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# PHOTOCATALYTIC DEGRADATION OF ACID RED 112 DYE IN AQUEOUS SOLUTION WITH HYDRODYNAMIC CAVITATION

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Abstract: Degradation of dye by Cavitation is an attractive technique which has been extensively studied for the treatment of complex wastewater due to its ability to generating the reactive free radicals, thereby initiating the chemical transformation required for dissociation and degradation of organic pollutants. Cavitation is the phenomena of formation, growth and subsequent collapse of the cavities/bubbles where this process occurs in few microseconds and at the multiple locations of the reactor and thus releasing the large magnitude of the energy. The degradation of organic pollutants can be enhanced in cavitation by combining it with other AOP's and chemical oxidants. In this work, the extent of degradation of Acid red 112 dye by the combination of hydrodynamic cavitation with photocatalysis is performed and studied. To our knowledge, no studies were found in the literature on the degradation of Acid red 112 dye using combination of HC and photocatalysis. The objective of this study is to make the analysis towards the efficiency of the hybrid advanced oxidation processes, i.e HC+ TiO2 photocatalyst dye in aqueous solution.

Index Terms - Pressure, pH, Cavitation, TiO2, Dyes, Acid red 112.

# I. INTRODUCTION

Water is essential living source. Its worth for living organisms on Earth is more than gold. No living system can stay alive without water. Without water crop and livestock will die. Our natural ecosystem is as the way that it can prevent water pollution by recycling unbearable materials at approximately specified rate at which they are produced. In the past, our streams, lakes and oceans were convenient place to intentionally dispose of undesirable material produced by human because of industrial activity. It was originally thought that this water would magically eliminate these undesirables via the process of dilution. Unfortunately the result of dumping these untreated wastes into aquatic ecosystem has increased the risk for diseases in human and death of aquatic life. [1].

Natural water is being contaminated by industrial effluent, domestic and agricultural waste. Waste water is any water that has been affected by human use or the water in which undesirable substance is added. This wastewater is classified and defined according to it's sources of origin. The term domestic wastewater refers to flows discharged principally from residential sources generated by such activities food preparation, laundry cleaning and personal hygiene. Industrial/commercial wastewater is flow generated and discharged from manufacturing and commercial activities such as printing, food and beverage processing and production to name a few. Institutional wastewater characterizes wastewater generated by large institutions such as hospitals and educational facilities.[2]

Wastewater is generally categorized based on solid or liquid substance which is suspended or dissolved in it. Examples are total organic carbon (TOC), total dissolved solids (TDS), turbidity, chemical oxygen demand (COD), biological oxygen demand (BOD), dissolved oxygen, hardness, pH, and color. These characteristics vary between industries and also within each industry. Due to this, Dissolved oxygen reduces in fresh water. Therefore, disposing these wastewaters, without treatment, to water sources can be harmful to aquatic life.[2]

Conventional methods like conventional physical, chemical, or biological processes, or in some cases a combination of these operations can be used to treat waste water. From this, solids, organic matter and, in some cases nutrients from wastewater can be eliminated.[3]

Physical treatment includes isolation or separation of the waste material from the mainstream. In this method, little or no degradation of the waste is involved, such as in coagulation, filtration, etc. However, there are some contaminants found in wastewater that are recalcitrant to several of the generally applied physical or chemical treatment processes. Chemical oxidation processes can be used to expand the current treatment processes, by transformation processes that can be used to destroy the recalcitrant compounds through oxidation and reduction reactions [3]

According to economic and ecologic superiority, biological method has been paid much attention. The biodegradation rate and biodegradation degree of the organic substance partly depended on the characters of the substance. Some of

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the organic pollutants like organic matters, organophosphorus pesticide, which have relativity high water solubility and low acute toxicity, are bioavailable and easy to be degraded It has been frequently observed that pollutants not amenable to biological treatments may also be characterized by high chemical stability and/or by strong difficulty to be completely mineralized. In these cases, it is necessary to adopt a process which is much more effective than conventional purification processes. [4].

A lot of researches have been done in the last decade pointing out the prominent role of a special class of oxidation techniques defined as advanced oxidation processes (AOP) which usually, operate at or near ambient temperature and pressure. Advanced oxidation processes are defined as those which involve the generation of hydroxyl radicals in sufficient quantity to affect water purification.[4].

The presence of additives results in intensification due to following possible mechanisms. Additives can provide additional nuclei so that the number of cavitation events in the system increases. Additives can promote enhanced generation of free radicals or generation of additional oxidizing species in the system. Additives can alter the physicochemical properties of the liquid medium thereby facilitating the ease of generation of cavitation events. Additives can alter the distribution of the reactants at the site of cavity collapse. Additives can have ability to scavenge unwanted species. Additives can provide active surface area for the bubbles to grow leading to an increase in the cavitational activity. Moreover along with all these properties, it is important that the selected additive does not lead to additional waste in the process and is efficiently utilized in the treatment. [6]

The primary focus of this present examination is to find best fit among different catalysts with hydrodynamic cavitation so as to degrade dye particles from waste water. Above all else, an excellent understanding of degradation of dye is very important in order to successfully and effectively apply the outline or process in this research.

# II. RESEARCH METHODOLOGY

#### 2.1 Materials

Ponceau S (3-Hydroxy -4- (2 -sulfo- 4- [4-sulfophenylazo] phenyl azo) -2,7 naphthalene di-sulfonic acid sodium dye), which is also called as Acid red 112 has been used in the present study as a model of sulfonated azo-dye. Ponceau S azo dye is used in dying industry of textile, leather and paper and also used in clinical laboratories as a protein-binding dye for staining of blood serum proteins. However, its biotransformation products have toxic effects against aquatic organisms and suspicious of being carcinogenic for humans. The solution of Acid red 112 was prepared in tap water for all the experiments which is acidic in nature with pH 3. Titatnium dioxide (TiO<sub>2</sub>), Ferrous oxide (Fe<sub>2</sub>O<sub>3</sub>), Calcium oxide(CaO), Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), Sodium hydroxide (NaOH) were of lab grade and procured from the Lanxess speciality chemicals. All the chemicals were stored at normal atmospheric temperature. Sulfuric acid was handled with special care and stored in dark bottle. All chemicals were used as received from the supplier without any further purification or treatment.

#### 2.2 Experimental Setup

An In-house fabricated laboratory hydrodynamic cavitation reactor of 10 L capacity was used for the degradation of acid red 112 dye. The laboratory scale hydrodynamic cavitation setup have positive displacement pump having the power rating of 1.1 kW was used for pumping the dye solution through the cavitating device (venturi). Feed tank consists of a cooling jacket to cool down the heat, which is liberated during cavitation and to maintain the desired temperature. The bottom of the feed tank connected to the suction side of the pump and the discharge section of the pump was divided into one main line and one by-pass line. A circular venturi (2 mm throat diameter) was incorporated at certain point in the main line as a cavitating device to generate the cavitation. Appropriate number of manual control valves and pressure gauge were placed in pipe lines to control the flow rate and to measure the fluid pressures respectively. In addition to this, a variable frequency drive (VFD) was accommodated to the pump to adjust the rpm of the motor such that the flow rate through the main line can be controlled directly. A manual control valve is provided in the by-pass line can be adjusted to maintain the desired flow rate in the main line. The pipe line is made up of stainless steel (SS-316), whereas cavitating device (circular venturi) is made up of brass. Visible light is mounted at the center of feed tank for the light irradiation.

# 2.3 Experimental procedure

The degradation using HC was carried out using circular venturi as a cavitating device. The hydraulic characteristic of venturi was first studied to establish the operating conditions for the venture by estimating the cavitation number at different inlet pressures. After the hydraulic study the degradation of Acid red 112 was conducted using HC alone at different inlet pressure (1–5 bar) for original pH of a dye solution i.e. 3. This is done to establish the optimum

condition of pressure for getting maximum possible degradation using HC. After achieving optimum condition of pressure ie 1 bar, pH is varied from 2 to 10. The pH of the solution was maintained by adding appropriate quantity of  $H_2SO_4$  and/or NaOH. The concentration of Acid red 112 was kept constant in all the cases at a level of 20 ppm. Five litre of Acid red 112 solution was subjected to hydrodynamic cavitation for a total circulation time of 60 min. The samples were regularly withdrawn at a regular interval of 5 min and analyzed for the extent of degradation. To correlate the observed data and to optimize various parameters, a pseudo first order degradation kinetics was fitted to the observed degradation data.

After the optimization of HC in terms pressure, same procedure is followed for varying pH  $\,^2$  to 10 and optimized pH is obtained. The degradation of Acid red 112 using the hybrid process of HC and catalyst was carried out with varying concentration of catalyst ie 0.2 to 1 gm/lit. with the optimized pressure and pH to arrive at the optimum concentration of Catalyst. This was done for selected catalyst  $TiO_2$ , CaO,  $Fe_2O_3$ . The efficiency of the hybrid processes of HC and optimized catalyst  $TiO_2$  was compared with the individual process of HC to estimate synergy factor.

Factors that were obtained as observation are Gaug pressure, absolute pressure(KPa), Vapour Pressure of liquid at  $25^{\circ}$ C, Density of liquid(Kg/m<sup>3</sup>), flow rate at variable pressure from 1 to 5 bar, Area of pipe.(Eq. 1 and 2)

Velocity of liquid = 
$$\frac{\text{Flow rate}}{\text{Area of pipe}}$$
 (1)

Cavitation number =  $\frac{\text{Absolute pressure-vapour pressure}}{0.5 \text{*density of liquid*}}$  (2)

# 2.4 Analytical procedure

The collected samples were analyzed using UV-Vis spectrophotometer to observe a change in the absorbance of Acid red 112 with time at a maximum absorbance wavelength of 460 nm. The concentration of Acid red 112 was then calculated using the calibration curve prepared for Acid red 112.

Percentage of Degradation (%) = 
$$\frac{\text{C0-C}}{\text{C0}} \times 100$$
 (3)

Where,  $C_0$  and C are the concentrations of dye solution at air-bubbling 0 and t time (min), respectively.

# **III. RESULTS AND DISCUSSION**

# 3.1 Effect of Pressure

The oxidation potential and the ability of HC in degrading organic pollutant depend on the type of cavitation produced which in turn depends on the operating inlet pressure and cavitation number. Initially, hydraulic characteristics of the circular venturi was studied by measuring the volumetric flow rate at different inlet pressure and then the cavitation numbers were calculated with the knowledge of the liquid velocity and area of the throat. Where, the cavitation number is a dimensionless number used to characterize the cavitating condition inside the cavitating device.

From table 3.1, the degradation rate was high at the inlet pressure from 1. Beyond 1 bar pressure, the rate of degradation shows the decreasing trend. Higher degradation at 1 bar pressure could be due to the increase in the inlet pressure increases the volumetric flow rate which leads to increasing the number of passages through the cavitating device for the same time of operation. Increasing the number of passages resulted into higher degradation rate of Acid red 112. Beyond 1 bar pressure, chocked cavitation may occur and leads to the generation of vaporous cloud consisting of bubbles. These bubbles are carried away along the flow without any collapse. This phenomenon could reduce the .OH radical's generation, therefore the rate of degradation reduced with further increasing of pressure beyond 1 bar. The maximum degradation of 26.030 % in 60 minutes and degradation rate constant of Acid red 112 at 5.02 ×10<sup>-3</sup> min<sup>-1</sup> was achieved at 1 bar pressure.(Table 3.2)

Table 3.1 Percentage of degradation at various inlet pressure

Inlet pressure(Bar)	% of degradation	
1	26.0303	
2	13.21	
3	6.6666	
4	4.5500	
5	10.3671	

Table 3.2: Rate constant k' and percentage of degradation at various inlet pressure using Hydrodynamic cavitation

Inlet pressure(Bar)	Volumetric flow rate(LPH)	Cavitation number	% of degradation	k' * 10 <sup>-3</sup> min <sup>-1</sup>
1	175	0.002011946	26.0303	5.02
2	260	0.001371462	13.21	2.35
3	325	0.001172126	6.6666	1.14
4	400	0.00096813	4.5500	0.77
5	425	0.001029735	10.3671	1.82

#### 3.2 Effect of pH

It is also one of the important parameter in determining the physical and chemical properties of the solution, which in turn affects the bubble dynamics. The generation of hydroxyl radicals under the effect of bubbling time generally depends on the operating pH and is favored under neutral conditions. To observe the effect of solution pH on the degradation rate of dye using hydrodynamic cavitation, experiments were performed at different pH, ranging from 2-10. All the experiments for the study of solution pH were conducted at optimized inlet pressure of 1 bar. Concentrated  $H_2SO_4$  and 1 N NaOH solutions were used to adjust the pH of the solution at the required value.

It has been observed that the superior degradation was obtained at conditions of pH 6, rather than other condition. The maximum degradation of 61.585 % was obtained at 20 ppm Acid red 112 at pH 6 at optimized pressure 1 bar and the percentage of degradation decreases as the pH increases.(Table 3.3)

At the interface cavities, the dye molecule is then subjected to the direct attack of OH- radicals, resulting into increased degradation rate. Whereas at higher pH, the dye molecules get ionized and become more hydrophilic in nature, thereby remain in bulk liquid. Because of recombination of OH radicals, small fraction of generated OH radicals can diffuse into the bulk which results in lower availability of OH radicals being used for oxidation of dye as only about 10% of OH radicals generated in the cavity can diffuse into the bulk solution.[23]

Table 3.3 Percentage of degradation at various pH

pН	% of degradation	
2	27.447	
4	35.537	
6	61.585	
8	33.264	
10	37.605	

#### 3.3 Effect of Catalyst Loading

Experiments have been carried out in order to check the efficiency of acid red 112 with addition of amount of solid particles such as CaO powder; TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> powder using hydrodynamic cavitation. The experiments performed with different amounts of nano-sized CaO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> particles in a range of 0.2-1 gm/L at optimized pressure 1 bar and pH 6. It has been observed that all the solid catalyst shows the same characteristics as in case of degradation. All the solid catalysts shows significant enhancement of degradation for acid red 112 with increasing catalyst loading.

#### 3.3.1 Effect of CaO loading

The degradation of Acid red 112 dye increases with addition of nano-sized CaO powder up to 0.6 gm/L and further addition of nano-sized CaO particles decrease percent of degadation of dye decreased to some extent. A steep variation is observed in the curve where the degradation of acid red 112 was maximum i.e. 91.5 % at the loading of 0.6gm/L of nano-sized CaO powder. (Table 3.4) This is due to the increase in the catalyst dosage, total active surface area increases, hence availability of more active sites on catalyst surface. Also, observed effect may be attributed to the adsorption characteristic of the Acid red 112 dye on the CaO powder; for higher extent of adsorption, more is the beneficial effect of degradation of dye molecule structure. The decline rate of degradation of dye with the addition of CaO particles might increase in turbidity of the suspension with high dose of CaO results into aggregation of molecules, lower number of active sites available, and hence minimal degradation of acid red 112 dye at higher catalyst loading. [26]

#### 3.3.2 TiO<sub>2</sub> Loading

In case of  $TiO_2$ , similar range of catalyst loading has been used to evaluate the optimal amount of catalyst from for the dye solution. The result from figure 5.12 clearly indicates that at 0.6 g/L of  $TiO_2$ , the percent of degradation of acid red 112 is highest 96%. (Table 3.4) Also, it has been observed that curve shows a continuous increase with linearity up to optimum level, afterwards excessive amount of  $TiO_2$  particles create mutual screening effects which will block some organic molecule from receiving the same amount of energy after the collapsing of cavities. The increase in the percentage of degradation of Acid red 112 up to 0.6 g/L due to the fact that the presence of solid catalyst (mostly  $TiO_2$ ) can enhance the dissociation reaction of  $H_2O$  molecules to increases the number of free radicals generated, thereby increasing the rate of degradation of dye.[26]

# 3.3.3 Fe<sub>2</sub>O<sub>3</sub> Loading

In case of Fe<sub>2</sub>O<sub>3</sub> solid particles, it has been seen that with increasing concentration of Fe<sub>2</sub>O<sub>3</sub>, the rate of degradation of Acid red 112 also increases. Higher percentage of degradation of Acid red 112, 94% has been obtained at 0.8 g/L of Fe<sub>2</sub>O<sub>3</sub>. (Table 3.4) The higher degradation of Acid red 112 is due to the fact that it provides additional nuclei for the cavitation phenomena and the hence the number of cavitation events occurring in the reactor is enhanced resulting in a subsequent enhancement in the cavitation activity resulting in increases in the number of free radicals.

Overall, it has been observed that the order of activities for better degradation of Acid red 112 is  $TiO_2 > Fe_2O_3 > CaO$  over a range of 0.2 to 1 g/L. The higher degradation of Acid red 112 using  $TiO_2$  may be dependent upon the characteristics of catalyst such as particle size, surface area etc.

# Table 3.4 Percentage of degradation at various catalyst loading

Catalyst loading(gm/L)	Percentage of degradation CaO	Percentage of degradation TiO <sub>2</sub>	Percentage of degradation Fe <sub>2</sub> O₃
0.2	68.58	53.46	84.36
0.4	90.07	69.6	92.92
0.6	91.5	96	93.1
0.8	73.56	82.2	94.02
1	70.1	75.23	70.6

#### IV. CONCLUSION

In this study hydrodynamic cavitation for dye degradation in aqueous solution was evaluated for the degradation of Acid red 112. It was found that the degradation of Acid red 112 using the HC depends on various parameters such as inlet pressure to the cavitating device, cavitation number and solution pH and it exhibits a maximum degradation rate at an inlet pressure of 1 bar. The degradation of Acid red 112 using HC enhances at pH 6.

Further, the combination of HC with catalyst TiO<sub>2</sub> gives greater degradation 96%. One suggest that these process should be used in combination HC and catalyst with each other rather than using them individually for getting enhanced degradation and higher degradation efficiency. This is more suitable than other process due to the later's higher oxidation potential and ability to dissociate into more reactive OH radicals under cavitating condition developed in HC.

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