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Preparation of a New Carbon/g-C_3n_4 Composite and Its Photocatalytic Activity

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Abstract*:*Novel activated carbon-graphitic carbon nitride (CNC) composite photocatalyst was synthesized by impregnation method. The resulting CNC composite photocatalyst was characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM) with EDAX, Fourier transform infrared spectroscopy (FT-IR). The photocatalytic activity of the novel photocatalyst was evaluated using Rhodamine B (Rh-B) as a target pollutant. The CNC composite sample exhibit enhanced photocatalytic performance under visible light irradiation than pure g-C_3N_4. The synergistic effect between carbon and g-C_3N_4 is found to be an improved photo generated carrier separation. The complete mineralization of Rh-B is determined by Chemical oxygen demand (COD) analysis.

Keywords: Activated carbon, Carbon nitride, Composite photo catalyst, Melamine, Rhoda mine B.

I. INTRODUCTION

Photocatalysis is an attractive yet challenging process to convert solar energy in to chemical energy. During the past 30 years, various inorganic semiconductors and molecular assemblies have been developed as catalysts for hydrogen production from water and as an environmental purification under visible light [1, 2]. However, the most stable metal oxide photocatalysts suffer from no or limited visible light absorption due to their large band gap [3]. For photocatalysis, precious-metal species must be used in most cases as extra co-catalysts to promote the transfer of photo induced charge carriers from bulk to the surface at which organic dyes are converted into CO_2 and H_2O. Synthetic polymer semiconductors have also been used for dye degradation; however they are active only in the ultraviolet region and have moderate performance. Here another simple polymer like semiconductor made of only carbon and nitrogen can function as a metal-free photocatalyst under visible light. Carbon nitride can exist in several allotropes with diverse properties but the graphitic phase is regarded as the most suitable under ambient conditions. The novel photocatalyst exhibits activity for the removal of organic pollutants under visible light irradiation [4], clearly demonstrating that the metal-free g-C_3N_4photocatalystposses an interesting electronic properties as well as high thermal and chemical stability, therefore making them valuable materials for photocatalysis applications. However, to date the photocatalytic efficiency of bare g-C_3N_4 is still limited due to the higher recombination rate of photo generated electron-hole pairs. Many methods have been used to extend the photocatalytic performance such as doping with metals or non-metal elementals [5] and coupling with other semiconductors [6]. These methods are effective because the high surface area and small particle size it enhance e^--h^+ pairs separation [7].

In this work, for the first time carbon/ g-C_3N_4 (CNC) composite was synthesized by impregnation method. The composite should increase the visible light harvesting efficiency by increasing the surface area and to evaluate the photo degradation of Rh-B under visible-light irradiation

II. EXPERIMENTAL

Melamine and activated carbon were purchased from Sigma-Aldrich. All other reagents were of analytical grade and were used as without further purification.

A. Synthesis of photocatalysts

The metal free graphitic carbon nitride (g-C_3N_4) powder was synthesized by thermal treatment of precursor melamine and it was placed in a crucible with a cover under ambient pressure in air. After dried at 80 °C for 24 h, the precursor was put in a Muffle furnace and heated to 550 °C for 3 h with a heating rate of 10 °C min^{-1}. The resultant yellow product was collected and ground into a powder for further use.

CNC composite photocatalyst was prepared as follows:

Carbon doped g-C_3N_4 composite photocatalyst was prepared by an impregnation method. Synthesis involved mixing calculated quantities of the above prepared CN powder and activated carbon in the ratio 10:1 was dispersed in 1 M HCl and deionized water.

The dispersion was magnetically stirred for 3 h. After that the product was collected by centrifugation and washed with ethanol and deionized water. Finally the sample was dried at 80 °C in an oven for 1 h. The obtained CNC was in the form of grey powder.

B. Characterization Techniques

X-ray diffraction (XRD) analysis was carried out at room temperature with a Bruker D8 advance diffract meter. The patterns were run with Cu Kα radiation at a scan rate of 20-80 °C. UV-Visible spectral data were collected over a spectral range 200-800 nm with Shimadzu UV-3101 PC spectrophotometer. Fourier transform-Infra red spectra (FT-IR) in transmittance mode were recorded for a solid mixture of sample and KBr in the form of pellets on a SHIMADZU FTIR spectrometer in the frequency range 4000 to 400 cm^{-1} with a spectral resolution of 16 (1 cm^{-1}) and an accumulation of 70 scans at room temperature. Scanning electron microscope (SEM) analysis was performed on platinum coated samples using a JOEL apparatus model JSM-5610 LV. Elemental analysis was performed by energy dispersive X-ray micro analysis (EDAX) using BRUKER-10498 model instrument.

C. Evaluation of photocatalysts

The photocoatalytic degradation of Rh-B was performed under the natural sunlight in the presence of CNC composite photocatalysts. In the photocatalytic treatment of dye, a known concentration of Rh-B dye solution 1×10^{-5}M (5 mg/l) was prepared in deionized water resulting in a solution with pH 7.6 was taken in a borosil glass beaker of 250 ml capacity. 0.1 g photocatalyst was added to the 50 ml (2 g/l) of dye solution. Before irradiation of the dye solution, the suspension was stirred for 30 min in the dark to realize adsorption-desorption equilibrium in the presence of catalyst. The dye solution was agitated with an electromagnetic stirrer at a constant speed of 790 rpm. The dye solution was directly exposed to sunlight radiation in an open atmosphere. At given intervals of irradiation a known volume of sample along with the catalyst particles were collected, centrifuged and then filtered through Millipore filter paper. The filtrates were analyzed by UV-Visible spectrophotometer. The determination wavelength is 553 nm for Rh-B dye, which is the maximum absorption wavelength.

The degradation efficiency of dye is calculated by the following equation

$$\text{Degradation efficiency (\%)} = \frac{C_0 - C}{C_0} \times 100$$

Where, C_0 is the initial concentration of dye before irradiation and C is the concentration of dye after a certain irradiation time.

III. RESULTS AND DISCUSSION

A. Characterization of metal free CNC composite sample

The crystalline and phase structure of the pure g-C_3N_4 is determined by X-ray diffraction (XRD) as shown in Fig.1. The X-Ray diffraction patterns of pure graphitic carbon nitride revealed an intense broad peak at 2θ value of 27.57°, indexed to (002) planes which is a characteristic indicator of layered stacking with a distance of 0.326 nm in the conjugated aromatic system and the inplanar repeating tri-s-triazine unit with a period of 0.675 nm can be observed from the diffraction peak at 2θ = 13.12°, indexed with (100) plane [8]. Furthermore in CNC composite two pronounced peaks at 2θ = 27.4° and 12.28° correspond to the characteristic diffraction peaks of pure g-C_3N_4 composite. This together with the appearance of small peaks at 2θ = 6.15°, 10.6°, 18.38°, 22.9°, 30.9° indicates the successful deposition of activated carbon on to the graphite carbon nitride layer to form CNC composite as depicted in Fig .2.

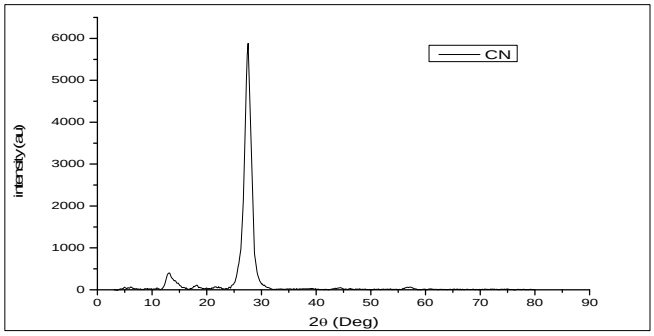


Fig.1 XRD pattern of CN composite sample.

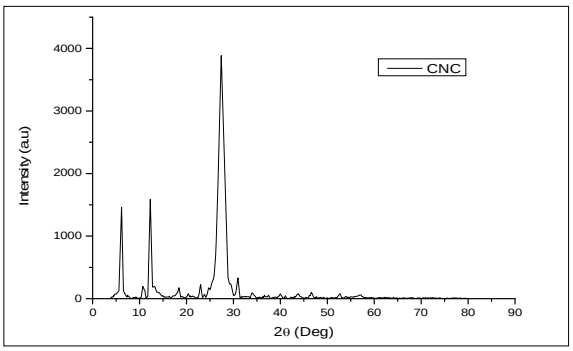


Fig.2 XRD pattern of CNC composite sample.

The molecular geometry, information about its functional groups and inter/intra molecular interactions of the resulting composite photocatalysts are characterized by FT-IR spectroscopy. Fig. 3 and Fig. 4 show the FT-IR spectra of pure g-C_3N_4 and CNC composite samples. The main characteristic peak of pure g-C_3N_4 can be assigned as follows: The main absorption bands at 1080, 1242, 1327, and 1411 cm^{-1} is allocated to aromatic C-N stretching and the conjugated C=N stretching can be seen at 1566 and 1627 [9]. The band at 810 and 887 cm^{-1} is attributed to out of plane bending modes of triazine units.

The FT-IR spectrum of CNC composite posses all bands similar to that of pure g-C_3N_4. Notably after doping activated carbon, a new band emerges at 1095 cm^{-1}, which likely originates from the coupling between C-N stretching vibration of g-C_3N_4 and the C-C stretching vibration of activated carbon and also the peak at 3263 cm^{-1}diminished due to the absorption of moisture on the surface of carbon.

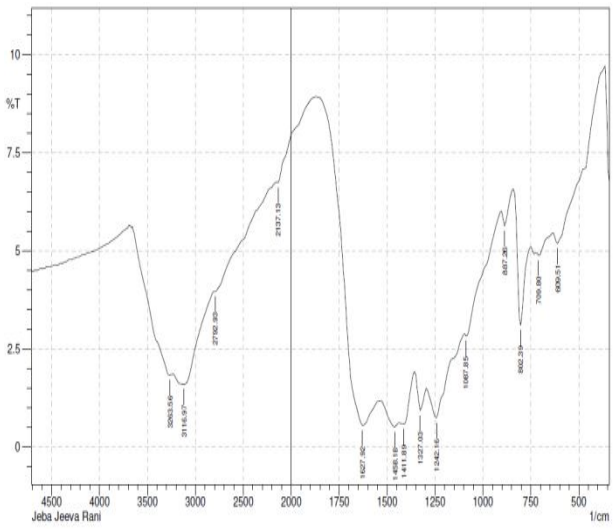


Fig. 3 FT-IR spectrum of CN Composite sample.

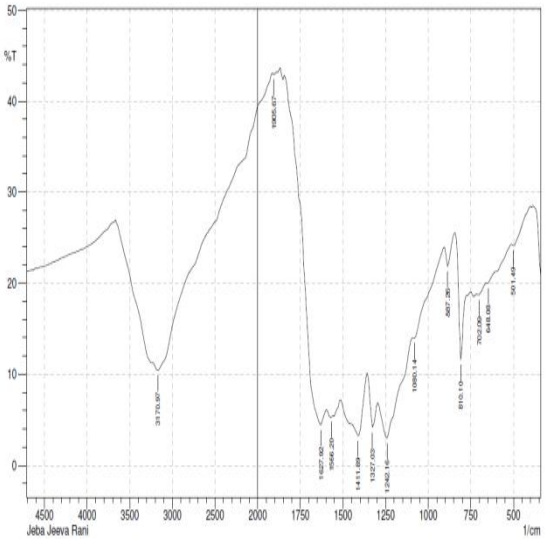


Fig. 4 FT-IR spectrum of CNC composite sample.

The morphology of pure g-C_3N_4 and CNC composite samples are investigated by SEM. Fig. 5 and Fig. 6 shows the SEM images of pure g-C_3N_4 and CNC. Both the samples display aggregated morphologies. The pure g-C_3N_4 sample appears to have aggregated particles which contain many smaller crystals with the crystallite size of 16-81 nm.

After introducing activated carbon, the carbon particles deposits on the surface of g-C_3N_4 and get the tubular structure with the particle size of 10.03 nm. However, the CNC surface morphology observed in Fig.6 exhibits many acicular and small granular structures.

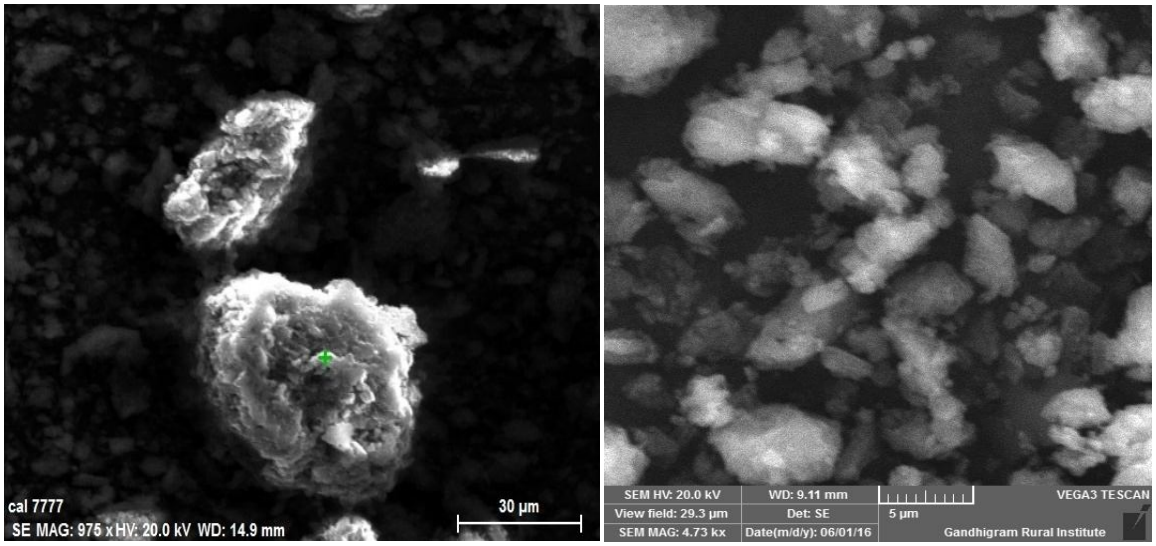


Fig. 5SEM micrographs of CN composite sample.

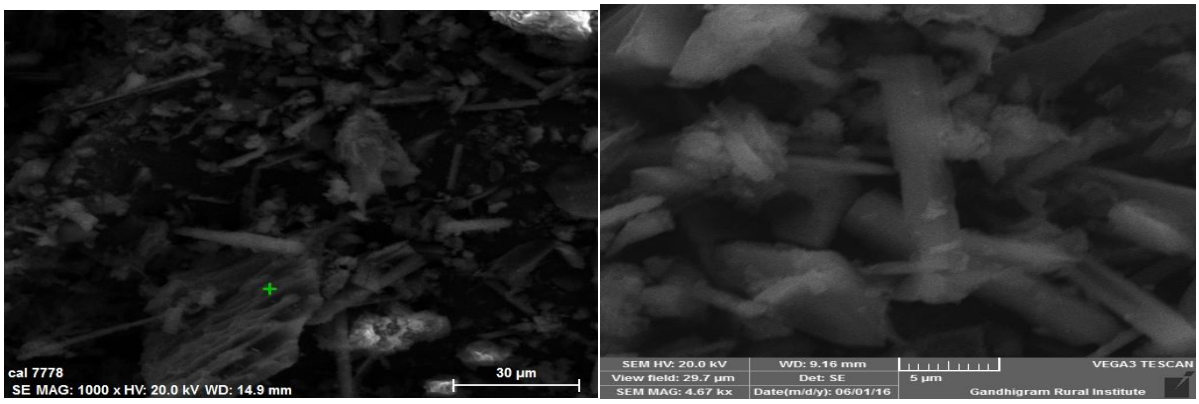


Fig. 6SEM micrographs of CNC composite sample.

The chemical stoichiometry of pure g-C_3N_4 and CNC composite samples are investigated with energy dispersive X-ray spectrometry analysis. It depicts as in Fig.7 and Fig.8, the results listed in Table 1 and 2. EDAX results confirmed the purity of the composite sample.

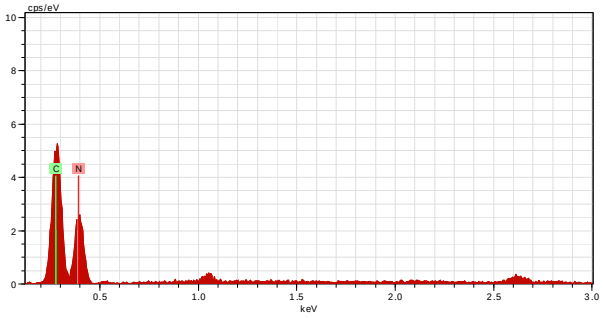


Fig. 7 EDAX spectrum of CN composite sample.

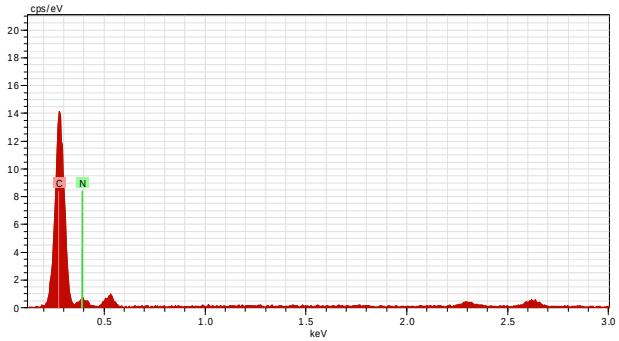


Fig. 8 EDAX spectrum of CNC composite sample.

Table -1Atomic and weight percentage of CN composite sample.

El AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at. %]	Error(1 Sigma) [wt.%]
N 7	K-series	58.27	58.27	56.55	17.98
C 6	K-series	41.73	41.73	43.45	8.69
	Total:	100.00	100.00	100.00	

Table -2Atomic and weight percentage of CNC composite sample.

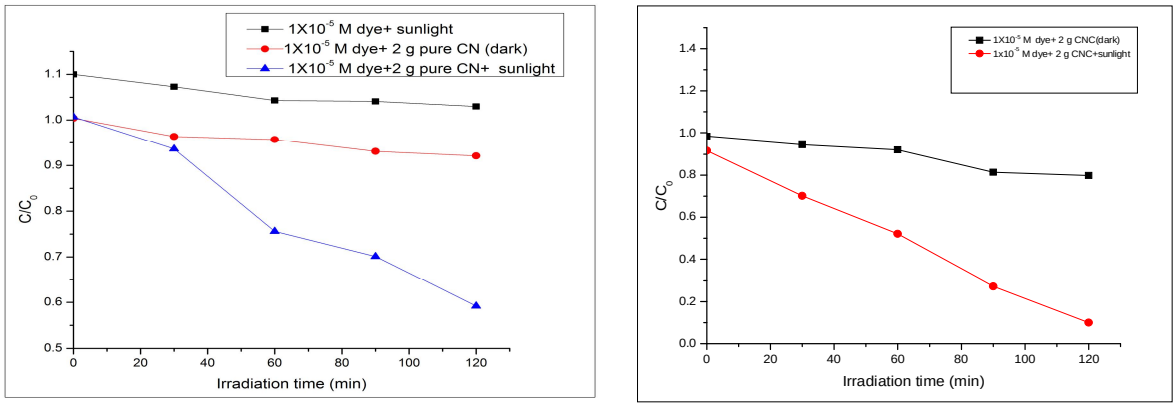
El AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at. %]	Error(1 Sigma) [wt.%]
C 6	K-series	68.34	68.34	71.57	9.73
N 7	K-series	31.66	31.66	28.43	9.07
	Total:	100.00	100.00	100.00	

B. Photocatalytic activities

The photocatalytic activities of pure g-C_3N_4 and CNC composite samples are investigated by choosing the photodegradation of Rh-B dye in aqueous solution under visible light irradiation ($\lambda > 420$ nm) at room temperature. The target molecule Rh-B is relatively stable in aqueous solution upon visible light irradiation. The characteristic absorption of Rh-B at $\lambda = 553$ nm is employed to

monitor the photocatalytic degradation process. Fig. 9 (a) & (b) shows the photocatalytic activities of pure g-C_3N_4 and CNC composite samples under visible light irradiation.

As a comparison, Rh-B photo degradation without the photocatalyst also performed and the results demonstrated that the degradation of Rh-B is very slow in the absence of the photocatalyst under visible light irradiation. Moreover, only 40.5 % Rh-B can be photo degraded by pure g-C_3N_4 under visible light in 120 min. However, significantly enhanced photocatalytic performance is observed in the CNC composite sample it shows the highest activity with Rh-B degradation rate of 89 %. This enhancement of photocatalytic performance can be attributed to the synergistic effect between non-metal carbon and g-C_3N_4, which has an important role in the separation of electron-hole pairs.



(a) (b)

Fig. 9Effect of incident light on the degradation of Rh-B over (a) CN (b) CNCcomposite samples.

The rates and efficiencies of photo assisted degradation of organic dyes are improved in the presence of oxygen. The effect of degradation of Rh-B dye in the presence of pure g-C_3N_4 and molecular oxygen is as shown in the Fig.10. From the graph it is observed that 45 % of dye degradation is achieved in the presence of oxygen bubbling at pH 7.6 within 120 minutes irradiation, whereas in the absence of molecular oxygen the degradation is about only 40 %. Fig.11 illustrates the degradation of Rh-B dye in the presence of CNC with and without oxygen bubbling at pH 7.6. It is observed that 94 % degradation is achieved in the presence of oxygen whereas only 89 % dye degradation is obtained without oxygen bubbling. The effect of molecular oxygen is an efficient conduction band electron trap, suppressing electron hole recombination because the conduction band of photocatalyst is nearly isoenergetic with the reduction potential of oxygen in inert solvents. So the absorbed oxygen enhances the photocatalytic activity [10].

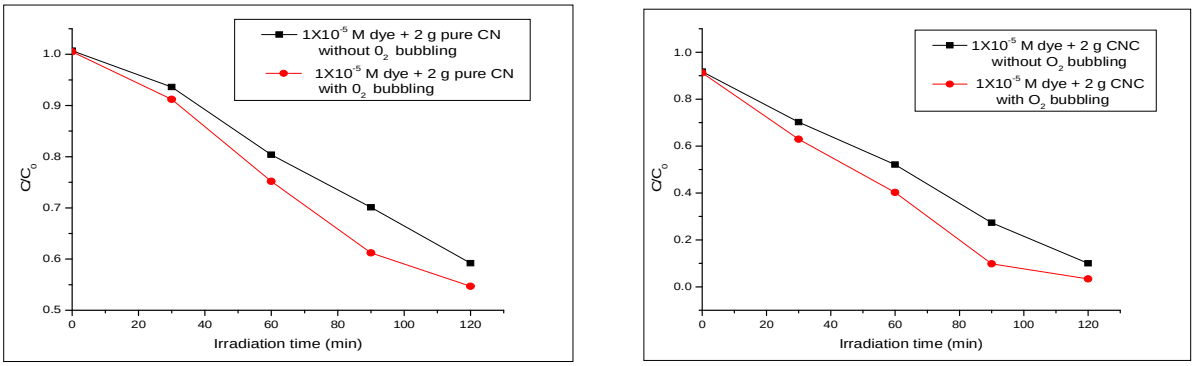
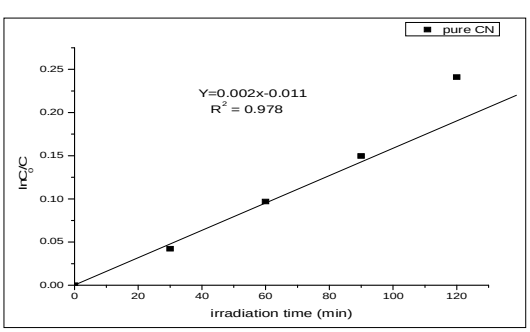


Fig. 10 and Fig. 11 Effect of O_2 bubbling on photo degradation of Rh-B over CN and CNC composite photocatalyst.

The experimental datas are fitted by applying a first order model in order to quantitatively investigate the reaction kinetics of the Rh-B degradation. Fig.12 and Fig.13 displays the pseudo first order plots for the photo degradation of Rh-B over pure g-C_3N_4 and CNC composite photocatalyst. The plot of the irradiation time (t) against ln(C_0/C) is nearly a straight line. The reaction constant *k*

can be used to evaluate the degradation rate. The CNC sample exhibits the highest photo degrading rate (k) of 0.018466 min^{-1}, which is about 4.1 times higher activity than that of pure g-C_3N_4. Therefore CNC catalyst shows excellent photocatalytic activity.



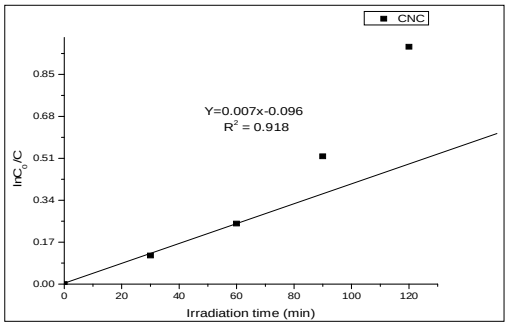


Fig. 12 and Fig. 13 First-order plots for the photo degradation of Rh-B using CN and CNC composite photocatalyst.

Each cycle, it is dried and reused for other cycles. Fig.14 illustrates the cyclic runs of Rh-B degradation using CNC photocatalysts. The Rh-B degradation rate slightly decreases after being irradiated for 360 min, which indicates sufficient stability of the CNC composite photocatalyst for Rh-B degradation.

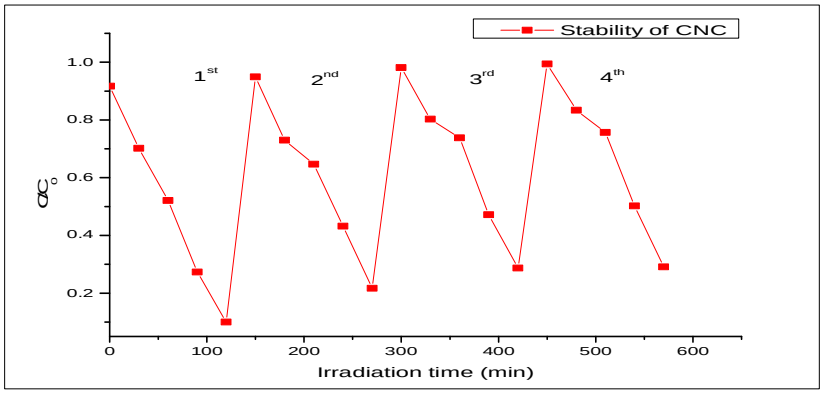


Fig. 14Recycling runs in the photodegradation of Rh-B using CNC composite photocatalyst.

C. COD analysis

To determine the organic strength of Rh-B dye, the COD of the dye solution before and after the treatment of pure g-C_3N_4 and CNC composites are estimated. For pure g-C_3N_4 the initial COD concentration of the Rh-B solution is 29.9 mg/l and after 2 h of visible light irradiation the COD concentration decreases to 19.9 mg/l. The COD value of Rh-B using CNC composite is found to be 5.8 mg/l after 120 min irradiation. The reduction in COD value of the treated dye solution indicates the mineralization of Rh-B dye molecule.

IV. CONCLUSION

Novel carbon-g-C_3N_4 composite photocatalyst is successfully prepared and characterized using XRD, FT-IR, SEM, EDAX and UV-Visible spectroscopy. The absorbance band intensity of CNC composite is found to be stronger than that of pure g-C_3N_4. Significantly, the photocatalytic activity of CNC under visible light irradiation increases 4.1 times for photo degradation of Rh-B under visible light irradiation in comparison to pure g-C_3N_4. It is indicated that the efficient photo generated electron-hole transfer and separation led to the improvement of photocatalytic performance. The effectiveness and activity of the catalyst over four cycles shows that the CNC composite photocatalyst can be recycled. The COD result shows that the large conjugated chromophore structure of Rh-B is destroyed under visible light, yielding smaller organic molecules which are non-toxic to the environment.

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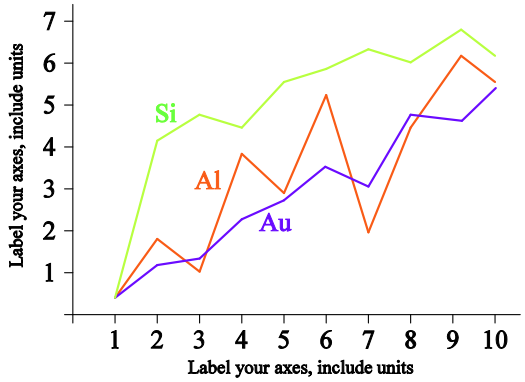

Fig.1 A sample line graph using colors which contrast well both on screen and on a black-and-white hardcopy

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REFERENCES

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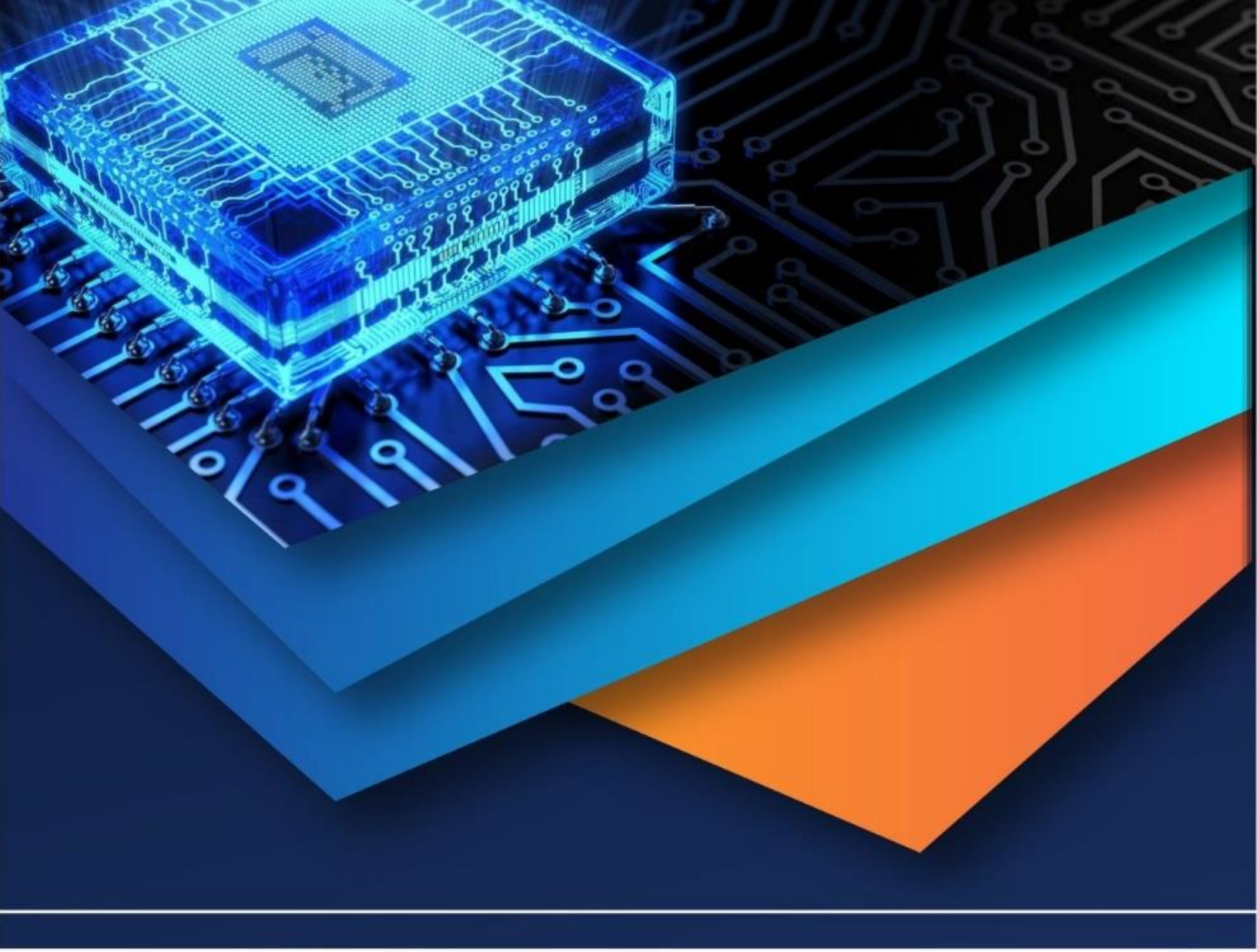

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