

# Catalytic Ozonation: A Rising Advanced Oxidation Technology for Textile Dyes

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**ABSTRACT:** Catalytic ozonation is a blooming technique in treating the dye effluents containing azo dyes, reactive dye, acidic dyes etc. Catalytic substances are used in ozonation provides an active surface for colour removal of dye. Aim of this paper is to prove catalytic ozonation as the better technique for treating industrial effluents. In this paper, different parameters enhancing the decolourization of different dyes using various types of catalyst are mentioned. Supportive research has been mentioned in this paper which uses various catalyst, (homogenous and heterogeneous catalyst) for treating all types of dyes. This paper emphasises on a comparative study of Catalytic ozonation, a branch of Advance oxidation process, with ozone system alone. Influence of different parameters like pH value, Ozone supply, catalyst concentration and temperature is observed in this review paper.

**KEYWORDS:** Catalytic ozonation, Decolourization, Advanced oxidation process, Homogenous, Heterogeneous.

## I. INTRODUCTION

Many advance technologies have been developed to treat the dye effluents from textile industries. This dye effluent contains high level of dye concentration, which is highly structured polymer causing harm to the organism. It has adverse effect on the environment, so the effluent needed to be decolourized and toxicity level should be minimized. For this purpose, different AOP's have been used like ozonation, catalytic ozonation, Fenton process, sonolysis, photo catalytic ozonation etc. for treating dye effluents. AOP's are the alternative techniques for the decolourization and detoxification of the dye effluent [2]. Some convention treatments like coagulation, flocculation, flotation, adsorption and membrane filtration were just seen to change the phase of the pollutants instead of destroying them [4]. AOP's came into picture as the future reliable technologies for waste water decolourization, detoxification, mineralization and recalcitrant waste reducer. Advanced Oxidation Process is defined as the oxidation process, which generate hydroxyl radicals in sufficient quantity to affect water treatment [8].

Ozonation is amongst the widely used AOP for treating the dye effluent. It has an excellent decolourization due to following reasons:

- (1) Decomposition of residual ozone in to water and oxygen
- (2) Less harm to humans
- (3) Negligible sludge residue
- (4) Experimental areal required is less
- (5) Easy in performing
- (6) Single step degradation and decolourization
- (7) Strong germicidal properties than any other process
- (8) Degradation of organic matter and micro pollutants

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Ozonation carried out in two ways: direct molecular ozonation reaction and indirect route leading to ozone decomposition, and hydroxyl radical (OH) generation. Hydroxyl radical plays an important role in ozone decomposition. OH radical is one of the most reactive free radical and strongest oxidant [8]. Ozone has an oxidation potential of 2.07V, which help in most of the organic matter degradation. Depending upon the molecular selection and decay ratio, ozone and hydroxyl radicals generated in aqueous solution can open the aromatic rings and oxidise organic and inorganic compounds to their highest oxidation state [5]. Components which do not react with ozone like aldehydes and carboxylic acids are produced during the reaction of ozone with organic matter. Ozonation reaction is strongly influenced by high pH, dye concentration, pressure, temperature and ozone flow rate, but uncontrolled decomposition is taking place. So for controlled decomposition, ozonation can be combined with catalytic ozonation, leading to effective hydroxyl radical formation at low pH too.

Many types of research have proven the high efficiency in removal of dye pollutants by catalytic ozonation. Catalyst ozonation process can be of two types:

- i. Homogeneous catalytic ozonation: - transition metal iron is used to catalyse ozone decomposition,
- ii. Heterogeneous catalytic ozonation: - the solid catalyst is used to catalyse ozone decomposition.

An organic pollutant used for studying catalytic ozonation includes those which are removed by a radical mechanism. The general idea concerning the mechanism is a transfer of an electron from supported metal to ozone molecule with the production of  $O_3^-$  and final OH $\cdot$  generation [4]. The homogeneous catalyst used in the homogeneous solution of ozone medium are the transition metals like Cr(III), Zn(II), Ag(II), Ni(II), Cd(II), Fe(II), Mn(II), Cu(II), etc. The catalyst used in heterogeneous catalyst ozonation reaction includes some metals like Fe, Cu, Pd, Co, Pt, Ru, etc. metal oxides like TiO<sub>2</sub>, MnO<sub>2</sub>, CeO<sub>2</sub>, SnO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, ZnO, MgO, etc. and/or the metal supported metal oxides like Al<sub>2</sub>O<sub>3</sub>, Cu/Al<sub>2</sub>O<sub>3</sub>, O<sub>3</sub>/Mn(II) (or MnO<sub>2</sub>), H<sub>2</sub>O<sub>2</sub>, UV/TiO<sub>2</sub>, FeOOH, Ru/Al<sub>2</sub>O<sub>3</sub>, Pt/Al<sub>2</sub>O<sub>3</sub>, etc. Minerals like activated carbon, ceramic honeycomb, cordierite, perovskite, zeolite, etc. A catalyst like manganese was believed to be one of the most effective transition metal for catalytic ozone decomposition in gases and also with same properties [15]. Two important activities should take place by a catalyst to reveal its catalytic activity viz. ozone adsorption and organic molecule on its surface should take place. The oxidative cleavage of chromophores also plays a vital role which results in the colour removal of the dye in ozonation process. C=C bond and other functional groups shift the adsorption spectra of the molecule out of the visible region [20].

The effect of various parameters and catalyst used in catalytic ozonation process for different dye effluents are studied. Research done on catalytic ozonation was studied under the parameters like pH value, ozone dosage, pressure, catalyst loading, dye concentration rate and temperature, etc. This numerous research came up with different mechanisms using various catalysts.

## II. RELATED WORK

Catalytic ozonation has proved to be a better technique in colour removal of different dyes. It is also effective for TOC and COD removal of the dye effluent. 90%-99% Decolorization was observed by Hong Sun et.al [1] with the use of Mn-Fe-honeycomb as a catalyst. Study for decolorization of two different azo dyes (C.I. Reactive Red 2 and Acid Orange 6) using MnO<sub>2</sub> catalyst in ozone system was done by Chung-Hsin Wu et.al [2]. Maximum colour removal was observed for the two dyes. (RR2- 90-95% and AO6- 90%). An experiment performed by K. Pachhade et.al [5] to see the catalytic action of metal ions on decolorization and degradation of Procion Red MX-5B resulted in about 95% of colour removal and COD and TOC removal as 80% and 75%. M. Khadhraoui et.al [9] studied discoloration and detoxification of Congo red. TOC removal was observed to be 32% and almost 90% to 99% colour removal by increasing the contact time [9]. Decolorization using MgO nanocrystals gave 90-95% colour removal with 20-25% TOC removal [10]. A comparative study was made by Saltuk Pirgalioglu et.al [11] under catalytic and non-catalytic ozone system. % TOC removal and % colour removal was observed for three different dyes showing maximum colour removal at ten pH. Decolorization of two dyes (Acid Red 1 and Acid Yellow 23) from dye effluent was experimentally observed by Hung-Yee Shu et.al [13]. Metal catalyst and metal oxide catalyst proved more effective than another

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catalyst according to the research. Manganese as a catalyst used for the degradation of atrazine was studied in the presence and absence of humid substances [15].

Advanced oxidation processes using ozone were studied by Bai Cuiping et.al [16]. This experiment under different system showed that UV/ozone system was more efficient in decolorization and COD removal rate (99.78% and 47.43% after 15 min ). Removal of colour and COD was observed by M.B. Mandake et.al.[23] under the catalytic action of zinc oxide.

### III. MECHANISM

In catalytic ozonation process the reaction steps taking place during catalytic ozonation is as follows:

- Adsorption
- Reaction of ozone decomposition
- Reaction of surface oxidation
- Desorption

Catalytic ozonation gives two mechanism types depending on the type of catalyst used:

1. **Mechanism of Homogeneous Catalysis:** In this process, hydroxyl radicals are produced due to the decomposition of ozone under metal ion as a catalyst. The oxidation of organic compounds results in the new complex compound formed with the metal ion, which is attacked by the ozone molecules.

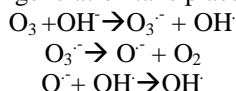
2. **Mechanism of Heterogeneous Catalysis:** Many physical and chemical process (temperature, pH of the solution, water composition with organic molecule properties, mass transfer, surface material, interfacial surface area) have an influence on the heterogeneous catalytic process.

i. Metal oxides and metal oxides with supports: Process supports the ozone adsorption producing active oxidative species and hydroxyl radicals.

ii. Alone metal on supports: Formation of  $\text{OH}^\cdot$  radical and  $\text{O}_3^\cdot$  takes place due to electron transfer from metal(supported) to ozone molecule.

Ozonation combining with catalytic ozonation produces synergetic effect resulting in decolourization of dyes and decomposition of organic compounds. The decolourization and decomposition take place by two routes.

At basic pH: Hydroxyl and other radical generation take place.

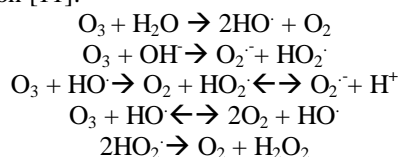


At acidic pH: Direct reaction with the ozone takes place, as ozone being stable at low pH [4].

In reaction, molecular ozone plays a crucial role due to its chemical properties. Its properties depend on its molecular structure. The resonance structure of molecular ozone is responsible for it being a dipole and electrophilic or nucleophilic agents. Ozone is highly unstable in water due to its high reactivity. When ozone reacts with water, there are some compounds which are responsible for initiation (initiators:  $\text{OH}^\cdot$ ,  $\text{H}_2\text{O}_2/\text{HO}_2^\cdot$ ,  $\text{Fe}^{2+}$ , formate, humic substances), promotion (promoters:  $\text{R}_2\text{-CH-OH}$ , aryl-(R), formate, humic substances,  $\text{O}_3$ ) and termination (terminators:  $\text{CH}_3\text{-COO}^\cdot$ , alkyl-(R),  $\text{HCO}_3^-/\text{CO}_3^{2-}$ , humic substances) of the radical chain reaction [8]. The oxidation potential of ozone helps it to oxidise organic and inorganic compounds.

The catalytic ozonation taking place with transition metals ions as catalyst gives the following mechanism:

The reaction of  $\text{O}_3$  decomposition [11]:



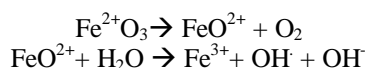
Formation of hydroxyl radicals from a metal ion (here taken as  $\text{Fe}^{2+}$ ) [8]:

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The decomposition of ozone in water takes place by two ways as follows [11]:

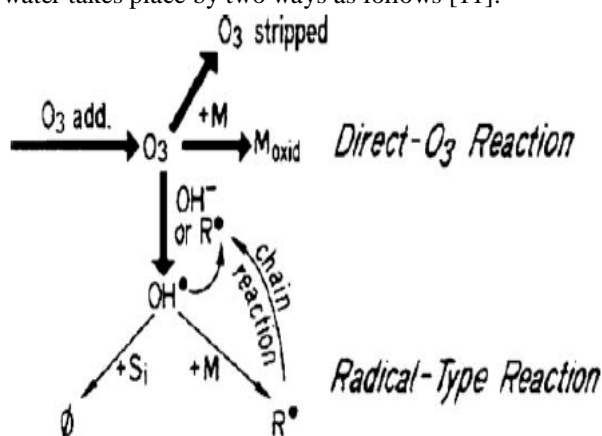


Fig 1: Ozone reaction type

Varieties of the mechanism are put forward by researchers using a different catalyst. Ozone got its properties due to the third or nascent oxygen molecule. These properties were enhanced by using a catalyst in catalytic ozonation process. The mechanism also sometimes includes catalytic ozonation using UV light and sound, for enhancing the process. According to the pH value range, catalyst concentration, reaction time, the ozonation process is seen to accelerate yielding greater results. The mechanism changes accordingly with the use of parameters too.

### IV. EFFECTS OF EXPERIMENTAL VARIABLES

#### EFFECT OF OZONE DOSAGE

Ozone dosage effect was studied for dye degradation by increasing the ozone supply in the catalytic environment. An experiment using X-3B wastewater, treated with Mn-Fe-ceramic honeycomb catalyst, resulted in 90%, 93%, 97.2% and 98.5%, for ozone supply of 0.5, 1.0, 1.5 and 2.0 g.h<sup>-1</sup> [1].

The effect of ozone **Chung-Hsin Wu et. al.** [2] only showed that the decolorization efficiency was 83% in ozone system, but later with the use of catalyst MnO<sub>2</sub> it was 92% for O<sub>3</sub>/MnO<sub>2</sub>(1g), 95% for O<sub>3</sub>/MnO<sub>2</sub>(2g), 95% for O<sub>3</sub>/MnO<sub>2</sub> respectively for Reactive Red 2. And for Acidic Orange 6 it was 67%, 91%, 90%, and 93%. It is cleared that ozonation alone helps in maximum colour removal, but with the help of a catalyst, the rate of decolorization is increased. According to **K. Pachhade et. al** [5] during ozonation, pH of the solution was greatly affected by showing the drop down from pH=11 at time=0min, to pH= 7.6 at time=15 min, and pH=3.52 at time=30 min. Ozone supply affects the dye concentration in not only the effluent solution but also the pH value of the solution changes.

Effluent gets its colour from the chromophore groups which are the organic compounds having the conjugate double bonds; these bonds can be broken by ozone in ozonation process, into smaller molecules causing the colour of the effluent to decrease. According to **Ensar Oguz et. al** [14] the colour removal efficiency of about 75% was observed by sample A and sample C, at high ozone dosage. The mass transfer should be enhanced by the increasing ozone dosage. However, it was observed that, when ozone dosage and ozonation time was increased, the ozone utilisation ratio decreased. [7].

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**Bai Cuiping et. al.** [16]. Reported that ozone dosage had greatly influenced the decolourization of Rhodamine B. The dosage of ozone in the dye solution containing rhodamine B, from 10ml/min to 30 ml/min resulted in decolourization of around 72.6% to 92.15%. The rate of decolorization was slightly decreased by the ozone dosage from 30ml/min to 40ml/min. This shows that at high ozone dosage the rate of decolorization is affected. This abnormality is seen due to the ozone transformation from gas to the liquid phase, and the OH radical are generated. Many researchers have concluded that ozone dosage has significant effect on the decolorization of dye effluent. The degradation is seen progressing at an expected rate but as the quantity of ozone dosage increases the decolourization decreases.

### EFFECT OF pH

The pH parameter influences the charge of the catalyst's poisons, charge on ionic and ionizable organic compounds and charge on active centres on the catalyst. Researchers have concluded that catalytic ozonation is highly affected by the variation in the pH value. pH value is seen to be influencing parameter for ozonation process. According to **Chung-Hsin Wuet.al.** [2] the variation in pH seem to be decreasing for RR2 and AO6 dyes. While treating both the dyes with ozone only, the pH values were seen decreasing consistently (RR2 changed from 6.7 to 4.5 and AO6 from 5.2 to 4.5). Some researchers have concluded in the greater decline of pH value, due to the formation of an acidic final product or intermediate product.

**Chung-Hsin Wu et. al.** [4] results showed the variation in pH value for systems like  $O_3/UV/TiO_2$  and  $UV/TiO_2/O_3$ , in decolourization of RR2 dye. The main Oxidation products like sulphate, nitrate, formate and oxalate were produced while treating azo dye in the  $O_3$  system, which caused pH decline of the solution [17]. The combination of some catalyst and compounds resulted in decomposition at neutral pH, and high efficiency noted at acidic pH. **B. Kasprzyk-Hordern et. al** [8] observed the pH variation, resulting alumina to be an ineffective catalyst for ozonation of 2-chlorophenol at neutral pH. The increase of about 83.7% TOC degradation noticed in acidic pH with  $Al_2O_3/O_3$  as a catalyst. In both heterogeneous and homogeneous medium manganese is the active catalyst [8].

**K. Pachhade et. al.** [5] studied decolourization by using ozone system, on the reactive dye, Procion Red MX-5B showed the decrease in pH value [5]. As the pH value was shifted from acidic to basic value, the colour removal was observed to reach 80%. The values for pH, as increases from 3.9, 5.5, 7.0, 8.5, 10, and 11 in ozone system resulted in increasing decolourization from 5%, 25%, 45%, 50%, 65% and 80% respectively. Decomposition is seen to be faster in the alkaline medium than in acidic medium because of the higher oxidation potential of OH radicals than the ozone molecules alone. The alkaline pH influences the reaction involving the opening of the aromatic ring by electrophilic attack of ozonation to form aldehydes and organic acids [19]. Contact time changing the pH value was observed by **M. Khandhraoui et. al** [9] in treating Congo red dye under ozonation system. As the contact time increases the pH changed from 8.7 to 3 (basic to highly acidic). Acidic pH value was the result of the formation of acidic by-products such as  $H_2SO_4$  from sulphonate groups and aliphatic acids and  $NH_4^+$ . Some dyes like reactive red 198 azo dye showed a different concept of degradation at low pH. **G. Moussavi et al.** [10] came up with the degradation of reactive red 198 azo dye in the presence of MgO nanocrystals as a catalyst, at low pH (acidic pH). It was observed that at pH 2 the decolourization was 91% and decreased to 85% as the pH was increased to 6 (reaction taking place by direct oxidation process). Increasing in pH to 10, brought degradation of dye at 97% (reaction taking place by indirect oxidation.).

**S. Pirgaltoglu et. al** [11] showed the degradation of three dyes AR-151, RBBR, RB-5, at pH range of 3, 7, and 10. AR-151 showed a decrease in degradation of pH value 3 to 7, but as the increase in pH value at 10 resulted proportionally increase in dye degradation. For RBBR dye no such significant change was observed. However, RB-5 showed drastic increase in its degradation rate for pH 3 to 10. Decolourization of mono-azo dye i.e. Acid yellow 23 in  $UV/H_2O_2$  system by **Hung-Yee Shu et. al.** [13] stated that increase in pH value should decrease the reaction rate.

**E. Oguz et. al** [14] came with a different phenomenon of Bomaplex Red CR-L dye decolourization at both (acidic and basic) pH values. The direct ozonation being predominant and selective leads to great decolorization at low pH.



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However, indirect ozonation by OH radical (OH radical being high in oxidation potential than ozone molecule) is preferred due to its fast reaction rate.

Decolourization of Rhodamine B is seen to be highest at low pH value, in the research done by **B. Cuiping et. al.** [16] Rate of decolourization in acidic medium is higher than that of basic medium because in alkali medium formation of free radical scavenger starts taking place as the pH increases. The decolourization rate reached to 85.64% at pH value 2 and decreased to 77.73 % at pH value 10.

Experiments have also resulted in the increasing rate of decolorization with increasing pH values, due to the catalysing action of hydroxide ions in the reaction. **TeramotoM et.al** [20] showed the drastic change in rate due to the pH change from 1 to 13.5. The rate at pH 13 was seen to be  $10^7$  times that of at pH 1. The decolourization if affected by pH change due to the property of the dye solution.

### EFFECT OF CATALYST ON OZONATION

Catalyst plays a vital role in enhancing the degradation rate of any dye. The homogeneous and heterogeneous catalyst used in dye degradation are metals, metal oxides, metals support on ametal oxide, minerals, etc. A great difference was observed by **Hong SUN et. al** [1] in degradation of X-3B wastewater using Mn-Fe ceramic honeycomb as a catalyst. The degradation rate was 3% in 60 min, which shifted to 93% in later 60 min. This showed that catalyst could bring about a significant change in X-3B degradation. Studies were done by **Chung-Hsin Wu et. al** [2] in decolourization of azo dye using  $MnO_2$  as a catalyst resulted in higher degradation of azo dyes. As the concentration of catalyst in ozone system was increased the degradation rate reached to 95% from 83% when treated under ozone system only.

**K. Pachhade et. al.** [5] reported the effect of metal ions as acatalyst on decolorization of Reactive dye Procion Red MX-5B. The metal ions like Zn, Fe, Co, Cu, Mn were used, which were inexpensive, low in toxicity level and easily available. Mn ion proved to be more efficient with its metal oxide as  $MnO_2$ , resulting in complete decolourization and 94% COD removal. The main parameters determining the catalyst property are acidity and basicity. According to **B. Kasprzyk-Hordern et. al** [8], hydroxyl group present on metal oxides surface behaves as Bronsted acids sites, which is said to be the catalyst centres of metal oxides. Hydrogen peroxide proved to be effective with ozone in colour removal of Congo red dye solution, according to **M. Khadhraoui et. al.** [9]. It was also observed that treating Congo red solution with ozone alone proved efficient than with ozone and hydrogen peroxide. This phenomenon is observed due to the hydrogen peroxide property to act as an OH radical scavenger. Recent studies done by **G. Moussavi et. al** [10] stated that MgO nano crystals were seen highly improving the colour removal of RR198 dye by increasing the concentration of catalyst. The reaction taking place at aconstant reaction time of 5 min and constant pH of 8 resulted from 53% colour removal (absence of catalyst) to 89% in 1g/L MgO powder catalyst. The increase in catalyst concentration to 5g/L MnO increased the colour removal to 99%. However, a further increase inthe concentration of catalyst did not affect the colour removal. Studying decolourization by **SaltukPirgaliogluet.al** [11], for AR-151, RBBR, and RB-5 using CuS as a catalyst, efficient decolorization was seen for RB-5 only. TOC removal was highly influenced by the CuS activity in the homogenous phase.**Jun Ma et.al** [15] reported that in degradation of atrazine with manganese catalyst, the ozone system was the most efficient catalyst than the  $MnO_2$  and Mn(II). Various researchers concluded the 100% decolorization of dye using a catalyst in heterogeneous and homogeneous phase in ozone system.

### COD AND TOC REMOVAL

Degradation of dyes using a strong oxidising agent as ozone is more relevant than any other oxidising agent. Ozone having the highest oxidation potential allows it to degrade organic compounds. **K. Pachhade et.al.** [5] studied the COD and TOC removal for Procion Red MX-5B, which came to be 80% and 75%, for the colour removal of 97%. Colour removal was seen to be faster rate than COD and TOC removal. COD removal was observed by changing the parameters like ozone dosage, pH value and initial dye concentration on therate of ozonation, by **Mehmet F Sevimli**

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et.al [7]. COD was seen to be increasing with the ozone dosage and Ph value, but the opposite graph was observed for initial dye concentration on therate of ozonation. TOC results were observed to be 25%, and COD removal was more than 50%, by **M. Khadhraoui et. al.** [9]. **M. F. Sevimi et.al.**[7] and **G. Moussavi et. al** [10] observed that MgO(catalyst) dosage increased the COD removal. COD removal was seen to be less than colour removal. **S. Pirgahoglu et. al.** [11] also studied the TOC removal rate varying with increasing pH value after the complete dye decolorization occurred. As the TOC removal was seen to be growing at high pH value, it can be said that TOC is high at basic pH and low at acidic pH value.

**E. Oguz et. al** [14] observed the effect of pH on COD removal for decolourization of Bomaplex Red CR-L using ozonation alone. The COD removal was increasing with the decreasing pH value. It reached only up to 54% at lowest pH value. Ozonation has proven to be inefficient in COD removal, so a need for acatalyst to increase COD removal was concluded. COD removal (50% and 40 %) was seen to be more efficient than TOC removal (30% and 25%) by **M. Koch et. al.** [17]. **M. B. Mandake et. al.** [23] studied that COD is a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. Depending upon the pH value the COD and TOC removal were seen to be changing. TOC Removal always showed a slower removal rate than COD removal. However, Colour removal always observed to be higher than the COD and TOC removal.

## V. CONCLUSION

The review paper presents general idea concerning the concept of catalyst introduction in an ozonation system for better results. The two ways ozonation enhancement process was studied, either by ozone decomposition accelerated by active radical production or by molecular ozone reaction acceleration, under various parameters. The effect of ozone dosage, pH value, catalyst concentration, COD and TOC removal was studied by different researchers using avariety of dyes under different ozone catalyst system. Ozone is being proved to be inefficient alone for dye decolourization and TOC removal, which concluded the researchers to make use of acatalyst for effective results. A mechanism using somecatalysts were put forward by the researchers for better understanding the decolourization and degradation process. The majority of the researchers suggested that maximum colour removal was observed with increasing the ozone dosage, reaction time, catalyst concentration and pH change.

Catalyst Used	Dye Used	% COD	% TOC	% COLOUR REMOVAL
MnFe ceramic honeycomb[1]	Reactive Red X-3B	-	-	90-99%
MnO <sub>2</sub> in O <sub>3</sub> system	C.I- Reactive Red-2 and Acid Orange-6 [2]	-	-	RR2-90-95% AO6-90%
TiO <sub>2</sub> , Mn (II), MnO <sub>2</sub> . [4]	C.I.Reactive Red 2	-	17-21%	95%
Metal ions[5]	Procian Red 2	80%	75%	98%
H <sub>2</sub> O <sub>2</sub> [9]	Congo Red	50%	25%	90-95%
MgO nanocrystals[10]	Reactive Red 198	20-25%		90-95%
Powder copper sulfide [11]	Acid Red-151 Reactive Black-5 Remazol Brilliant Blue-R	-	98% 90% 80-90%	Max at 10 pH

Table 1: Different catalyst used for various types of dyes.

The decolorization using catalyst always gives better colour removal. About 20% increase is seen in catalytic ozonation systems than in a system using ozonation alone.

The main aim of the review paper is to activate the ozonation process by using numerous heterogeneous and homogeneous catalyst. Researchers experiments proved catalytic ozonation a better technique for dye effluent

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treatment. Due to the extraordinary properties possessed by ozone and the synergetic effect produced by catalyst introduction in the system, makes it the technique gaining significant attention.

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