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SYNTHESIS OF NANO BIOCL, CHARACTERIZATION AND APPLICATION IN PHOTOCATALYTIC DEGRADATION OF TOLUIDINE BLUE IN SOLAR LIGHT

Brijesh Pare[*1], Vaishali Joshi[2] & Satish Piplode[3]

[*1,2&3]Laboratory of Photocatalysis, P. G. Department of Chemistry, Govt. Madhav Science P. G. College, Ujjain, India 456010

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ABSTRACT

Nanoparticle technology is becoming an increasingly popular choice for treatment of hazardous and toxic wastes, and for remediation of contaminated water. The smaller particle size of nanoparticles and their large specific surface area corresponds to enhanced reactivity for wastewater remediation. Recent trends in nanoparticle synthesis and production have resulted in substantial cost reductions and increased availability of nanoscale BiOCl for large scale applications. In this work, a simple hydrolysismethod of nano BiOCl synthesis at room temperature and its characterization is highlighted. The average particle size of as-prepared nano BiOCl was obtained to be 45 nm. Activity of nano BiOCl for treatment oftoluidine blue dye was studied for different reaction conditions.

KEYWORDS: Nano BiOCl, XRD, SEM, Photocatalytic Degradation, Solar Light.

I. INTRODUCTION

Now a days, scientists are focused on synthesis and applications of nanomaterials in various fields. Nanoparticles show completely new properties as compared to their bulk counterparts. Many nanomaterials possess distinctive mechanical, magnetic, optical and catalytic properties as a result of their subcolloidal size and unique molecular and atomic structures, which contributes in the promising applications in various fields(1). Recently synthesis of bismuth oxychloride has attracted much attention among scientific communities, due to its nontoxic nature, stability and low cost. Nano BiOCl possess high surface to volume ratio, which results in greater availability of active sites and hence lead to good catalytic and photocatalytic properties.These unique properties of nanoparticles are size and shape dependent. Nanotechnology has been applied to the photocatalytic degradation of pollutants in waste water treatment, In a typical process, semiconductor photocatalysts absorbs radiations higher than bandgap and generates electron hole pairs(2,3).

In this study nano BiOCl has been synthesized in aqueous solution at room temperature and employed for the photocatalytic degradation of an environmentally hazardous dye toluidine blue. This work has been carried out in solar light to make the process more cost effective and easy to handle.

II. MATERIALS AND METHODS

Synthesis of Nano BiOCl

All the reagents (analytical grade) were commercially available and used as received. In the process, 1.26 g $BiCl_3$ (4 mmol) was dissolved in 2 mL HNO_3 (65 wt %) and then was diluted to 100 ml using deionized water which contains a proper amount of L-Lysine. After further stirring for 5 min, the pH of the solution was adjusted to 9 by adding aqueous ammonia (5 wt.%) dropwise with constant stirring. After another 5 min of stirring, the resulted white precipitate was collected, and washed several times with distilled water and absolute ethanol. The product was then dried (at 80 °C for 4 h)(4).

Measurment of Photocatalytic activity

Dye solution was freshly prepared by dissolving in double distilled/deionized water. Prior to light experiments, dark (adsorption) experiments were carried out to know the extent of adsorption of the dye on the catalyst. For solar experiments, dye solution of 100 mL was taken in a double walled beaker with water circulation system, to this solution a known amout of catalyst was added and this complete setup was placed on a magnetic stirrer and

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illuminated in solar light of optimistic intensity, in clear weather. Water circulation system maintains the temperature and controls evaporation of reaction mixture. To measure the concentration changes due to dye degradation, the 3 ml aliquot was periodically withdrawn from the reaction mixture and centrifuged at 3500 rpm to remove BiOCl particles. The spectrophotometer 166 of Systronics was used for measuring absorbance at different time intervals. The light intensity was measured by a Lux meter (Lutron LX-101). The pH of the solution was constantly been monitored using a pH meter. pH was adjusted by the addition of either NaOH or H_2SO_4.

III. RESULTS AND DISCUSSIONS

Structural studies

Fig.1 shows the XRD patterns of the as prepared nanoBiOCl sample. No other diffraction peaks were detected, indicating the high purity of BiOCl. The intense and sharp diffraction peaks suggested that the as synthesized product was well- crystallized. From the x-ray patterns the broadening of the diffraction peaks of the nanoparticles is obvious(5)which is characteristic of nanosized. By applying Debye- scherrer formula(6).

$$D = \frac{0.9\,\lambda}{\beta COS\theta}$$

Where D is the mean particle size, λ is the wavelength of incident X-ray (1.5406 ˚A), θ is the degree of the diffraction peak , and β is the full width at half maximum (FWHM)of the XRD peak appearing at the diffraction angle θ, the average particle size of nano BiOCl was calculated as 45 nm Fig. 6 shows the XRD pattern of nano BiOCl, and Fig. 2 shows the SEM image of the as-prepared nano BiOCl. It can be clearly seen that the BiOCl is present in plate like structures with smooth surfaces.

Photocatalytic Studies

Effect of pH

The pH of reaction mixture was varied from 4 to 11. It was observed that degradation rate constants were found higher at 4 pH as well as 11 pH as 5.18×10^{-4} s^{-1} and 6.25×10^{-4} s^{-1} respectively. These high rate constants at both these pH values is attributed to the fact that toluidine blue has two pKa values (2.4 and 11.6). Hence the degradation rate constant values decreased with the increase in pH from 4 to 8 and further increased with increase in pH from 9 to 11. Thus highly acidic and highly alkaline pH favours the toluidine blue degradation(7). The effect of pH on the photocatalytic degradation of toluidine blue is presented in Fig. 3.

Effect of Catalyst Loading

The catalyst concentration greatly influences the photocatalytic reactions. In this study the concentration of BiOCl nanoplates was varied from 10 mg to 60 mg/100 ml in solar light and all other factors were kept constant. It was observed that with an increase in amount of catalyst from 10 mg to 30 mg/100 ml the rate was found to be increased from 4.52×10^{-4} s^{-1} to 6.25×10^{-4} s^{-1}because of an increase in the number of active sites available on the catalyst surface.When the catalyst concentration was increased from 40 mg to 60 mg/100 ml in next few sets further decreased the rate as 5.75×10^{-4} s^{-1} to 3.22×10^{-4} s^{-1}. It is because with a higher catalyst loading, tendency of agglomeration of photocatalyst increases which leads to the less available surface area of catalyst, higher catalyst dosage may also result into increased turbidity of the solution which hinders the passage of light(8). The photocatalytic degradation of toluidine blue for different concentration of nano BiOCl is shown in Fig. 4.

Effect of Initial Dye Concentration

The impact of dye concentration greatly alters the degree of photoatalysis with time. It was studied by changing the concentration of dye and keeping all other factors constant. The concentration of dye has been varied from 1.0×10^{-5} mol L^{-1} to 7.0×10^{-5} mol L^{-1}. The experimental data shows that an increase in concentration of toluidine blue dye from 1.0×10^{-5} mol L^{-1} to 4.0×10^{-5} mol L^{-1} increased the degradation rate constant from 3.37×10^{-4} s^{-1} to 6.25×10^{-4} s^{-1} because with an increased dye concentration more molecules of dye was available for energy transfer via excitation, as a result of which a gradual increase in the rate was noticed. On increasing dye concentration from 5.0×10^{-5} mol L^{-1} to7.0×10^{-5} mol L^{-1} reduced the degradation rate constant values from 5.33×10^{-4} s^{-1} to 3.49×10^{-4} s^{-1}. The cause of this loss in the rate may be given as, dye molecules in their higher concentrations shield the desired light intensity to reach the semiconductor photocatalyst(9).The results are summarized in Fig 5

IV. CONCLUSION

In summary we conclude the facile and green method to synthesize BiOCl nanoparticles and its use in photocatalytic degradation of toluidine blue. The different characteristics of synthesized nanoparticles were studied by XRD and SEM analysis. The purity and high crystallinity of nano BiOCl was confirmed by XRD. SEM image showed the plate-like morphology of nano BiOCl. The photocatalytic efficiency of nano BiOCl were tested against various reaction parameters for toluidine blue dye in solar light.The possible mechanisms of effect of various reaction parameters like pH, photocatalyst concentration and initial dye concentration were also discussed.

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Graphs and Figures:

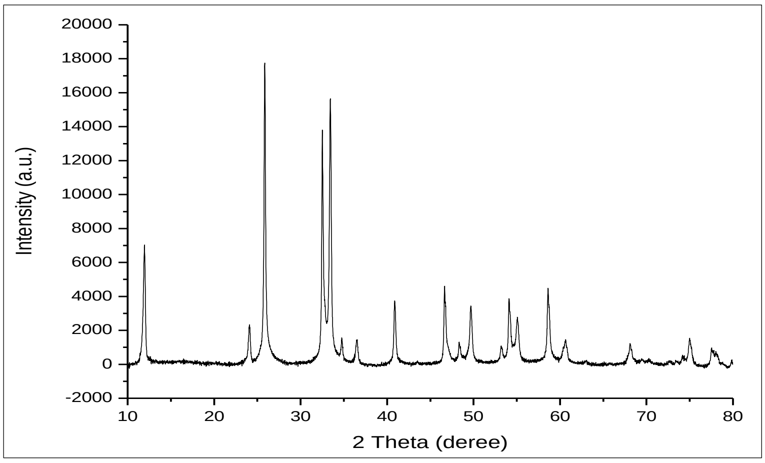

Fig 1 : XRD Pattern of nano BiOCl

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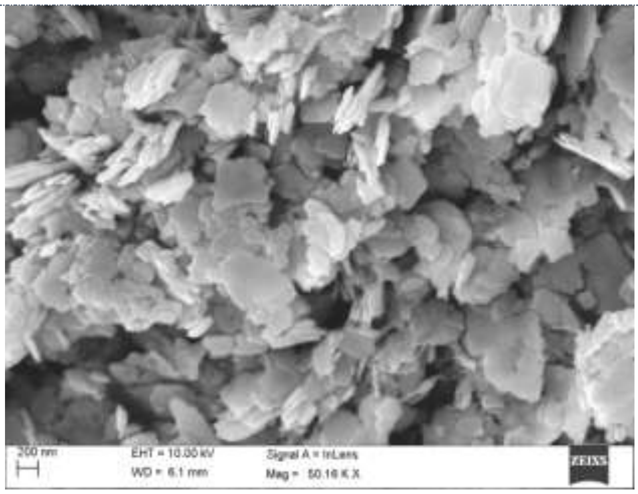

Fig. 2: SEM Image of Nano BiOCl

Effect of Initial pH: [TB] = 4×10^{-5}mol L^{-1}, BiOCl NPs = 30 mg/100 ml

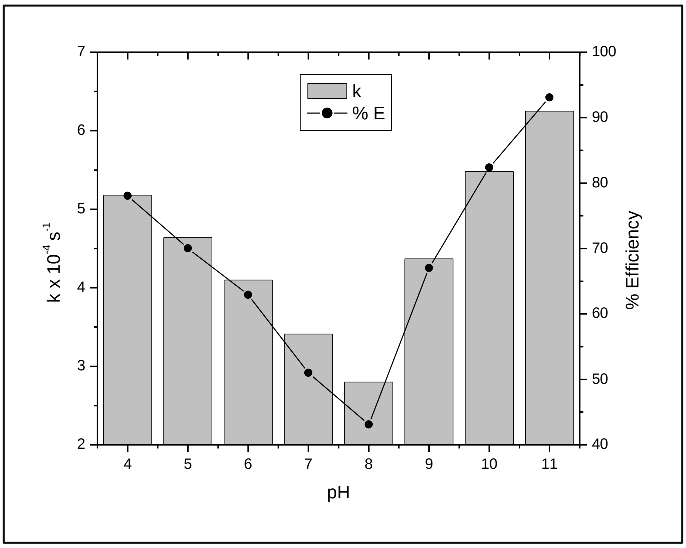

Fig. 3 : Effect of Initial pH

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Effect of Catalyst Loading: [TB] = 4 × 10^{-5}mol L^{-1}, pH = 11

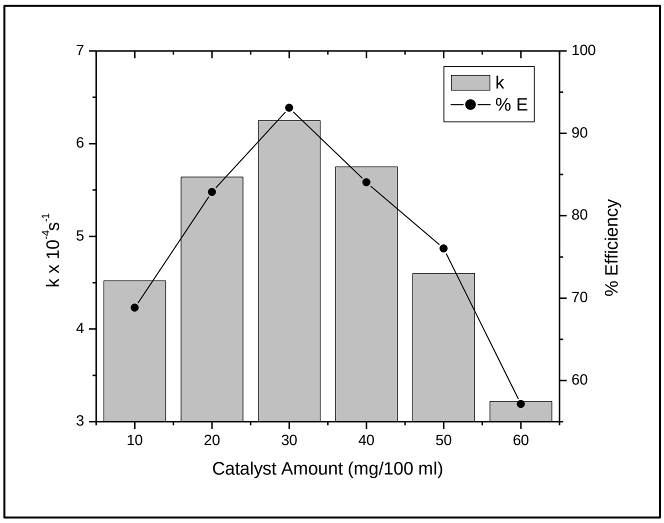

Fig. 4 : Effect of Catalyst Loading

Effect of Initial Dye Concentration: pH = 11, BiOCl NPs = 30 mg/100 ml

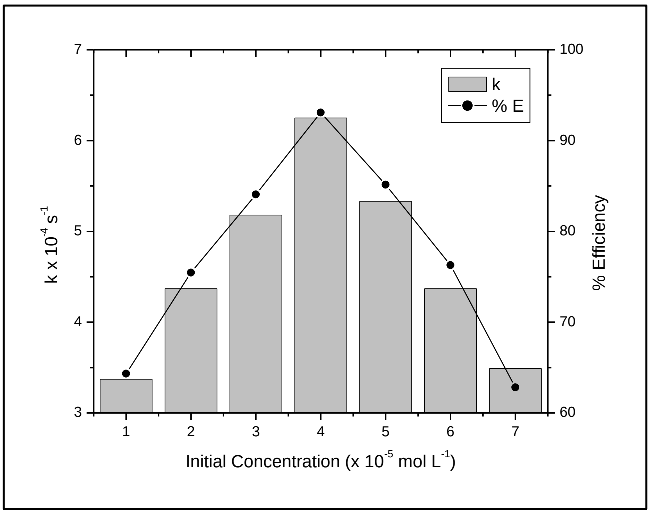

Fig. 5 : Effect of Initial Dye Concentration

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