

Photocatalytic Degradation of MB Using Sn-Doped Graphitic Carbon Nitride Assisted by Graphene Oxide

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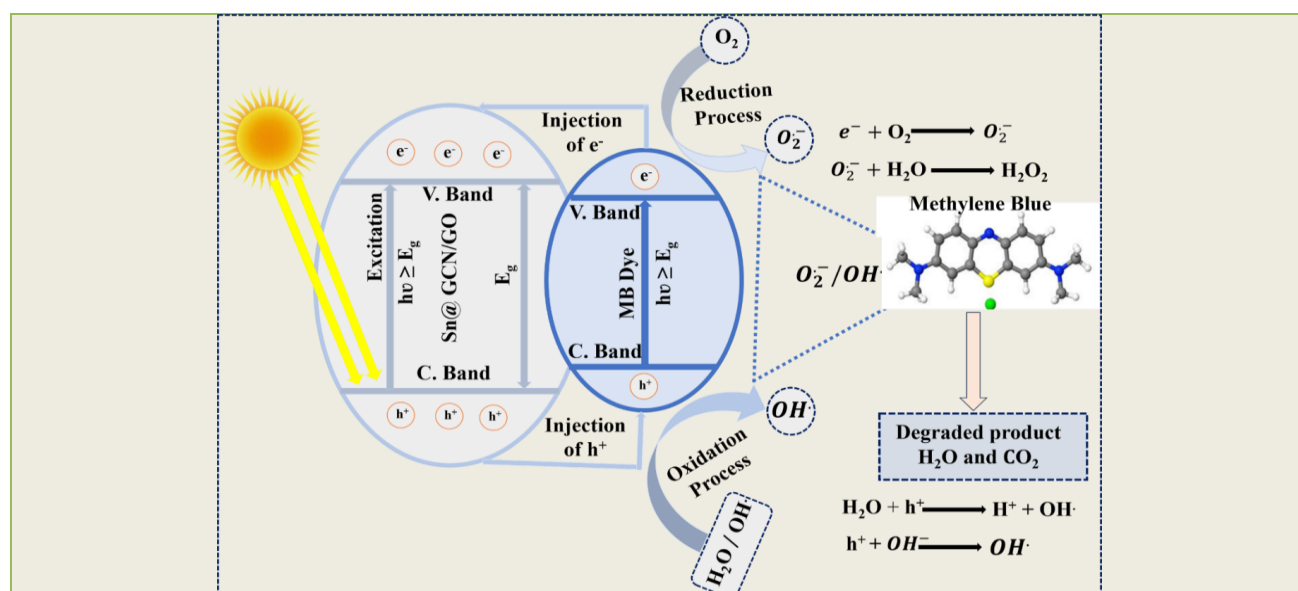
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Abstract

Review Article



The increasing contamination of aquatic systems by synthetic dyes like MB demands sustainable, efficient, green and promising remediation approach, like photocatalysis. These studies focus on engineering a novel photo catalyst Sn-doped g-C₃N₄/GO by using the hydrothermal method, aiming to enhance catalytic efficiency under natural sunlight. The morphology, optical properties, elemental composition, particle size, band gap, and functional group detection were done by using spectral techniques like FTIR, SEM/EDX, UV Visible spectroscopy and XRD. The lower angle peak shift in XRD confirms the doping of Sn⁺² in g-C₃N₄. The maximum degradation of MB was about 94% at 6 pH after 90 min under visible light as compared to Sn-doped g-C₃N₄ was about 70% degradation efficiency increased by supporting graphene oxide. The statistical analysis for the interaction of different parameters was performed by response surface methodology.

Keywords: Graphitic carbon nitride; Graphene oxide; g-C₃N₄/GO composites; Photocatalytic degradation; Methylene blue.

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1. INTRODUCTION

The prime natural resources fulfilling our needs are the precious asset [1]. It is the essential element and

component in the beginning, continuation, and evolution of life on Earth because of its special and extremely important qualities. Since water is present everywhere,

including in living things, it is essential to studying ecology and biodiversity, protecting the environment, and comprehending the cosmos [2]. Despite its abundance, less than 1% of the water on Earth can be used to suit human needs. Water may seem bountiful, but only under 1% of the Earth's water can be used to meet human demands. We must act to preserve and utilize every drop of water.

Water pollution is the main issue of the present time especially water effluent from the industry has many organic dyes that are harmful not only to humans as well as plants and aquatic animals[3]. To color the fabrics in the textile industry different dyes are used, these dyes have adverse effects such as they are carcinogenic in nature[4], mutagenic[5], causing irritation in eyes and skin, and also harmful to aquatic animals as these dyes are not degraded by the natural process as dyes are persisted in nature[6]. This wastewater needs to treat before flushing into fresh water[7].

Wastewater treatment is a critical problem that creates serious health issues if it is not treated properly[8]. Conventional methods are being used for wastewater treatment that may be physical, biological, or chemical[9]. The conventional techniques include[10] coagulation, osmosis, membrane separation ion exchange, flotation, and adsorption. The conventional methods are useful for small scale but have a high cost. Although, the efficiency for degradation of dye present in wastewater of textiles is less due to this reason conventional methods are less preferable [11]. Innovative advancements in Nanomaterials increase the use of nanotechnology for wastewater treatment[12]. Currently, the photocatalysis method is used due to its simple operation, non-selectivity, complete degradation capability, energy-saving, and environmental friendliness[13].

Titania has high efficiency for dye removal, inexpensive, nontoxicity, oxidizing capacity, and photostability[14]. The main issue is that Titania has 3.2eV band gap [15] therefore degradation of dyes needs UV light[16]. As the solar spectrum is consist of 46% visible light and just 4% ultraviolet light there is a need of a material that could harness the visible light[17]. The use of graphitic carbon nitride in wastewater treatment introduces a new way to study the g-C₃N₄ photocatalytic activity as it has low cost, green photocatalyst, easy synthesis method high chemical stability, two-dimensional nanostructure structure bandgap band gap .2.7eV[18]. The pure form of g- C₃N₄ had its defects like low specific surface area less absorption and more recombination. Metal doping can overcome these defects, among all metals doping of tin was more satisfactory as tin has low-cost precursors such as SnCl₂.2H₂O. By doping of GCN with tin altered the structure, microstructure and optical ability of GCN[19]. It decreased the band gap and increase the surface area

of GCN because of this reason maximum absorption in visible light occurred, by reducing the recombination of electron and hole pairs[20]. The removal rate of methylene blue also increased by using tin doping as compared to pristine GCN

The degradation efficiency of tin doped graphitic carbon nitride improved by using a support like GO. GO used as support because of its extraordinary ability like 2D structure, more chemical activity, high electrical conductivity, more ability of charge separation, high electron mobility, and strong [21]. Graphene oxide acts as a good support and increase removal of MB as it was studied in literature that Nano TiO₂ combination with GO increased the advancements in photocatalysis[22]. As graphene oxide was used as support in nano TiO₂ efficiency of degradation of methyl blue increased up to 93% which was 20% more than simple TiO₂[23].

As inspired from above consideration and reported literature on Sn doping, herein we report the detail study of tin doped GCN by using graphene oxide as support. The hydrothermal method was selected for synthesize the Sn@ GCN/GO and the precursor of tin was SnCl₂.2H₂O. The prepared composite Sn doped graphitic carbon nitride supported by graphene oxide was characterized by using techniques like FTIR, SEM/EDX, XRD, and UV-Visible Spectroscopy detects the functional group, the structure of crystalline material and morphology, elemental identification of the compound, and optical properties respectively [24]. The removal of MB was checked at different degradation parameters such as time, oxidant dose, catalyst dose, IDC and pH. The degradation potential of Sn@g-C₃N₄ was more than pristine graphitic carbon nitride and efficiency was again increased up to 94% by using graphene oxide as support.

2. EXPERIMENTAL PROCEDURE

2.1 Chemicals

Sodium nitrate (NaNO₃, 98%) and melamine (99%) acquired from DAEJUNG sodium hydroxide (NaOH pellets, 98%), (KMnO₄ > 99%), (H₂SO₄, 98%), (HCL, 35% w/w), (H₂O₂; 35% w/w) were taken from Sigma Aldrich and graphite powder (99%) was purchased from Scharlau. From Fischer scientific company methylene blue (92%) was taken. Tin chloride dehydrates (SnCl₂.2H₂O, 99%) was perched from GLENTHAM. Analytical graded chemicals were used and no more purification was done. Distilled water utilized throughout the all the reactions taken during research.

2.2 Synthesis of graphene oxide

In a conical flask specific amount of sodium nitrate and graphite powder was mixed with sulphuric acid. In a water bath with constant stirring the mixture of reaction was positioned. Under the ice bath by adding the potassium permanganate pinch by pinch into the reaction

after two hours. Making a paraffin oil bath and hot plate to maintain the temperature of the mixture. With the help of sucker with constant stirring the water was added. After that hydrogen peroxide was added with constant stirring and stirring was continued for three days until the dark chocolate color was produced. To maintain pH water used the mixture was dried in an oven. The sheet of graphene oxide was prepared [25].

2.3 Preparation of GCN

In the furnace condensation of melamine was used to prepare the GCN. Under the vacuum condition 5g of melamine was put in the crucible at 550°C for three hours. After that yellow colored final product was obtained [26].

2.4 Preparation of GO/g-C₃N₄ composites

The graphene oxide sheet was sonicated till uniform solution obtained. Similarly, the fine powder of graphitic carbon nitride was sonicated. After that the sonicated solution of GO and GCN was stirred for one hour separately then the solution of GCN and GO was mixed and again stirred to get uniform mixture. The mixture autoclaved for whole night at 120°C. The solution is filtered and washed. The final mixture was

poured into dish and dried in oven at 60°C. Finally the composites of GCN/GO was grinded [27].

2.5 Synthesis of 3% Sn@ GCN

The calculated amount of GCN was sonicated in 100ml of water for 30min. After that the sonicated mixture of GCN was stirred for one hour. The 1.05g of SnCl₂.2H₂O was mixed in 10ml of distilled water and added in mixture stirring was done. The solution was autoclaved at 180°C overnight. After that solution was filtered and dried at 60°C overnight in oven. Finally the 3% Sn doped graphitic carbon nitride was synthesized and grinded to get fine powder [28].

2.6 Synthesis of 3% Sn doped GCN/GO

1g of graphitic carbon nitride was sonicated and graphene oxide was also sonicated in 50ml of distilled water. Both solutions was stirred. After that the mixture was mixed and stirred for 30min then SnCl₂.2H₂O was mixed in 10ml of distilled water and poured to the above solution and again stirred for one hour. The mixture was autoclaved at 180°C overnight and then filtered and washed. The filtered solution was dried at 60°C for one night. The Sn doped GCN/GO grinded in the pastel mortar to get a fine powder [29].

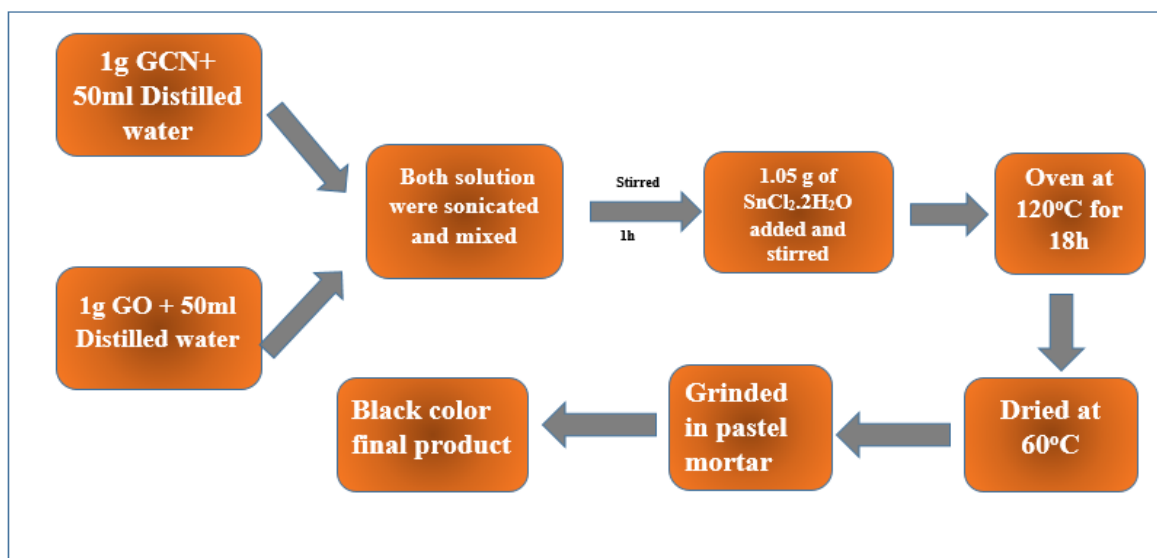


Fig. 1: The systematic diagram for the synthesis of 3% Sn doped GCN/GO

3. Photocatalytic activity assessment

The rate at which MB broke down in an aquatic solution when exposed to sun radiation was used to assess the photocatalytic activity (PCA) of the produced nano catalysts. For every experiment, a 200 mL glass beaker containing 60 mL of stock solution and 10 mg of nano catalyst was used to form an aqueous reaction suspension. Prior to sun irradiation, the suspension was agitated for ten minutes to bring the dye particles and photocatalyst surface into balance. Five milliliters of the decolorized solution were extracted at ten-minute intervals and utilized for UV-vis spectroscopy to

measure the breakdown of MB, as indicated by the decrease in absorption intensity at $\lambda_{\text{max}} = 665$ nm. To extract the photocatalyst for a subsequent test, centrifuge the mixture for five minutes at 4000 rpm. The following expression was used to assess the photoactivity efficiency [30].

$$\text{Degredation efficienncy (\%)} = \left(\frac{C_0 - C}{C_0} \right) \times 100\% \quad (1)$$

Where C denotes the final absorbance (at time = t) and C₀ denotes the beginning absorbance (at t = 0) [31-33]. To evaluate the reusability of NPs, the degraded

pollutant solution was treated by centrifuge at 4000 rpm for 12 min to recover the induced catalyst. Recovered NPs were dried in a furnace at 80 °C (3h) to be reused as photocatalyst for additional MB disintegration tests. Four experimental cycles were additionally conducted to assess the reusability of the catalysts. After each test cycle, the photocatalyst was subjected to centrifugation, heating, and then employed for the subsequent photoactivity.

4. The reaction's photochemistry

Figure 1 illustrates the semiconductor electronic band structure and displays the PCA of the photocatalysts used in this study. Here, solar radiation from sunshine causes an electron (e⁻) to move from the valence band (VB) to the conduction band (CB), which is separated by the high energy band gap. By leaving a hole (h⁺), this transition causes e⁻ to stay in the higher state, signifying an e⁻ deficiency. The production of excites (e⁻ and h⁺) causes the synthesized molecule to go through an oxidation-reduction reaction, which is then absorbed by the photocatalyst's surface. Only semiconductors can use this technique; in the case of insulators, photon absorption takes place through the equivalent bulk material. Photocatalysts with electronic band structures are exempt from this limitation. For example, the single chemical product can be used as a photocatalyst in solid form, but it does not enable the band structure described above. A further reaction may occur at specific localized areas of the photocatalyst when the bulk material is used for photo absorption [34-36].

5. Reaction kinetics

The photoexcited e⁻s and h⁺s trigger a redox reaction that ultimately controls the reaction kinetics. The method used determines the exciton recombination kinetics. For example, the first-order rate equation in Eq. 2 can be used to represent the process of excitation and exciton recombination.

$$-\frac{dA}{dt} = k(A) \quad (2)$$

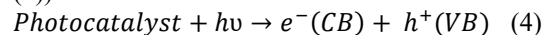
The time rate equation of second order is valid if many excitons are produced concurrently within the photocatalyst, as shown by equation (3).

$$-\frac{dA}{dt} = k(A)^2 \quad (3)$$

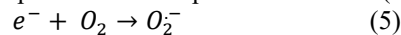
The rate constants (k-recombination and k-redox), which are the same as "e⁻" and "h⁺" and their recombination, also influence the total rate of reaction in the reaction cycle. However, as the process produces no chemical products, there is no method to calculate k (recombination).[35].

Regarding the mechanism, the nano catalyst is triggered by photons (E = h) with energy equal to or

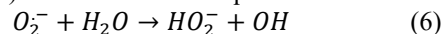
greater than a parallel band gap of the nano catalyst (eq (4)).



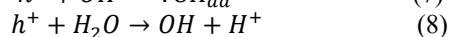
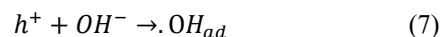
O₂, which was initially the photocatalyst's surface absorbent, may freely absorb the resulting e⁻, leading to the production of superoxide radicals (O₂⁻).



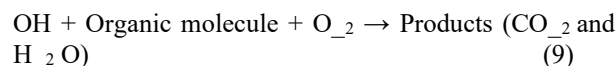
Moreover, hydroxyl radicals (OH•) and hydroperoxyl radicals (H₂O•), which act as oxidizing agents to degrade organic contaminants, are produced when O₂⁻ interacts with the equivalent H₂O.



The hydroxyl (OH⁻) groups on the surface of NPs may quickly interact with the h⁺s generated by irradiation to form OH•.



The organic molecules eventually undergo oxidation to provide CO₂ and H₂O, as shown below:



A little degree of exciton recombination occurs momentarily, hence photocatalysis of the nano catalyst may be temporary [33, 37].

6. Characterization

The prepared composite Sn doped graphitic carbon nitride supported by graphene oxide was characterized by different techniques like FTIR, XRD, SEM/EDX, and UV-Visible Spectroscopy detects the functional group, the structure of crystalline material and morphology, elemental identification of the compound, and optical properties respectively[24].

6.1 UV-Visible spectroscopy of the composite

UV-Vis DRS was used to show the opto-electronic characteristics of pristine GCN and GO/GCN nanocomposites. At 454 nm, a significant absorption of g-C₃N₄ was found, suggesting a 2.7 eV energy gap.

The absorption wavelength of GO/ GCN nanocomposites was somewhat red shifted in comparison to bulk g-C₃N₄. The energy gap has a value of 2.67 eV. And the bandgap of 3% GCN was about 2.4eV that showed more absorption in visible region due to doping of the Sn to the graphitic carbon nitride. By using the graphene oxide as support the bandgap more decreased to the 2.25eV. The combined results showed improved catalytic activity and good absorption in visible region [38]. The figure of the spectrum is given below:

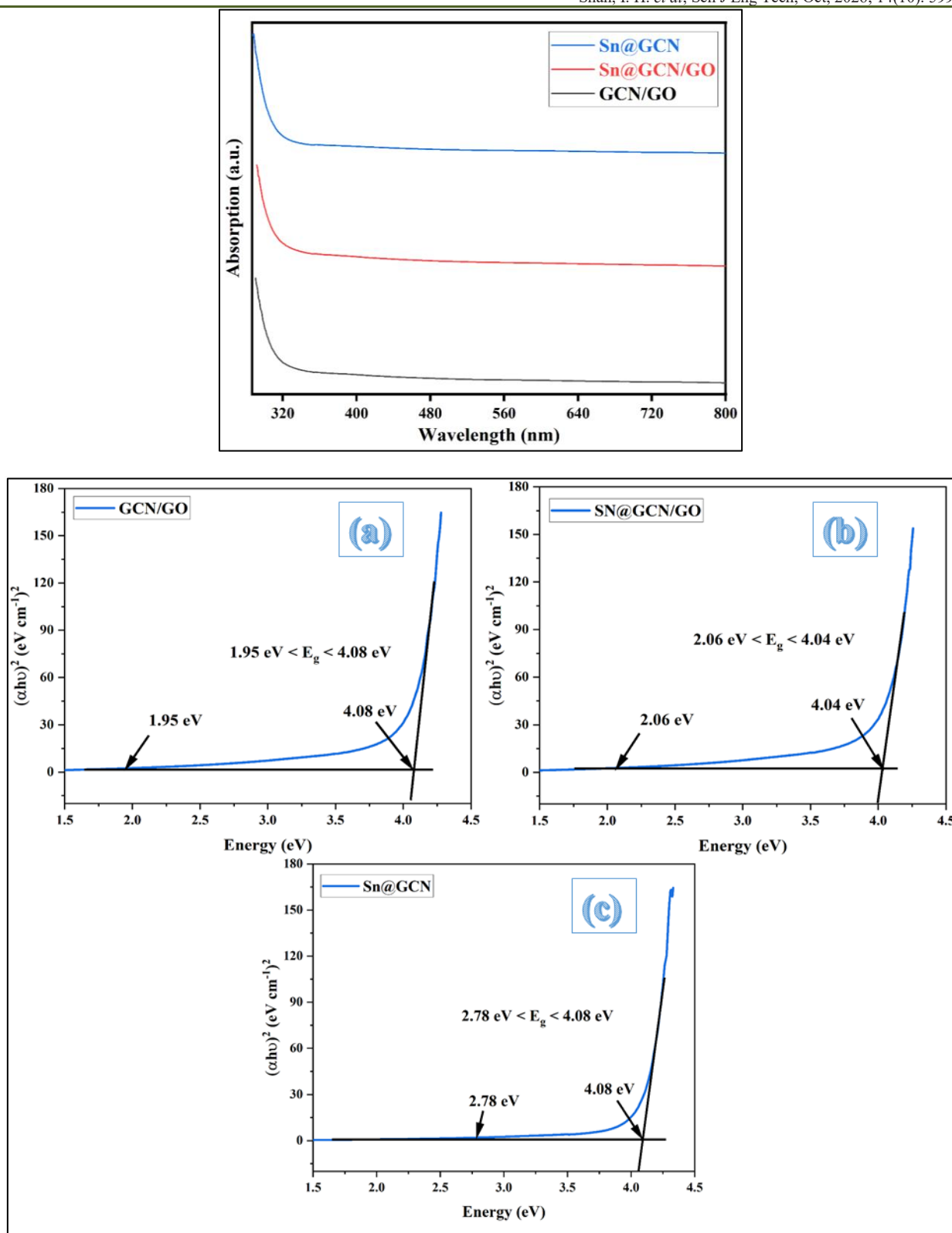


Fig 2: UV- VIS Spectra of the GCN/GO (a), 3%Sn@ GCN/GO (b) and 3%Sn@ GCN(c).

6.2 XRD characterization

The XRD of GCN/GO, Sn@GCN and Sn@ GCN/GO

At 10.64° which corresponds to an interlayer spacing (d) of 0.817 nm with indices, a diffraction peak of GO was seen (001). In comparison to pure graphite, which emerges at $2\theta = 26^\circ$, a prominent peak of GO

occurred at $2\theta = 11.06^\circ$. At 26.3° , a further broad diffraction peak was seen, which corresponded to an interlayer spacing of approximately 0.34 nm and an index of (002). In the case of dried GO, the primary peak (002) of graphite is completely gone. The graphene with decreased layers is shown by this peak, which was wider

and much less intense than natural graphite [39]. The XRD spectra of combined nanosheets of g-C₃N₄ and GO was at 11.9° and 27.7°, respectively and the (002) peaks of GO and g-C₃N₄ respectively. The GO sheets may have completely exfoliated in the mixed suspension, according to this indication.[40]. The phase and structure development of the GCN, GO, and GO/ g-C₃N₄ hybrid nanocomposites as they were manufactured were shown using p-XRD analysis. The synthetic GCN-p-XRD pattern's hexagonal form and the diffraction peaks at $2\theta = 13.1^\circ$ and 27.8° (0.683 nm and 0.326 nm), which represent the (100) and (002) reflection planes, respectively, are clearly visible. The (001) lattice planes were responsible for the peak of the GO p-XRD pattern at 11.2° . The successful synthesis of hybrid nanocomposites is demonstrated by the GO/g-C₃N₄ p-XRD pattern, which demonstrates that the peaks pertain to pure GO and g-C₃N₄ and that any extra crystalline impurities can be detected. Using the Scherer equation and a reflection of the (002) orientation, the average crystallite size of pure GCN and GO/g-C₃N₄ hybrid

nanocomposites could be determined. The XRD of Sn@GCN shows the peaks of GCN at (002) reflection planes and angle were about 2θ 26.4 for Sn the angle 2θ 26.5, 37, 51.25, 63.1 and 79.7 peaks with (110), (101), (211), (103) and (100). The hexagonal structure and doping of tin do not alter the structure of graphitic carbon nitride, but they do increase the surface area of the tin by making it more porous and slightly shift the peak of the g-GCN to the left, according to the miller indices of Sn@GCN. Similarly, the novel composite Sn@GCN/GO shows the similar peaks for tin doping on GCN as discussed above with additional peak of graphene oxide that was used as support material for the enhancement of the photocatalytic activity of the Sn@GCN that was (001) and (100). The doping of the tin that shifted the peaks to the left side increased the visible light harvesting ability of Sn@GCN/GO. The spectrum of XRD peaks of Sn@GCN, GCN/GO, Sn@GCN/GO and combined XRD spectrum is showed in 1, 2, 3 and 4 in below diagram:

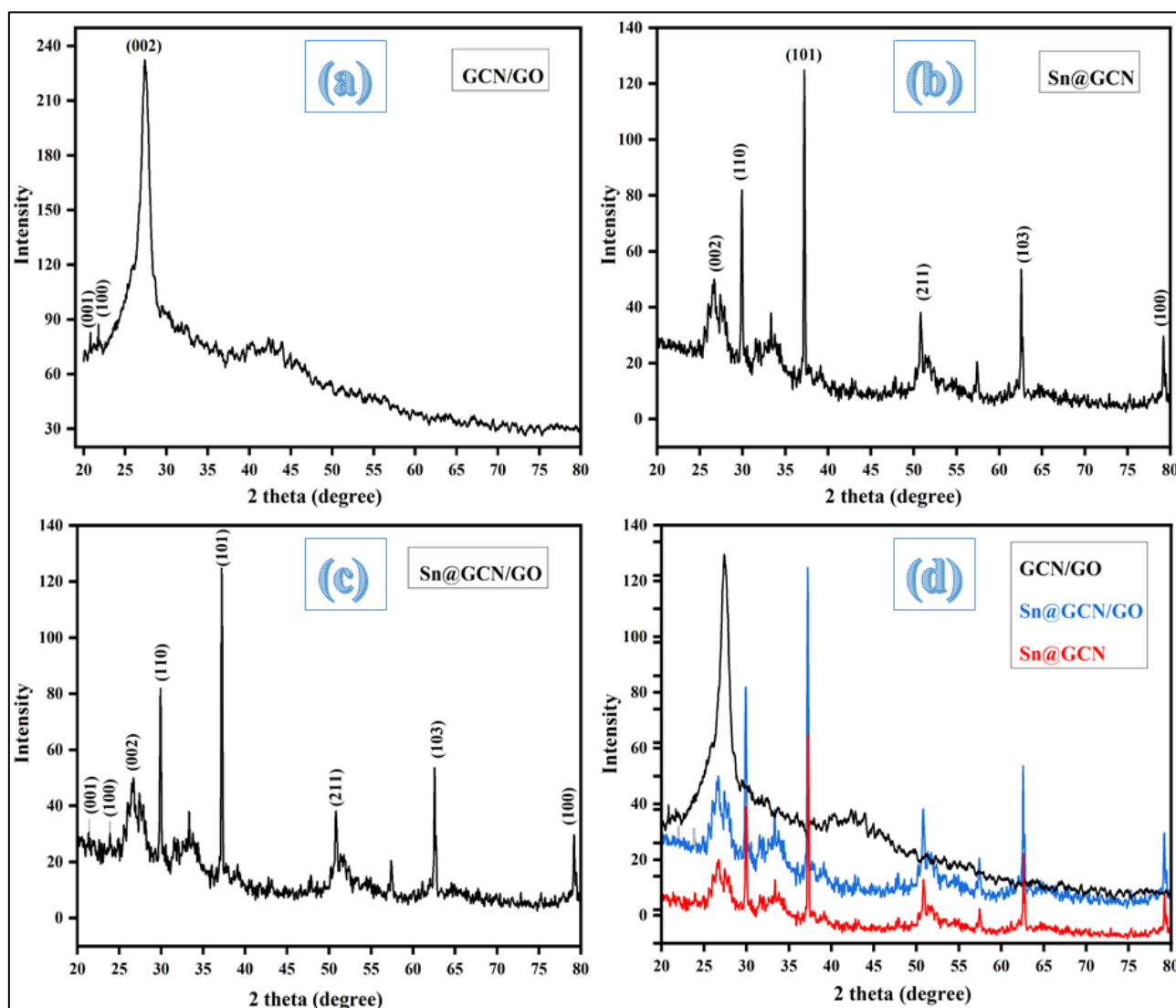


Fig. 3: The XRD analysis figure of GCN/GO, Sn@GCN and Sn@GCN/GO composites.

6.3 SEM/EDX of the composites

SEM with an EDX spectrum was used to characterize GO/ g-C₃N₄ hybrid nanocomposites and their forerunners in order to display the surface morphology. The as-synthesized GO is depicted in SEM images with a significant amount of sheet-like shape and a smooth surface. The structure of g-C₃N₄ was a multilayer, platelet-like aggregation. The GO/ g-C₃N₄ hybrid nanocomposites' SEM micrographs demonstrate that g-C₃N₄ was firmly adhered to the GO surface. The EDX analysis shows that C, N, and O atoms co-exist in the GO/ g-C₃N₄ hybrid nanocomposites with weight percentages of 39.86, 54.84, and 5.30 percent,

respectively. This shows the great purity of the materials. The structure of the Sn@ GCN was complex sheet like and doping of Sn clearly visible in scanning electron microscope like flower form with Weight percentage C, N, O and Sn was about 32.95, 43.15, 9.13 and 12.55 respectively A. The graphitic carbon nitride sheet, graphene oxide sheet, and flower-shaped tin doped material make up the ternary hybrid of Sn @GCN/GO, which has an aggregation-like structure. According to the EDX analysis, the weight percentages of C, N, O, and Sn are approximately 24.69, 4.09, 22.43, and 46.65, respectively.

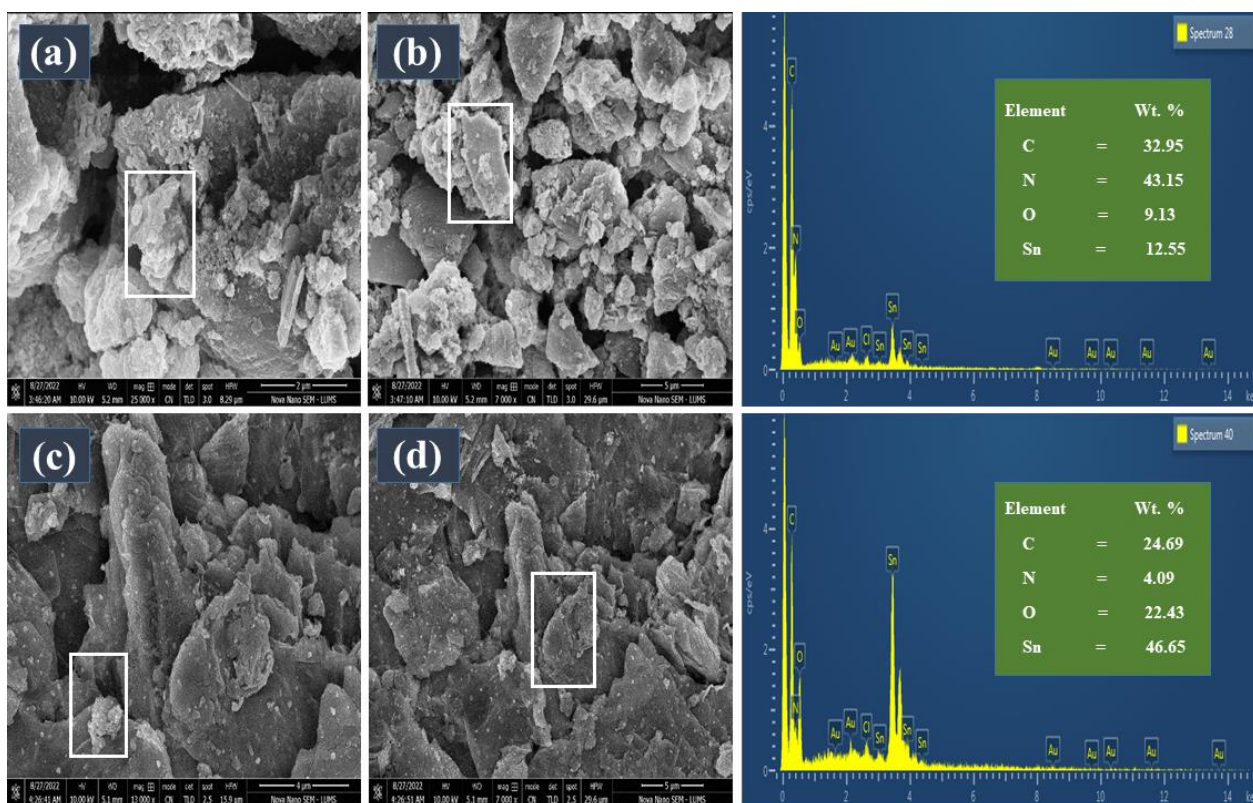


Fig. 4: (a& b) SEM images of bulk Sn@ GCN and its EDX and (c & d) exfoliated Sn@ g-C₃N₄/GO and its EDX spectrum

6.4 FTIR of Graphitic Carbon Nitride and 3% Sn doped GCN

The stretching vibration in N-H bonds is the cause of the broad peak band of absorption in pure graphitic carbon nitride, which is located between 3300 and 3000 cm⁻¹. This band is mostly caused by inadequate amino acid condensation. The stretching bond in the heterocyclic ring of C-N at 1700 cm⁻¹ to 1200 cm⁻¹. The strong peak at 1613 cm⁻¹ best represents the crystal structure and C=N bond existence of graphitic carbon nitride. The s-triazine modes at 801 cm⁻¹ and 1470 cm⁻¹ are shown by C-N at 1335 cm⁻¹. Peak shifting from 718 cm⁻¹ to 707 cm⁻¹ indicates that graphitic carbon nitride has been doped with tin.

[41].The FT-IR spectra of graphene oxide generated in these steps demonstrates that the graphite

has been successfully oxidized. The fact that there is oxygen functions of various types in graphene. Graphene oxide was found in a variety of places 3448 cm⁻¹ is the highest point. The stretching and water vibrations of the C-OH groups are caused by the O-H at 1561 cm⁻¹. The sharp at 1416 cm⁻¹ there is a strong peak of CO-carboxylic is responsible for this peak [42].

6.5 FTIR of GCN/GO composite and Sn- GCN/GO

The molecular structure data was provided along with a comparison of the FT-IR spectra of g-C₃N₄, GO, and GO/g-C₃N₄ hybrid nanocomposites. The typical C-N and C=N stretching modes of heterocyclic compounds are shown by absorption peaks for pure g-C₃N₄, which were seen between 1200 cm⁻¹ and 1650 cm⁻¹. The breathing mechanism of the tri-s-triazine units is also responsible for the prominent and abrupt peak at

808 cm⁻¹. The broad absorption peak at 3000-3600 cm⁻¹ is caused by the N-H surface adsorbed by H₂O molecules. The stretching vibrations of C-O, C-OH, and C=O are represented by the FT-IR peaks of the GO molecules at 1039 cm⁻¹, 1420 cm⁻¹, and 1728 cm⁻¹, respectively. As a result, the peak at 1627 cm⁻¹ represents the skeletal vibration of non-oxidized graphitic domains (C=C stretching vibration of epoxide

groups). As a result, the FTIR spectra, along with the positive XRD data, support the production of the GO/GCN hybrid nanocomposites. The existence of peak at 707cm⁻¹ shows the doping of tin on graphitic carbon nitride [43]. Diagrams E, F, G, and H below show the FTIR of the GCN/GO Sn@, GCN/GO, Sn@ GCN, and combined FTIR of Sn@ GCN/GO and GCN/GO, respectively.

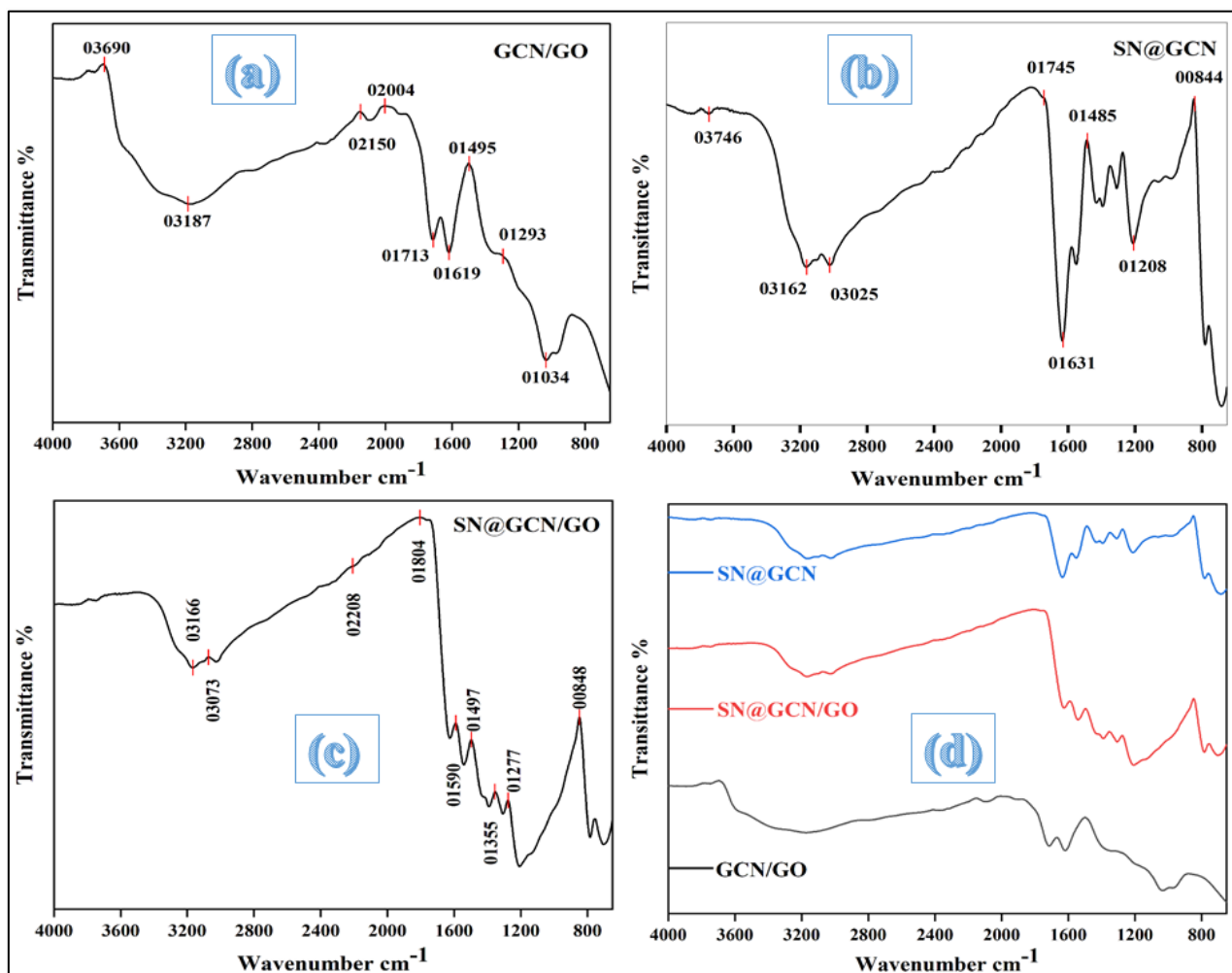


Fig. 5: FTIR of the GCN/GO (a) Sn@ GCN (b), Sn@ GCN/GO (c), and combined FTIR of Sn@ GCN, Sn@ GCN/GO and GCN/GO (d)

Optimizing different parameters that effects photocatalytic activity

The dye's degradation is influenced by a number of variables, including pH, time, catalyst dose, oxidant dose, and initial dye concentration. All these parameters is listed below that affect directly removal process of the MB.

Preparation of the dye solution

As methylene blue is a cationic dye and thus water soluble. By mixing 1g of methylene blue with 1L of distilled water, a stock solution containing 1000ppm of methylene blue was created. Farther dilution was done to get 100ppm solution and then desired dilution was obtained to perform farther experiment.

Calibration curve of methylene blue

A methylene blue solution was made at various ppm levels, including 2, 4, 6, 8, 10, 12, 14, and 16 ppm. The reference was distilled water. Using a visible spectrophotometer, the absorbance of every stock solution was measured at its highest wavelength. All the degradation parameters were done in 10ppm solution of methylene blue. The percentage removal was checked by this formula % degradation= $(1 - C_t/C_o) \times 100$

Effect of oxidant

The main role of oxidant in the degradation of the dye is to make hindrance for the combination of the holes and electron pairs by capturing the electron [44].

Different milli-molar solution of H_2O_2 was prepared [45] such as 5,10,15,20,25,30,35,40,45,50mM in 100ml of distilled water and then variation in dye degradation was noted and show is Fig.6a. The amount of oxidant at pH=6, 30mg catalyst dose in 100ml of methylene blue dye solution was about 94% shown by Sn@GCN/GO. Similarly at pH=7, 40mg/100ml catalyst dose and 20Mm the maximum degradation about 70% was shown by Sn@GCN and 51% dye removed by GCN/GO at pH=8 by using 50mg/ 100ml catalyst dose and 30mm of the oxidant dose.

Effect of catalyst

To remove MB, different amounts of catalyst were utilized. [46]. The catalyst amount mainly play vital role in degradation of dye [47]. The catalyst dose is different for degradation of dyes. The removal of the dye decreased after optimum condition [48] due to overlapping of particles on surface of catalyst [49]. The concentration of color, pH, and oxidant dosage were all held constant and different dose of catalyst 10, 20, 30, 40,50,60,70 and 80mg in 100ml of methylene blue solution [50]. The degradation of dye with all catalyst dose was noted after 30, 60 and 90min. The maximum degradation of the MB was noted by taking 10ppm initial dye concentration by using 30mg/100ml, 40mg/100ml and 50mg/100ml of Sn@ GCN/ GO, Sn@ GCN and GCN/GO illustrated in Fig.6b.

Effect of initial dye concentration

To assess the catalyst's effectiveness, the dye's concentration was recorded [7] The photon's path length shrank [51] as concentration of the dye increased [52] of the photocatalyst used for degradation of the dye was kept constant [53]. The maximum degradation of the methylene blue was noted for Sn@ GCN/GO, GCN/GO and Sn@ GCN at 10ppm

Effect of the time

For initiating any photocatalytic degradation light plays important role as it provides optimum energy for degradation [54]. By fixing all parameters such as time, oxidant dose, catalyst dose [55] and initial dye concentration was checked [56]. As the time increased removal efficiency was also increased the color of the dye was almost gone after 30 min but degradation was completed in 90minute maximum degradation was about 94%, 70% and 51% of Sn@ g-C₃N₄/GO, Sn@ g-C₃N₄ and GCN/GO respectively.

Effect of pH

The pH factor, which also influences the pace at which the dye in the wastewater decolorizes, is used to gauge the photocatalyst's activity[57]. As at higher pH and pH=7 the hydroxyl ion acts as key oxidation potential specie and at lower pH the holes works[58]. For measuring the point of zero charge [59] of catalyst the pH drift method was used[60]. It is a charge when the adsorbent surface charge is zero. 0.1M of NaCl was prepared and de-gas the solution by placing it on hot

plate. Next, 0.1M HCl and 0.1M NaOH were added to the NaCl solution to maintain the pH of 2, 4, 6, 8, and 10. Each beaker was filled with the calculated 75 mg of catalyst. The beakers were then put in a UV chamber for a full day while covered with aluminum foil. After that noted the pH of each beaker and draw the plot of initial and final pH[61]. The point interaction was the point zero of composite which was 5.1, 4.3 and 4.5 for Sn@ GCN/GO, GCN/GO and Sn@ GCN respectively. When $pH > pH_{PZC}$ negative charge is present on the surface of the catalyst[62] similarly when $pH < pH_{PZC}$ catalyst is positively charged[63]. The removal of MB was by varying the pH[64]. The maximum degradation of the methylene blue was noted at pH=6 about 94% for Sn@ GCN/GO, at pH about 51% for GCN/GO and pH = 7 about 70% for Sn@ GCN.

7. REUSABILITY

The heterogeneous photocatalyst in this work exhibits exceptional efficiency even after numerous cycles of reuse. Catalyst was used to check its variation in photo activity [65]. Same catalyst was used for five cycles and after washing and drying in oven at 60°C due to its thermal stability and strength of the catalyst it can sustain different extreme condition. Therefore, prepared tin doped graphitic carbon nitride/ graphene oxide was used to degrade the methylene blue for 5 times at 6 pH. 30mg catalyst dose, 10ppm dye concentration and 10mm oxidant dose after 90 min following degradation efficiency was noted as given below in fig part d.

8. Effect of radical trapping scavenger on degradation

Electrons, holes, hydroxyl radicals, and other reactive groups were used to break down methylene blue [66]. The experiment includes trapping radicals in order to identify the probable radical implicated in the breakdown of methylene blue [67] 5 milliliters from 10 mm scavenger solutions, such as ascorbic acid[68] for O₂, potassium dichromate for electron trapping, ethylenediamine tetraacetate for holes, and dimethylsulphoxide for OH. As DMSO and K₂Cr₂O₇ reduced the degradation of MB so it was included that hydroxyl ion and electrons are involved in degradation of dye. The diagram of the radical scavenger effects is given in C part of figure.

9. Kinetics

The kinetics of reaction tells us about order of the reaction [69] to find out that how many factors control the removal of MB [70]. The quantitative of reaction kinetics was done by using different models of kinetics [71]. The formula for first order [72] is given as $\ln C_t / C_o = kt$

And for second order kinetic the formula[73] is that $1/C_t - 1/C_o = kt$

As the value of R² is greater in first order reaction the kinetics of novel photocatalyst is first order.

The diagram given below show the graphical representation of the kinetics and calibration curve of MB in part A and B.

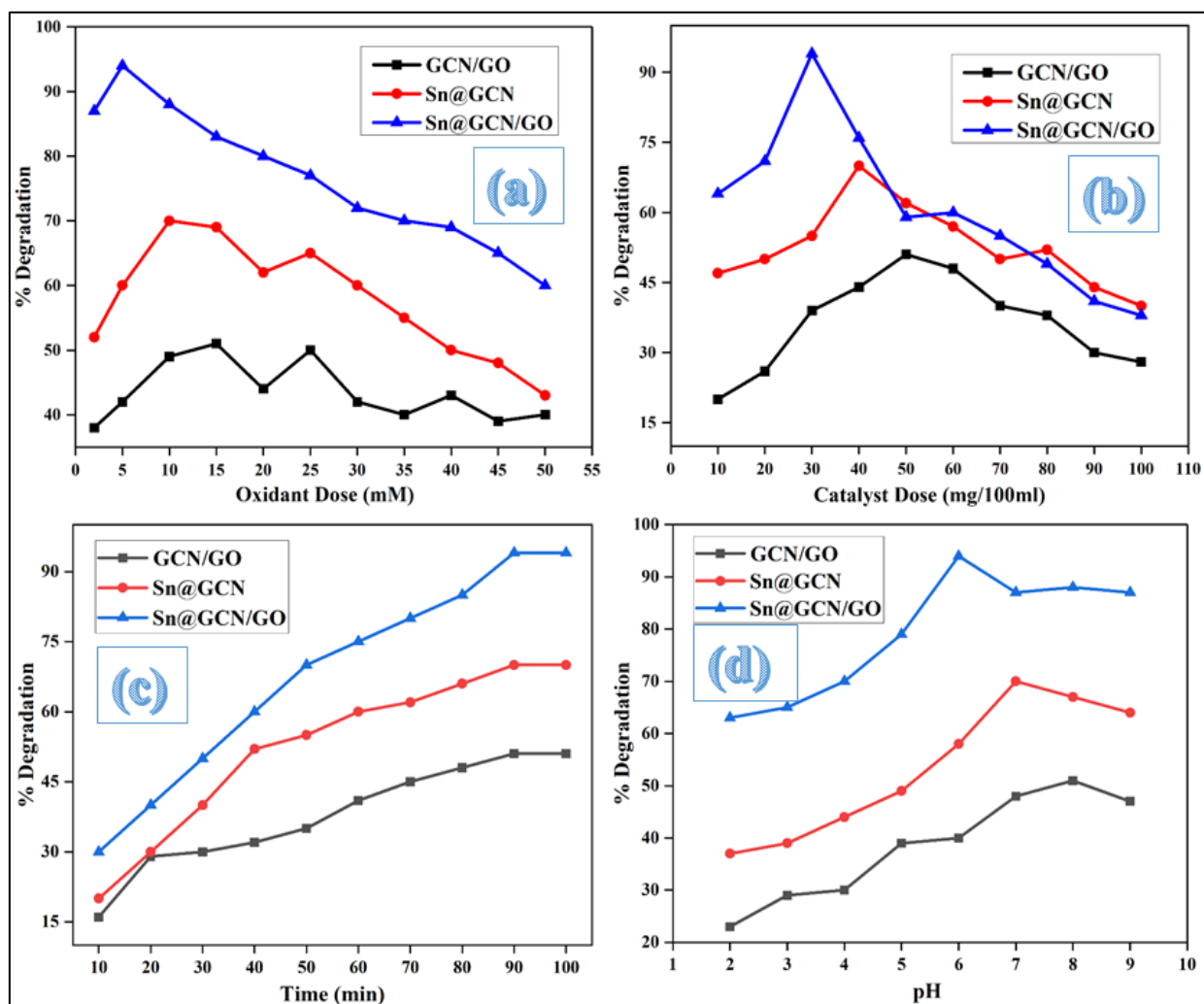


Fig. 6: The diagram of effects of oxidant dose (a), catalyst dose(b), time(c) and pH (d) on the composites of Sn@ GCN, GCN/GO, Sn@ GCN/GO

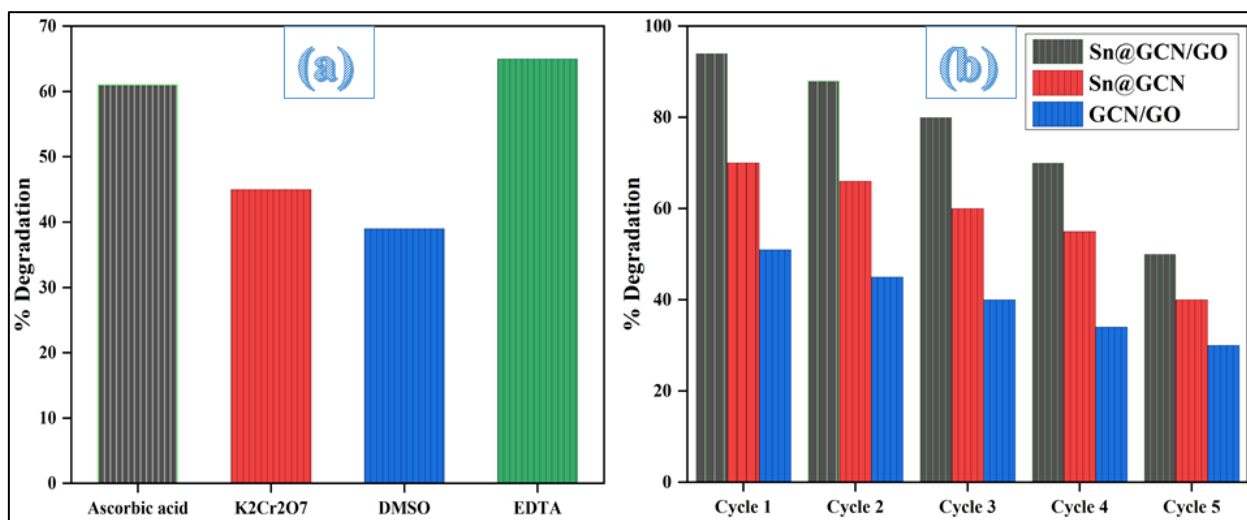


Fig. 7: The diagram of first order reaction of Sn@ GCN/GO (X), Calibration Curve of MB (Y), Scavengers effects on Sn@ GCN/GO (A) and Reusability (B).

Statistical analysis

Response surface methodology[74] was used to monitor the removal of MB [75]. The impact of specific parameters is tracked in the suggested study under the supervision of a design specialist on the elimination of MB dye. By controlling independent variables removal efficiency was noted [76]. The ternary composite Sn@GCN/GO for the removal of methylene blue was prepared by using hydrothermal method. Response surface technique study was conducted using a central composite design [77]. Thirty experiments was performed according to this model. The significance of the model obtained is high which is showed by analyzing the plot the fit summary[78]. The value of R^2 must be one[79]. This model of quadratic displayed association of the %degradation of the dye and selected variables. To influence the significant effect on the degradation effect, 3 factors catalyst dose, oxidant dose and pH were noted. For interpretation the three factors the regression model employed the 1st order polynomial equation. Analysis of variance was used to assess the model's efficacy. The

Model F-value of 53.01 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, BC, A^2 , B^2 , C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The Lack of Fit F-value of 1.38 implies the Lack of Fit is not significant relative to the pure error. There is a 36.76% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit. Fit statistics The Predicted R^2 of 0.8962 is in reasonable agreement with the Adjusted R^2 of 0.9610; i.e. the difference is less than 0.2. Coefficients in Terms of Coded Factors.

Analysis of variance ANOVA

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2719.81	9	302.20	53.01	< 0.0001	Significant
A-pH	886.29	1	886.29	155.47	< 0.0001	
B-Catalytic dose	43.36	1	43.36	7.61	0.0202	
C-Oxidant dose	138.27	1	138.27	24.26	0.0006	
AB	3.13	1	3.13	0.5482	0.4761	
AC	6.13	1	6.13	1.07	0.3244	
BC	45.22	1	45.22	7.93	0.0183	
A^2	1280.99	1	1280.99	224.71	< 0.0001	
B^2	151.20	1	151.20	26.52	0.0004	
C^2	386.97	1	386.97	67.88	< 0.0001	
Residual	57.01	10	5.70			
Lack of Fit	33.01	5	6.60	1.38	0.3676	not significant
Pure Error	24.00	5	4.80			
Cor Total	2776.82	19				

The terms for coded factors

Table 4.15: Coefficients in Terms of Coded Factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	95.09	1	0.9738	92.92	97.26	
A-pH	8.06	1	0.6461	6.62	9.50	1.0000
B-Catalytic dose	1.78	1	0.6461	0.3423	3.22	1.0000
C-Oxidant dose	3.18	1	0.6461	1.74	4.62	1.0000
AB	-0.6250	1	0.8441	-2.51	1.26	1.0000
AC	0.8750	1	0.8441	-1.01	2.76	1.0000
BC	-2.38	1	0.8441	-4.26	-0.4966	1.0000
A^2	-9.43	1	0.6289	-10.83	-8.03	1.02
B^2	-3.24	1	0.6289	-4.64	-1.84	1.02
C^2	-5.18	1	0.6289	-6.58	-3.78	1.02

Combined effect of catalyst dose and pH through RSM was evaluated. On the optimized condition catalyst dose 30mg, 10Mm oxidant dose at 6 pH maximum degradation of methylene blue was shown. Total surface area increased by increasing catalyst

amount but up to optimized condition after that degradation rate decreased. Increased the oxidant dose decreased the degradation due the OH radicals recombination[80].

Factor Coding: Actual

Degradation (%)

Design Points:

● Above Surface

○ Below Surface

54.08 97.05

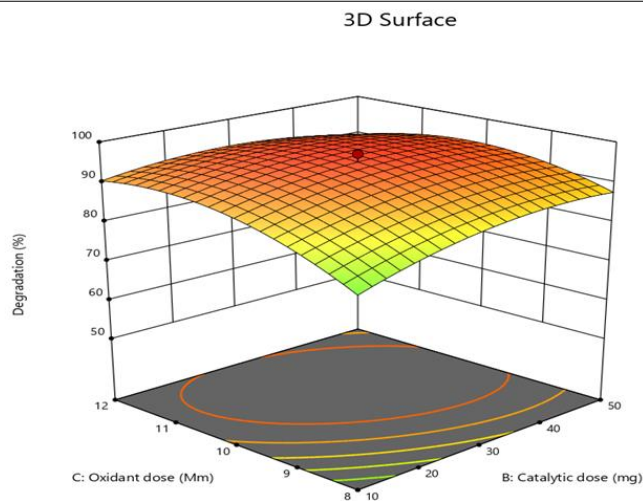
X1 = B

X2 = C

Actual Factor

A = 6

(a)



Factor Coding: Actual

Degradation (%)

Design Points:

● Above Surface

○ Below Surface

54.08 97.05

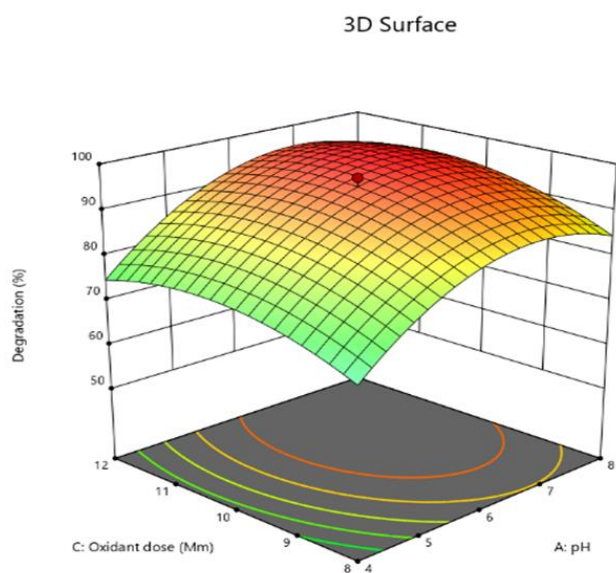
X1 = A

X2 = C

Actual Factor

B = 30

(b)



Factor Coding: Actual

Degradation (%)

Design Points:

● Above Surface

○ Below Surface

54.08 97.05

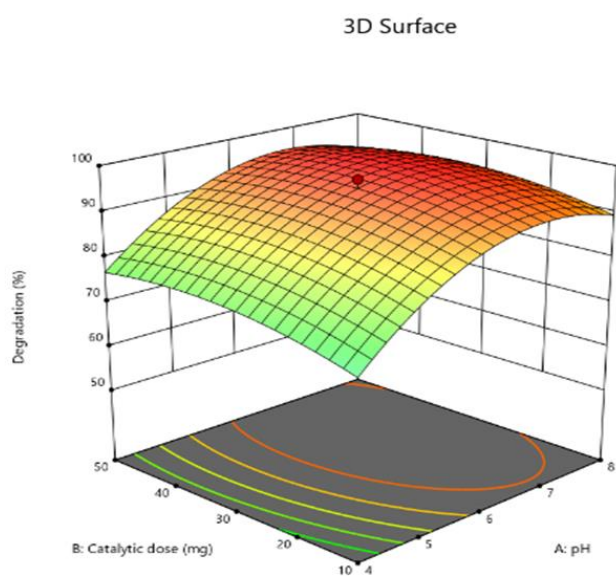
X1 = A

X2 = B

Actual Factor

C = 10

(c)



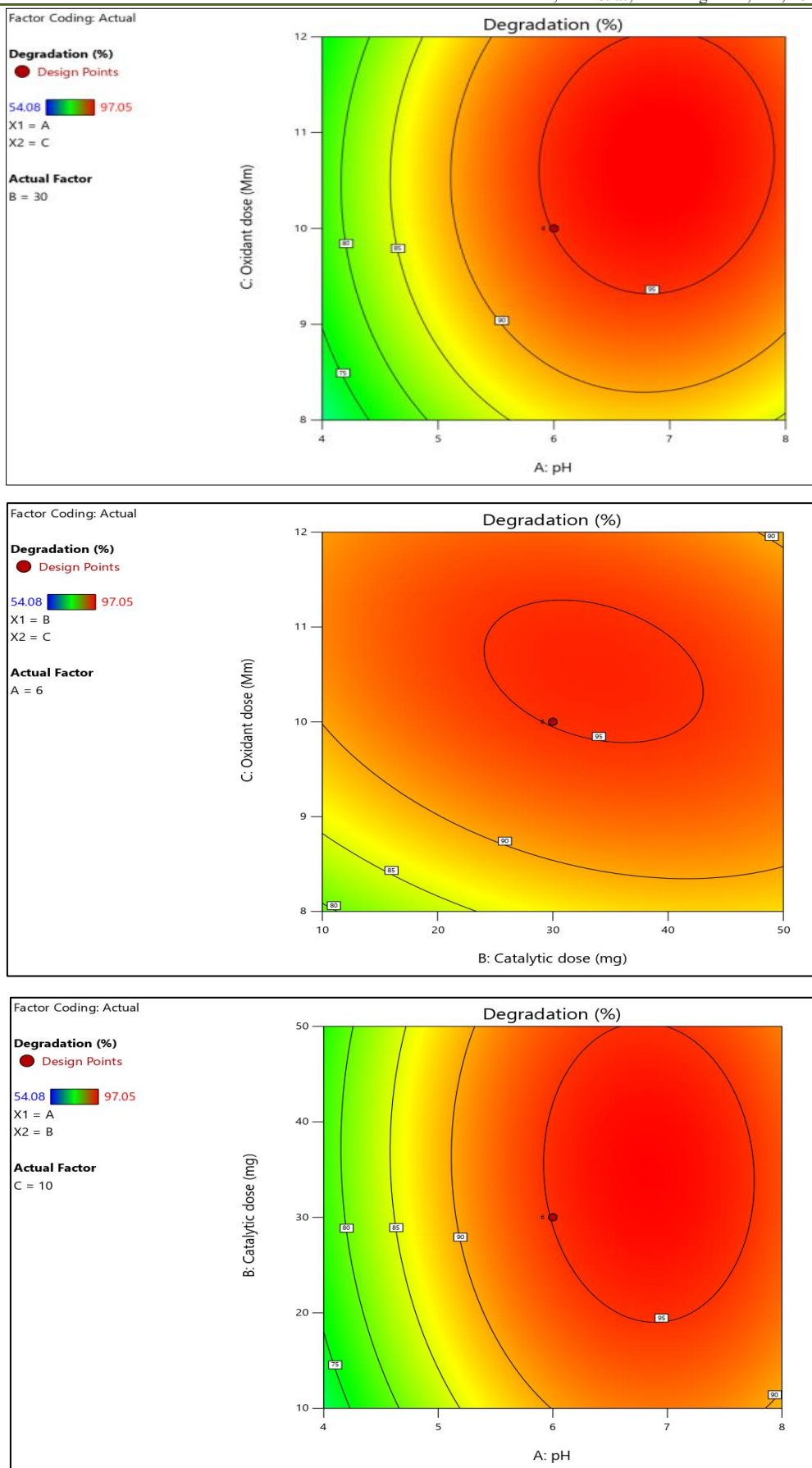


Fig. 8: The 3D diagram of Interaction of oxidant dose and catalyst dose, catalyst and Ph, and oxidant and pH of Sn@ GCN/GO

CONCLUSION

Sn@ GCN/GO was synthesized using the hydrothermal method, Sn@ GCN and GO/GCN adapted Hammer's method, and graphene oxide and graphitic carbon nitride were synthesized using the thermal polymerization method, respectively. Among above three photocatalyst GO/GCN, Sn@ GCN and Sn@ GCN/GO the photocatalytic degradation of the methylene blue by using Sn@ GCN/GO was more up to 94%. Different characterization such as XRD, UV-Visible Spectroscopy, FTIR and SEM/EDX was performed to check the structural properties, band gap and absorption, functional group detection and element percentage weight respectively. After those various parameters such as time, catalyst dose, oxidant dose and pH trails was done to check the maximum degradation standard for each composite. The statistical analysis was performed by Response Surface Methodology for checking interaction of the different parameters and 3D surface diagram clearly showed the interaction of parameters such as pH and oxidant dose of Sn @GCN/GO.

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