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Combinative treatment of chocolaterie wastewater by a hybrid up-flow anaerobic sludge blanket reactor and solar photo-Fenton process

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ABSTRACT

The study aims to treat chocolaterie wastewater by combining the primary anaerobic treatment and solar photo-Fenton process. A laboratory scale anaerobic treatment was conducted in a hybrid up-flow anaerobic sludge blanket reactor at organic loading rates (OLRs) ranging from 0.413 to 18.2 kg COD/m^3/d over a period of 320 d. The optimum OLR of the anaerobic reactor was found to be 16.5 kg COD/m^3/d, and the corresponding chemical and biochemical oxygen demand (COD and BOD, respectively) removal at this OLR were 88% and 91%, respectively. A maximum biogas production of 30 mL/d was achieved. The effluent from the anaerobic treatment was further treated through the solar photo-Fenton process. At the optimised conditions, photo-Fenton treatment achieved COD and BOD removals of 83% and 85.4%, respectively. The predicted optimum parameters for the solar photo-Fenton process were 0.25 g/L of Fenton, 0.85 g/L of H_2O_2, and 30 min of solar exposure. Combining solar photo-Fenton with primary anaerobic treatment resulted in a COD removal of 96%, which completed the wastewater treatment.

Keywords: Chocolaterie wastewater; Anaerobic treatment; HUASB; Fenton oxidation; Biogas

1. Introduction

Energy recovery during wastewater treatment has acquired greater importance [1], with many studies focused on this area. Anaerobic treatment is a common method for treating wastewater with high organic content. In addition to wastewater treatment, it produces the fuel gas, methane [2]. Among the various anaerobic treatment processes, the hybrid up-flow anaerobic sludge blanket (HUASB) reactor is widely employed in the treatment of domestic and industrial wastewater [3]. The advantages of using HUASB include better gas–solid separation, prevention of the formation of a dead area inside the reactor, and the attached growth process [4,5]. A limitation of the HUASB treatment is that the effluent cannot achieve the disposal standards, which necessitates further treatment of HUASB-treated wastewater [6–9].

Advanced oxidative processes (AOPs), such as those using TiO_2 and Fenton, can serve as effective posttreatment options [10,11]. Among these, Fenton oxidation is efficient and can rapidly mineralise or break down organic substances [12]. Fenton oxidation is improved by UV-light provided as solar energy, which reduces the operational cost [13]. The response surface methodology (RSM) was followed to study, investigate, and predict the results of the solar Fenton process. The RSM model has been used to optimise the Fenton treatment of various wastewaters, such as those from petroleum refineries [14], the hexogen industry [15], coking [16], paper milling [17], textile production [18], and car washing [19]. RSM is a widely

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accepted statistical method of analysing and optimising the Fenton process without the need to conduct many experiments [20]. Coupling AOP with primary anaerobic treatment solves the problem of the high organics presence in the effluent stream. The combined treatment has been successfully applied to wastewaters such as those from poultry farming, olive milling, dairy, landfill leachate, and domestic applications [21,22].

In this study, HUASB was combined with the solar photo-Fenton process to treat chocolaterie wastewater, which is classified as a high-strength organic wastewater that contains non-toxic strong organic substances without any hazardous particles [23], and high total solids (TS), biochemical oxygen demand (BOD), and chemical oxygen demand (COD). Processes such as harvesting, cleaning, fermentation, drying, roasting, grinding, pressing, pulverising, and mixing consume water, thereby generating wastewater [24]. Per the literature survey, activated sludge treatment is a widely adopted conventional method for treating chocolaterie wastewater; however, it has a major disadvantage in that it requires a larger area and high financial support. A novel attempt to reduce this issue has been made by combining HUASB and solar photo-Fenton treatment for high-strength organic chocolaterie wastewater. The objective of this study is to assess the operational parameters for the primary anaerobic treatment by HUASB reactors, optimise the parameters of the posttreatment process (solar photo-Fenton treatment), and investigate the overall treatment efficiency of the combined process.

2. Materials and methods

2.1. Wastewater sampling

The chocolaterie wastewater was generated by a chocolate and sweet candies manufacturing industry – Chennai Sweets, Tenkasi, Tirunelveli, Tamilnadu, India. It was preserved by refrigeration at 4°C. The physicochemical characteristics of the chocolaterie wastewater were determined following the standard procedure and are summarised in Table 1.

2.2. HUASB reactor

The HUASB reactor was fabricated from plexiglass with an internal diameter and height of 11 and 88 cm, respectively, with a working volume of 5.9 L and a gas headspace of 1.5 L above the effluent mark. A screen was placed at a height of 60 cm to prevent floating carriers from being washed away, which consisted of 150 plastic cut rings. A peristaltic pump (Model: PP 20, Miclins, Chennai) was used to feed wastewater into the reactor. The effluent pipeline was connected to a water seal to prevent gas from leaking, and the gas outlet was connected to a wet gas meter. The schematic representation of the HUASB reactor was given by Sokkanathan et al. [12]. This HUASB reactor was operated under mesophilic conditions.

Table 1
Initial characterization of chocolaterie wastewater

Parameters	Before treatment	After HUASB treatment
Total suspended solids (mg/L)	860	NA
BOD (mg/L)	1,400	145
TCOD (mg/L)	4,090	480
SCOD (mg/L)	1,350	NA
Alkalinity (mg/L)	550	2,020
pH	6.5	6.8

BOD, biochemical oxygen demand; HUASB, hybrid up-flow anaerobic sludge blanket; NA, not applicable; TCOD, total chemical oxygen demand; SCOD, soluble chemical oxygen demand.

2.2.1. Start-up phase

During the start-up phase, the HUASB was operated at a COD of 1,000 mg/L and an organic loading rate (OLR) of 0.413 kg COD/m^3/d. The highest applied OLR in the start-up phase was 4.13 kg COD/m^3/d, which was achieved by increasing the flow rate from 100 to 1,000 mL/h over a period of 210 d.

2.2.2. Treatment phase

During the treatment phase, the HUASB reactor was operated at a constant flow rate of 1,000 mL/h, and the organic load was varied from 2,000 to 4,000 mg/L with the respective OLR varying from 8.27 to 16.55 kg COD/m^3/d. At the end of the treatment phase, OLR was increased by increasing the flow rate from 1,000 to 1,100 mL/h, with a respective OLR of 18.2 kg COD/m^3/d.

2.3. Solar photo-Fenton treatment of biologically treated retting-pond wastewater

The HUASB-treated chocolaterie wastewater was subjected to posttreatment by the solar photo-Fenton process in triplicate. To conduct solar photo-Fenton treatment in the batch mode, 500 mL Erlenmeyer flasks were utilised. Approximately 300 mL of chocolaterie wastewater was added to the flask with 0.2–0.3 g/L and 0.8–0.9 g/L of Fenton and hydrogen peroxide, respectively. The flask was then kept open in an air shaker under solar radiation at 150 rpm for 120 min. Samples were collected periodically, and the treatment efficiency was determined. The solar photo-Fenton treatment was optimised through RSM, with a central composite design created with Design-Expert software 8. During analysis, three independent variables were examined: time, H_2O_2 dosage, and Fenton dosage. The COD removal efficiency was considered as a response variable and was fitted using the predictive function of variables. The general form of COD-removal efficiency (%) can be written as the following quadratic equation:

$$X = \beta o + \beta_1 Y_1 + \beta_2 Y_2 + \beta_3 Y_3 + \beta_{12} Y_1 Y_2 + \beta_{23} Y_2 Y_3 + \beta_{31} Y_3 Y_1 + \beta_{11} Y_1^2 + \beta_{22} Y_2^2 + \beta_{33} Y_3^2 \quad (1)$$

where X is the response (COD removal efficiency in %); Y_1, Y_2, and Y_3 are the coded levels of the variables; and β_o, β_1, β_2, β_3, β_{11}, β_{22}, β_{33}, β_{12}, β_{23}, and β_{31} are the model coefficients estimated from the observed data. The response and variable values were studied by the response surface function to determine the values of the coefficients in Eq. (1). The interactive effects of these three independent variables were also explored.

2.4. Analytic study

As described in the American Public Health Association standard methods [25], the COD, BOD, TS, suspended solids, alkalinity, and volatile fatty acids (VFA) of raw and treated wastewater were analysed. The methane content of the biogas was measured by gas chromatography (Model: GC 1000, Chemito, Mumbai) equipped with a flame ionisation detector using a Porapak Q column.

2.5. Statistical analysis

The solar photo-Fenton process was analysed in triplicate, and the results were expressed as the average of three values. One-way analysis of variance (ANOVA) was used to test the significance of the results; statistical significance was considered at $p < 0.05$.

3. Results and discussion

3.1. Anaerobic treatment

The HUASB reactor efficiency was regulated by various operational parameters, such as flow rate, hydraulic retention time (HRT), and OLR [26]. Of these parameters, OLR was a major influential parameter on the efficient operation of HUASB [27,28]. Anaerobes play a vital role in oxidising the organic matters, thereby enhancing biogas generation. Dextrose was used as a nutrient to maintain the culture over solid media. Fig. 1 presents the removal of COD during the anaerobic treatment of chocolaterie wastewater by HUASB. The figure shows that the anaerobic treatment efficiency improved with increasing OLR during the start-up phase. The COD removal increased as HUASB operation continued. For example, on day one, the COD removal efficiency by the HUASB reactor was 45%. It then increased to 90% at the end of the start-up phase at an OLR of 4.13 kg COD/m^3/d. During the treatment phase (day 210–300), that is, up to an OLR of 16.55 kg COD/m^3/d, the COD removal efficiency was between 88% and 91%. Borja et al. [29] reported a similar COD removal efficiency range for a HUASB operating at OLR values of 12–17 kg COD/m^3/d. At this OLR, the influent COD loading was between 4,150 and 4,250 mg/L. When the OLR was increased from 16.5 to 18.2 kg COD/m^3/d, the COD removal efficiency decreased from 88% to 51%, and the corresponding effluent COD levels were approximately 480–1,989 mg/L.

The influence of OLR on BOD and its removal efficiency throughout the chocolaterie wastewater treatment is shown in Fig. 2. In the start-up phase, the BOD removal from wastewater increased slowly as the OLR increased. During the treatment phase, the removal rate increased, reaching an OLR of 16.55 kg COD/m^3/d. The influent BOD remained within a range of 2,440–2,480 mg/L. The maximum BOD removal of 91% was obtained at an optimised OLR of 16.55 kg COD/m^3/d and a 5.8 h HRT, resulting in an effluent BOD concentration of 200–230 mg/L. When the OLR was increased beyond 16.55 kg COD/m^3/d, there was a gradual decrease in process efficiency. The BOD value increased from 200 to 1,200 mg/L when the OLR was increased from 16.55 to 18.2 kg COD/m^3/d.

VFAs are important metabolites of anaerobic fermentation and are produced by acetogenesis during wastewater treatment [30]. The VFA concentration in the wastewater was stable up to an OLR of 16.5 kg COD/m^3/d and was varied in the range of 350–550 mg/L. When the OLR was increased to 17.3 kg COD/m^3/d, the VFA concentration increased as shown in Fig. 3. At the highest applied OLR (18.2 kg COD/m^3/d), the VFA concentration was approximately 1,500 mg/L. At this concentration, souring of the HUASB reactor can occur [10,31]. The produced VFA should be utilised immediately by methanogens to prevent it from building up during anaerobic reactions. If it is unused by methanogens, then it must be buffered; otherwise, the reactor will fail [11,32,33]. Alkalinity can act as a buffering agent in an anaerobic digester when the VFA to alkalinity ratio is within the range of 0.4–0.2, which prevents the pH inside the reactor from decreasing. Up to an OLR of 16.5 kg COD/m^3/d, the alkalinity was within the recommended level for the anaerobic digestion of effluent. However, when the OLR was increased to 17.3 kg COD/m^3/d, the alkalinity gradually reduced. At the highest applied OLR, the VFA to alkalinity ratio was 1.32, which indicates a lower buffering capacity.

The influence of OLR on biogas generation and cumulative biogas production is shown in Fig. 4. For effective anaerobic treatment, the pH must be neutral [3,34]. During the start-up phase, the pH of the wastewater was between

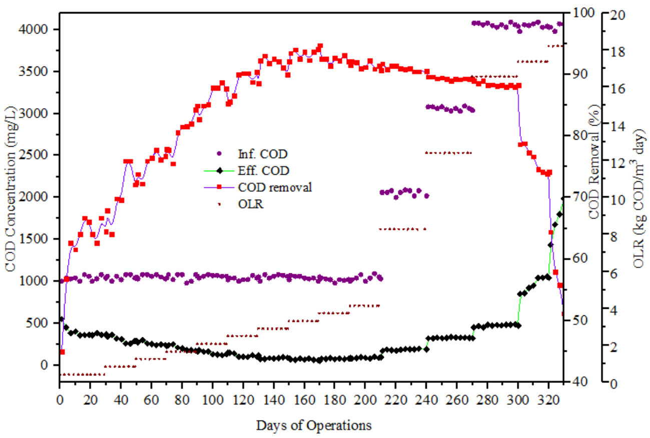

Fig. 1. Influence of OLR on COD and COD removal throughout the treatment of chocolaterie wastewater.

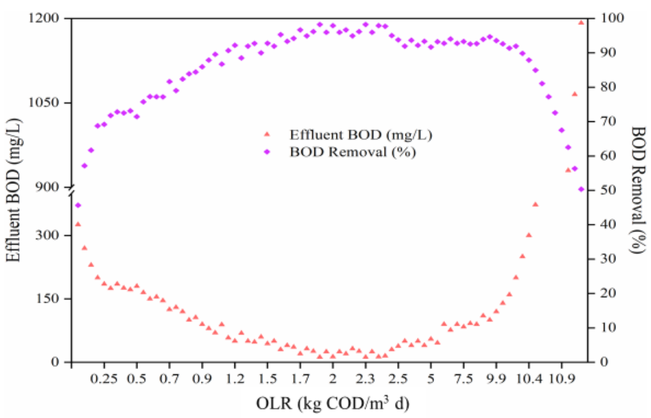

Fig. 2. Influence of OLR on BOD and BOD removal throughout the treatment of chocolaterie wastewater.

6.5 and 6.8. This favours the anaerobic process up to an OLR of 3.3 kg COD /m³/d for 170 d. The pH increased to 7 at an OLR of 4.13 kg COD/m³/d. Within the period of 210 d, the biogas increased from 0.3 to 8.2 mL/d as the OLR increased from 0.413 to 4.13 kg COD/m³/d. Biogas generation increased as the reaction progressed. During the treatment phase, the pH was ranged from 6.8 to 8.5, promoting anaerobic degradation. A maximum biogas generation of 29.5 to 30 mL/d was achieved for an OLR of 16.55 kg COD/m³/d, with an 88% COD removal efficiency. As the OLR exceeded 16.55 kg COD/m³/d, the pH of the wastewater decreased to 5.3 after 300 d of operation. This was due to the lower alkalinity and higher accumulation of acid, which affected anaerobic digestion. A cumulative biogas yield of 700 mL was achieved during the biological treatment of chocolaterie wastewater. Thus, an OLR of 16.55 kg COD/m³/d is considered favourable for anaerobic treatment at 5.8 h HRT.

3.2. Solar photo-Fenton treatment

3.2.1. Optimisation of process parameters for photo-Fenton (post) treatment by the response surface method

The RSM was used to analyse the independent parameters that were involved in the stimulation of Fenton oxidation, and a graph of this was plotted. The coded factors were generated during the RSM analysis. Using these coded factors, the interactive and significant effects of parameters towards a response were predicted in the form of an empirical model. The effect of the reaction was determined by the coefficient of an unknown factor; the single factor coefficient indicated the effect by that factor alone. The second-order double factor coefficient indicated an interactive effect between the two factors. Table 2 shows the experimental run of the solar photo-Fenton process using design experts. Eq. (2) shows the interaction between the response factor (i.e., COD-removal efficiency) and parameters:

$$\begin{aligned}\text{COD removal efficiency} = &-1172.96 + 1732.45\,A + 2276.58\,B \\ &+ 2.26\,C - 1050\,AB + 0.75\,BC \\ &+ 2.08\,CA - 1630.78\,A^2 \\ &- 1206.52\,B^2 - 0.04\,C^2\end{aligned} \tag{2}$$

where A, B, and C are the time (min), H_2O_2 dosage (g/L), and Fenton dosage (g/L), respectively. The model competence was evaluated by R^2, the *p*-value, and lack of fit.

The model *F*-value of 4,850.09 indicates that the model is significant. There is only a 0.01% chance that an *F*-value this large could occur due to noise. *p*-values below 0.05 indicate that the model's terms are significant. In this case, A, B, C, AB, AC, BC, A^2, B^2, and C^2, are significant model terms. The interactive effects of time, H_2O_2 dosage, and Fenton dosage are indicated as AB, BC, and AC. The Table 3 shows that the *p*-values of the interactive parameters AB, BC, and AC were below 0.05. This indicates that the interactive effects of time, H_2O_2 dosage, and Fenton dosage have a major influence on Fenton oxidation and promote efficient degradation. Values that are greater than 0.1 indicate that the model terms are not significant.

The lack of fit *F*-value of 2.12 indicates that this parameter is not significant relative to pure error. There is a 21.47% chance that a lack of fit *F*-value this large could occur due to noise. Analysis and validation of the model by ANOVA determined the correlation coefficient R^2. The predicted R^2 of 0.9984 is in reasonable agreement with the adjusted R^2 of 0.9996; the difference is less than 0.2. Therefore, the R^2 indicated goodness of fit. The ANOVA results for the quadratic model with the COD-removal efficiency as a response variable is shown in Table 3. The coefficient of variance (CV) represents the ratio of the standard error of the estimate to the mean value of the observed model (represented as %). The CV value was 1.31%, indicating that the precision and reliability of the experiment were high. Adequate precision measures the signal-to-noise ratio, and a ratio greater than 4 is desirable. The ratio of 179.234 indicates an adequate signal. Therefore, this model can be used to navigate the design space, and the response surface models could be used to predict, analyse, and optimise the solar photo-Fenton process.

A 3D plot showing the interactive effects of independent variables and responses during the solar photo-Fenton process is presented in Fig. 5. At the effective acidic pH of 3, the maximum COD removal efficiency during Fenton treatment of 83% was achieved. The corresponding effluent COD concentration was 76.5 mg/L. A higher removal efficiency was achieved at the optimised condition of 0.25 g/L of Fenton,

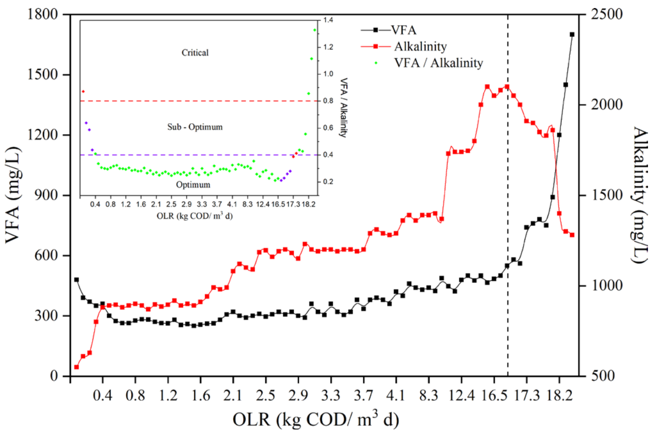

Fig. 3. Variation of VFA, alkalinity, and VFA/alkalinity throughout the treatment of chocolaterie wastewater.

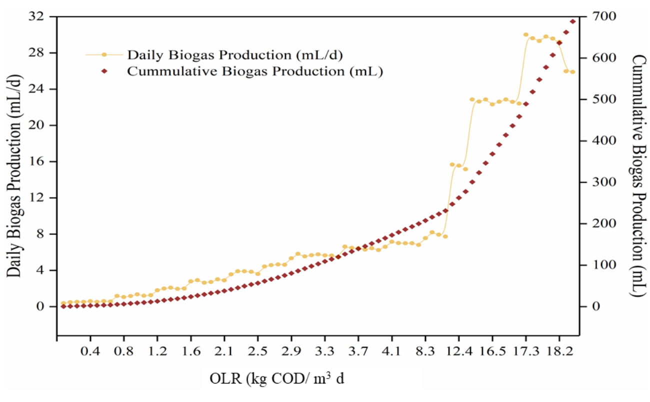

Fig. 4. Plot showing the biogas production during biological treatment of chocolaterie wastewater.

Table 2
Experimental run of design expert in optimizing solar photo-Fenton process

Std.	Run	Factor 1	Factor 2	Factor 3	Response 1
		A-Fenton dosage (g/L)	B-Hydrogen peroxide dosage (g/L)	C-Time (min)	COD removal efficiency (%)
10	1	0.33409	0.85	30	75
3	2	0.2	0.9	0	–9
16	3	0.25	0.85	30	82
18	4	0.25	0.85	30	83
19	5	0.25	0.85	30	81.5
4	6	0.3	0.9	0	–12
12	7	0.25	0.93409	30	76
13	8	0.25	0.85	20	64
1	9	0.2	0.8	0	–10
11	10	0.25	0.76591	30	71
7	11	0.2	0.9	60	78
2	12	0.3	0.8	0	–3
17	13	0.25	0.85	30	83
20	14	0.25	0.85	30	82.5
14	15	0.25	0.85	80.4538	30
5	16	0.2	0.8	60	64
9	17	0.16591	0.85	30	66
6	18	0.3	0.8	60	76
15	19	0.25	0.85	30	82.7
8	20	0.3	0.9	60	79

COD, chemical oxygen demand.

Table 3
ANOVA analysis for quadratic response surface model regarding COD removal efficiency

Source	Sum of squares	df	Mean square	*F*-value	*p*-value	
Model	24,163.45	9	2,684.83	4,850.09	<0.0001	Significant
A-Fenton dosage (g/L)	75.62	1	75.62	136.61	<0.0001	
B-Hydrogen peroxide dosage (g/L)	22.19	1	22.19	40.09	<0.0001	
C-Time (min)	15,406.10	1	15,406.10	27,830.80	<0.0001	
AB	55.13	1	55.13	99.58	<0.0001	
AC	10.13	1	10.13	18.29	0.0016	
BC	78.13	1	78.13	141.13	<0.0001	
A^2	238.60	1	238.60	431.03	<0.0001	
B^2	130.60	1	130.60	235.93	<0.0001	
C^2	15,449.21	1	15,449.21	27,908.68	<0.0001	
Residual	5.54	10	0.5536			
Lack of fit	3.76	5	0.7521	2.12	0.2147	Not significant
Pure error	1.78	5	0.3550			
Correlation total	24,168.99	19				

ANOVA, analysis of variance; COD, chemical oxygen demand.

0.85 g/L of hydrogen peroxide, and a reaction time of 30 min. During the solar photo-Fenton process, Fenton acts as a catalyst, stimulating the oxidation of hydrogen peroxide and causing the generation of a hydroxyl radical [35]. This generated hydroxyl radical has a high oxidative potential in the degradation process. The following equations detail the oxidation of organic compounds and generation of the hydroxyl radical during the Fenton process [36].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + \cdot OH \tag{3}$$

$$Fe^{2+} + \cdot OH \rightarrow Fe^{3+} + OH^- \tag{4}$$

$$RH + \cdot OH \rightarrow H_2O + R. \tag{5}$$

$$R. + Fe^{3+} \rightarrow R^+ + Fe^{2+} \tag{6}$$

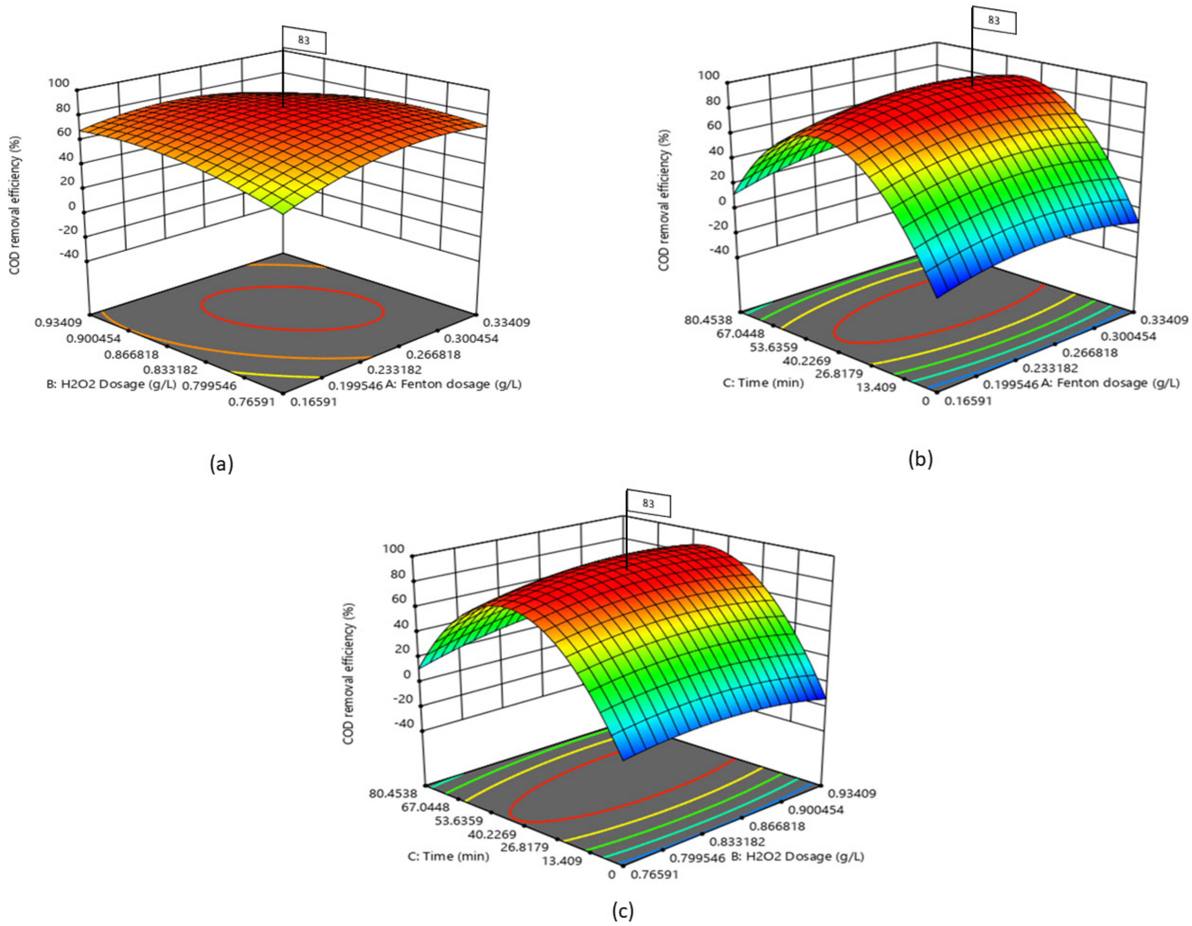

Fig. 5. 3D response and contour diagrams depicting the effects of various independent factors over the response factor.

This might be because the OH radical can degrade the organic matter in the wastewater by photo-Fenton oxidation, during which Fe^{2+} converts H_2O_2 into a hydroxyl radical, which in turn oxidises organic compounds.

3.2.2. Optimisation of the solar photo-Fenton process using the removal profiles of COD and BOD

The COD and BOD removal efficiency of the solar photo-Fenton treatment of chocolaterie wastewater is shown in Fig. 6. The COD and BOD concentrations decreased when the reaction duration increased from 0 to 60 min under optimum Fenton and hydrogen dioxide dosages (0.25 and 0.85 g/L, respectively). When the exposure time was 30 min, the COD and BOD removal efficiencies increased gradually. This was due to the occurrence of a rapid reaction between Fenton and hydrogen peroxide, leading to the generation of a hydroxyl radical, which degrades and mineralises organics. After further increasing the exposure time beyond 30 min, the COD and BOD removal efficiencies were stable due to the formation of radicals, such as per hydroxyl radicals. These additional radicals reduce the concentration of hydroxyl species and oxidation potential, affecting the Fenton reaction rate [13]. The maximum COD and BOD removal efficiencies were 83% and 85.4% with concentrations of 76.5 and 21.5 mg/L, respectively. Advanced Fenton methods are effective for organic degradation. Therefore, 30 min was considered as the optimum reaction time for the solar photo-Fenton process. During the disposal of wastewater, the pH needs to be adjusted to prevent sludge formation and protect the aquatic ecosystem.

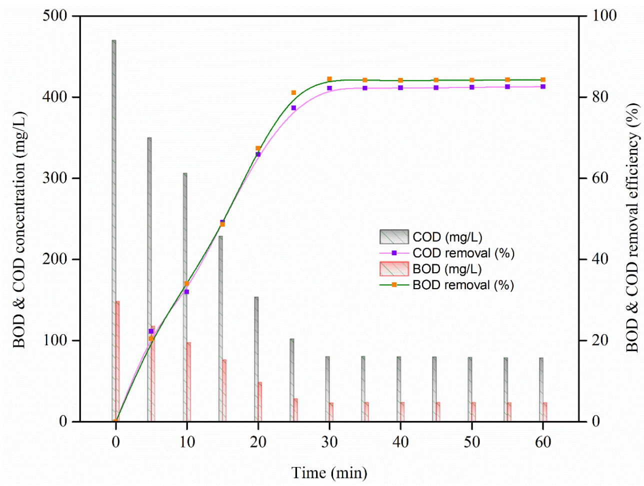

Fig. 6. Plot showing the COD and BOD concentration with its removal efficiency during photo-Fenton treatment of biologically treated chocolaterie wastewater.

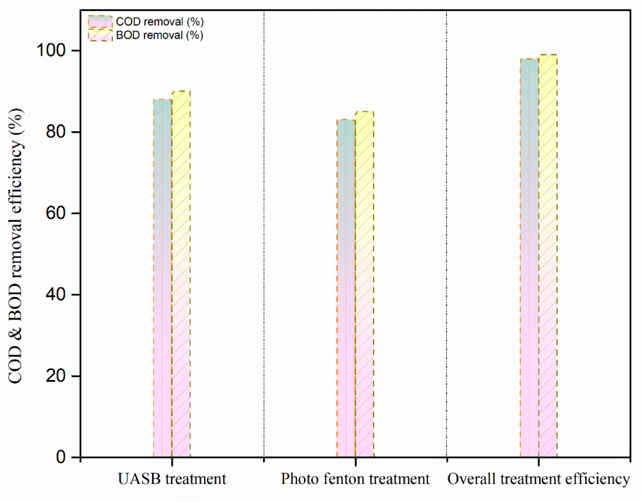

Fig. 7. Plot showing the overall treatment efficiency of the combined UASB and photo-Fenton treatment.

3.3. Overall efficiency of the integrated HUASB and solar photo-Fenton process

The combined treatment of chocolaterie wastewater achieves removal efficiencies of 98% and 99% for COD and BOD, respectively. This will assist in the safer disposal of wastewater. Individual treatment methods exhibited lower efficiencies than the combined treatment, as shown in Fig. 7. The solar photo-Fenton process was an effective posttreatment method for HUASB. This combined treatment is eco-friendly and can be applied in the field.

4. Conclusion

The HUASB treatment of chocolaterie wastewater at an OLR of 16.55 kg COD/m^3/d yielded COD and BOD removal efficiencies of 88% and 91%, respectively. The VFA/alkalinity ratio of the reactor governed the treatment efficiency of HUASB, with a maximum biogas production of 4.2 L/d recorded for an OLR of 16.55 kg COD/m^3/d. The wastewater at treated at an OLR of 16.55 kg COD/m^3/d was further treated by the solar photo-Fenton process. The RSM modelling indicated that 0.25 g/L of Fenton, 0.85 g/L of H_2O_2, and a reaction time of 30 min were the optimum conditions for treatment. The coupling of HUASB with photo-Fenton treatment resulted in overall COD and BOD removal efficiencies of 98% and 99%, respectively.

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