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Comparative study of advanced oxidation treatment of tannery effluent in multi-orifice oscillatory baffled column with sono-catalytic treatment using Titanium Dioxide

Saravanan Jegnanthan[1] Kannan Kandasamy[2,*] and M.D. Duraimurugan alias Saravanan[3]

[1]Department of Chemical Engineering, Mohamedsathak Engineering College, Kilakarai, Ramanathapuram, Tamil Nadu, India

[2]Department of Chemical Engineering, Kongu Engineering College, Perunduari, Erode, Tamil Nadu, India

[3]Department of Chemical Engineering, Department of Chemical Engineering Dhanalaxmi Srinivasan Engineering College (Autonomous)

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*to whom all correspondence should be addressed: e-mail: kannank@kongu.ac.in

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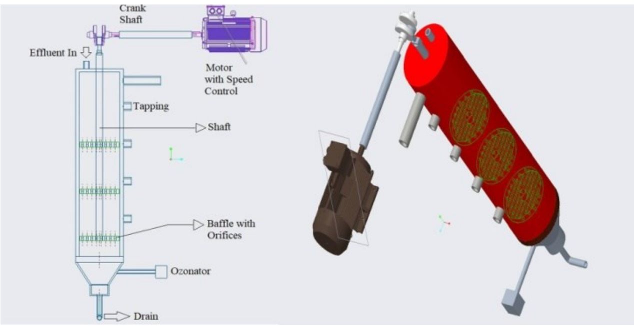
Graphical abstract



Abstract

Treatment of tannery effluent is a key issue that requires novel research. The presented work is an attempt to carry out advanced oxidation process in a Multi-orifice Oscillatory Baffled Column using ozone as oxidizer and compare the extent of treatment with sono-catalytic treatment with TiO_2as catalyst. Studies have been carried out to understand the effect of treatment time, concentration of effluent and oscillation frequency upon ozonation and the effect of power of ultrasound, time of treatment and catalyst loading for sono-catalytic treatment. For ozonation the COD, BOD and TDS reduction obtained are 88.8%, 84.01%, 90.73% respectively and for sono-TiO_2 treatment the reduction of COD, BOD and TDS obtained are 91.2%, 91.5%, 94%. Optimization and analysis of variables is carried out to identify the effective factors for treatment.

Keywords: Tannery effluent, MOBC, Sono-Cataltic Treatment, TiO_2, COD, BOD and TDS

1. Introduction

Industrialization is major key to the economical growth of a country. Among many sectors, process industries like tannery, textile, and petrochemicals play a major role on economical development of it**.** Due to the industrialization the usage of process water for the product conversion increases and also the discharge of effluent is much higher to the environment. Especially the effluent from Tannery industry produces wastewater containing toxic chemicals both organic and inorganic which has an adverse effect on aquatic life when disposed into water bodies with contaminants, it also affects the soil and ground water when disposed into land (Ambigai and Annadurai 2017). Chrome tanning is the process of converting raw skin to leather to enhance its shelf life which uses a lot of chemicals such as lime, sodium sulfide, chrome salts, etc (Cooman *et al.* 2002). Some existing effluent treatment techniques practiced are sedimentation, electro flotation, filtration, membrane filtration, precipitation, coagulation, adsorption, ion-exchange and biological methods(Syed Waqas Ahmad *et al.* 2019). These existing methods utilize chemicals like lime, sodium sulfide, ammonium sulfate, sodium chloride, alum and release the same into the environment after the treatment process. These chemicals will increase the load of organic and inorganic contaminants in the water bodies. Tannery effluent treatment by above methods is not meeting out the environmental standards (Robina Farooq *et al.* 2013). Therefore, there is a need to develop advanced technologies to achieve zero liquid discharge in sustainability perspective (Tirzhá Lins Porto Dantas *et al.* 2003).

Usages of new and eco-friendly adsorbents from wastes and bio-sorption of heavy metals have been fruitful in effluent treatment (Ugya*et al.* 2018). Low-cost adsorbents like neem bark and cotton shell were used to remove contamination from textile effluent in a continuous column. However, the technology for desorption and regeneration of adsorbents is yet to be developed (Kannan *et al.* 2012). The above literatures indicate that the conventional treatment methods suffer from ecological demerits of secondary effluent generation. Hence a new technology that is capable of treating the effluent without secondary pollutants generation is essential. Among upcoming technologies, advanced oxidation process is found to be viable for degradation of pollutants in the tannery effluent (Shivendu Saxena *et al.* 2018; Xiaobin Zhou *et al.* 2016). Reagents like fenton, gases like ozone etc are being utilized for advanced oxidation of different effluents. Fenton and Photo-fenton processes have shown reduction of COD and BOD in simulated tanning effluents (Sivagami*et al.* 2018). Ozone is an excellent oxidizing agent which has been used to disintegrate contaminants present in various effluents like textile, tannery etc. The ozonation treatment of effluent improves the biodegradability and the extent of ozonation depends on the dosage of ozone, treatment time and effluent characteristics (Balakrishnan *et al.* 2002).

Many contactors like bubble columns, packed columns, sparged agitated vessels have been used for operating gas liquid and liquid-liquid systems and mass transfer has been evaluated (Preethi *et al.* 2008; Sivakumar Venkatachalam *et al.* 2011). New techniques like baffling and pulsating have been studied for effective contacting, the Oscillatory Baffled Column is one which (OBC) combines the effects of baffling and oscillation thereby reducing the bubble size and increasing the contact area and the multi-orifice will increases the rate of mass transfer between multiple streams (Raf Dewil*et al.* 2017). Studies indicate that the mass transfer enhances by utilizing Oscillatory Baffled Columns for multi-phase systems as it facilitates mixing in microscopic scale by breaking the vapor liquid fragments into finer bubbles increasing the contact area (Safaa*et al.* 2018, McDonough *et al.* 2019). Ozonation is found to be intensified when it is carried out in a Multi-orifice Oscillatory Baffled Column for water and alkaline water systems utilizing the oxidizer completely (Marco S. Lucas *et al.* 2016).

Sonochemicalprocess is an emerging technology for the degradation of effluent that aids breaking and formation of chemical bonds, purification, extraction, elimination of suspended particles etc (Suslick*et al.* 1990). Sonochemical systems exploit the phenomena of cavitation, which causes extreme conditions that bring about the decomposition of contaminants (Cuiling Gong and Douglas P. Hart 1998). Conventional catalytic reactions can be intensified by ultrasound assistance, which reduces the addition of chemicals for treatment process (Armin Tauber and Heinz-Peter Schuchmann 2000). Sono-chemical treatment is found to be fruitful in treating diversified streams that contain dyes and heavy metals (Entezari and Sharif Al-Hoseini 2007, Laurent *et al.* 2009). Ultrasound assisted electrolysis of tannery effluent is found to be successful giving out 80% of chromium removal (Syed Waqas Ahmad 2019). Combined sono - TiO_2 induced advanced oxidation of tannery effluent indicated more than 85% removal of COD, BOD and TDS (Kannan Kandasamy *et al.* 2017).

The objective of the present work is to treat the tannery effluent using combinedAOPs in MOBC and sonocalaytic process. The effect of time, ozone concentration, frequency of oscillation and dosage of sonocatalyst on reduction of COD, BOD and TDS were analysed, and optimization ofabove mentioned process parameters is carried using Central Composite Design (CCD). The optimizationof process parameters is found to be effective in CCDapproach with three factors at three levels.

2. Experimental

2.1. Multi-orifice oscillating baffled column

The experimental column consists of column with baffle, motor and coupled with ozone generator. The baffle column was made up of polyacrylonitrile fibre and the multi orifice baffle plates were made of stainless steel and fitted in the stainless-steel pipe in equal spacing which is connected with the motor shaft. The MOBC internal diameter is 140 mm, height is 450 mm, the total volume was 6.175L with a working volume (V) of 5.675 L was used in this work. All experiments have been carried out at atmospheric pressure and room temperature. Ozone from ozone generator was fed from the bottom using a sparger with diameter of 0.6 mm. Q_{gas} was controlled by a ball valve. The effluent in the MOBC was kept at a constant volume, with the free liquid surface always kept well above the top of the baffle in order to avoid air entrapment from the headspace. To facilitate the oscillatory motion of the baffles, a piston is attached to the top of the column, and it is driven by a motor with variable frequency drive control. The column is equipped with 3 baffles with 80mm and the orifice having the diameter of 2mm with triangular pitch. The baffles were designed to fit closely to the column wall with the clearance of 10 mm. The diagram is shown in Figure 1.

The effluent is fed into the reactor and ozonized continuously with a retention time from 40 to 120 min, Ozone dosage 100 to 500 mg/hr and Oscillation frequency of 1-2 hz.

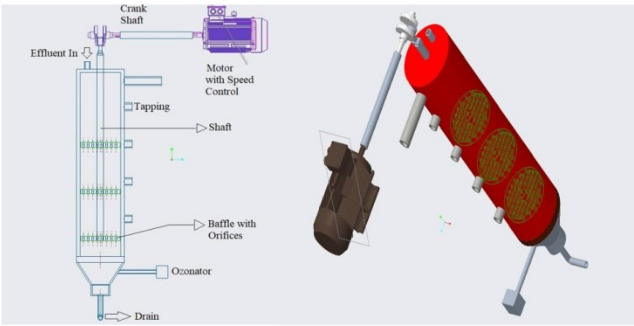


Figure 1. Diagram of Multi-orifice Oscillatory Baffled Column

2.2. Sonocatalytic reactor

Sonication of the tannery effluent was done using make Heilscher UP 200 HT, Germany. sonication probe. Sonotrode which is attached with a flowcell. A frequency of 26 kHz and a maximum power of 200W is maintained throughout the experimental study. TiO_2 catalyst was synthesized and the characterized. The preparedTiO_2 catalyst was loaded in the reactor with dosages varying from 0.05 to 0.25 g.The characterized tannery effluent was introduced into the sonocatalytic reactor varying the time of treatment between 40 and 120 mins and with the power of ultrasound varying from 20 to 100 W.

2.3. Characterization of Effluent

For thepresent experimental study, wastewater samples were collectedfrom a tannery in and around Erode district of Tamil Nadu in South India. The lab grade reagents were used throught the study. The methods used to measure parameters like BOD by microbiological titration, COD by closed reflux colorimetric method, turbidity (NTU) was measured by digital nephlometer, gravimetric method was used to measure total solids, total dissolved solids and total suspended solids (Table 1).

Table 1. Characterization of Tannery effluent

Parameter	Unit	Value
BOD	ppm	2600
COD	ppm	7700
pH	-	11.65
Turbidity	NTU	1486
Hardnss	mPa	917
Conductivity	Ω.m	46.7
TS	ppm	8550
TSS	ppm	1533
TDS	ppm	

2.4. Preparation of TiO_2 Nano Catalyst:

Sol gel method is easier method to prepare TiO_2 (Suslick*et al.* 1990). 60 ml of $TiCl_4$ was added with 200 ml of ethanol under fume hood with continuous strring. $TiCl_4$ is easily release the HCl when the Ethanol is added to it. A yellow Colored Precipitate is formed and dried at 85°C for 15h. At last, TiO_2 nano catalyst was obtained. It is then calcined to activate the surface at 200 to 800°C for 2h.

2.5. Characterization of TiO_2:

The prepared TiO_2 characterized by SEM, XRD and FTIR to confirm that the size of TiO_2 forms nano structure. The SEM analysis of sample is done in Field Emission Scanning Electron Microscopy (QUANTA - 250 FEG). to study the morphology of the catalyst prepared. The catalyst is analysed in 500 nm and size of the particle are in the range of 15-27 nm (Figure 2).

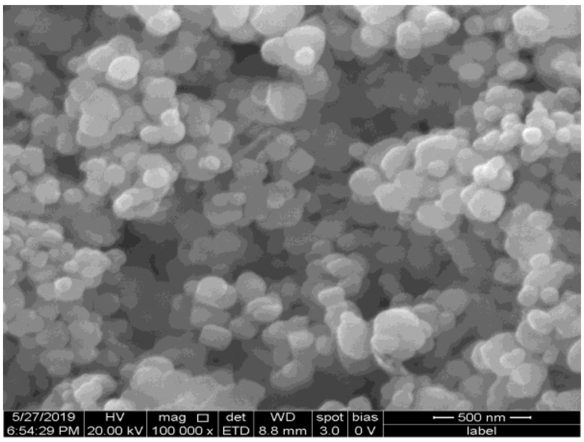


Figure 2. SEM Image of Titanium Dioxide

XRD of the synthesized catalyst sample done with X-Ray Diffractometer (Rigaku - ULTIMA -IV) and it confirms that the TiO_2 nano sized particle which a peaks at 25.33°, 32.69°, 48.08° of anatase phase. Where 25.33° having maximum peak value. The crystalline nano particle is shown by the intensity of the beam. The average crystallite size (D) for the samples is calculated using Debye-Scherrer formula (Figure 3) (Table 2).

$$D = \frac{0.9}{(FWHW*Cos)} \quad (1)$$

Table 2: FWHM data of Titanium Dioxide XRD Spectra

2theta	ƛ	FWHM	Diameter (nm)
25.33	3.512	0.199	16.27
32.693	2.73	0.121	21.21
48.085	1.89	0.130	14.33

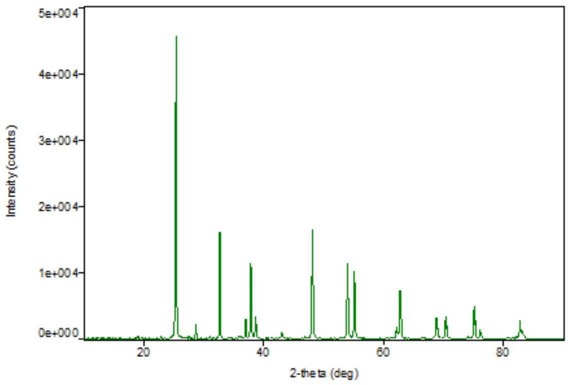


Figure 3. X-ray Diffraction Spectra of Titanium Dioxide

3. Result and discussion

In this work, The Treatment of tannery effluent by Ozonation in Multi-orifice Oscillating baffled column and Sono catalysis. The results of individual process are discussed and compared below,

3.1. Effect of MOBC – Ozonation:

Continuous ozonation of tannery effluent was carried out in the multi orifice oscillatory baffled column. Initially, the study was conducted with constant oscillation frequency, varying the ozone concentration and retention time and further the effect of oscillation frequencies was studied at a constant retention time of 2 hours and constant ozone dosage of 500 mg/hr. Since ozone is a first-grade oxidizing agent the contaminants causing the oxygen demands were considerably disintegrated and oxidized. Closer examination of the process indicated that the reduction of COD increased with increase in the ozone dosage. It was observed that the percentage Reduction of COD was maximum of 88.8% at 500 mg/hr of ozone at 120 min and 21.3 % at 100 mg/hr of Ozone at 40 min. Hence it is evident that time is also an influencing factor for the reduction of COD. It is evident that the removal of COD was observed as maximum at the time of 120 min after that the removal attains saturation. The frequency of oscillation of the baffles also has a direct influence on the reduction of COD. When oscillated at a frequency of 2 beats per second, which is equivalent to 0.033 Hz, the COD reduction was found to be maxiumum. The results obtained indicate that the ozonation is much superior to conventional electro-oxidation (Jeremi Naumczyk and Małgorzata Kucharska 2011). The observed reduction in COD is more than that is achieved at higher concentration of ozone with alkali treatment (Zabihollah Houshyar*et al.* 2012). The results obtained are exceeding the extent of removal obtained in photo-Fenton assisted electrocoagulation (Módenes*et al.* 2012). On comparison with the ozonation studies carried out using a packed bed column, the ozonation carried out in a Multi-orifice Oscillatory Baffled Column is more effective. The reduction of COD is more in the MOBC for similar conditions (Balakrishnan*et al.* 2002). This is due to the improved contact between the effluent and the ozone while treating it using a MOBC. Figure 4 shows the 3D plot of COD removal which depends on the Ozone dosage and increase the time and dosage of ozone will increases the COD removal.

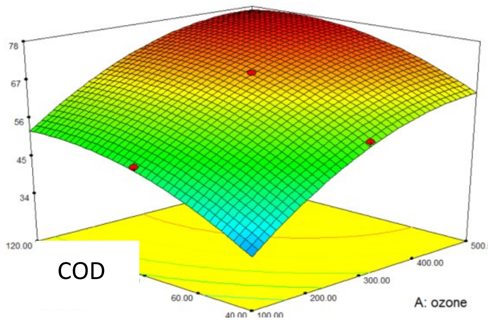


Figure 4. Effect of Ozonation on COD of tannery effluent

COD reduction = +68.76 + 13.42 A +7.91 B +3.05 C -1.25 A B -1.78 A C - 1.57 B C – 6.60 A2 – 5.36 B2 - 1.14 C2 (2)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001.Analysis of variables (ANOVA) data Ozone dosage (A), Time(B), Oscillation frequency (C) are significant and these parameter very well interact with eachother.The predicted R squared of 0.9494 is in reasonable agreement with adjusted R squared of 0.9876

BOD of effluent is depends on the bio degradeable substance which affect the environment. While treatng the effluent by MOBC, 84.01 % of removal at 500 mg/hr of Ozone with 2 hr of time and having minimal of 37.2 % at 100 mg/hr of ozone with 40 min of treatment. The results obtained for reduction of BOD is found to be improved than the ozonation in a packed bed (Balakrishnan *et al.* 2022). The BOD is found to reduce more than that of the pre alkalized treatment (Zabihollah Houshyar *et al.* 2012). This once again supports the intensification of mass transfer when it is ozonated with MOBC. 3D plot shows the BOD Reduction which depends on Ozone and Time (Figure 5).

BOD Reduction = + 74.45 + 7.41 A + 11.10 B + 2.84 C - 1.90 A B - 1.24 A C - 1.70 B C - 2.37 A2 - 8.73 B2 - 1.09 C2 (3)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001.Annalysis of variables (ANOVA) data Ozone dosage (A), Time(B), Oscillatrion frequency (C) are significant and these parameter very well interact with eachother.The predicted R squared of 0.9562 is in reasonable agreement with adjusted R squared of 0.9892.

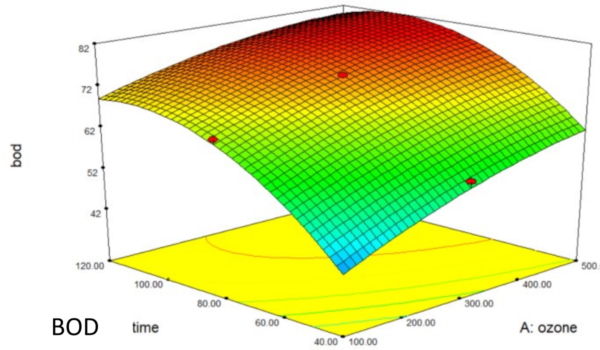


Figure 5. Effect of Ozonation on BOD of tannery effluent

TDS is produced because of the chlorides and sulfides used in the Tanning process which is more responsible for the increasing of turbidity of the effluent. By the usage of MOBC, due to the oscillation which makes the smaller size particle into bigger which reduces the TDS as well as the Ozone will enhance TDS reduction. 21.3% of TDS is reduced in 100 mg/hr of ozone at 40 min and 90.73 % of reduction in 500 mg/hr at 120 min as a minimum and maximum reduction respectively. The TDS reduction obtained is also found to be better than the ozonation in packed bed. The findings can be concluded that MOBC is a better contactor for ozonation than the conventional devices. The 3D plot shows that the ozone and time will have a impact on TDS reduction (Figure 6).

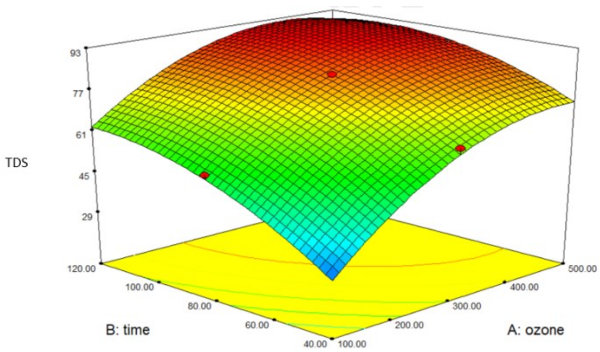


Figure 6. Effect of Ozonation on TDS of tannery effluent

TDS Reduction = + 82.96 + 17.45 A + 12.60 B + 3.38 C - 4.20 A B - 1.81 A C - 1.50 B C - 11.84 A2 - 7.57 B2 - 1.02 C2 (4)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001. An analysis of variables (ANOVA) data Ozone dosage (A), Time(B), Oscillatrion frequency (C) are significant and these parameter very well interact with each other.The predicted R squared of 0.9635 is in reasonable agreement with adjusted R squared of 0.9929.

3.2. Effect of Sono-catalytic treatment

The Sono-catalytic treatment was carried out using a probe with TiO_2 catalyst. The power of ultrasound was varied along with the quantity of catalyst added and treatment time. Since ultrasound is a powerful tool for breaking down macro-molecules, the study was carried out to understand the effect of power of ultrasound upon reduction of COD. The Sonolysis of tannery effluent will give the COD Reduction maximum 91.2% at 0.25g of catalyst for 120 min at a frequency 26 kHz with 100W and minimal amount 53.9% at 0.05g of catalyst fot 40 min at 20 W. The rate of removal of COD is gets linearised at 0.25g of catalyst loading and gets saturated at 120 min. The results obtained for the reduction of COD is better than the photo-catalytic treatment of tannery effluent using TiO2 in a parabolic trough reactor (Surya Pratap Goutam *et al.* 2018). This proves that the phenomena of cavitation is as good as the photo-excitation for tannery effluent treatment. The COD reduction obtained are better the sono-catalytic treatment of tannery effluent at 42 kHz (Kannan Kandasamy*et al.* 2017). This suggests the influence of power of ultrasound upon the treatment. 3D plot shows removal of COD which depends on the Catalyst loading and increase the time and catalyst will increases the COD removal at 100 W (Figure 7).

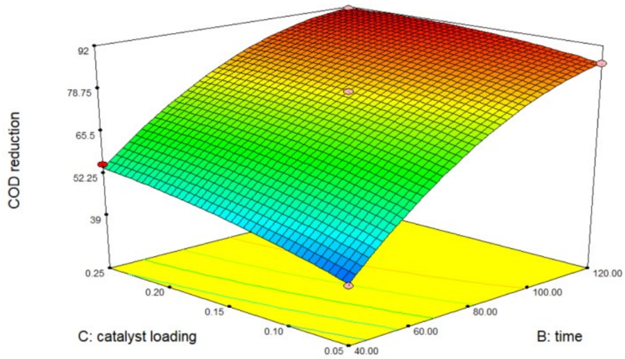


Figure 7. Effect of Sono-catalytic treatment on COD of tannery effluent

COD reduction = + 74.98 + 2.62 A + 20.99 B + 5.41 C + 0.046 A B - 0.65 A C - 2.42 B C + 0.64 A2 - 8.30 B2 - 1.91 C2 (5)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001.Analysis of variables (ANOVA) data Power (A), Time(B), Catalyst loading (C) are significant and these parameter very well interact with eachother. The predicted R squared of 0.9799 is in reasonable agreement with adjusted R squared of 0.9963.

The rate of reduction of BOD increases with increase in dosage of catalyst. For the 0.05 g catalyst at 40 min removal of BOD is 40.7 % whereas the 91.5 % is achieved when catalyst of 0.25g at time of 2h. The maxiumum reduction of BOD obtained was around 87% for the treatment at 42 kHz (Kannan Kandasamy *et al.* 2017). The influence of power of ultrasound and increased dosage of catalysts, intensifies the cavitation thereby bringing out the required decomposition of contaminants at a lower frequency of 26 kHz.The 3D graph shows the BOD removal is directly depends on catalyst and time (Figure 8).

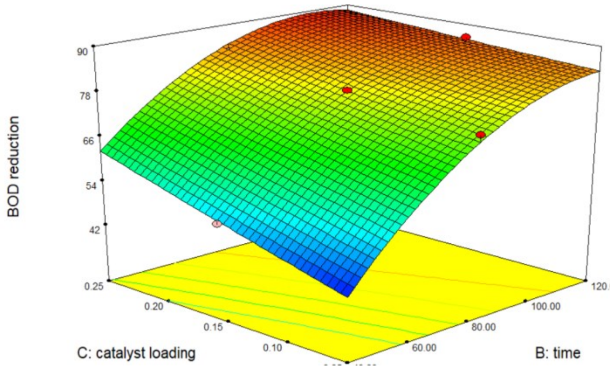


Figure 8. Effect of Sono-catalytic treatment on BOD of tannery effluent

BOD reduction = + 78.39 + 2.12 A + 16.80 B + 6.14 C - 0.29 A B-0.15 A C - 3.65 B C + 0.61 A2 - 9.26 B2 + 0.19 C2 (6)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001. An nalysis of variables (ANOVA) data Time(B), Catalyst loading (C) are significant where the Power is not significant to the system, which shows that the BOD removal is only depends on catalyst and time. The predicted R squared of 0.9639 is in reasonable agreement with adjusted R squared of 0.9927.

The percentage reduction of TDS was very high by treating the effluent in sono catalyst as shown in the 3D plot. Reduction in TDS increases with increase in catalyst and time of reaction. The reduction efficiency is 94% for 0.25 g and 51.8% for 0.05 g. at time of 120 min and 40 min respectively. The showcased improvement in reduction of TDS is due to the increased catalyst dosage. The increased catalyst dosage is found to give effective treatment under comparitively lower frequency (Kannan Kandasamy *et al.*, 2017). For the reduction of TDS the power of ultrasound is not found to be influential. During the sono-catalytic treatment the contaminants are broken down and the reduction of BOD and COD indicate the same. But the broken down contaminants seem to remain in the dissolved form, which can be separated by evaporation. Since the BOD and COD are reduced the bio-degradability of the effluent is improvised (Figure 9).

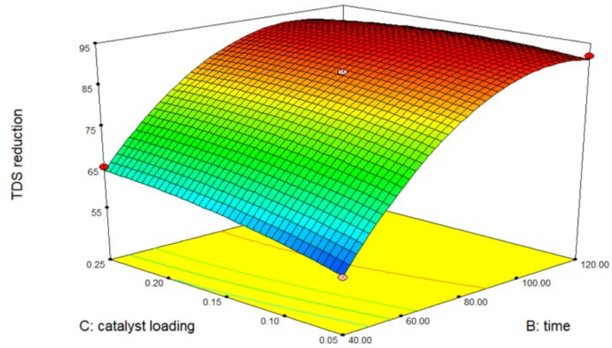


Figure 9. Effect of Sono-catalytic treatment on TDS of tannery effluent

TDS reduction = + 87.10 + 1.69 A + 16.11 B + 2.10 C - 0.59 A B + 0.038 A C - 2.39 B C + 0.13 A2 - 11.85 B2 - 1.70 C2 (7)

The Sequential Model Sum of Squares is Quadratic and it is significant the Prob>f is less than 0.0001. An analysis of variables (ANOVA) data Time(B), Catalyst loading (C) are significant where the Power is not significant to the system, which shows that the TDS removal is only depends on catalyst and time. The predicted R squared of 0.9701 is in reasonable agreement with adjusted R squared of 0.9942.

3.2.1. Comparison of MOBC-ozonation & Sono-catalytic treatment:

The reduction of contamination using 2 novel treatment techniques showcased improved results. The following graph compares the reduction of COD, BOD and TDS by both techniques. The ozonation in MOBC is found to be a little inferior with respect to the reduction of contamination compared with sono-catalytic treatment. This substantiates the effectiveness cavitation phenomena in addition with catalysis. Considering the real time treatment of tannery effluent, Sono-catalytic treatment is defective as the technique can't be scaled up easily. The consumption of power is also high for ultrasonic treatment. Ozonation can be easily scaled up and consumes less power (Figure 10).

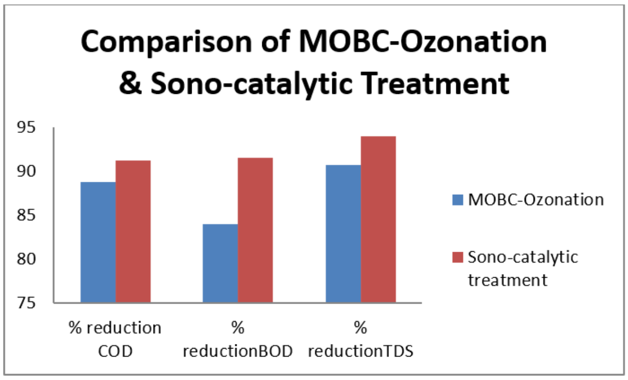


Figure 10. Comparison of MOBC-ozonation and Sono-catalytic treatment of tannery effluent

4. Conclusion

In this work, tannery effluent is treated by using sono-catalysis and ozonation by MOBC. The removal of COD, BOD, TDS in Ozonation and Sonication is 88.8%, 84.01%, 90.73%, and 91.2%, 91.5%, 94% respectively. The obtained results indicate that both treatment techniques are superior on comparison with the other research works and conventional techniques. Ozonation in MOBC is good for large scale treatment of tannery effluent, but the biodegradability of the treated effluent is low. There is a scope for improvement of the treatment when the design features of MOBC is modified and studied. Ultrasound assisted catalytic treatment shows good treatment, but it cannot be scaled up. Multiple catalysts can be subjected for the sono-catalytic treatment and the effects can be compared. A feasible solution can by hybridization of ozonation in MOBC with sono-catalytic treatment, which will bring profound results for nearly complete degradation of contaminants present in tannery effluent in large scale.

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