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Enhanced Catalytic Ozonation for Wastewater Treatment using Diclofenac Sodium and Acephate

Anand Jaiswal

Chemical Engineering Department, Mahatma Gandhi Mission's
College of Engineering and Technology,
Kamothe, Navi Mumbai, Maharashtra, India

Abstract

The execution of ozone operated independently and in amalgamation with a catalyst for commencing the realizable synergistic effects for the degradation of Diclofenac sodium (CN103145574A - anti-inflammatory drug) and Acephate (CN101074241A - organophosphate pesticide) has been studied using different approaches. The reliance of expanse of degradation on maneuver parameters like concentration (over the range of 20ppm-80ppm), pH (3-9), catalyst as ZnO, and oxidizing agent as H_2O_2 (loading of 20-80/L) and ozone flow rate of diclofenac sodium (100-400mg/h) and Acephate (30-100mg/h) have been established to intensify the efficacy of ozonation induced degradation.

Only by using the ozonation method, maximum degradation is achieved for diclofenac sodium. Whereas through combined approaches such as O_3+ ZnO + H_2O_2, O_3+ ZnO, O_3+ H_2O_2 degradation has been scrutinized under optimized conditions and observed to be more efficient. The maximum degradation products were further analyzed through COD with a quite eminent reduction.

Keywords: *Wastewater Treatment, Ozonation, Combined approaches, Diclofenac sodium, Acephate*

1. Introduction

Anthropogenic applications and industrial yields are leading to an increased level of emerging contaminants into the ecosystem [1] Among the emerging contaminants, pharmaceuticals (hormones, antibiotics, and others), cosmetics, synthetic dyes, and pesticides are the major concern in many areas around the world [2] . Also, emerging pollutant (EP) were founded in rivers according to literature [3] the presence of many pharmaceuticals compounds in the Suquía River basin, in Córdoba province, Argentina. Atenolol, carbamazepine, and diclofenac were the most frequently detected compounds, with concentrations ≤ of 1µg/L. These presence of hazardous compounds and toxic wastes in the water streams can result in hazards to human, animal and plant life. [4] In recent years pharmaceutical drugs and agricultural yield demand have increased due to which pharmaceutical and agricultural leftovers have emerged as a novel class of water contaminants [5] [6] In the aquatic environment, Diclofenac sodium (DS) is one of the most frequently detected pharmaceuticals [7] it is a phenyl acetic acid derivative [8] non-steroidal anti-inflammatory drug, widely used as an analgesic, antiarthritic, and antirheumatic [9] [10] DS has a short half-life of 1-2h and should be administered frequently at a high dose, which leads to severe undesirable effects and rises the possibility for missing a dose [11] Similarly acephate is an organophosphate insecticide which is used in agricultural sectors as it is easily soluble in water-soluble and up taken by the plant [12] which helps into control a wide range of chewing and sucking insect pests like aphids, thrips, sawflies, leaf miners, leafhoppers, cutworm of cotton, paddy, soybean, sugarcane, chilies, maize, tobacco, etc. [13] However, the acephate have high to medium acute oral or medium inhalation toxicity to mammals [14] [15] [16] [17]. It also hurts the nervous and respiratory system, and causes eye and gastrointestinal problems in humans [18] [19]. Thus, it is an alarming issue to degrade and remove acephate and DS in the plants and environment.

To improve the quality of wastewater before being discharged or reused, different purification methods have been applied. WWTPs generally employ a primary treatment (removal of suspended solids), a secondary treatment (removal of dissolved and suspended biological matter) and an optional tertiary treatment, which are commonly used to produce higher quality discharged water for certain purposes, such as water reuse; however, these treatments are always associated with high cost. [20] Several recent studies have revealed that this conventional water

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treatment processes cannot remove some drugs completely from water sources [21] [22] [23], so the adoption of advanced oxidation processes (AOPs) in the tertiary treatment section of existing UWTPs can significantly contribute to this reduction [24] [25] [26] Advanced oxidation processes (AOP) that have vast impact on the degradation of biorefractory organic compounds mainly including cavitation [27] [28], photolysis [29], Fenton [30], Photo Fenton [31], ozonation etc. Ozonation is one of the advanced oxidation processes (AOPs), which is applied in wastewater pretreatment as a strong oxidant as it generates the single atomic oxygen (O) and hydroxyl radicle (OH•) with the strong oxidation capacity, to decompose the organic compounds in water instantly [32] The main advantages of the implementation of AOPs over solo conventional treatment processes are as follows: (a) they have higher effectiveness at removing resistant organic compounds, (b) they almost completely mineralize organic contaminants into carbon dioxide, (c) they only have a minor susceptibility to the presence of toxic chemicals, (d) they produce a minor amount of harmful by-products and (e) they have better microbial disinfection [33]

There have been few studies reported for degradation of DS and Acephate using ozonation process with various combined approaches like [34] showed investigation of the efficiency of O_3 and US and also of their combined application (US + O_3) for the degradation and potential mineralization of diclofenac in a water matrix leading to 22% of mineralization for O_3 and 36% for US after 40 min of treatment. Also, in [5] showed that ozonation and H_2O_2/UV systems proved to be effective in inducing diclofenac degradation, ensuring a complete conversion of the chlorine into chloride ions and degrees of mineralization of 32% for ozonation and 39% for H_2O_2/UV after a 90 min treatment. Similarly, for acephate degradation in [35] combination of both ultraviolet-visible (UV-Vis) spectroscopic and Raman spectroscopic techniques were used results show that ultrasonication and ozonation have a synergistic effect in the combined system and the degradation efficiency of acephate increases from 60.6% to 87.6%. similarly, with .OH and e-aq produced by 60Co-c irradiation and electron pulse radiolysis was studied in [36] for acephate. An overview of literature studies suggested that degradation of acephate and diclofenac sodium was not combinedly studied. Thus, this study investigates the combined effect and efficacy of AOP's with combined approaches for acephate and diclofenac sodium.

2. Materials and Methodology

Materials: Primary impurities diclofenac sodium and acephate was bought from Simson Pharma Ltd, Mumbai. With the use of freshly prepared distilled water solutions was prepared. Other chemicals such as potassium iodide, photocatalyst titanium dioxide, hydrogen peroxide, ferrous sulphate etc. was obtained from SD fine chemicals, Mumbai. All the chemicals obtained were lab grade and used without purification i.e. as received from the suppliers.

Experimental setup:

Ozonator:

A standard laboratory-scale ozone generator (model-ZY-H103) with 600L capacity was used. Valve was attached to the outlet pipe to regulate the ozone flow during the experimental work.

Experimental methodology:

Below given figure1 and figure2 [4]; gives the schematic representation of the experimental setup used in experiments with ozone individually and combinedly with catalysts. During experimentation, the chemical reactor was filled with 500ml of aqueous CN103145574A and aqueous CN101074241A solutions individually in set of 5. The different parameters were studied one by one for both CN103145574A and CN101074241A under optimum conditions.

For both impurities, experimental aqueous aliquots of CN103145574A and CN101074241A were prepared by using fresh distilled water. For scrutinization of degradation of initial concentrations over the range of 20-100ppm concentrations were prepared and after each 30 min samples were withdrawn up to 2 hr which was further used for HPLC analysis. pH was initially studied at 7pH (neutral) and for further experimentation, pH was varied through the use of 0.1 M H_2SO_4 and 0.1 M NaOH solutions over the range of 3-9.

In the case of ozonation experiments, ozone was bubbled in the reactor through an ozonator with control ozone flow rate. Different ozone flow rates were used for the scrutinization of the impurities. Over the range of 100-400mg/hr ozone flow rate was used for CN103145574A whereas 30-100mg/hr ozone flow rate was used for CN101074241A. Combined approaches were also used for studying the extent of degradation. For CN103145574A two combined approaches O_3+ZnO and O_3+ H_2O_2 were used whereas for CN101074241A only O_3+ZnO method was used. In the range of 20-80mg/L concentrations were analyzed for the extent of degradation.

All the experiments were performed for a fixed treatment of time i.e. 120 min and samples withdrawn after each 30 min was analyzed using the HPLC method with an injection value of 10 μL. The temperature was maintained constant at 30 ± 2 ℃ by cooling water circulation using a peristaltic pump. The data reported in figures after each experimentation was the average of results obtained

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for each parameter run. Graphical representation has been shown for variation in data obtained.

Analysis: Withdrawn impurities samples were analysed using HPLC unit procured by Agilent Technologies (1200 infinity series), USA. HPLC unit comprised of Auto sampler, HPLC dual pump, UV detector and HPLC column eclipse plus C-18 column with dimensions of 4.6*250mm. The mobile phase used in the analysis consists of 60% methanol and 40% water mixture at flow rate of 1ml/min with wavelength of 278nm for UV detection for diclofenac sodium whereas in contrast, for acephate mobile phase used for analysis was 95% acetonitrile and 5% water with wavelength of 220nm for UV detection. The degradation products were also analysed using chemical oxygen demand (COD).

3. Result and Discussion

Effect of ozone concentration:

The obtained results for the effect of ozone on the extent of degradation over ozone flow range of 100-400mg/hr for diclofenac sodium and 30-100mg/hr for acephate under constant conditions of natural pH (7), initial concentration of 20 ppm have been depicted in graph 1 and graph 6 and the obtained kinetic rate constants have been given in table 1 for 2 hr. Almost complete degradation (99.98%) was achieved at 300-400mg/hr ozone flow for pharmaceutical whereas at 100mg/hr for acephate i.e. 94.59%. results also depict that the degradation efficiency increases with an increase in ozone flow rate at a logarithmic rate for DS whereas decreasing but fluctuating results were obtained for acephate.

Effect of initial concentration:

The extent of degradation at different initial concentration was observed under specific conditions (temperature 30±2, neutral pH, 100mg/hr ozone flow rate). Different concentrations of 20,40,60 and 80mg/L was used for detection of degradation of initial concentration. Table 2 shows comparative results obtained after the experimentation for 2 hr. Decreasing extent of degradation were obtained for pharmaceutical as seen in graph 2 whereas very high fluctuating results were obtained for pesticide with highest degradation at 20mg/L concentration as shown in graph 7. The results clearly depicted that at high concentration degradation of diclofenac sodium decreases.

Effect of initial pH:

The effect of initial pH was studied at different pH values ranging from 3-9; obtained results are shown in graph 3 and graph 8. Solutions H2SO4 and NaOH of 0.1M were used for maintaining the acidic and basic conditions. The rate of degradation was seen to be decreasing in both acidic(> 7)and basic (7 <) conditions. Maximum degradation was observed at neutral pH(7) for both diclofenac sodium (68.31%) and acephate (94.59%). The variation showed in graphs also depicts that degradation of impurities were favourable in acidic conditions after maximum degradation obtained. Comparison of acephate and diclofenac sodium % degradation on different initial pH with 100mg/hr ozone flow rate along with fixed regular interval of time is shown in Table 3. Comparative results showed that % degradation of initial pH for acephate was higher than diclofenac sodium.

Effect of combine approaches:

O_3 + ZnO

In the combined approach O_3 + ZnO, the catalyst ZnO was used as it exhibits exceptional physical, chemical, and optical properties that allow complete mineralization of organic pollutants. Under optimum conditions at 100mg/hr ozone flow rate, the results were scrutinized starting from 20mg/L till 80mg/L concentration. The results obtained clearly depict that there was variation in % of degradation as shown in Graph 4 and Graph 9. Combined results obtained up to 2 hr of experimentation is shown in Table 4. The results observed depicted that a combined approach with ozone and catalyst was favorable for acephate. The highest degradation achieved was at 60mg/L for diclofenac sodium whereas for acephate at 40mg/L catalyst concentration.

$O_3 + H_2O_2$ / O_3 + ZnO + H_2O_2

Combined approach $O_3 + H_2O_2$ and O_3 + ZnO + H_2O_2 was only used for pharmaceutical impurity diclofenac sodium (DS). Various concentrations ranging from 20mg/L-80mg/L were used for studying extent of degradation. As H_2O_2 is used as a key intermediate for hydroxyl radical generation it helps in speeding up the OH- yield in the ozone transformation process also it doesn't produce any secondary pollution. The studies were scrutinized in specific limited conditions were ozone flowrate of 100 mg/h, initial concentration of Diclofenac sodium at 20 mg/L, initial pH at 7 and ZnO loading at 60 mg/L was used as shown in Graph 5. The results found were in the range of 80-90% degradation as shown in table 5. Whereas when treated with 200mg/hr ozone concentration degradation was nearby to maximum i.e. 96.28%. Whereas when treated with O_3 + ZnO + H_2O_2 highest degradation obtained at 100mh/hr ozone flow rate was 89.52%

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Table 1: Comparison of % degradation of Acephate and Diclofenac Sodium (DS) at different ozone concentration

% degradation of Acephate at different Ozone concentration @ Acephate & DS Concentration 20ppm			
Initial Ozone Concentration (mg/hr)	Time	% degradation of Acephate	% degradation of DS
30 for Acephate 100 for DS	0	0	0
	30	21.71	25.777
	60	28.32	40.68
	90	34.57	53.87
	120	42.76	68.31
60 for Acephate 200 for DS	0	0	0
	30	27.85	32.41
	60	32.41	55.28
	90	42.18	81.65
	120	64.82	96.28
90 for Acephate 300 for DS	0	0	0
	30	34.15	69.74
	60	48.61	89.12
	90	61.52	95.88
	120	82.24	99.98
120 for Acephate 400 for DS	0	0	0
	30	41.47	71.15
	60	59.38	91.56
	90	78.47	96.27
	120	94.59	99.98

Table 2: Comparison of % degradation at different initial concentration

Comparision of % degradation at different initial concentration & ozone @ 100mg/hr			
Initial Acephate & DS Concentration (mg/L)	Time	% degradation of Acephate	% degradation of DS
20	0	0	0
	30	41.47	25.77
	60	59.38	40.68
	90	78.47	53.87
	120	94.59	68.31
40	0	0	0
	30	38.33	19.33
	60	54.82	28.39
	90	69.68	36.79
	120	83.43	45.83
60	0	0	0
	30	31.53	15.03
	60	42.28	18.98
	90	54.46	24.15
	120	68.37	32.53
80	0	0	0
	30	28.91	10.6
	60	40.55	13.84
	90	51.32	16.76
	120	60.62	19.27

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Table 3: Comparison of % degradation at different pH concentration

Coparision of % degradation at different initial pHconcentration & ozone @ 100mg/hr 20ppm)			
Initial pH	Time	% degradation of Acephate	% degradation of DS
3	0	0	0
	30	32.88	19.45
	60	49.35	31.63
	90	71.52	42.46
	120	81.47	51.23
5	0	0	0
	30	37.11	23.81
	60	53.97	34.55
	90	74.63	44.17
	120	89.19	55.78
7	0	0	0
	30	41.47	25.77
	60	59.38	40.68
	90	78.47	53.87
	120	94.59	68.31
9	0	0	0
	30	35.84	7.64
	60	51.75	14.38
	90	73.11	21.88
	120	88.75	29.22
12	0	0	0
	30	32.66	6.71
	60	48.24	10.37
	90	70.84	13.48
	120	82.95	15.19

Table 4: Comparison of % degradation at different initial ZnO2 concentration

Comparision of % degradation at different initial ZnO2 concentration (ozone @ 100mg/hr 20ppm 7pH)			
Initial ZnO2 Concentration (mg/L)	Time	% degradation of Acephate	% degradation of DS
20	0	0	0
	30	45.19	38.81
	60	62.72	49.27
	90	79.39	60.47
	120	96.38	72.39
40	0	0	0
	30	47.51	40.69
	60	65.77	53.19
	90	83.46	65.25
	120	99.32	78.51
60	0	0	0
	30	44.22	41.88
	60	60.58	55.17
	90	78.31	69.42
	120	95.68	80.47
80	0	0	0
	30	34.17	37.82
	60	52.44	49.89
	90	69.83	61.76
	120	89.44	73.36

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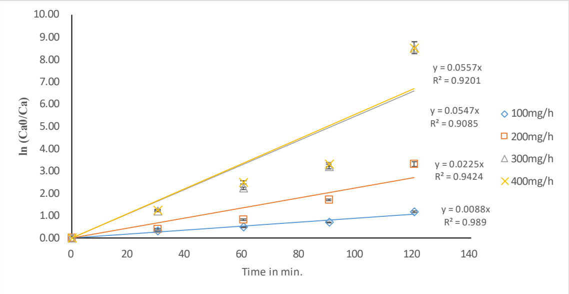
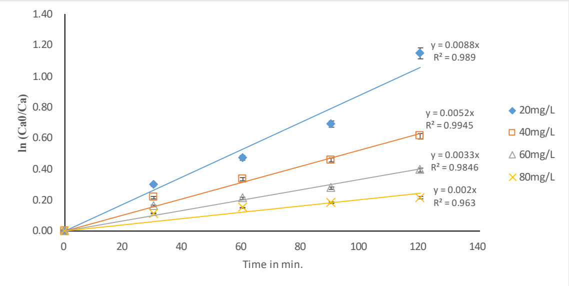
Table 5: % degradation of diclofenac sodium at different H2O2 concentration

% degradation of DS at different H2O2 concentration (ozone @ 100mg/hr 20ppm 7pH 60mg/L ZnO2)		
Initial H2O2 Concentration (mg/L)	Time	% degradation
20	0	0
	30	47.53
	60	60.29
	90	71.72
	120	82.28
40	0	0
	30	49.37
	60	62.74
	90	74.86
	120	85.44
60	0	0
	30	52.38
	60	66.04
	90	78.85
	120	89.52
80	0	0
	30	51.17
	60	63.76
	90	75.88
	120	87.93

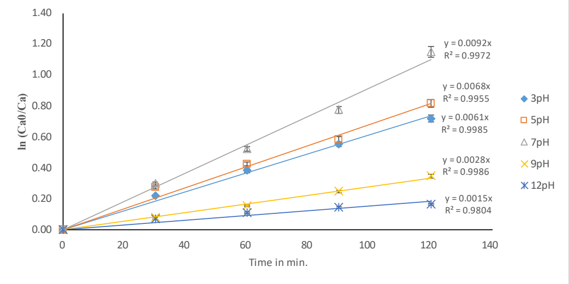
Graph 1: Effect of % degradation of diclofenac sodium at different ozone concentration



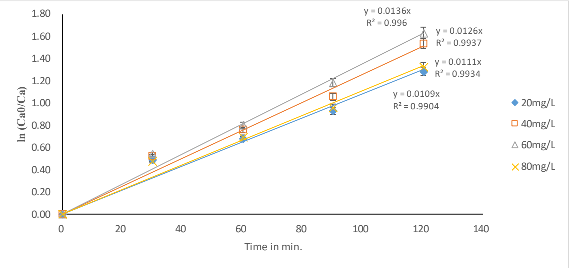
Graph 2: Effect of % degradation of diclofenac sodium at different initial concentration



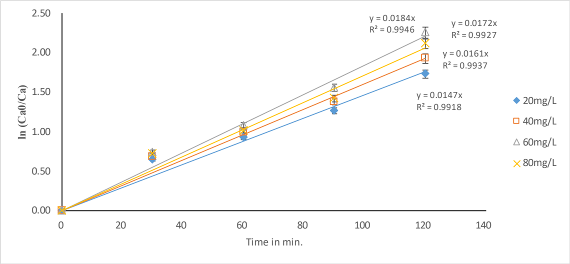
Graph 3: Effect of % degradation of diclofenac sodium at different initial pH



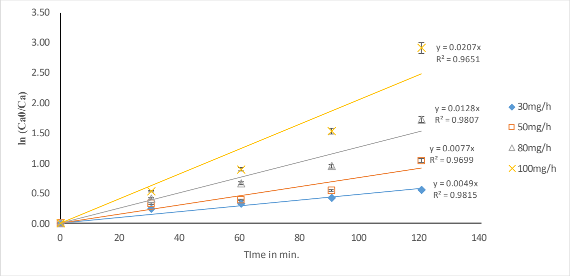
Graph 4: Effect of % degradation of diclofenac sodium at different initial ZnO2 concentration



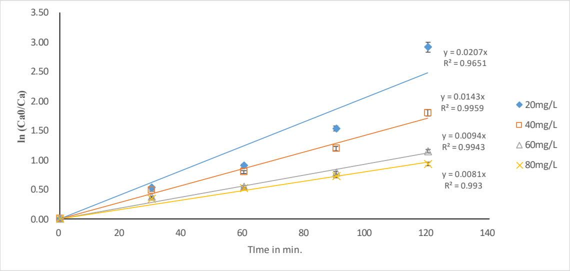
Graph 5: Effect of % degradation of diclofenac sodium at different H2O2 concentration



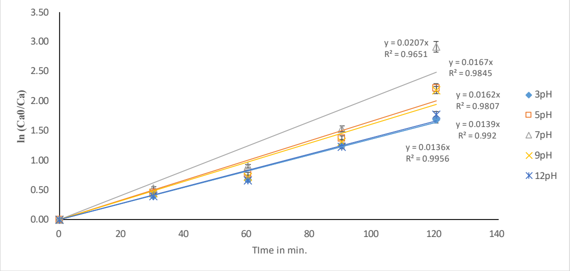
Graph 6: Effect of % degradation of Acephate at different ozone concentration



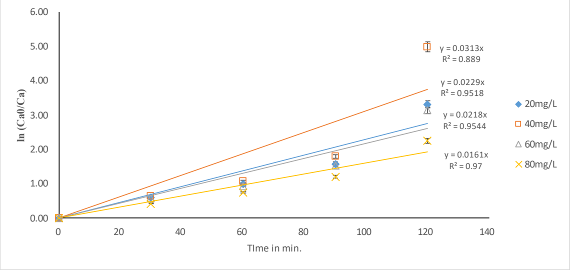
Graph 7: Effect of % degradation of Acephate at different initial concentration



Graph 8: Effect of % degradation of Acephate at different initial pH



Graph 9: Effect of % degradation of Acephate at different initial ZnO2 concentration



5. Conclusions

In the present study, the degradation of CN103145574A and CN101074241A has been investigated using various treatments strategies involving the use of ozonation, HPLC etc. The degradation was depended on parameters like pH of the solution, catalyst(ZnO) and oxidising agent (H_2O_2) loading time, initial concentration of diclofenac sodium and Acephate and ozone concentration.

a) The time critical studies for all the parameters signifies that the highest extent of degradation was observed at time of 120 min i.e. 2hr.

b) The pH was found to be influential factor on the degradation of impurities. The highest degradation was achieved at neutral pH for both impurities (Diclofenac Sodium and Acephate)

c) Catalyst ZnO proved to be an influential factor the degradation of impurities increased for all different concentrations used. But for diclofenac sodium highest degradation was achieved at 60mg/L concentration whereas for Acephate highest degradation was achieved at 40mg/L concentration.

d) The degradation efficiency increases with an increase in ozone flow rate for both the impurities.

e) In the combine approach with O_3 + H_2O_2, with increasing initial concentration decrease in degradation rate with fluctuating results was achieved with 100mg/h ozone flow rate whereas, high degradation was achieved at 200mg/h for Diclofenac Sodium.

f) In the combined process of O_3 + ZnO + H_2O_2 the maximum degradation was achieved at 89.52% at 100mg/h of ozone flow rate for diclofenac sodium.

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