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Removal of Color and Polyphenols from Sugarcane Molasses Ethanol Vinasse by Hydrodynamic Cavitation

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The hydrodynamic cavitation process using orifice plates was applied to remove color and polyphenols from sugarcane molasses ethanol vinasse, with a treatment time of 1 h. The raw vinasse was diluted with tap water in a volume ratio of 1:10 in an acidic medium (pH 2). A 2^k factorial analysis was carried out to evaluate the effect of the inlet pressure of 2 and 3.6 bar. The results indicate that the interaction between the inlet pressure and the number of orifices has a significant effect on the color and polyphenol removal efficiency. The plate with 9 orifices under the condition of an inlet pressure of 3.6 bar was more efficient, achieving a maximum color removal of 32.71% and polyphenol removal of 88.62% during a 20 min treatment, with a cavitation yield of 0.00365 mg J^{-1}. The requirement of a longer treatment time did not favor the removal efficiency of color and polyphenols. Hydrodynamic cavitation significantly improves the removal of color and polyphenols, being a viable alternative to reduce toxicity in alcoholic vinasse, contributing to environmental improvements for the alcohol industry.

1. Introduction

The ethanol distillery produces large amounts of dark brown wastewater, known as stillage, distillery wastewater, distillery effluent or vinasse, depending on the sugar source used for fermentation (Nagarajan & Ranade, 2020). Approximately 12 - 15 liters of vinasse are produced per liter of alcohol (Shinde et al., 2020). Vinasse shows remarkable characteristics that explain its environmental effects, including high chemical oxygen demand (COD) and biochemical oxygen demand (BOD), low pH, high total dissolved solids, unpleasant odor, and complex phenolic compounds, including melanoidin and polyphenols. Melanoidins and polyphenols are mainly responsible for the persistent color in wastewater resulting from the distillation process; both are recalcitrant (Singh et al., 2020). The high organic load content and recalcitrant contaminants in vinasses make them an ecological threat and make their final disposal in soil or water resources difficult.

There are several treatment methods to remove pollutants present in vinasse. Nevertheless, most of these treatments are time-consuming and expensive which hinders their scalability. Consequently, new treatment methods, such as hydrodynamic cavitation-based technology, are required to replace traditional methods, which are costly and fail to meet sustainability goals. Hydrodynamic cavitation has been shown to be a cost-effective and sustainable process in wastewater treatment due to its low energy consumption and high efficiency in contaminant removal (Nieto et al., 2021). Cavitation is the formation, growth, and collapse of microbubbles or cavities, resulting in the local generation of high pressures, shear stresses, and high temperatures (Capocelli et al., 2014). These cavities are created using restriction devices such as Venturi tubes and orifice plates (Gawande et al., 2024). Some studies have investigated the effectiveness of cavitation in the treatment of distillery wastewater (Nagarajan & Ranade, 2020; Padoley et al., 2012). However, there are no specific studies have been published on the removal of contaminants in ethanol vinasse using hydrodynamic cavitation with orifice plates. Therefore, this study focused on evaluating the effect between the inlet pressure to the orifice

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plate and the number of orifices in that plate, and its impact on the removal efficiency of color and polyphenols in sugarcane molasses ethanol vinasse, to minimize the contaminant load of the treated vinasse, facilitating its final disposal or its potential reuse in agricultural or industrial processes.

2. Materials and methods

2.1 Vinasse collection and analytical methods

The sugarcane molasses ethanol vinasse used in this study was collected from the effluent of an alcohol distillery located in the Lambayeque region of Peru. The sample was transported in 20 L plastic containers. The pH of the vinasse was adjusted to 2 with sulfuric acid (1M) before being refrigerated (4 °C), for later use.

The vinasse refrigerated at 4 °C was allowed to stand until it reached room temperature before proceeding with analytical measurements. COD, total solids, and total volatile solids analyses were performed on the raw vinasse using APHA standard methods 5220 D, 2540 B, and 2540 E, respectively (Rice, 2022). pH and electrical conductivity were measured with the HANNA HI5221 and HANNA HI2300 meters, respectively. For the analysis color and total polyphenols, raw vinasse diluted with distilled water in a volume ratio of 1:10 (España-Gamboa et al., 2017) was used at a pH adjusted to the value of 2. The intensity of the color of the diluted vinasse was measured at 475 nm (Singh et al., 2020). Polyphenols were analyzed using the Folin-Ciocalteu method and expressed as mg gallic acid equivalents per liter of diluted vinasse (mg GAE L^{-1}), using a standard curve (50 - 2000 mg GAE L^{-1}). Polyphenol absorbance was measured at 765 nm (Correa-Mahecha et al., 2022). Both measurements were recorded using a Thermo Scientific GENESYS 30 visible spectrophotometer.

2.2 Experimental procedure

The experimental module consisted of a 15 L storage tank, equipped with a water-cooling system to maintain the temperature in a range of 30 °C to 48 °C. This tank is connected laterally, through a pipe, to the inlet of a 1.5 kW peripheral water pump. The pump outlet pipe is divided into a main line and a bypass line, as shown in Figure 1a and 1b. The pipes and fittings are constructed of stainless steel, with a nominal diameter of 1 inch. The orifice plate, also constructed of stainless steel, has a thickness of 2 mm, and each orifice has a diameter of 1.5 mm, arranged in a quadrangular pattern, as shown in Figure 1c. For vinasse treatment, 1 L of raw vinasse was diluted in 10 L of tap water, using the recommended dilution for fertigation (España-Gamboa et al., 2017). To prevent foam formation, 2 ml of Defoamer 605 antifoam was added to the solution. Adjusting the pH to 2. The cavitation treatment was performed for 1 h, with sampling every 20 min.

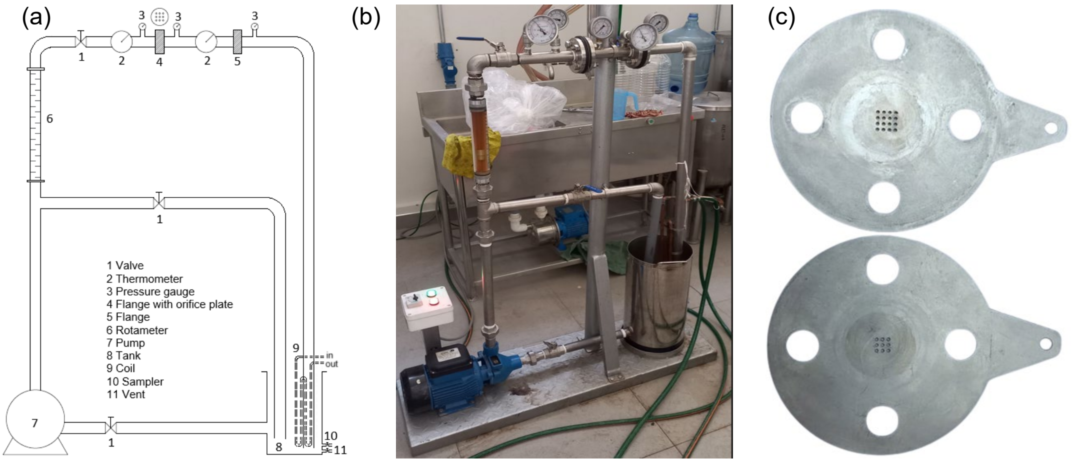

Figure 1: (a) schematic representation of module, (b) experimental module, (c) orifice plate

2.3 Statistical analysis

A two-factor factorial design with two levels each was used. The factors considered were the inlet pressure to the orifice plate (2 and 3.6 bar) and the number of orifices in the plate (9 and 16 orifices). The results were reported as mean ± standard deviation and the experiments were carried out in triplicate. For data analysis, analysis of variance (ANOVA) with a significance level of 5% was used, followed by Duncan's post hoc test. Statistical analysis was performed with the RStudio software and graphs were generated with the Origin Pro 2022 software. The color removal efficiency, polyphenol removal efficiency, and cavitational yield were calculated using Equations 1, 2 and 3, respectively.

$$\text{Color removal efficiency (\%)} = \frac{Abs_0 - Abs_f}{Abs_0} * 100\ \% \quad (1)$$

$$\text{Polyphenol removal efficiency (\%)} = \frac{C_0 - C_f}{C_0} * 100\ \% \quad (2)$$

$$Y = \frac{V*(C_0 - C_f)}{\Delta P*Q*t} * 100\ \% \quad (3)$$

Where Abs_0 and Abs_f are the initial and final absorbances; C_0 and C_f are the initial and final concentrations in mg L^{-1}; V is the volume in m^3; Q is the volumetric flow in $m^3\ s^{-1}$; ΔP is the pressure drop in kPa, t is the treatment time in seconds and Y is the cavitation yield in mg J^{-1}.

3. Results and discussion

3.1 Physicochemical properties of sugarcane molasses ethanol vinasse

The physicochemical characteristics of the vinasse are presented in Table 1.

Table 1: Physicochemical characteristics of sugarcane molasses ethanol vinasse

Properties	Units	Average value	(Singh et al., 2020)	(Prazeres et al., 2019)
COD	mg L^{-1}	106137 ± 3303	147400 ± 850	30633 – 31600
Total solids	mg L^{-1}	85177 ± 784	149100 ± 480	21500 – 22200
Total volatile solids	mg L^{-1}	27957 ± 191	–	13900 – 15200
pH	–	4.62 ± 0.017	4.1	4.27 – 4.32
Electrical conductivity (EC)	dS m^{-1}	29.66 ± 0.072	–	8.21 – 8.56
Polyphenols	mg GAE L^{-1}	338.54 ± 14.44*	13970 ± 1660	–
Color	–	dark brown	dark brown	dark brown
	Abs	2.307 ± 0.0021*	–	–

*: diluted vinasse: 1:10 ratio, Abs: absorbance

The total solids, COD, and total polyphenol content (TPC) shown in Table 1 are below the values reported by Singh et al. (Singh et al., 2020) and other authors. The COD of the vinasse was reported to be 110065 ± 11486 mg L^{-1}, the TPC reached 10834 ± 1476 mg L^{-1}, and the pH was recorded at 4.39 ± 0.006 (España-Gamboa et al., 2017). On the contrary, the TPC was found to be in the range of 230 and 390 mg of GAE L^{-1} (Paz-Pino et al., 2014). Other authors observed lower levels of COD, total solids, pH, and EC than those found in this work (Prazeres et al., 2019). The differences observed in the physicochemical characteristics of vinasse can be attributed to changes in the composition of the raw material, the operating conditions of the distillation column and the process in general (Shinde et al., 2020). Furthermore, the different varieties of sugarcane and the degrees of sugarcane maturity used in the sugar industry contribute to these differences (Paz-Pino et al., 2014).

3.2 Statistical analysis

Statistical analysis indicates that both inlet pressure and number of orifices, as well as the interaction between both variables, significantly influence ($p < 0.05$) the efficiency of color and TPC removal from alcoholic vinasse during 1 h of hydrodynamic cavitation. It was observed that the inlet pressure of 3.6 bar was more effective than the pressure of 2 bar in color and TPC removal. Regarding the number of orifices, the plate with 16 orifices showed a statistically significant effect on color removal compared to the plate with 9 orifices. However, in TPC removal, the increase in the number of orifices did not have a statistically significant effect ($p > 0.05$).

3.3 Effect of inlet pressure and cavitation number

Inlet pressure and cavitation number (CV) are determining factors that influence both the formation of cavities and the pressure at which they collapse or implode (Katiyar et al., 2024). Table 2 shows that an increase in inlet pressure leads to an increase in the volumetric flow rate in the main line and the velocity in the orifice plate, resulting in a decrease in the CV. It is also observed that for plates with 9 and 16 orifices, the CV decreases from 0.43 to 0.36 and from 0.93 to 0.69, respectively, as the inlet pressure increases from 2 to 3.6 bar. However, when the number of orifices increased at the same inlet pressure, the CV tends to increase. The CV, which indicates the tendency of the liquid to cavitate under certain conditions, does not always reflect the final efficiency of the process. Its effectiveness also depends on other factors, such as physicochemical properties and geometrical parameters of the cavitating device (Rajoriya et al., 2017). The CV of 0.36, obtained at an inlet pressure of 3.6 bar with the 9-orifice plate, effectively removed color and TPC. This is because a lower number of cavitation leads to a higher number of hydroxyl radicals and improves the efficiency of organic pollutants (Rajoriya et al., 2017). Katiyar et al. (Katiyar et al., 2024) studied the influence of increasing inlet pressure on the CV using different orifice plate configurations, finding values ranging from 0.027 to 1.723 at inlet pressures in the range of 3 bar to 15 bar.

Table 2: Flow characteristics for plates

Inlet Pressure (bar)	Orifices	Q (L min^{-1})	A (m^2)	v (m s^{-1})	CV
2	9	17	1.59e-5	17.61	0.43
2	16	24	2.83e-5	14.13	0.93
3.6	9	21	1.59e-5	22.01	0.36
3.6	16	34	2.83e-5	20.14	0.69

Q is flow rate, v is the fluid velocity at the orifice plate and CV is cavitation number (Petkovic et al., 2019).

3.4 Effect of inlet pressure on color and TPC removal

At 20 min of treatment, it is observed that the highest color removal efficiency is achieved to the inlet pressure at 3.6 bar compared to 2 bar, for both configurations. The values achieved are 32.71% ± 0.067 and 31.05% ± 0.37, respectively. This high efficiency is attributed to the formation, development, growth, and implosion of cavities in the fluid, a phenomenon induced by pressure variation and the increase in velocity due to the cavitation (Nieto et al., 2021). On the other hand, at the inlet pressure of 2 bar, the efficiency is significantly lower, reaching 26.13% ± 0.034 of color removal at 20 min with the 16-orifice plate, as shown in Figure 2a. The initial increase in efficiency is explained by the generation of a sufficient number of cavities, the collapse of which produces reactive •OH radicals, which attack the compounds responsible for the color, generating intermediate products that continue to react with the •OH radicals until their complete mineralization (Poblete et al., 2020). However, after 60 min, a general decrease in color removal efficiency is observed for both configurations, being more pronounced in the 2 bar and 9 orifices condition, with a reduction of 2.45% ± 0.067. This behavior can be attributed to the fact that, initially, these gases facilitate the generation of cavities. However, as the process progresses, they are eliminated from the medium, which decreases the effectiveness of cavitation (Thanekar et al., 2018).

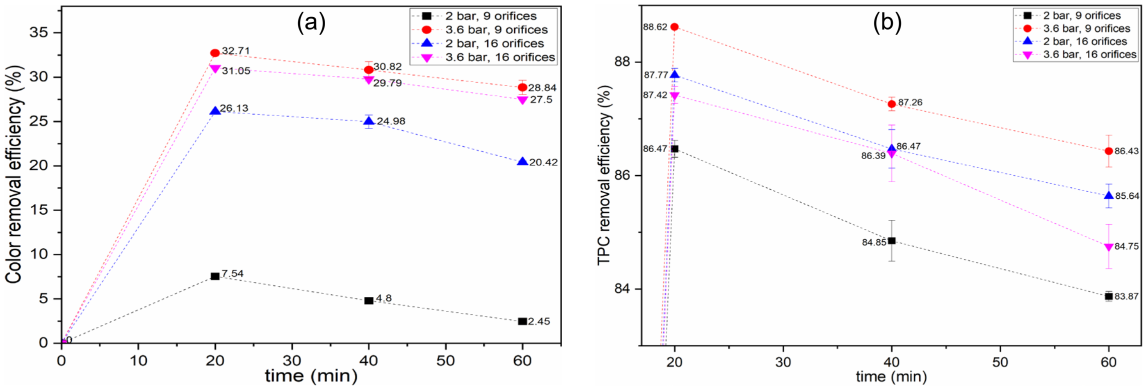

Figure 2: Effect of inlet pressure on removal (a) color and (b) TPC

The maximum TPC removal at 20 min is 88.62% ± 0.035 at 3.6 bar and 9 orifices, while with 16 orifices a similar value of 87.77% ± 0.12 is reached with 2 bar, as shown in Figure 2b. On the other hand, at the pressure of 3.6 bar and 16 orifices, the TPC removal efficiency decreases to 87.42% ± 0.15, because, at higher inlet pressure, the liquid carries larger bubbles, which can escape from the cavitation unit without experiencing complete compression and expansion cycles, thus reducing the overall intensity of cavitation, decreasing its effectiveness (Baradaran & Sadeghi, 2024). Furthermore, this behavior can be explained by the nature of phenols and their solubility in water, factors that determine their interaction with cavitational bubbles and generated radicals. More soluble and simple-structure phenols are more susceptible to degradation, while complex or less soluble phenolic compounds can inhibit process efficiency (Nagarajan & Ranade, 2020). At 60 min, efficiencies decrease for all configurations, being the lowest under 2 bar and 9 orifices conditions (83.87% ± 0.086), which is consistent with the trend observed in color removal. On the other hand, oxidation and polyphenol removal showed a positive influence on color removal, since there is a significant correlation between polyphenol removal and color (Poblete et al., 2020), indicating that polyphenol removal directly contributes to the decrease in color intensity. The study confirmed previous results showing 41% color removal in 25% diluted distillery wastewater after 10 min at 5 bar (Padoley et al., 2012), and 68.8% phenol (20 mg L^{-1}) at 6 bar with CV of 0.23 at 60 min (Baradaran & Sadeghi, 2024). These findings confirm that inlet pressure is a critical factor in optimizing cavitation for wastewater treatment. The discrepancies observed may be attributed to the type of cavitator used or to the properties of the treated effluent.

3.5 Effect of orifice number on color and TPC removal

Figure 3a shows that removing color efficiency is higher than the TPC removal efficiency when the number of orifices increases from 9 to 16 orifices at the inlet pressure of 2 bar with 20 min of treatment. The results indicate that the increase in removal efficiency at the same inlet pressure is due to the expansion of the hydrodynamic cavitation region caused by the appropriate increase in the number of orifices (Yi et al., 2021). However, at an inlet pressure of 3.6 bar, the removal efficiency decreases in both cases. This behavior is due to the formation of large bubbles by cavity fusion, which, instead of effectively imploding, tend to escape from the liquid, thus reducing the cavitational performance (Lu et al., 2019). Furthermore, when the number of orifices increases, the pressure at each orifice decreases, resulting in a decrease in the cavitation intensity (Yi et al., 2021). Similar results have been reported in the literature on the effects of orifice plate geometric parameters on the removal of Bisphenol (Lu et al., 2019) and Norfloxacin (Yi et al., 2021), with different orifice plate configurations.

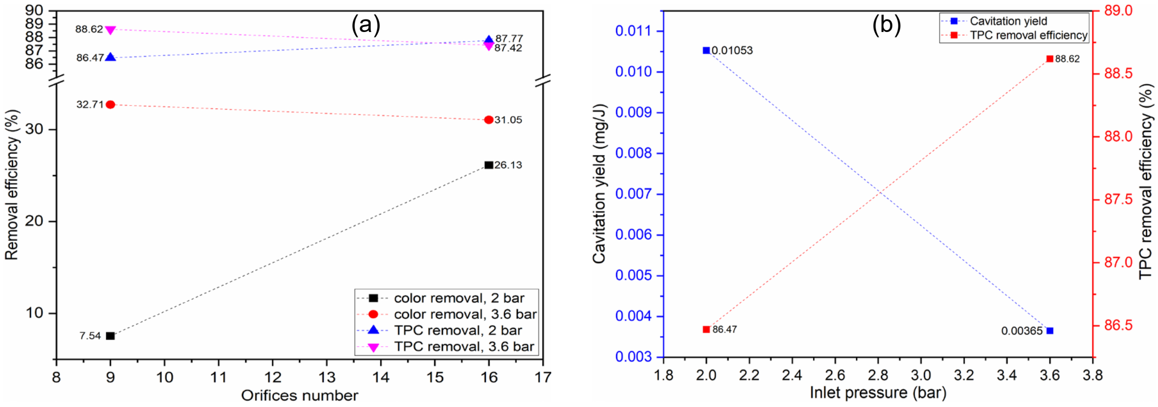

Figure 3: effect of (a) the number of orifices on color and TPC removal (b) the inlet pressure on cavitation yield

3.6 Effect of inlet pressure and cavitation yield

Figure 3b shows that the cavitation yield decreases from 1.053×10^{-2} to 3.65×10^{-3} mg J^{-1} by increasing the inlet pressure from 2 to 3.6 bar, while the TPC removal efficiency increases, using a 9-orifice plate for 20 min of treatment. This indicates that at higher pressures, the cavitation process is less efficient in terms of the energy needed to generate cavitation. On the contrary, the process remains highly effective in removing TPC under higher pressures. The results of this study are consistent with previous research that reported a cavitation yield of 6.25×10^{-5} mg J^{-1} for oseltamivir phosphate removal, using a Venturi cavitator and orifice plates (Katiyar et al., 2024). However, the cavitation yield obtained in this work was significantly higher than that of studies that using different orifice plate configurations (Katiyar et al., 2024; Lu et al., 2019).

4. Conclusions

Inlet pressure showed a more significant effect than number of orifices on the removal of color and polyphenols from sugarcane molasses ethanol vinasse. The highest color and polyphenol removal efficiency was achieved with an inlet pressure of 3.6 bar and a 9-orifice plate with a diameter of 1.5 mm, arranged in a quadrangular arrangement, with a cavitation number of 0.36 and a cavitation yield of 0.00365 mg J^{-1} during 20 min of treatment. Increasing the number of orifices had a significant effect on color removal, while no significant effect was observed on polyphenol removal. These findings provide a solid basis for optimizing contaminant reduction in vinasse through hydrodynamic cavitation using orifice plates, facilitating safer removal with reduced environmental impact.

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