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Treatment of pulp and paper industry wastewater by ozonation and solar Photo Fenton Process

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ABSTRACT

Paper Industry is one of the highest polluting industries in India and is highly water intensive. Relatively large wastewater has been discharged from the paper industry which releases high pollution load to the environment. Necessary Steps are been taken to reduce the pollution causing discharges, especially water which is an integral part of the pulp and paper industrial functioning. In the paper, industry water is required for cleaning and washing purposes also. Among all the industries in the world paper industry provide the highest water polluters to the Environment. The paper industry is the largest user of energy resources from the Environment. The main aim of the paper is to reduce the effects caused to the Environment due to the disposal of wastewater from the paper industry. In the manufacturing process, a large number of lignin compounds and non-bio degradability substance present in the wastewater released to the environment which causes lethal effects to the aquatic organisms, reduce the toxicity and improve the biodegradability.

Keywords— *Paper industry, Wastewater, BOD, COD, Ozonation, Photo Fenton process*

1. INTRODUCTION

From the environment, the pulp and paper industry consumes a large number of natural resources and it finally discharges pollutants to the environment. Pollutants are characterized that (BOD_5), (COD), suspended solids, toxicity and colour. A Large number of lignin compounds in the wastewater resist the biodegradation process because of its toxicity. The pulp and paper industry is considered as the largest polluter in India. The effluents from the industry cause various effects such as slime growth, scum formation, colour problems, and loss of aesthetic beauty in the environment. Therefore prior to disposal, the wastewater must be treated which are identified as a major source of environmental pollution and also it creates disposal problems.

The treatment of this kind of effluent by chemical oxidation, especially with advanced oxidation processes (AOPs), is able to oxidise bio refractory pollutants into a more easily biodegradable form and to reduce the high concentration of organic matter. The AOPs are defined as oxidation processes in which hydroxyl radicals are the main oxidants involved. This type of radical is a very powerful oxidant which leads to a very effective oxidation process. In addition to their efficiency, these technologies have another important advantage no dangerous or persistent by-products are formed as a consequence of the reduction of the oxidizing agent. The hydroxyl radical is produced from single oxidants such as ozone (O_3), or from a combination of strong oxidants such as O_3 and hydroxide (OH^-), O_3 and hydrogen peroxide (H_2O_2), or ferrous or ferric ions (Fe^{2+}/Fe^{3+}) with H_2O_2 and combining these processes with UV radiation. The combination of $Fe^{2+}$and H_2O_2 is called the Fenton reaction. Ozone in water can follow two pathways are direct oxidation of compounds by molecular ozone and indirect oxidation through hydroxyl free radicals produced during the decomposition of ozone and from reactions between ozone and some organic and inorganic species in water found that under acidic conditions, direct oxidation with molecular ozone is of primary importance. Under conditions favouring hydroxyl free radical production, such as high pH (O_3/OH^-), exposure to UV or addition of hydrogen peroxide (O_3/H_2O_2), the hydroxyl oxidation starts to dominate. As well as hydroxyl radicals, other radicals are generated such as superoxide, ozonide and hydroperoxide radicals. Ozonation and ozone-related AOP’s have been used for the removal of organic compounds from paper and pulp industry wastewaters

The Fenton oxidation process is based on the use of a combination of hydrogen peroxide and ferrous or ferric ion to form active hydroxyl radicals in acidic solutions in a very simple and cost-effective way. In the photo-Fenton process (PF), additional reactions occur in the presence of light that produces hydroxyl radicals or increase the production rate of hydroxyl radicals.

2. MATERIALS AND METHODS

The wastewater was collected from pulp and paper mill at Karur. The wastewater has been collected in cans and carried to the lab and it was stored at 4°C, and the initial characteristics of the pulp and paper were done by the standard methods.

Ozonator, Tray reactor, wastewater, Phosphate buffer solution, magnesium sulphate, calcium chloride Ozonator, ferric chloride, manganese sulphate, alkali iodide azide, concentrated sulphuric acid, sodium thiosulfate, starch indicator, potassium dichromate, mercuric sulphate, ferrous ammonium sulphate, ferroin indicator, analytical grade iron sulphate and hydrogen peroxide were used. In both cases, sulphuric acid was used for carbonate stripping and pH adjustment. For ozonation experiments, pure oxygen was used for ozone generation and N_2 to stop the reaction by removing residual dissolved O_3 present in samples.

Fig. 1: Pulp and paper mill, Karur

Table 1: Standard and TNPL effluent standards

S. no.	Parameters	Unit	TNPCB	TNPL
1	TSS	mg/l	100	40-45
2	TDS	mg/l	2100	1600-1900
3	COD	mg/l	250	180-200
4	BOD	mg/l	30	5-10

3. EXPERIMENTAL STUDY

3.1. Ozonation Treatment

Ozone-based treatments were performed in a spherical reactor (2 L) made of Pyrex glass. Ozone was produced using an ozone generator (Fischer Model 500). The ozone and oxygen mixture was continuously introduced into the reactor through a bubble diffuser contactor placed at the bottom.

Inlet ozone partial pressure in the gas mixture 0.82 kph) and 1.5 L of wastewater was treated for 150 min. The gas flow rate was 50 LO_2/h, resulting in an input ozone concentration of 1.9 g O_3/L (O_3 generated). Ozone not transferred into the process water during contact was released from the reactor as not consumed O_3 (O_3 not consumed). This not consumed O_3 was routed to two ozone destruct units containing 250 mL of a 2% KI solution. When the ozone reacted with this KI, it was reduced to oxygen and released to the atmosphere as an innocuous element. The ozone residual dissolved in the sample (O_3 residual dissolved) was lower than 0.1 mg/L for all assays in accordance with other authors. This value can be considered insignificant for the calculation of the ozone consumed (O_3 consumed) by the sample, which was obtained as the difference between O_3 generated and O_3 not consumed. The ozonation efficiency was calculated according to the following expression: ozonation efficiency (%) = (O_3 consumed/O_3 generated)*100. In the O_3/H_2O_2 process (Peroxone), the selected H_2O_2 concentrations were 1017 and 2000 mg/Taking into account two criteria. The molar ratio [O_3/H_2O_2] = 0.75 and the weight ratio [COD/H_2O_2] = 3 Furthermore, the influence on the methodology of the addition of H_2O_2 was studied: (i) one addition at the initial time and (ii) stepwise addition. In the photo-oxidative treatments (O_3/UV and $O_3/UV/H_2O_2$), the reactor was equipped with a low-pressure mercury lamp (light intensity of 660 W/m^2, Helios Italquartz) emitting nearly monochromatic light at 254 nm. In the O_3/UV/H2O2 process, a concentration of 1017 mg/L of H_2O_2 was used. Ozone-based treatments were carried out at different pHs: real pH of wastewater (pH=8), 9 and 10. In the O_3/H_2O_2 and $O_3/UV/H_2O_2$ processes, some experiments with control of the pH (pH=7) during the reaction time were carried out. According to (Steeling and Hoagie) the lower limit for the effectiveness of the H_2O_2/O_3 process is in a pH range of 5–9. In each experiment, samples were periodically taken at regular time intervals from the reaction vessel and immediately analyzed to avoid further reaction. All experiments were carried out in room temperature.

Fig. 2: Experimental setup of ozonation treatment

Table 2: Optimization of ozonation treatment

Sample	Operating Parameters	Optimization	Parameter
Pulp and paper WW (250ml)	Ozonation Intensity	60%	SCOD and SS and colour reduction
	Time (min)	0 – 90 min	

3.2. Solar Photo Fenton Treatment

The photo-Fenton experiments were assisted with natural solar radiation and carried out in batch Systems in quartz flasks of 500 ml. The assays were performed during sunny days in our lab, Namakkal, using natural solar light between 11:00–1:00 hours.

The conditions were selected taking into account the previous results of the photo-Fenton process in the solar chamber that demonstrated its effectiveness in the treatment of effluents from the pulp and paper industry.

The experimental runs were reproduced using natural solar irradiation in the combination of factors that maximize the yield of the removal of organic matter, concentration Fe^{2+}510 mg/L and hydrogen peroxide dosage in an interval of 50-125% (established as a percentage of the stoichiometric ratio in weight between the H_2O_2 and the COD, R = H_2O_2/COD = 2.125 The wastewater was stirred before the addition of hydrogen peroxide and during the treatment at 150 rpm. All experiments were conducted at the natural pH of the synthetic samples during 30 min, which ensured total consumption of the H_2O_2 concentration employed. In order to detect possible Interferences, a control sample without the addition of Fenton reagents was used in each solar photo-Fenton experiment to quantify losses by volatilization and other causes. Each experiment was carried out.

Fig. 3: Experimental Setup for Solar Photo Fenton Treatment

Table 3: Optimization of solar photo fenton treatment

Sample	Operating Parameters	Optimization	Parameter
Pulp and paper we (250ml)	SPF Intensity	50:1 ratio	SCOD, Colour reduction
	Time (min)	75min	

4. RESULTS AND DISCUSSION

Table 4: Initial characteristics of waste water

S. no.	Parameters	Units	Value
1	pH	-	9.4
2	Total Dissolved Solids (TDS)	mg/L	3900
3	Total Suspended Solids (TSS)	mg/L	2850
4	Total Chemical Oxygen demand (TCOD	mg/L	2050
5	Soluble Chemical Oxygen demand (SCOD)	mg/L	2500
6	Total Solids (TS)	mg/L	1800
7	Chlorides	mg/L	89.2
8	Alkalinity	mg/L	450.86
9	BOD_5	mg/L	365.19

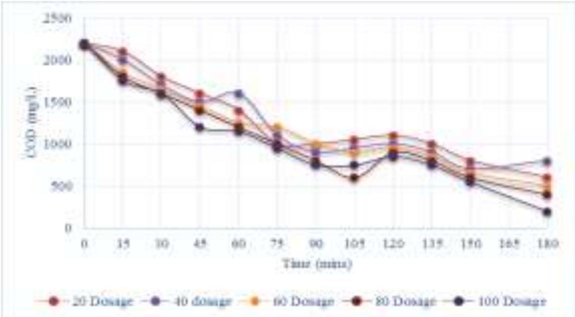

Fig. 4: Effect of ozone on SCOD release at various time intervals

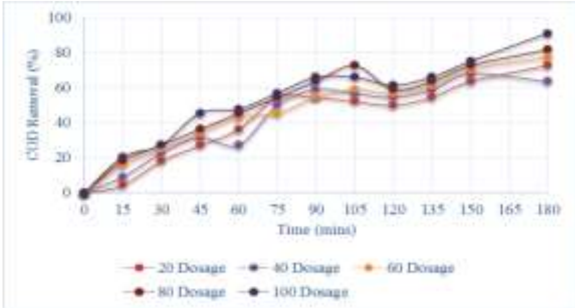

Fig. 5: Effect of ozone on COD solubilization

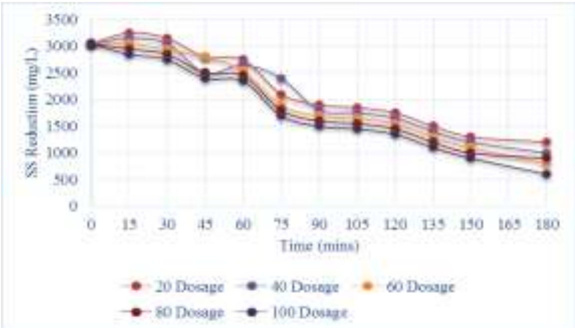

Fig. 6: Effect of ozone on SS reduction

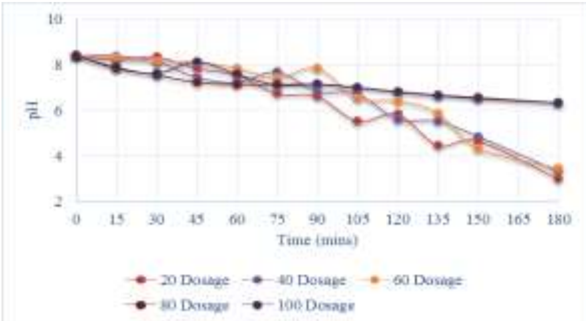

Fig. 7: Effect of ozone treatment on SS concentration

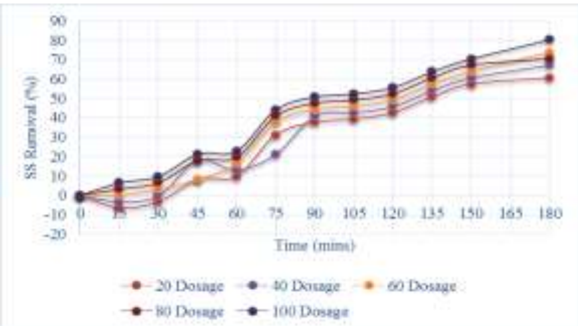

Fig. 8: Effect of pH on ozone treatment

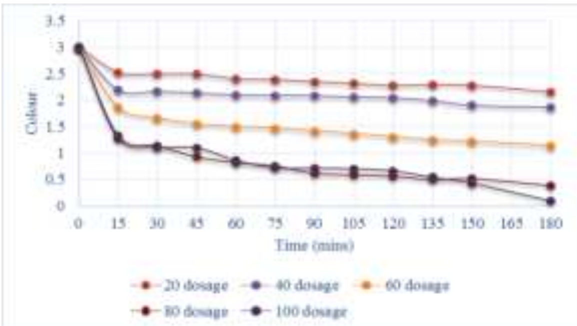

Fig. 9: Effect of colour on ozone treatment

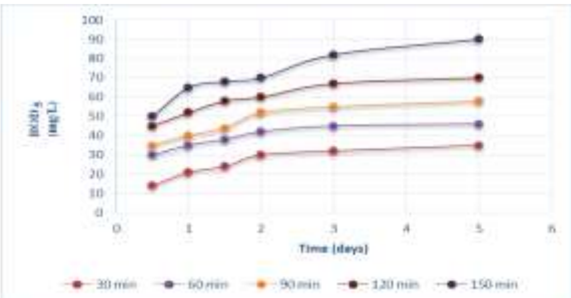

Fig. 10: Variation of BOD with ozonation time

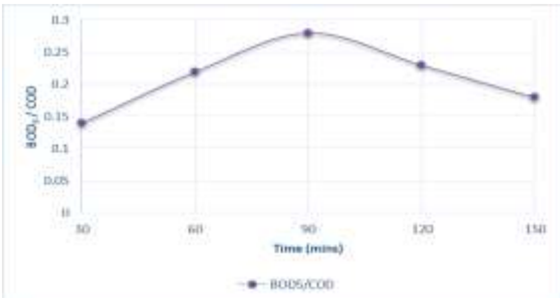

Fig. 11: Variation of BOD/COD ratio with ozonation time

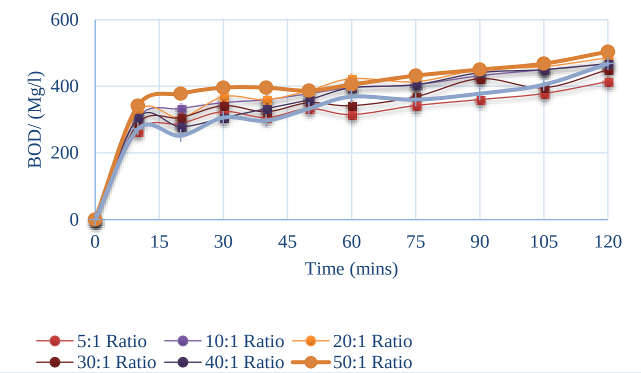

Fig. 12: Effect of biodegradability

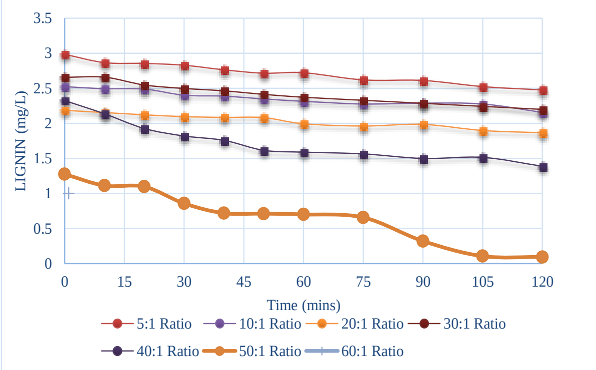

Fig. 13: Effect of lignin

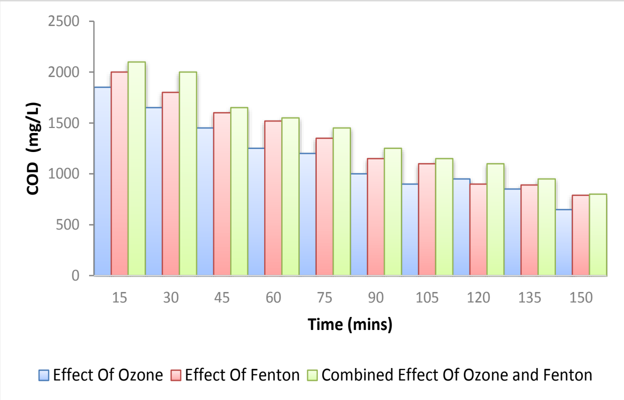

Fig. 14: COD removal on combined treatment

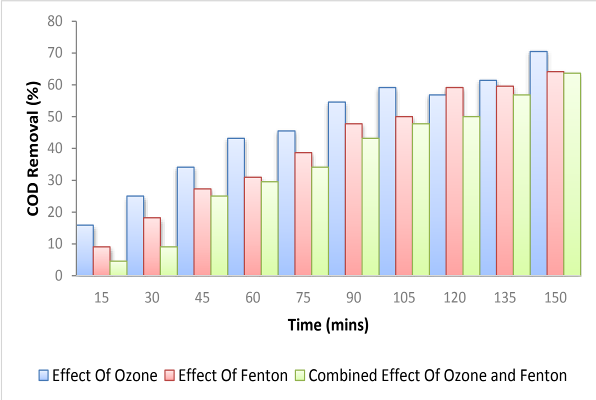

Fig. 15: COD removal efficiency on combined treatment

5. CONCLUSION

During Ozonation treatment, operating variables such as ozone dose and the reaction time were optimized. For Ozonation treatment, the dose was varied between 20-100% and the reaction time was varied between 0 to180 minutes. The efficiency of wastewater treatment was measured in terms of COD solubilization and SS reduction. COD solubilization was found to increase and SS reduction was found to decrease with an increase in ozonation dose and reaction time, respectively. Thus, maximum solubilization (65%) and SS reduction (50.8%) were found effective at 60% ozone dose for 90min, Based on the energy consumption, 60% ozone dose and 90 min reaction time similarly for solar photo Fenton treatment the maximum COD solubilization is 60% and optimum dose is 50:1 ratio at 75 mins of reaction were selected an effective parameter for combined zonation and solar photo Fenton treatment.

The colour is removed successfully for 100% of wastewater and adjusted pH =3 is the effect for the maximum removal of COD, lignin compounds present in the wastewater is removed. The combined treatment of ozonation with Fe^{2+}+H_2o_2 enhances the performance of biodegradability of wastewater. Finally, it is concluded from that when compared with the ozonation process and solar photo Fenton process it proves that the combined effect is essential for the maximum COD removal and efficiency.

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