Green synthesis of alumina/graphene oxide nanocomposites for photodegradation of imidacloprid

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Imidacloprid is a common pesticide, which can leach into groundwater and cause lethal diseases in humans. So, it is needful to degrade the imidacloprid from water via a green approach. So, the Al_2O_3/GO nanocomposites (Al_2O_3/GO NCs) were synthesized through a green hydrothermal approach. The alumina (Al_2O_3) nanoparticles were prepared using *Cymbopogon citrus* (Lemongrass) leave extract and Graphene oxide (GO) was prepared with the Hummer's process. The prepared samples were characterized by XRD, and FTIR to determine composition and functional groups, respectively. The photocatalytic degradation of imidacloprid from water carried under sunlight and was monitored by a UV-visible spectrophotometer. The impact of parameters such as contact time, temperature, pH, catalyst dose, oxidant, and pesticide dose were also observed during photocatalytic degradation of imidacloprid. At optimized values of all parameters (i.e., Temperature: 340K, pH: 3, Al_2O_3/GO: 90 mg/L, H_2O_2: 8 mM, imidacloprid: 4 ppm), the 98.09% imidacloprid degradation was noted after 90 minutes. Thus, the Al_2O_3/GO NCs are excellent photocatalysts with high degradation ability, stability, and reusability.

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1. Introduction

The population of human beings is increasing day by day. According to a United Nations (UN) report in 2021, the population was nearly 7.9 billion, and there is a chance to cross 9.7 billion by 2050 [1]. Agricultural lands are being turned into malls, housing schemes, etc. Migration trends towards urban areas have increased. Due to these reasons, food shortage has become a significant challenge for human beings, and the burden on farmers to grow crops from available agricultural lands has increased [2]. To overcome food shortages, farmers are using pesticides. Pesticides are organic/inorganic chemical compounds that increase crop production by controlling pests and plant diseases. Pesticides help in increasing crops yield, improving quality of produce, and cost efficiency. Usage of pesticides is increasing more than 20 lac tons per year. Despite benefits, there are many drawbacks of pesticides such as non-target effects, human especially farmer health concerns, and environmental pollution [3]. Water is important constituent for life. Without water it is impossible to survive. Quality of water is compulsory for excellent working of biological cell functions. Water pollution is increasing with more usage of pesticides. When pesticides are not treated properly, they can leach into ground water, lakes, water bodies and rivers and cause of water pollution [4].

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Imidacloprid is a neonicotinoid insecticide mainly used to control insects and pests in agriculture and pest control in parks, lawns, and other places. Imidacloprid act on the nervous system of insects by binding to nicotinic acetylcholine receptors, causing excitation of the nerves, which eventually leads to paralysis and death of insects [5]. A significant portion of imidacloprid is discharged into the air or leaches into groundwater. When living organisms drink imidacloprid-contaminated water, they face many diseases and health concerns, such as neurological symptoms, heart diseases, kidney failure, gastrointestinal irritation, and effects on other organs, which may even cause death. Many reports indicate the adverse effects of imidacloprid in mammals, such as immunotoxin effects, teratogenic, mutagenic, neurotoxin, endocrine disruptor, and reproductive toxicant [6].

So, to overcome the issues mentioned above regarding this pesticide, it is necessary to degrade imidacloprid from water so people can drink clean water with no imidacloprid. Nanocomposites (NCs) are a suitable choice for the degradation of imidacloprid [7]. Nanocomposites are engineering materials made by combining NPs with other materials which can include polymers, ceramics, or metals to produce a composite which has superior properties as compared to NPs used alone [8]. The use of NCs for degradation is a superior method as this technique has many advantages, such as considerable interaction between NCs and pesticides due to high surface area, the combination of different nanoparticles (NPs) can increase catalytic ability, high adsorption ability, many NCs can degrade only selective pesticide in this way interaction with non-target reduced, several NCs can be reused, photodegradation becomes possible and less harmful by-products are produced [9].

Alumina NPs have been used in capacitors, water treatment, fire hindrance, filtration, and ceramics due to nontoxicity, large surface area, adsorption capacity, mechanical strength, inertness to most bases and acids, and thermal stability [10]. Pseudo-first-order kinetics and single-layer adsorption study of Al_2O_3 NPs proved their promising adsorbent ability. Moreover, Al_2O_3 NPs and alumina-based nanocomposites have proven promising in photocatalysis or degrading organic/inorganic pollutants from water [11]. Researchers previously focused on the chemical synthesis of Al_2O_3 NPs. Alumina nanoparticles were prepared using laser ablation, ball milling, ultrasonic assisted hydrothermal approach, sol-gel, and pyrolysis technique [12]. These techniques are unsuitable because they involve toxic chemicals, high temperatures, pressure, energy requirements, and dangerous by-products. Therefore, green synthesis of Al_2O_3 NPs using plant extract is the best choice, as it is eco-friendly, inexpensive, non-toxic by-products, and versatile [13].

Many researchers used *Cymbopogon citrus* (Lemongrass) plant extract for the synthesis of NPs due its unique properties. *C. citrus* belongs to *Gramineae* family and enriched with vital oil ingredient. Lemongrass is widely available in tropical and sub-tropical areas of Arica, America and Asia. *C. citrus* has been using globally as medicinal supplement to cure analgesic diseases, green tea, and insecticide. It has no harmful impact on the environment. [14]. This plant extract is used in present research because it contains naturally occurring reducing agents like terpenoids, polyphenols, flavonoids, Phyto steroids, tannins, steroids and carbohydrates in higher concentrations which can reduce metal ions into their respective NPs [15].

Graphene oxide (GO), the graphene derivative, has also been vital in this field. It comprises one-layer sp^2 carbon atoms with oxygen-containing functional groups on the edges. GO is prepared using graphite, a cheap and readily available raw material. GO has attracted research attention recently due to its versatile characteristics such as heat transmission, large surface area, electron mobility, stiffness, and unique chemical and physical properties [16]. Many GO-based NCs have been prepared and utilized for the remediation of contaminants from water and air due to their adsorption properties. Moreover, GO can hinder the recombination of photogenerated electron-hole pairs of Al_2O_3. Also, GO provides a large surface area and honeycomb-like structure where NPs can adjust themselves [17].

Numerous techniques are available for pesticide degradation, but photocatalytic degradation is one of the most suitable techniques. Pesticides that are less biodegradable, complex, and present in higher concentrations can be degraded with the photocatalytic method [18]. In this technique, light sources provoke the electrons of photocatalysts. These electrons are captured by oxygen, hydroxyl group, and water and generate reactive species. These reactive species react with pollutants and cause degradation [19]. In the current study, Al_2O_3/GO NCs were prepared and utilized as a

photocatalyst for the degradation of imidacloprid. It appeared to be an excellent photocatalyst at optimum conditions (i.e., 98.09% degradation was noted after 90 mins).

2. Experimental section

Synthesis of Al_2O_3 NPs was carried out using *Cymbopogon citrus* extract. Lemongrass (*Cymbopogon citrus*) Leaves were collected from Fazal Nursery Faisalabad. After washing the leaves with tape water, they were cut into smaller pieces with stainless steel scissors. Distilled water was used to remove impurities and was spread on a piece of cloth for drying for 15 days. Dried leaves were floored to a fine powder with an electric grinder. After this, a small amount (20g) of powder and distilled water (200 mL) was transferred into a beaker (250 mL). The mixture was heated with a hot magnetic stirrer at 90 ºC for 90 minutes and allowed to cool slowly at room temperature. After filtering the solution, the filtrate was stored in air-tight bottles to prepare alumina NPs [20].

An appropriate quantity of *Cymbopogon citrus* extract (25 mL) and distilled water (100 mL) was transferred into a beaker (250 mL). The mixture was heated on a hot magnetic stirrer at 105 ºC for 3 hours, adding 50 mL of aluminium nitrate (0.1 M) during heating. The thick paste was formed in the beaker, which was transferred to the crucible and was further heated in an oven at 250 ºC for 4 hours. Yellowish-brown precipitates (PPTs) of alumina NPs were obtained. After filtration, the PPTs were centrifuged and purified by washing with methanol and distilled water and further centrifugation at 13000 rpm for 10 minutes. Finally, Al_2O_3 NPs were dried at 70 ºC for 2 to 3 h and stored in an airtight bottle [21].

Hummer's method was applied for the synthesis of GO. H_2SO_4 (250 mL) and $NaNO_3$ (3g) were taken in the flask (500 mL) while mechanically agitating in an ice bath. A minute quantity of graphite powder (2g) and $KMnO_4$ (1g) was slowly added to the flask. The temperature was maintained at 4 ºC for 2 hours and increased to 36 ºC for the next 3 hours. After adding distilled water (44 mL), the temperature was raised to 96 ºC for 35 min. After that, the reaction mixture was cooled to 42 ºC, followed by the addition of 6 mL H_2O_2 (30%) and 50 mL distilled, which resulted in the development of the yellow color solution. The solution was centrifuged and washed with HCl (6%) and purified water. After freeze-drying, GO was obtained in solid form [22].

Al_2O_3/GO composites were synthesized through a green hydrothermal approach. 3g GO was added in 40 mL distilled water and sonicated for 70 minutes. After this, 2 g Al_2O_3 NPs were transferred to the GO solution through constant agitation for 70 minutes. The obtained mixture was kept in a 200 mL beaker to autoclave and placed in an oven; the temperature was maintained at 155 ºC for 350 minutes. Black precipitates of Al_2O_3/GO formed and purified by washing with methanol and water through centrifugation. Then, the sample was kept in an oven at 55 ºC temperatures for 23 hours, and the final yield of Al_2O_3/GO NCs was obtained and stored in an airtight bottle for further analysis and study [23].

The prepared Al_2O_3/GO NCs were characterized with FTIR and XRD. The FTIR was used to identify various functional groups present in the compounds. FTIR analysis (apodization: Happ-Genzel, sample and background scans: 40, resolution: 4, and range: 4000-650 cm^{-1}) was performed in the Department of Chemistry, University of Agriculture Faisalabad. The crystallinity and phase of samples were measured by using the XRD technique. XRD analysis of samples was performed in the Central Hi-Tech Lab, Government College University Faisalabad.

Synthesized Al_2O_3/GO NCs were utilized for the photodegradation of imidacloprid in sunlight. In the current research work, various 100 mL solutions of imidacloprid were used for the degradation study. UV-visible spectrophotometer (Perkin Elmer) in the Central Hi-Tech lab, Government College University Faisalabad, was used to note the absorbance of samples separately at regular intervals. Various parameters such as the effect of contact time (15-90 min), effect of temperature (290-360 K), imidacloprid dose (1-16 ppm), Al_2O_3/GO dose (0-150 mg/L), oxidant dose (2-14 mM), and pH (1-10) were optimized for improving degradation of pesticide. The reusability test for catalyst was also performed. All parameters were measured separately, while other parameters were

constant. The absorbance of various samples was noted, and a degradation (%) graph was plotted [24]. The following equation was used to calculate percentage degradation.

$$\text{Percentage degradation} = (Co - C_t / C_o)\ 100$$

where C_o = Absorbance without catalyst at reaction time (t =0)
C_t = Absorbance in the presence of catalyst after reaction time (t = t).

Eley Rideal scheme was used for the kinetic study of degradation reaction. The equations used are as follows:

First order kinetics

$$\ln \frac{C_o}{C_t} = k_1.t \quad (1)$$

Second order reaction

$$\frac{1}{C_t} = k_2.t + \frac{1}{C_o} \quad (2)$$

C_o, C_t, t, k_2, and k_1 are the concentration of imidacloprid at time "0", at a time "t," time, and rate constant for second-order and first-order reactions.

3. Result and discussion

3.1. Structural analysis

The formation of the Al_2O_3/GO composite was verified with vibrational and stretching peaks through FTIR analysis, as shown in Figure 1a. Alkoxy groups (C - O – C), ketone group (C=O), and epoxy groups (C-O) of graphene oxide showed the bands at 1021, 1628, and 1244 cm^{-1}, respectively. SP^2 hybridization in graphene oxide showed absorbance at 1545 cm^{-1}. The H-O-H (water) is indicated at 3530 cm^{-1} [25]. Successful decoration of Al_2O_3 NPs on

GO was confirmed with smooth and broad absorption in the 501 to 999 cm^{-1} wavenumber range. The Vibrational peaks at 526, 576, 733, and 882 cm^{-1} are due to Al-O-Al Stretching vibration [26].

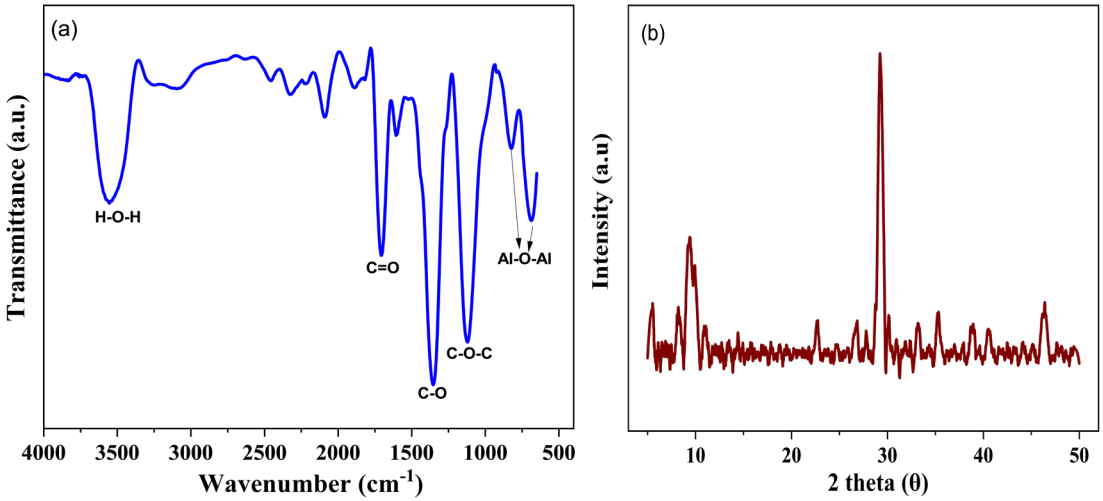


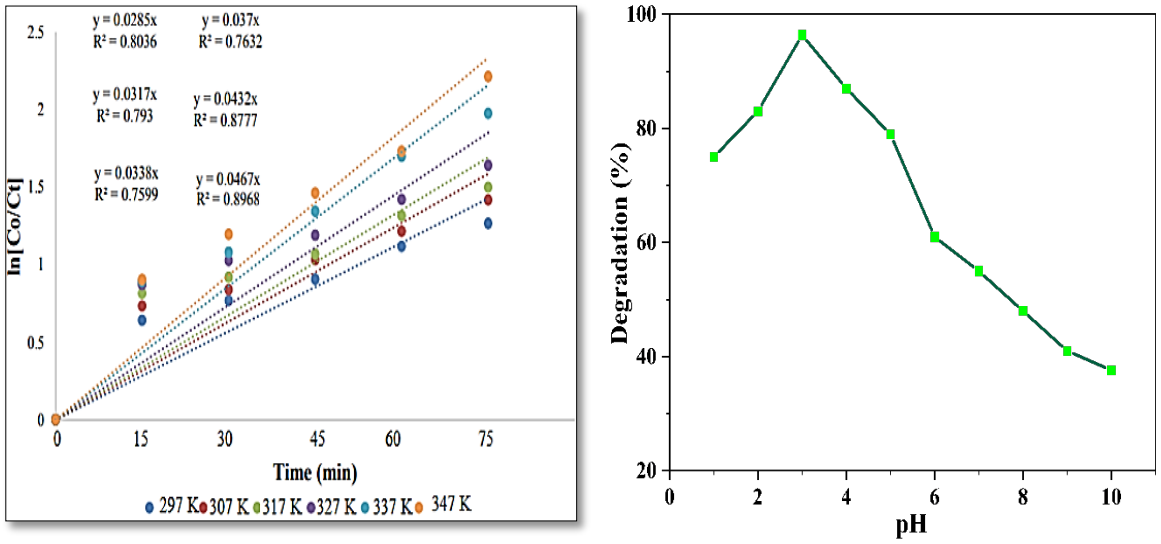
Fig. 1. (a) FTIR pattern (b) XRD pattern of Al_2O_3/GO NCs.

Figure 1b represents the XRD study of prepared Al_2O_3/GO NCs. The planes of cubic phase of gamma alumina showed at (441), (512), (401), (223), and (312) corresponds to 20.5°, 32.0°, 38.7°, 40.6°, and 46.9° values of 2 theta respectively [JCPDS no.10-0425] [27]. The purity of alumina NPs was confirmed with the absence of diffraction of any other substance. In addition to these peaks, [(002) at 10.65° and (001) at 29.4°], characteristic peaks of graphene oxide were also noted in the XRD pattern of the composite, which showed the successful deposition of Al_2O_3 NPs on GO. The peak (002) of graphene oxide was broader with lower intensity than graphite [28].

3.2. Photocatalytic degradation of imidacloprid

The prepared Al_2O_3/GO NCs were used to degrade imidacloprid in sunlight. The degradation of imidacloprid using Al_2O_3/GO followed a pseudo-first-order reaction, as presented in Figure 2a. Initially, the reaction rate is very high as imidacloprid is in significant excess, and there is more chance of collision between pesticide and catalyst. As the reaction proceeds, the reaction rate slows down gradually, indicating the degradation of imidacloprid.

Maximum degradation (96.38%) was noted at 3 pH while minimum degradation (37.63%) at 10 pH, as shown in Figure 2b. Hence, 3 pH is the optimum pH for the degradation of imidacloprid by Al_2O_3/GO NCs. Degradation declined after 3 pH was due to more $Al(OH)_3$ formation, as this can surround the catalyst and decrease photocatalytic ability. Besides this, OH^- ions started to auto-decompose at higher pH and will no longer be available for degradation.



(a) *(b)*

Fig. 2. (a) Degradation of imidacloprid using Al_2O_3/GO; first-order reaction (b) Effect of pH on degradation of imidacloprid.

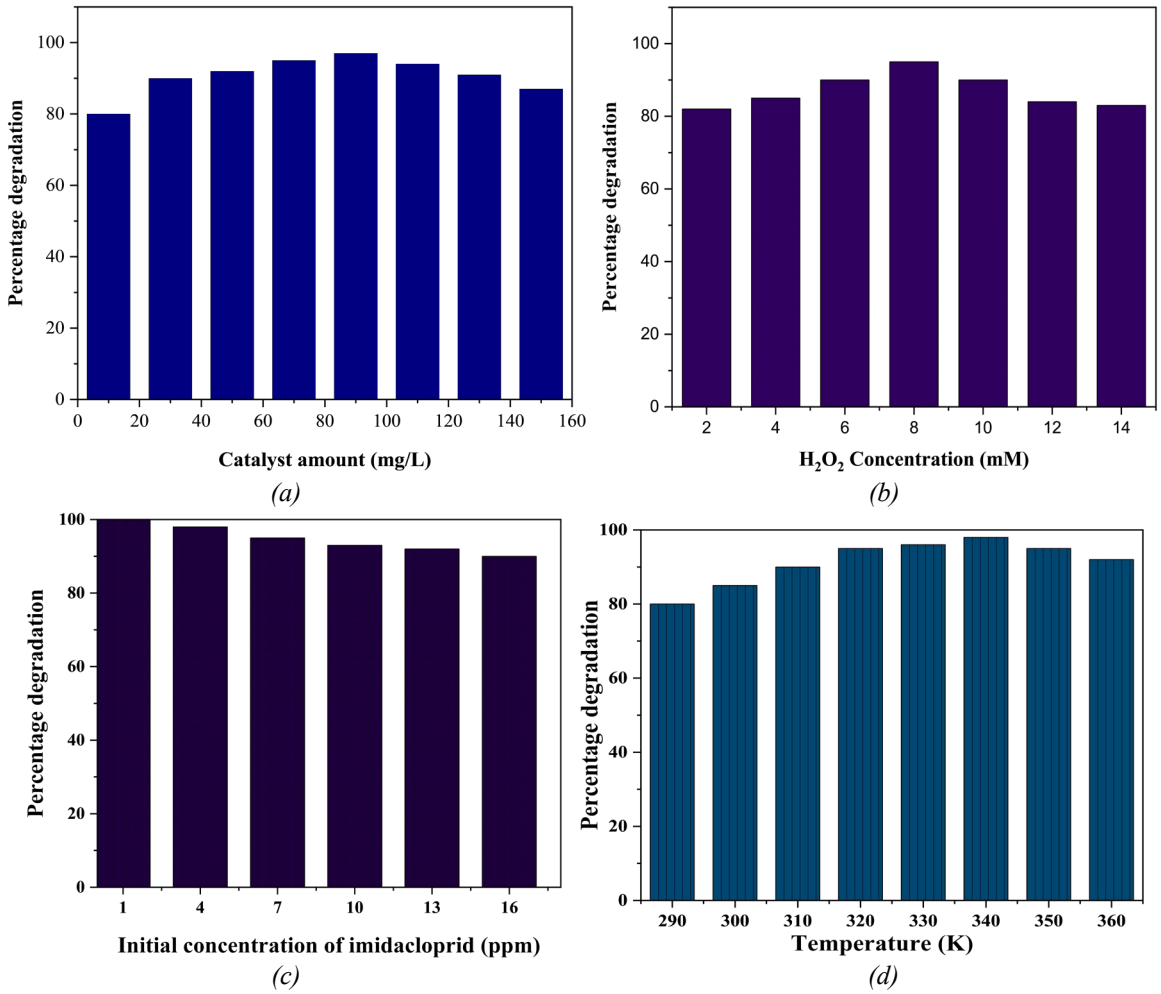
In this experimental work, the quantity of Al_2O_3/GO photocatalyst was changed from 10 to 150 mg/L for the degradation of imidacloprid. The Results obtained are shown in Figure 3a. Maximum degradation (97.06%) was observed with 90 mg/L of nanocomposites. As the amount of catalyst increased from 10 to 90 mg/L, more active sites were created for interaction with pesticide; hence, degradation efficiency improved gradually. At higher doses (90 to 150 mg/L), degradation (%) was slightly reduced due to catalyst agglomeration, blockage of photon absorption to a solution, and lessened active sites available [29].

Hydrogen peroxide acted as an accelerator in photocatalysis reactions. The role of H_2O_2 in current experimental work was also studied by changing its concentration from 2 to 14 mM. The obtained results are shown in Figure 3b. The degradation ability of Al_2O_3/GO increased with increasing concentration of H_2O_2. The enhancement in degradation efficiency was due to the

absorbance of photons by H_2O_2 and its conversion into OH free radicals in the presence of aluminum ions. The free radicals improved the degradation of imidacloprid. The optimum value of 8 mM of H_2O_2 was noted. However, degradation efficiency declined gradually from 8 to 14 mM due to reactions between OH free radicals and H_2O_2 [30].

As shown in Figure 3c, the initial concentration of imidacloprid greatly affected the degradation rate. Various samples of different concentrations of imidacloprid (1 to 16 ppm) were treated using Al_2O_3/GO for degradation. The nanocomposite degraded (90%) 16 ppm solution of imidacloprid, while 98% degradation was observed for 1 to 4 ppm solutions. The reasons for low degradation at higher doses of imidacloprid are (a) active sites are no longer available for further doses of imidacloprid, (b) more photons were captured by pesticide, and less interaction of photons with the catalyst, hence hindering the photocatalytic efficiency of the catalyst [31].

The degradation (%) of imidacloprid was also observed at different temperatures during experimental work. Results are shown in Figure 3d; the degradation rate increased with increasing temperature because when temperature increases, the kinetic energy of the Al_2O_3/GO catalyst and imidacloprid molecules increases, and hence the chance of collision increases. Maximum degradation was observed at 340 K. It was also noted from the result that degradation increased with increasing temperature until a limit; when the temperature limit was crossed above 340 K, the degradation rate decreased due to the denaturation of the catalyst [32].



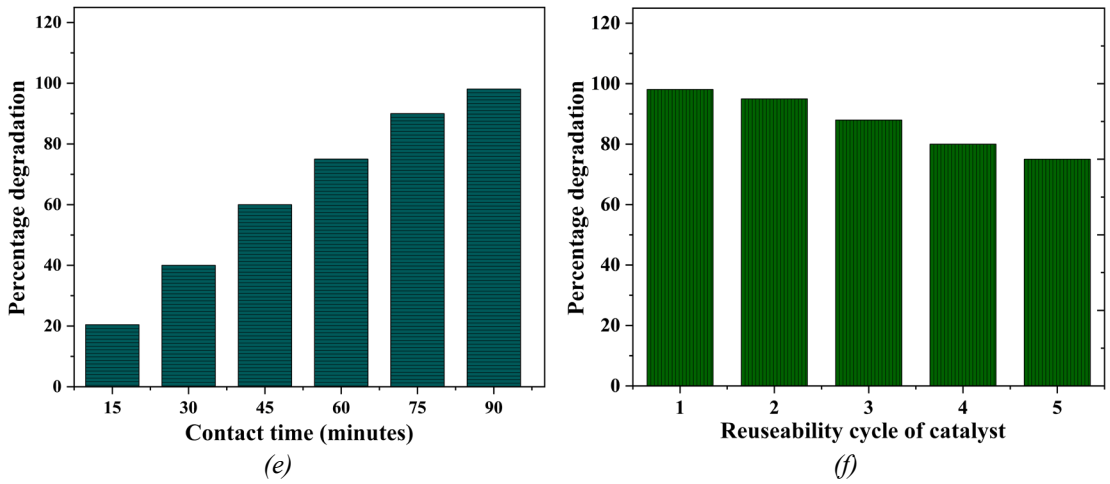


Fig. 3. Optimization of (a) catalyst amount, (b) H_2O_2 concentration, (c) Initial concentration of imidacloprid, (d) temperature, (e) contact time, and (f) reusability of Al_2O_3/GO nanocomposite for photodegradation of pesticide.

The effect of contact time between imidacloprid and Al_2O_3/GO was also investigated, while other parameters were kept at optimized values. Results, as shown in Figure 3e, reported minimum degradation (20.44%) after 15 minutes and maximum degradation (98.09 %) after 90 minutes. The increase in degradation (%) by increasing contact time was due to a more significant number of formations of hydroxyl ions [33].

This experimental work was also investigated for the reusability and stability of Al_2O_3/GO NCs. After the degradation process, the catalyst was removed from the solution, washed with ethanol and water, dried at 80 ^oC in the oven, and reused for untreated samples of imidacloprid. The process repeated for five cycles, shown in Figure 3f. The photocatalytic ability of the catalyst was not reduced too much (75% degradation), even in the fifth cycle. After the reusability test, the leaching of aluminium in water was investigated, and the results are shown in Table 1; very minute concentrations of aluminium in water were reported, and these leaching concentrations below 0.20 mg/L (maximum Al limit in drinking water in accordance to U.S. EPA) are not harmful to human health [34].

Table 1. Al_2O_3/GO photocatalyst stability in terms of Al leaching.

Cycle	Degradation (%)	Al leaching (mg/L)
1st	98.09	0.18
2nd	95.2	0.15
3rd	87.09	0.13
4th	80	0.08
5th	75	0.05

4. Conclusion

In this recent experimental work, alumina (Al_2O_3) nanoparticles were prepared through a green synthesis approach using extracts of leaves of the *Cymbopogon citrus* (Lemongrass) plant. Graphene oxide (GO) was prepared with the help of Hummer's process. A green hydrothermal approach was applied to synthesize the Al_2O_3/GO NCs. GO enhanced the catalytic efficiency of Al_2O_3 NPs by exposing its active sites to oxidants and pesticides. The prepared Al_2O_3/GO NCs were characterized with XRD and FTIR.

The effect of alumina/graphene oxide Al_2O_3/GO NCs for the photodegradation of imidacloprid pesticide in water was investigated with a UV-visible spectrophotometer. Various parameters, such as the effect of contact time, pesticide dose, catalyst dose, oxidant dose, and pH, were optimized to improve the degradation of the pesticide. At optimized values of all parameters, 98.09 % degradation was noted after 90 minutes. The photodegradation followed a pseudo-first-order reaction. So, this approach is easy, inexpensive, eco-friendly, and can be adopted on a large scale. The Al_2O_3/GO has proven to be an excellent photocatalyst as it has high degradation ability, stability, less aluminum leaching, and reusability. The 100% degradation of pesticides can be achieved with Al_2O_3/GO NCs by enhancing the light penetration. This could be improved by focusing on acoustic cavitation, light, and advanced oxidation techniques.

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