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Use of Graphene Oxide-Zinc Oxide Composite in Photocatalytic Degradation of Victoria Blue B

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ABSTRACT

The photocatalytic activity of graphene oxide (GO) and zinc oxide composite was explored. The composites were prepared in three different ratios: GO:ZnO (1:1), (1:2) and (2:1) by mechanochemical method (grinding) of the two components. Graphene oxide was synthesized by three different methods namely Hummer's, modified Hummer's and Tour's method. It was observed that composite of GO-ZnO (2:1) gave better results. The photocatalytic efficiency of the composite was evaluated using Victoria blue B as a model pollutant. It was found to be even better than zinc oxide. The reusability of the composite was also confirmed.

1. Introduction

Organic dyes present in water bodies can cause deteriorating effects on the aquatic life and thus, as a consequence affect human health also [1]. Advanced oxidation processes (AOPs) are evolved as very efficient techniques to resolve the problem of not only water pollution but also air pollution. Thus, AOP serves as a very advantageous technique [2]. The degradation of dyes using nanosized semiconductor photocatalysts like TiO_2, CdS, ZnO, $BaCrO_4$, etc. is a promising solution for waste water treatment. The advantage with nanosized particles is based on the fact that they provide a large surface to volume ratio, which ensures high UV absorption and effectively separating electron and hole pair. Efforts were made to modify the properties of the photocatalytic material to make them more efficient [3-6].

Graphene oxide (GO) has gained importance as new nanostructured particles. They can be easily prepared by oxidation of graphene sheets, low cost, easily processed and compatible with various substrates [7]. The band gap of graphene oxide may vary depending on the level of oxidation. If graphene oxide is fully oxidized, it acts as an insulator, but if it is partially oxidized, then it behaves as a semiconductor [8]. Just like graphene, graphene oxide has a special surface with hydroxyl and carboxyl groups, which get attached to the other component during synthesis of GO-containing composites. Since graphene oxide is a photo-generated electron transmitter, it can not only extend the light absorption range, improves the adsorption of pollutant on its surface but also charge transportation and separation [9, 10].

Vadivel et al. [11] prepared graphene oxide-BiOBr composite and observed that degradation efficiency of the composite GO-BiOBr (5%) towards methylene blue and rhodamine B was 95-98%) as compared to pure BiOBr (40-50%). A composite of graphene oxide/chitin was synthesized by Wang et. al. [12] and used it for degradation of different dyes. This photocatalyst was fabricated by *in situ* synthesis of Cu_2O in the regenerated chitin (RC)/graphene oxide (GO) composite film. The rate of photocatalytic activity was enhanced drastically due to the introduction of Cu_2O in the Cu_2O/GO/RC film.

Guo et al. [13] synthesized graphene oxide-based hydrogels (GO/polyethyleneimine; PEI) and found it an efficient dye adsorbent in waste water treatment. The adsorption capacity of hydrogels is due to GO sheets, where PEI facilitates in the gelation process of GO sheets. Nano-TiO_2 functionalized graphene oxide was also used by Wang et. al. [14] for the degradation of methylene blue. Ag/graphene oxide was synthesized by Haldorai et al. [15] and used it for degradation of rhodamine 123 dye and acetaldehyde. Kashinath et al. [16] synthesized zinc oxide flower grown on graphene oxide sheets via microwave assisted facile hydrothermal method. It was reported that the structure and morphology played a key role in enhancing the photoresponse.

Huo et al. [17] successfully synthesized N-ZnO/CdS/Graphene oxide composite by hydrothermal method. This photocatalyst had a strong photocatalytic activity towards degrading ciprofloxacin, tetracycline, oxytetracycline hydrochloride and levofloxacin. The role of GO in the composite was as an electron conductor, and it also enhances the separation rates of electron and holes. Efficient degradation of methyl orange dye was reported by Esmaeili and Entezari [18] using the composite Ag/AgBr/graphene oxide. It was observed to have highest photocatalytic activity due to high adsorption capacity and enhanced charge transfer. Bare TiO_2 with graphene oxide was selected as a photocatalyst by Cruz et al. [19] for degrading pesticides like diuron, alachlor, isoproturon and atrazine. Diphenhydramine and methyl orange were photodegraded by graphene oxide-P25 photocatalyst by Morales-Torres et al. [20]. They observed that reduction of GO by thermal treatment in contact with TiO_2 resulted in enhancement of photocatalytic rate.

Umukoro et al. [21] photocatalytically degraded acid blue 74 in water using Ag-Ag_2O-ZnO nanostructures anchored on graphene oxide. It was observed that Ag, Ag_2O and graphene oxide improved the visible photocatalytic properties of ZnO. Percentage dye removal was found out to be ZnO (75%), Ag–Ag_2O–ZnO (85%) and Ag–Ag_2O–ZnO/GO (90%).

Surface modified TiO_2 and graphene oxide composite photocatalyst was prepared by Pastrana-Martínez et al. [22] and used for degradation of water pollutants under near-UV/Vis and visible light. They compared photocatalytic activity of TiO_2 catalyst synthesized by a modified sol–gel method (ECT), TiO_2 nanoparticles surface modified with organic shell layer (m-TiO_2) and a graphene oxide-TiO_2 composite (GOT-3.3). Graphene oxide based CdSe photocatalyst was synthesized by Ghosh et al. [23]. They used it for efficient photodegradation of rhodamine B and texbrite MST-L dye. Graphene oxide and reduced graphene oxide platelets were pillared with carbon nanotubes (CNTs) by Zhang et al. [24] using chemical vapor deposition with acetonitrile as the carbon source and nickel nanoparticles as the catalyst. The CNT-pillared RGO composite materials exhibited an excellent visible light photocatalytic performance in degrading Rhodamine B because of the unique porous structure and the exceptional electron transfer property of graphene.

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2. Experimental Methods

2.1 Preparation of Graphene Oxide

Graphene oxide was synthesized by three different methods namely Hummer's [8], modified Hummer's [8] and Tour's method [25]. It was observed that best results for photocatalytic degradation of dyes was found with composite of ZnO with GO (prepared by modified Hummer's method). Therefore, further experiments were carried out using graphene oxide synthesized by modified Hummer's method in the composite with ZnO.

2.2 Preparation of Composites

A composite of GO and ZnO was prepared by simple solid state mechanochemical method by grinding these two components using agate mortar and pestle. Three different composites of GO-ZnO were prepared based on different ratios of graphene oxide and zinc oxide (GO:ZnO) (1:1), (1:2), and (2:1). These composites were then used to degrade Victoria blue B dye.

3. Results and Discussion

3.1 Characterization of Composite

X-ray diffraction patterns of the composite GO-ZnO was taken using X-ray Diffractometer Panalyticals XPert Pro individually for GO-ZnO (1:1, 1:2 and 2:1) composites and graphene oxide. The results are shown in the Figs. 1-4 respectively. A peak around 2θ = 10.65° is attributed to graphene oxide sheets and is the characteristic peak of graphene oxide [26]. The average particle size of the crystalline composites and graphene oxide was calculated using Debye –Scherrer equation and are summarized in the Table 1. It was observed that as the amount of ZnO was increased in the composite, the average particle size also increases.

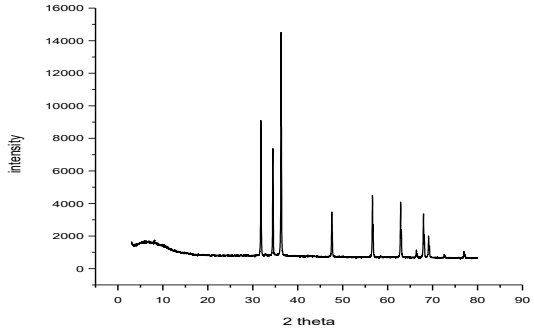

Fig. 1 X-ray diffraction pattern of 1:1 GO-ZnO

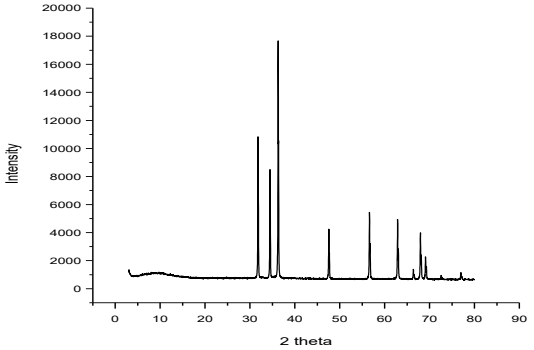

Fig. 2 X-ray diffraction pattern of 1:2 GO-ZnO

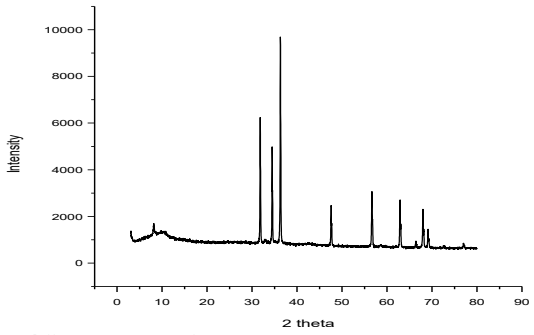

Fig. 3 X-ray diffraction pattern of 2:1 GO-ZnO

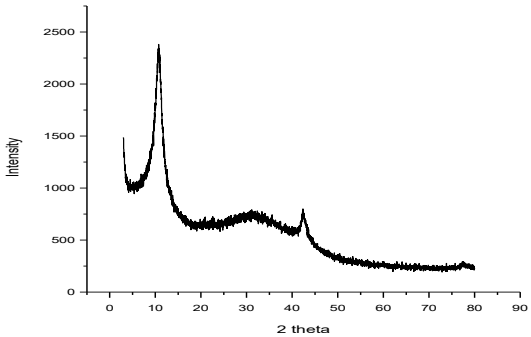

Fig. 4 X-ray diffraction pattern of graphene oxide (GO)

Table 1 Average particle size

Composite	Crystallite Size
GO-ZnO (1:1)	48.33 nm
GO-ZnO (1:2)	49.41 nm
GO-ZnO (2:1)	46.89 nm
Graphene oxide	4.717 nm

Fourier Transform Infrared (FTIR) spectra was recorded using F.T. Infra-Red Spectrophotometer Model RZX (Perkin Elmer). The IR spectra of GO and GO-ZnO (2:1) composite are given in the Figs. 5 and 6 respectively.

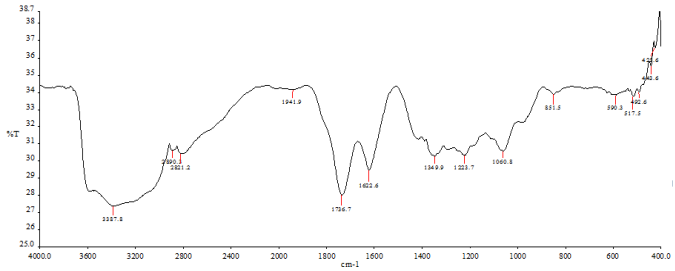

Fig. 5 FTIR spectrum of GO (Graphene oxide)

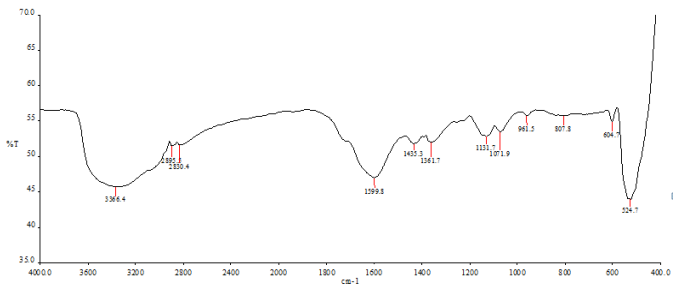

Fig. 6 FTIR spectrum of GO-ZnO composite

Different vibration bands were observed for GO, which may be attributed to alkoxy C=O stretching (1060.8 cm^{-1}), phenolic C-OH stretching (1223.7 cm^{-1}), carboxyl O-H stretching (1349 cm^{-1}), C=O stretching vibrations of carboxyl and carbonyl functional groups (1736 cm^{-1}) and hydroxyl hydrophilic group (3387 cm^{-1}).

A broad peak at 3366.4 cm^{-1} shows O-H stretching vibrations of the C-OH groups and water. [26] A peak around ~500 cm^{-1} (524 cm^{-1}) suggests the Zn-O bond vibrations in GO-ZnO composite. EDX analysis was conducted to detect the elemental composition of the composites and graphene oxide. The results are reported in Table 2.

Table 2 Elemental composition

Elements	1:1 (GO : ZnO)	1:2 (GO : ZnO)	2:1 (GO : ZnO)	Graphene oxide
	Atomic (%)	Atomic (%)	Atomic (%)	Atomic (%)
O	31.35	21.50	25.04	29.93
Zn	42.67	54.39	33.39	_
C	25.98	24.11	41.56	70.07

3.2 Photocatalytic Degradation

The photocatalytic activity of GO-ZnO composites was evaluated by degrading Victoria blue B in aqueous solution. The light source used for the experiment was 200 W lamp (Philips). 0.0506 g of the dye was dissolved in 100 mL doubly distilled water to prepare a stock solution of the concentration 1.0×10^{-3} M. This stock solution was diluted for making working solutions as and when desired. The wavelength of maximum absorption (λ_{max}) for the Victoria blue B dye was observed using

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spectrophotometer (Systronics model 106) and it was found to be 592 nm. A water filter was used in order to cut-off thermal radiations.

The dye solution of a particular concentration was poured equally in four different beakers. The first beaker having the Victoria Blue dye solution was kept in dark. The second beaker was simply exposed to light. The third beaker containing dye solution and GO-ZnO composite was placed in dark while fourth beaker containing dye solution and composite was exposed to light. After 4 hours of exposure, the absorbance in all four beakers was measured. It was observed that there was a decrease in the absorbance of fourth beaker while there was negligible or no change in absorbance of remaining three beakers. It was observed that the presence of composite (GO-ZnO) and light energy, both are necessary for the degradation of the dye.

The light intensity was measured by solarimeter (Suryamapi CEL 201). pH was measured using a digital pH meter (Systronics) pH of solution was varied by adding 0.1 N H_2SO_4 and 0.1N NaOH solution. The absorbance (A) of the dye solution was measured by UV visible spectrophotometer (Systronics Model 106). The experiment was conducted to observe photocatalytic degradation of Victoria blue dye using three different ratios of the composite GO:ZnO. The optimum conditions were obtained for all the three composites and are reported in Table 3. It was found that the reaction follows a pseudo-first order kinetics as evident from a linear plot between log A and time (Table 4 and Fig. 7). The rate constant can be calculated as k = 2.303 × slope.

Table 3 Optimum conditions

	GO:ZnO (1:1)	GO:ZnO (1:2)	GO:ZnO (2:1)
pH	7.0	6.5	5.5
[Victoria Blue B] (M)	5.0×10^{-5}	5.0×10^{-5}	5.0×10^{-5}
Amount of composite used (g)	0.10	0.05	0.06
Light intensity ($mWcm^{-2}$)	50.0	70.0	70.0

Table 4 Typical runs

Time (min)	1:1 (GO:ZnO)		1:2 (GO:ZnO)		2:1 (GO:ZnO)	
	Absorbance (A)	1 + log A	Absorbance (A)	1 + log A	Absorbance (A)	1 + log A
0	0.676	0.8299	0.485	0.6860	0.591	0.7716
10	0.591	0.7716	0.457	0.6599	0.501	0.6998
20	0.484	0.6848	0.418	0.6211	0.412	0.6149
30	0.409	0.6117	0.389	0.5900	0.363	0.5599
40	0.386	0.5866	0.348	0.5416	0.292	0.4654
50	0.331	0.5198	0.329	0.5172	0.269	0.4298
60	0.309	0.4900	0.301	0.4786	0.219	0.3403
70	0.262	0.4183	0.282	0.4502	0.184	0.2648
80	0.224	0.3502	0.258	0.4116	0.167	0.2227
90	0.201	0.3032	0.236	0.3729	0.153	0.1847
	$k (1:1) = 2.30 \times 10^{-4} sec^{-1}$		$k (1:2) = 1.34 \times 10\text{-}4 sec^{-1}$		$k (2:1) = 2.770 \times 10^{-4} sec^{-1}$	
	$k (ZnO) = 9.21 \times 10^{-5} sec^{-1}$		$k (ZnO) = 1.04 \times 10^{-4} sec^{-1}$		$k (ZnO) = 7.93 \times 10^{-5} sec^{-1}$	

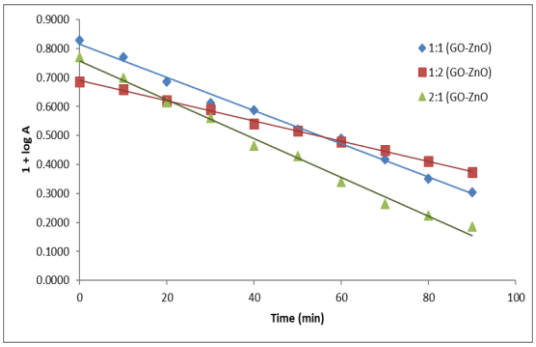

Fig. 7 Typical runs

Table 5 Effect of pH

pH	$k \times 10^4 (sec^{-1})$ (1:1)	$k \times 10^4 (sec^{-1})$ (1:2)	$k \times 10^4 (sec^{-1})$ (2:1)
3.0	0.70	0.48	1.54
3.5	0.77	0.56	2.09
4.0	0.86	0.90	2.31
4.5	0.90	1.03	2.57
5.0	1.02	1.22	2.58
5.5	1.10	1.23	2.77
6.0	1.15	1.25	2.56
6.5	1.17	1.34	2.40
7.0	2.30	1.18	2.23
7.5	1.67	0.36	2.18
8.0	1.15	0.35	1.64

3.2.1 Effect of pH

Photocatalytic degradation of any dye solution may be affected by the pH of the medium. pH was varied from 3.0 to 8.0. The effect of pH on the dye degradation with three different ratios of the composite is summarized in the Table 5. A general trend was observed in all the three cases. An increase in rate of degradation was observed up to a certain pH, and after reaching an optimum value, rate of degradation starts falling.

As the pH was decreased, there was a corresponding decrease in the rate of degradation of Victoria blue B. This may be explained on the basis that in acidic pH range, the dye is present in cationic form and the surface of the composite is positively charged due to adsorption of protons. Hence, there will be repulsion between two positively charged species resulting in lowering of rate.

3.2.2 Effect of Dye Concentration

The dye concentration was varied from 3.0×10^{-5} M to 6.0×10^{-5} M. A regular increase in the rate of dye degradation was observed till dye concentration reached 5.0×10^{-5} M and thereafter, there was a gradual fall in rate (Table 6). The photocatalytic activity increases with the increase in the concentration of the dye as a greater number of dye molecules are available for excitation and energy transmission and thus, an enhanced photoactivity is observed. But with further increase in the dye concentration above this limit, the dye starts behaving as a filter and will not allow incident light to reach the surface of composite and a decrease in photoactivity was observed.

Table 6 Effect of dye concentration

$[VB B] \times 10^5$ M	$k \times 10^4 (sec^{-1})$ (1:1)	$k \times 10^4 (sec^{-1})$ (1:2)	$k \times 10^4 (sec^{-1})$ (2:1)
3.0	1.56	0.74	1.72
3.5	1.70	0.86	1.95
4.0	2.04	0.96	1.99
4.5	2.13	1.01	2.75
5.0	2.30	1.34	2.77
5.5	1.70	1.26	1.92
6.0	1.60	0.89	1.80

3.2.3 Effect of Amount of Catalyst

The amount of photocatalyst may also affect the photocatalytic degradation of Victoria blue B. The amount of the catalyst was varied from 0.01 g to 0.1 g. It was observed that the rate of photodegradation of Victoria Blue B increases with an increase in the amount of catalyst to a certain extent reaching a maximum value. Any further increase in the amount results in a decrease of the rate of degradation. The results are represented in the Table 7. It can be explained on the basis that as the amount of composite was increased, the exposed surface area also increases. As a result, the rate of photodegradation increases. With a further increase in the amount of composite beyond this limit, the exposed surface area will not increase but the excess amount will form multilayers of the composite. These multilayers of the composite will interact with each other resulting in the recombination of generated electron-hole pairs. Thus, number of effective electron-hole pairs decreases and the photodegradation activity is declined.

Table 7 Effect of amount of catalyst

Catalyst (g)	$k \times 10^4 (sec^{-1})$ (1:1)	$k \times 10^4 (sec^{-1})$ (1:2)	$k \times 10^4 (sec^{-1})$ (2:1)
0.01	0.81	1.09	1.10
0.02	0.87	1.18	1.18
0.03	0.88	1.24	2.05
0.04	1.02	1.26	2.13
0.05	1.20	1.34	2.50
0.06	1.23	1.27	2.77
0.08	1.83	1.20	1.85
0.10	2.30	1.17	1.64

3.2.4 Effect of Light Intensity

The intensity of light also influences the rate of photodegradation. The light intensity was varied from 20.0 to 70.0 $mWcm^{-2}$ and the results are tabulated in the Table 8. The data revealed that the rate of degradation increases with an increase in the light intensity, but a further increase in light intensity was found to decrease the rate of photodegradation. An increase in photoactivity may be due to an increased number of photons particles striking per unit area per unit time. But with further increase in intensity there could be chances of thermal reactions resulting in a fall in photoactivity and therefore, higher intensities were avoided

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Table 8 Effect of light intensity

Light intensity ($mWcm^{-2}$)	$k \times 10^4$ (sec^{-1}) (1:1)	$k \times 10^4$ (sec^{-1}) (1:2)	$k \times 10^4$ (sec^{-1}) (2:1)
20.0	1.72	0.65	0.90
30.0	1.86	0.66	1.39
40.0	2.16	0.80	1.39
50.0	2.30	0.82	1.68
60.0	2.17	0.85	2.25
70.0	1.45	1.34	2.77

3.3 Mechanism

From all these observations, a tentative mechanism for photocatalytic degradation of Victoria blue B can be proposed. A light of suitable wavelength was absorbed by the dye and it gets excited to its first excited singlet state. This first excited singlet state of the dye undergoes inter system crossing (ISC) and gets converted to its triplet state. Composite (GO-ZnO) also absorbs the incident light energy and excite its electron from the valence band to the conduction band, leaving a hole in the valence band. This hole may extract an electron from the hydroxyl ions to produce hydroxyl radicals. The hydroxyl radical will behave as the active oxidizing species and convert the dye to its leuco form, which may ultimately degrade to smaller products. The confirmation of hydroxyl radical (•OH) as an active oxidising species was done by using isopropyl alcohol (2-propanol) (a hydroxyl radical scavenger). Here, a significant decrease in the rate of dye degradation was observed supporting the participation of •OH as an oxidant. This mechanism is operative in the composite (1:1 ratio).

$$^1VBB_0 \xrightarrow{h\nu} {}^1VBB_1 \quad (1)$$

$$^1VBB_1 \xrightarrow{ISC} {}^3VBB_1 \quad (2)$$

$$SC \xrightarrow{h\nu} SC\,[h^+ (VB) + e^- (CB)] \quad (3)$$

$$h^+ + OH^- \longrightarrow {}^\bullet OH \quad (4)$$

$$^3VBB_1 + {}^\bullet OH \longrightarrow \text{Leuco VBB} \quad (5)$$

$$\text{Leuco VBB} \longrightarrow \text{Products} \quad (6)$$

Here VBB = Victoria Blue B. The first three steps in the mechanism are similar to the above-mentioned mechanism with the composite ratio (1:1) but an electron generated in the third step is captured by the dissolved oxygen forming oxygen anion radical. This superoxide anion radical is unstable in the acidic medium and hence, reacts with H^+ ions forming $HO_2^\bullet$ radical. Now this $HO_2^\bullet$ radical will behave as an active oxidising species and convert the dye to its leuco form, which ultimately degrade to smaller products. This mechanism is operative with composites having (1:2 and 2:1 ratios) as the optimum pH was in acidic range.

$$^1VBB_0 \xrightarrow{h\nu} {}^1VBB_1 \quad (7)$$

$$^1VBB_1 \xrightarrow{ISC} {}^3VBB_1 \quad (8)$$

$$SC \xrightarrow{h\nu} SC\,[h^+ (VB) + e^- (CB)] \quad (9)$$

$$e^- + O_2 \longrightarrow O_2^{\bullet-} \quad (10)$$

$$H^+ + O_2^{\bullet-} \longrightarrow HO_2^\bullet \quad (11)$$

$$HO_2^\bullet + {}^3VBB_1 \longrightarrow \text{Leuco VBB} \quad (12)$$

$$\text{Leuco VBB} \longrightarrow \text{Products} \quad (13)$$

4. Conclusion

Among the three different methods, composite with graphene oxide prepared by modified Hummers method was found to be more active for photocatalytic degradation of Victoria blue B. Among the three ratios of composites (GO-ZnO) (1:1, 1:2 and 2:1), the composite with ratio 2:1 was highly active than the remaining two composites. Scavenger studies indicated that the photoactive oxidizing species in case of ratio 1:1 (GO-ZnO) was •OH radical while for the ratio (1:2) and (2:1), the active species was $HO_2^\bullet$ radicals. This composite could be reused 4 times without any significant loss in its photoactivity. Graphene oxide and its composites found various applications in the field of energy storage devices, sensors, biosensors, antibacterial activity, electronic devices, biomedical and optical applications etc. apart from its use as a photocatalyst.

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