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Green synthesis of magnesium oxide nanoparticles using leaves of *Iresine herbstii* for remediation of reactive brown 9 dye

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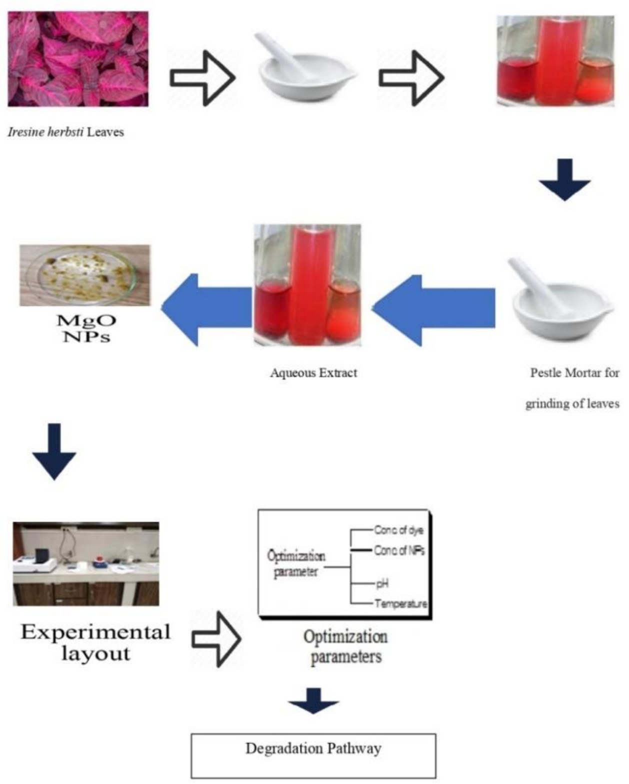
Graphical abstract



Abstract

Nanotechnology is the newest and one of the most promising areas of research in modern medical sciences. Metal nanoparticles possess extraordinary optical, thermal, chemical properties and are being widely used in industrial, electronics, and biomedical fields. It has presented its potential to contribute to solving one of the greatest problems of the worldwide wastewater treatment issue. The approach of green synthesis of metal oxide nanoparticles seems to be a cost-efficient, eco-friendly, and easy alternative approach. The current study deals with the synthesis and characterization of Magnesium Oxide nanoparticles (MgO-NPs) using leaves extract of *Iresine herbsti*. The characterization was done by XRD and SEM. Then MgO-NPs were applied for the remediation of Reactive Brown 9 dye following the optimization of reaction parameters (conc. of dye, conc. of nanoparticles, pH, and temperature). The maximum decolorization (95.8%) was obtained at 0.02% dye conc., 0.003 mg/L conc. of MgO-NPs, at pH 4, and temperature 40°C. TOC and COD were used for mineralization assessment of studied dye samples and their values were found to be 88.56% and 85.34%, respectively. The magnesium oxide nanoparticles could be applied successfully for the treatment of other problematic dyes as well.

Keywords: Nanotechnology, MgO-NPs; *Iresine herbstii*, XRD and SEM, TOC and COD.

1. Introduction

On this earth, water is a basic need. With the human population and industrial work, water supplies are being rapidly polluted. For their proper functioning, each nation demands clean water (Godoy *et al.*, 2020). Pollution caused by synthetic dyes has become one of the most serious environmental issues. By rapidly developing industrial units, effluents having synthetic dyes are directly or indirectly being discharged into the environment. Elimination of these toxic dyes is very necessary as it destroys the aquatic environment and become a source of many diseases (Kiran *et al.*, 2020). The methods of water treatment are very limited so far, but with the passage of time, advanced technology and human skills are creating new avenues for different eco-friendly and effective wastewater treatment methods. As water is considered the primary solvent in manufacturing, agricultural and household operations, water should be well handled and made suitable for further use (Kiran *et al.*, 2019a,b; Kumari *et al.*, 2019).

Nanotechnology is the emerging and most thrilling field of study in various applied fields. In food, nanomaterials have been broadly applied for storage to progress food security and food value (Kaung *et al.,* 2020). Nanotechnology is used in agriculture to increase the food production, with nutritional value, quality and protection being equal or even higher. The most effective ways to increase crop production are the effective usage of fertilizers, pesticides, plant growth factors and herbicides. Nanotechnology has made a major contribution to the healthcare, pharmaceutical, clothing, fabrics, information technology and renewable energy sectors (Acharyaa *et al*., 2020). There are two methods for the synthesis of nanoparticles. One is top-down and other one is bottom-up approach. The "top-down" approach is the industrial method that reduces the size of higher molecules. The "bottom up" method is the production of individual components (atoms or molecules) by its own contribution to do so, using physical and chemical techniques (gas- and liquid-phase) (Francisco & Estepa, 2018). Nanoparticle preparation is based on size reduction of starting material through various physical and chemical processes, which is not chosen because it is an expensive approach that requires a lot of energy, and produces a small amount of material. At the industrial level, high output at low cost is required for successful adoption of this technology (Jamkhande *et al*., 2019).

Iresine herbstii is a perennial herb with purple or deep purple to violet, multicolored leaves. It is native to tropical South America and has now spread across the globe. In traditional Brazilian medicine, it is used topically for treating eczema and for wound healing. It is traditionally used in China for the treatment of anemia and is used in Thailand as a tonic (Nencini *et al.,* 2006). *I. herbstii* is readily available and inexpensive one which is contributing healthy and productive fresh secondary metabolites. Significant examples of these metabolites are alkaloids, coumarins, terpenes, flavonoids, anthraquinones and naphthoquinones (Andleeb *et al*., 2019).

The magnesium oxide nanoparticles (MgO-NPs) are ionic in nature. Magnesium oxide nanoparticles have extremely high surface area and transparent morphologies (Dobrucka, 2018). Owing to its crystal morphology it retains optical, magnetic, electronic, thermal and mechanical properties (Ramanujam & Sundrarajan, 2014). Numerous biochemical procedures have been accepted for the synthesis of magnesium oxide (MgO) nanoparticles, for example laser evaporation, precipitation, hydrothermal, precipitation, sol-gel combustion, micro-emulsion, chemical gas phase installation, and laser evaporation practices (Nassar *et al*., 2016). Removal of toxic dyes present in textile effluent by nanoparticles is the cost effective way and a greener approach (Kiran *et al.,* 2017; Acharyaa *et al.*, 2020).

So keeping in view the importance of metal oxide nanoparticles, the current study was planned to synthesize the magnesium oxide (MgO) nanoparticles using leaves of *Iresine herbstii* and then the synthesized MgO nanoparticles were applied for the remediation of Reactive Brown 9 dye following the optimization of experimental parameters.

2. Materials and methods

The chemicals and solvents were purchased from Public Scientific Store, Jinnah colony Faisalabad, Pakistan. Reactive Brown 9 dye which was used for research analysis was also purchased from the same chemical store whose structure is given below (Figure 1).

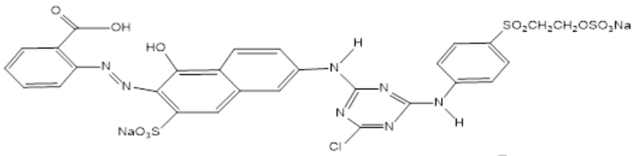


Figure 1. Chemical structure of Reactive Brown 9 dye (C.I. 12225-66-0).

2.1. Sample collection and preparation

Fresh and healthy leaves of *Iresine herbstii* free from disease were collected from Punjab Forest Department, Faisalabad Pakistan. The leaves were carefully washed with tap water following washing with distilled water till no external material was left. The freshly washed leaves were left to dry for 10 days in a locked era (25-28°C). To attain a powdered form, the dried leaves of *Iresine herbstii* were placed in a purified electrical mixer (Andleeb *et al*., 2019).

2.2. Aqueous extract preparation using leaves extract of Iresine herbstii

25 g of finely chopped leaves were taken in a reaction vessel having 250 mL distilled water and was boiled for 20 minutes to prepare aqueous extract of *Iresine herbstii* leaves. By using whattmann filter paper 42 the extract was filtered and refrigerated at 4˚C for further use (Dipanker and Murugan, 2012).

2.3. Preparation of MgO-NPs

25 g powdered sample was taken and 250 mL distilled water was added in it. After that, the solution was put into a hot plate and boiled for 20 minute at 60°C. 8 g of magnesium nitrate was added in it and boiled for 4 hours at 80°C. 0.1M sodium hydroxide was added till the colour change happened. The appearance of brown color showed the development of the MgO-NPs which were separated by centrifugation. The synthesized MgO-NPs were placed in microwave oven at 60^{0} C for a day. The dried sample was stored in an eppendorf tube for further usage (Dipanker and Murugan, 2012).

2.4. Characterization of synthesized magnesium oxide nanoparticles

The MgO-NPs were characterized by XRD and SEM. The XRD pattern shows the cubic structure and the crystallite size. Magnesium nitrate was used as a source material and the prepared samples were calcined at 400°C for 3 hours and micro-structural properties were got studied (Balakrishnan *et al*., 2020).

2.5. Scanning of λ_{max}

Scanning of λ_{max} was done with different dilutions of dye solution and UV-Visible double beam spectrophotometer was used to measure the absorbance.

2.6. Experimental protocol

50 mL of Reactive Brown 9 dye solution (0.01%) was taken in reaction vessel and MgO-NPs (3 mg) were added in to it and pH of this solution was checked and it was adjusted at 5 by adding 1M HCl/1M NaOH into it and was stirred it on hot plate with magnetic stirrer at 40°C for 90 minutes and noted the absorbance after every 15 minute to check the absorbance. Only one parameter was altered while the other remains constant.

2.7. Optimization of different parameters for decolorization of Reactive Brown 9 dye

For the decolorization of Reactive Brown 9 dye solution, magnesium oxide concentration (MgO-NP) (1-5 mg/L), dye concentration (0.01-0.05), pH (4-7), and temperature (30-80°C) were optimized.

2.8. Chemical analysis

All the decolorization experiments were done thrice. The dye absorbance was observed at 390 nm wavelength using double beam spectrophotometer. The effective percentage of decolorization of all the parameters was estimated by measuring absorbance of solution. To get the percentage of decolorization, the following equation was applied

$$\text{Decolorization (\%)} = [(I-F)/I] \times 100$$

Here I = initial absorbance at zero time, F = final absorbsance of dye solution.

2.9. Mineralization study

The chemical oxygen demand (COD) is a measure of water and wastewater quality. COD is the amount of oxygen consumed to chemically oxidize organic water contaminants to inorganic end products. Total organic carbon (TOC) which utilizes a catalytic oxidation combustion technique at high temperature to convert organic carbon into CO_2. The treated and untreated dye samples were evaluated by water quality parameters like COD and TOC (Greenberg *et al*., 1985)

2.10. Degradation study

The decolorization of Reactive Brown 9 dye was evaluated by cracking of bonds and development of new compounds in different steps (Nadeem *et al*., 2020).

2.11. Statistical analysis

All experiments were run in triplicate and results were computed as an average of means. Standard error of means was calculated (Steel and Torrie, 1997).

3. Results and discussions

3.1. Characterization of synthesized Magnium oxide nanoparticles by XRD

The synthesized MgO nanoparticles were characterized by X-ray diffraction to evaluate its form and crystal orientation from the diffraction peaks. In the XRD pattern (Figure 2), no other impurity step was found. It tests and studies micro-structural properties (Balakrishnan *et al*., 2020). 14.23° is the angle which shows maximum peak clearly demonstrates the formation of MgO nanoparticles with a polycrystalline cubic phase (Kujur *et al*., 2018).

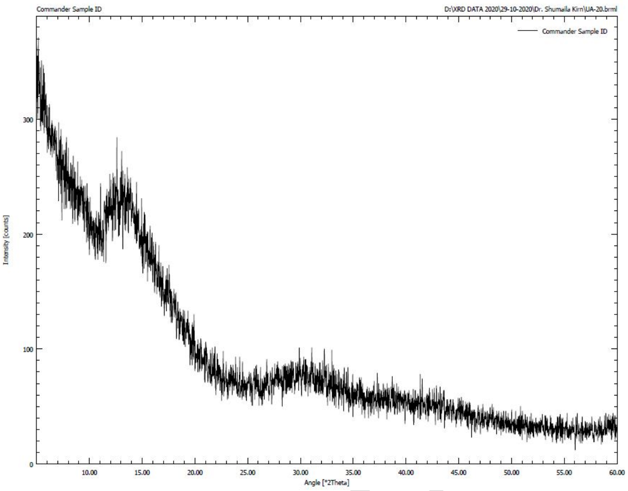


Figure 2. XRD Image of MgO nanoparticles.

3.2. SEM analysis of synthesized MgO nanoparticles

The synthesized MgO-NPs were characterized by scanning electron microscopy. The size and shape of synthesized MgO nanoparticles was screened by SEM. The MgO nanoparticles powder exactly illustrates the shape of spherical MgO nanoparticles (Figure 3), uniform, dense and translucent with accumulation (Balakrishnan *et al*., 2020).

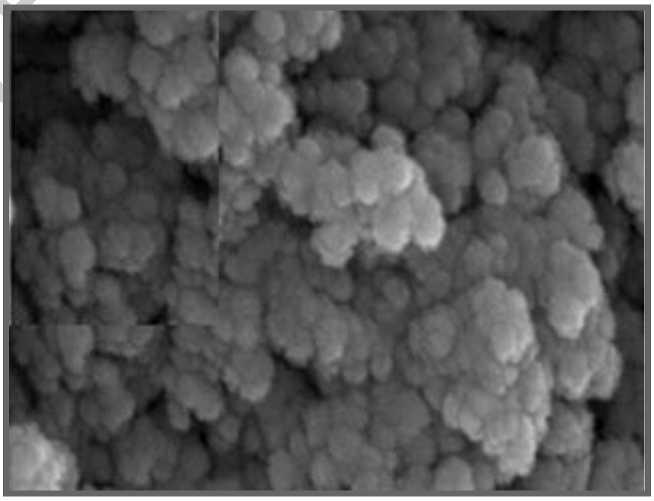


Figure 3. SEM image of MgO nanoparticles.

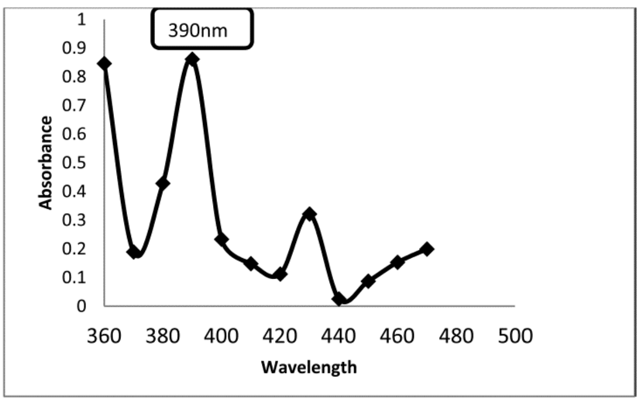


Figure 4. Scanning of λ_{max} for reactive brown 9 dye solution.

3.3. Application of MgO-NPs for remediation of Reactive Brown 9 dye

3.3.1. Determination of λ_{max} for Reactive Brown 9 dye

In the visible region of 360 to 470 nm range of wavelength, in the intervals of 20 nm wavelength, absorbance was noted in order to find the absorbance at maximum level. In order to calculate the wavelength having highest absorbance, the obtained data was graphed. The maximum wavelength was 390 nm (evident from the graph below in Figure 4).

3.3.2. Optimization of experimental parameters for remediation of Reactive Brown 9 dye

The decolorization rate of Reactive Brown 9 dye was assesses by measuring the various experimental parameters like Reactive Brown 9 dye concentration (0.01-0.05%), concentration of the MgO-NPs (1-5mg), pH (4-7), Temperature (30-80°C).

3.3.3. Effect of dye concentration

Dye as emerging pollutant was established in current years with respect to different dye handling procedures. The influence of many primary applications of dye on catalytic decolorization has been examined from 0.01-0.05%. The dye decolorization went on increasing from 56.54% to 86.65% in 45 min. as dye concentration was raised from 0.01 to 0.02%. The extreme decolorization has been happened at concentration of 0.02% i.e 86.65% as shown in the Figure 5a (Nassar *et al*., 2019). The lowest decolorization value was observed at 0.05% concentration i.e 29.54% at 45 min. Huge application of dyes in water is very toxic, as it is dangerous for the marine atmosphere (Kiran *et al*., 2020). At the high dye concentration there is significant amount of the dye molecule instead of catalyst and this as well, can decline catalytic proficiency which in turn can hinder the reaction (Kale and Kane, 2017; Gulzar *et al*., 2017; Kamranifar *et al*., 2018).

3.3.4. Effect of magnesium oxide nanoparticles

Magnesium oxide nanoparticles (MgO-NPs) concentration is a fundamental factor which needs to be optimized. With rising catalyst concentration, which is property of heterogeneous catalysis, the dye degradation rises. The capacity of the catalyst and the accumulation of catalyst particles in its high amount also affect the dye degradation (Nassar *et al*., 2016). Series of experiments were done by varying the magnesium oxide nanoparticles concentration (0.001, 0.002, 0.003, 0.004, 0.005g/100 mL) to find out the optimum catalyst amount. Beyond 0.003 g/L of the dose of MgO nanoparticles, there is no significant increase in dye decolorizationas as shown in Figure 5a (Kiran *et al*., 2020). For successive experiments, from 0.001 to 0.005g/L, 0.003 g/L of MgO nanoparticles was therefore observed as an optimal level of MgO nanoparticles for degradation of the Reactive Brown 9 dye and its decolorization value was observed to be 92.65% (Figure 5b). The lowest decolorization value was observed at 0.001g/L value of MgO Nanoparticles at 90 min. The rise in the rate of degradation as catalyst packing increases because net active site region increases i.e. the accessibility of high catalyst activeness surface sites (Nassar *et al*., 2016; Kamranifar *et al*., 2018; Ghaffar *et al*., 2021).

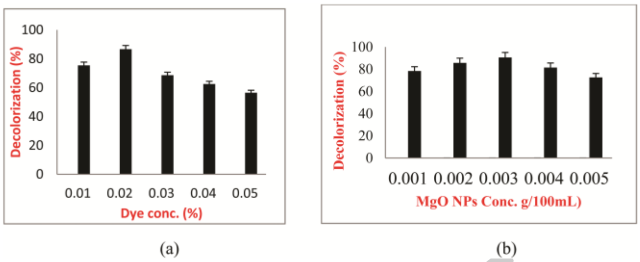


Figure 5. Effect of Reactive Brown 9 dye concentration on its decolorization (a) and Effect of different amounts of MgO-NPs on the decolorization of Reactive Brown 9 dye (b).

3.3.5. Effect of pH on decolorization of Reactive Brown 9 dye

pH of solution has a very direct effect in the efficiency of decolorization. With varying in pH, the decolorization of dyes is effected considerably (Kiran *et al*., 2020). Highest decolorization was observed at 95.7 % for MgO catalyst at pH 4.0 for 45 min. as shown in the Figure 6a. The lowest decolorization was observed at pH 8 and its decolorization value was recorded as 18.54%. At this point, the capability of MgO-NPs to decolorize the dye was near to end. The pH 4 has consequently been taken as an optimum pH value and this shows catalysts color removal abilities as a function of pH (Naseer *et al*., 2016; Shahid *et al*., 2016). Optimum pH gives the catalyst a way to work efficiently. For lightly acidic pollutants, the rates of reaction increases at lower pH (Silva *et al*., 2019). The perfect interaction stage of dyes was nominated as 40-50 minutes as at that time the process of adsorption knockout the balance and remained constant (Nassar *et al*., 2016; Kiran *et al*., 2021).

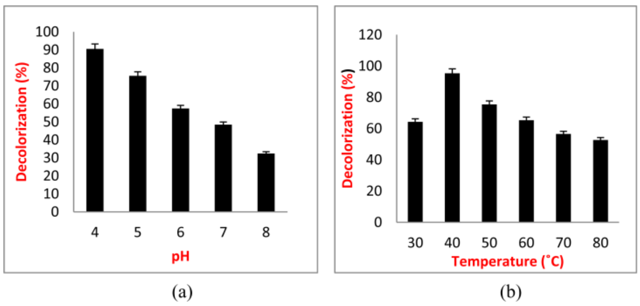


Figure 6. pH effect on Reactive Brown 9 dye decolorization with MgO nanoparticles (a) and Temperature effect on decolorization of Reactive Brown 9 dye engaging MgO-NPs (b).

3.3.6. Temperature effect on decolorization of reactive brown 9 dye

The degree of Reactive dyes decolorization increases till optimum temperature and then steady decrease in degradation of dye (Satar and Husain, 2009; Kiran *et al*., 2018). The effect of temperature (30-80°C) on decolorization (%) of Reactive Brown 9 dye was studied. The complete experimentation done keeping the previously optimized parameters (like pH was 4, conc. of MgNPs 0.003g/L, Reactive Brown 9 dye dose 0.02 %)

constant. It was observed that maximum decolorization value was 95.76% at 40˚C (Figure 6b). In this treatment process, decolorization ability of Reactive Brown 9 dye slowly concentrated as the temperature increased (Rafique *et al*., 2021). There is a reduction in adsorption efficiency at higher temperatures, possibly because of the phase conversion of catalyst with temperature promotion, which may decay in the surface area of catalyst (Lafta, 2015).

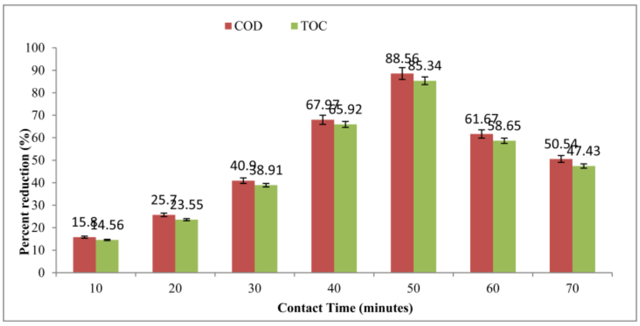


Figure 7. Effect of contact tome on percent reduction of chemical oxygen demand (COD) and total organic carbon (TOC).

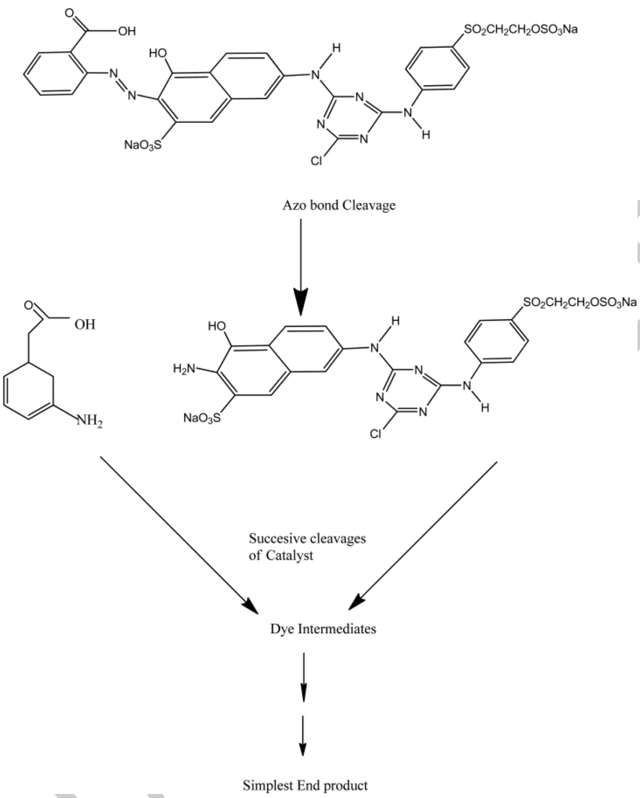


Figure 8. Degradation Pathway of Reactive Brown 9 dye.

3.4. Mineralization study

To find the COD and TOC values of Reactive Brown 9 dye treated samples using MgO nanoparticles as a catalyst, experiments were run for varying time intervals (10-70 minutes). The results have been presented in Figure 7. It is evident from Figure 7 as the time was increased; the percent reduction of both water quality parameters (COD & TOC) went on increasing up to 50 minutes of contact time. COD and TOC reduction (%) values were found to be 88.56% and 85.34%, respectively at 50 minutes of contact time. Therefore, it may be expected that MgO nanoparticles not only get rid of color simply, but additionally lessen the COD and TOC of the dye solution to ensure mineralization progress (Kale and Kane, 2018; Kiran *et al*., 2020; Rafique *et al*., 2021).

3.5. Degradation pathway of reactive brown 9 dye

The primary stage of degradation is the oxidative breakage of azo (N=N) bond in catalytic step by hydroxyl group functioning as a radical. Result of the breakage of azo bond, there is a formation of compounds called dye intermediates. The carbon-sulphur containing intermediates were formed to discharge SO_3 in the second step (Figure 8). The successive steps finally resulted in the formation of simplest products like CO_2, H_2O and by products (Gautam *et al*., 2019; Kiran *et al*., 2020).

4. Conclusions

Agrowaste is waste generated as a result of agricultural activity. Agro-waste accumulation can be hazardous to one's health, safety, and the environment. As a result, this is a problem that necessitates the use of a safe and environmental friendly way to address environmental concerns. Nanotechnology plays an important role in this regard. The use of nanoparticles in many industries such as energy, medicine, and nutrition has grown at a rapid pace. This recent research work was the synthesis of MgO nanoparticles from the leaves extract of *Iresine herbstii* and its application for the removal of Reactive Brown 9 dye. After the synthesis of MgO-NPs, the characterization was done by SEM and XRD. For the remediation of reactive brown 9 dye various parameters was optimized. The maximum decolorization of dye (95.8%) was observed at dye concentration 0.02 %, pH 4 and catalyst dose 0.003 g/L at 40 °C. Percentage reduction values of COD and TOC were 88.56% and 85.34%, respectively. The degradation pathway of dye under study confirmed the formation of non-toxic end-products. It can be concluded from current study that other toxic dyes could be eliminated out using greenly synthesized MgO nanoparticles.

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Declaration of conflicting interests

All the authors of this paper declare no conflict of interest.

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