	Yes
R10	12.2	3.2				
R11	18.5	4.8				
R12	6.1	1.6	4	25	3.8	Yes
R13	8.9	2.3				
R14	12.2	3.2				
R15	18.5	4.8				

3. Results and discussion

3.1. COD removal efficiency

In this work, a total of 15 distinct SBR operating conditions were evaluated to determine the viability of using SBR technology for the treatment of WW, focusing on the COD removal efficiency and effluent quality (i.e., COD and TSS). After obtaining a steady-state in each tested condition, the influence of OLR and F/M ratio on the COD removal efficiency was assessed (Figure 1). In nutrient deficient conditions, (i.e., without nutrient supplementation), the COD removal efficiency exhibited a decreasing trend, from 97% to 70%, with increasing OLR and regardless of the initial sludge concentration (Figure 1a). However, in assays with nutrient supplementation, the COD removal efficiency remained above 91% with OLR up to 4.8 g_{COD} L^{-1} d^{-1} (Figure 1a). Similarly, the increase of the F/M ratio resulted in a reduction of the COD removed, in assays without nutrient supplementation (Figure 1b). Moreover, at similar F/M ratio, COD removal efficiency was considerably lower in assays performed with 4 g_{VSS} L^{-1}, suggesting the occurrence of mass transfer limitation at higher sludge concentration. However, this limitation was not observed in nutrient-supplemented assays. In fact, COD removal efficiency of 97% could be sustained for F/M ratios up to 1.6 g_{COD} g_{VSS}^{-1} d^{-1}, which is higher than F/M ratios commonly applied in conventional activated sludge process [16]. It has been suggested that F/M ratios up to 1.4 g_{COD} g_{SS}^{-1} L^{-1} can be applied for the treatment of high-strength organic wastewater in SBR process [17]. In this work, COD removal efficiency of 91% was attained at F/M ratio of 2.4 g_{COD} g_{VSS}^{-1} d^{-1} with nutrient supplementation.

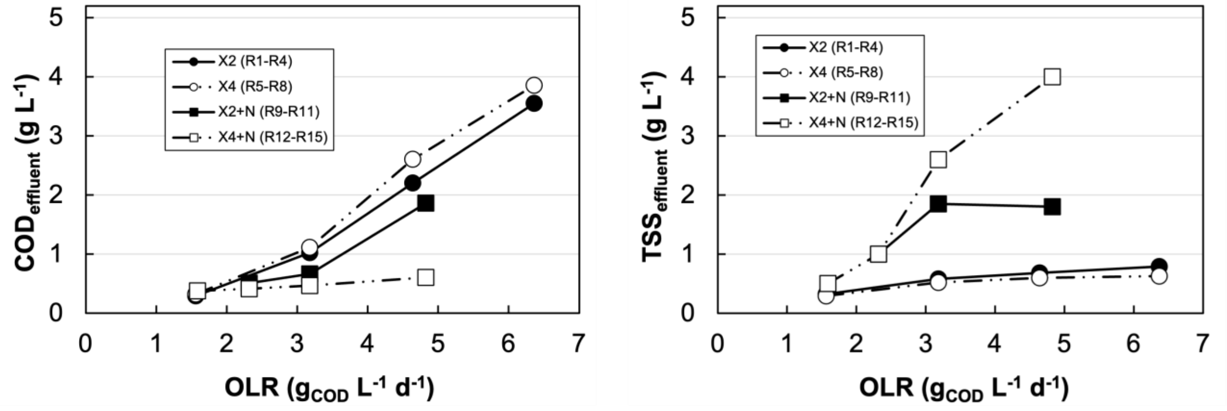


(**a**) (**b**)

Figure 1. COD removal efficiency achieved under increasing: (**a**) OLR and (**b**) F/M ratio. Assays were performed using two sludge concentration (X_2 = 2 g_{VSS} L^{-1}; X_4 = 4 g_{VSS} L^{-1}) and nutrient-supplemented conditions (X_{2+N}; X_{4+N}).

3.2. *Effluent quality and sludge settling properties*

The final treated effluent quality of each assay was evaluated in term of $COD_{effluent}$ and $TSS_{effluent}$ concentrations (Figure 2). In assays performed without nutrient supplementation, a clear degradation of the effluent quality was observed with the increase of the OLR applied, as shown by the increase in $COD_{effluent}$ concentration (Figure 2a). At the highest OLR tested, the $COD_{effluent}$ concentration reached 3.5 g L^{-1} at SBR steady-state. Nonetheless, at an OLR of 1.6 g_{COD} L^{-1} d^{-1}, the $COD_{effluent}$ concentration was below the legal discharge limit (i.e., 0.150 g_{COD} L^{-1}) without the need of nutrient supplementation and with both sludge concentration (Figure 2a). On the other hand, in nutrient-supplemented assays, the $COD_{effluent}$ concentrations were rather stable, remaining for the most part below 0.5 g L^{-1}, with the only exception observed in the assay performed at an OLR of 4.8 g_{COD} L^{-1} d^{-1} and 2 g_{VSS} L^{-1} of sludge (Figure 2a). In this condition, the $COD_{effluent}$ reached 1.67 g L^{-1}, due to mass transfer limitation caused by the high F/M ratio (i.e., 2.4 g_{COD} g_{VSS}^{-1} L^{-1}), as discussed previously (Figure 1b; Figure 2a).

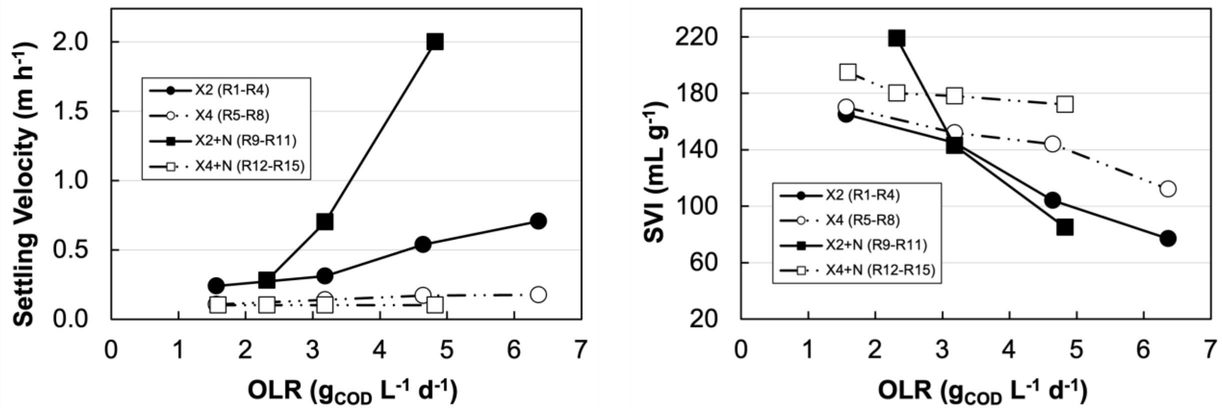


(**a**) (**b**)

Figure 2. Effect of increasing OLR on the effluent quality in terms of: (**a**) $COD_{effluent}$ and (**b**) $TSS_{effluent}$ concentration.

Nutrient supplementation had a negative impact on effluent quality in terms of the $TSS_{effluent}$ concentration, which increased significantly with the increase in organic loading (Figure 2b). These results suggests that, in nutrient-supplemented assays, the higher availability of nutrients stimulated sludge growth. The newly formed sludge was unable to settle fast enough in order to remain inside the system, contributing to the increase of $TSS_{effluent}$ concentration (Figure 2b). In nutrient-deficient assay, $TSS_{effluent}$ concentration increased slightly, yet remaining below 0.6 g L^{-1} (Figure 2b).

The sludge settling properties was monitored in all reactors, in terms of sludge settling velocity and sludge volume index (SVI) (Figure 3). Assays performed with a sludge concentration of 2 g_{VSS} L^{-1} exhibited the highest settling velocities across all organic loading conditions. Furthermore, in nutrient-supplemented assays, the highest sludge settling velocity (i.e., 2 m h^{-1}) was reached at OLR of 4.8 g_{COD} L^{-1} d^{-1}, which was 4 times higher than in nutrient-deficient assays for similar organic loading (Figure 3a). Assays performed with sludge concentration of 4 g_{VSS} L^{-1} exhibited low settling velocities (i.e., < 0.14 m h^{-1}), regardless of the OLR applied (Figure 3a). These results suggest that in assays with a high sludge concentration, the settling velocity was hindered due to the development of sludge with a poor floc structure. In all conditions tested, the SVI decreased with the increase in organic loading, varying from 219 mL g^{-1} to 77 mL g^{-1} (Figure 3b). A steeper improvement in the sludge settling properties was observed in assays performed with 2 g_{VSS} L^{-1} of activated sludge and in nutrient-supplemented conditions. In activated sludge systems, severe sludge bulking problems may occur when SVI is above 250 mL g^{-1}, while values over 150 mL g^{-1} generally indicate sludge with poor settling properties [18]. In this study, assays performed with 4 g_{VSS} L^{-1} and nutrient supplementation, the SVI values endured above 170 mL g^{-1} (Figure 3b). Furthermore, limited bulking condition (above 150 mL g^{-1}) were observed in the other assays, particularly at OLR below 3 g_{COD} L^{-1} d^{-1}, although severe sludge bulking problems were not detected in this study (Figure 3b).



(**a**) (**b**)

Figure 3. Effect of increasing OLR on the sludge settling properties in terms of: (**a**) settling rate and (**b**) sludge volume index (SVI).

4. Conclusions

WW was efficiently treated using an aerobic SBR process. High COD removal efficiencies (> 97%) was attained at an OLR of 4.8 g_{COD} L^{-1} d^{-1} in nutrient-supplemented assays performed with 4 g_{VSS} L^{-1} of sludge. While nutrient supplementation allowed high COD removal efficiencies across all OLR applied, the effluent quality was unable to meet legal requirements for safe wastewater discharge. Further work can be carried out adjusting the SBR cycle length in order to improve the effluent quality in high-rate organic loading conditions and/or adding a chemical process as a tertiary treatment.

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