Abstract

This study reports solution combustion synthesis of magnesia nanoparticles (nMgO) using magnesium nitrate as oxidiser and glycerol as fuel. Size, morphology, crystal structure and surface properties of synthesised nMgO were analysed by PXRD, SEM, TEM, FTIR and Point of Zero Charge. The XRD pattern of nMgO confirmed prepared samples were single cubic-phase without any impurities. TEM analysis proved nMgO was in nano regime with an average particle diameter of 20–40 nm. FTIR spectra show the presence of characteristic peaks of nMgO and support the XRD results. The prepared nMgO was employed as an adsorbent for the removal of two anionic dyes viz. Indigo Carmine (IC) and Orange G (OG). Furthermore, various adsorption isotherms and kinetic models were performed to understand the kinetics and mechanism of the adsorption process. Experimental results demonstrated that the adsorption equilibrium data fit well to Sips isotherm ($R^2 > 0.98$) and the saturated adsorption capacities of nMgO were found to be 262 mg g$^{-1}$ for IC and 126 mg g$^{-1}$ for OG. Adsorption kinetics analysis revealed that the adsorption followed pseudo-first-order model, with both film and pore diffusion governing the rate of adsorption. Excellent adsorption capacity combined with efficient regeneration proved the potential of the prepared nMgO as an adsorbent for the removal of harmful dyes from industrial effluent.

1. Introduction

Clean water is one of the vital components for all living organisms to sustain life on the planet earth [1]. But overconsumption of water resources due to the drastic increase in population growth, industrialisation, and urbanisation is leading to water crisis worldwide. Water pollution due to anthropogenic, industrial, and agricultural activities has become a major global issue, and posing a severe threat to both ecosystems and various living forms [2–5]. Water pollutants generated from various sources are generally categorised as organic (dyes, pesticides, aromatic compounds, antibiotics, etc) and inorganic (heavy metals) pollutants [6, 7]. Among many chemical pollutants, synthetic dyes have been reported as the most harmful substances [3, 8, 9]. Mass production of synthetic dyes is increasing significantly due to increasing demand for these dyes in various industries such as textile, paper, paint, plastic, tannery, pharmaceutical, and food industries, and more than 10$^8$ different types of commercial dyes are available with an annual production rate of 7 × 10$^5$ tons. The release of large amounts of dye effluents into water bodies severely affects aquatic organisms and human life owing to toxic, carcinogenic, mutagenic and allergic properties of synthetic dyes [10–14]. Efficient removal of these noxious dyes from wastewater before it is released to the environment helps in improving the quality of aquatic ecosystems [2]. Numerous treatment methods such as incineration, precipitation, coagulation, flocculation, ion exchange, membrane filtration, reverse osmosis, electrochemistry, photoelectrochemistry, advanced oxidation and bioremoval methods have proven various levels of removal efficiencies in the amputation of dyes [3, 13, 15–18]. Amongst these techniques, the adsorption process is widely used in the removal of dyes because it is a simple,
flexible, low-cost technique with better removal efficiency, selectivity, and feasibility. The advantages of adsorption are closely related to the properties of the adsorbent employed in the dye removal process [2, 19]. Therefore, the necessity for the development of adsorbents that are sustainable, economical with high removal rates and efficiency is crucial.

Due to their unique physical and chemical properties, the scholarly interest in nanoadsorbents is overgrowing worldwide in the past few decades. Reduced size, increased surface area, higher surface area to volume ratio, high porosity, and availability of active surface enables nanoadsorbents to sequester pollutants of varying properties effectively. Nanoadsorbents not only work rapidly but also possess better binding capacities and good regeneration ability [1, 3, 20, 21]. Among several nanoadsorbents, magnesium oxide nanoparticles/nano magnesia (nMgO), an alkali earth metal oxide has attracted greater attention as a potential adsorbent in the removal of various pollutants. The significant consideration of nMgO as a destructive adsorbent is due to its chemical stability, economic viability, non-toxic nature and the high point of zero charge, which is best suited for adsorption of anionic dyes. nMgO also finds applications as optoelectronic materials, catalysts, electrochemical sensors etc due to its excellent optical, electrical and mechanical properties [22–26].

Among the various wet chemical methods employed for the synthesis of nanoadsorbents, solution combustion synthesis (SCS) technique has gained widespread attention. SCS method has various advantages such as (i) requirement of simple equipment (ii) low cost (iii) high surface area and highly porous products obtained which are suitable for adsorption (iv) low-temperature technique since the energy required for phase formation is obtained from the combustion reaction itself. Further, many process parameters of SCS technique influence the product properties are fuel nature, Fuel to Oxidiser ratio (F/O ratio), adiabatic flame temperature, reaction temperature etc [27–30].

In the present study, we have reported the utilisation of inexpensive glycerol as fuel for solution combustion synthesis of nano magnesia and also the influence of F/O ratio on the phase formation of nano MgO. The removal of two anionic dyes Indigo carmine (IC) and Orange G (OG) from synthetic wastewater using nMgO as adsorbent has been attempted. Various parameters influencing the adsorption process such as pH, adsorbent dosage, initial dye concentration, time along with isotherm and kinetic models are studied in detail.

2. Materials and methods

2.1. Materials
Analytical grade magnesium nitrate hexahydrate (Mg(NO₃)₂ · 6H₂O), indigo carmine (C₁₆H₁₀N₂Na₂O₇S₂), orange G (C₁₆H₁₀N₂Na₂O₆S₂), glycerol (C₃H₈O₃), sodium chloride, hydrochloric acid and sodium hydroxide were purchased from S D Fine-Chem Ltd, India, and used without further purification. Double distilled water was used throughout the studies.

2.2. Synthesis of nanoparticles
For glycerol mediated synthesis of nMgO, required stoichiometric quantities of magnesium nitrate hexahydrate and glycerol was dissolved in 10 mL of double-distilled water. Fuel to oxidiser ratio was varied between 0.5 and 2 to optimise the size and yield of the nanoparticles. The quantity of the fuel was calculated based on the following equation:

\[
\frac{F(\text{Fuel})}{O(\text{Oxidizer})} = \frac{\sum \text{Reducing valency of oxidiser} \times \text{Mass of Oxidizer/Molecular Weight of Oxidizer}}{\sum \text{Oxidising valency of fuel} \times \text{Mass of Fuel/Molecular Weight of Fuel}}
\]

The redox mixture was stirred on a magnetic stirrer for 1 h to obtain a homogeneous solution. The resulting homogenous mixture was kept in a preheated (500 ± 10 °C) muffle furnace. Thermal dehydration followed by flame type combustion occurred, resulting in the evolution of a large amount of gases. Milky white fluffy mass of nMgO obtained was stored for further studies.

2.3. Characterisation of nanoparticles
Phase formation of nMgO was determined by Powder x-ray diffraction (PXRD) (1.5406 Å; Rigaku’s, Smartlabs, Japan) using rotating anode Cu Kα, 40 mA and 30 kV of current and accelerating voltage, 2θ range of 10° to 70° with a step size of 0.02°. Surface morphology and particle size was characterised by SEM (JEOL (JSM–840A) and TEM (JEOL, JEM–301) respectively. Surface functional groups were characterised by FTIR (Perkin Elmer spectrometer (Spectrum 1000)). Point of zero charge was determined by solid addition method [30].

2.4. Batch adsorption studies
Performance of the nMgO adsorbent was evaluated by batch adsorption experiments conducted in 250 mL conical flasks with IC, and OG solutions (50 mL) agitated at 130 rpm in a thermostatic shaker. Post adsorption,
the adsorbent was removed by centrifugation at 15000 rpm for 5 min and absorbance of supernatant solution recorded at 609 nm for IC and 480 nm for OG, respectively using a UV–vis spectrophotometer (Shimadzu, UV1800). Effect of process parameters such as adsorbent dosage, pH, initial dye concentration, and time were systematically investigated. 0.1 N NaOH and HCl were used to adjust the pH. The percentage dye removal, adsorption capacity at equilibrium \((q_e)\) and at time \(t\) \((q_t)\) are determined by the following equations:

\[
\text{Dye removal efficiency} \% = \frac{C_0 - C_e}{C_0} \times 100 \quad \text{or} \quad \frac{C_0 - C_t}{C_t} \times 100.
\]

\[
q_e \left( \frac{\text{mg}}{\text{g}} \right) = \frac{(C_0 - C_e)V}{m}.
\]

\[
q_t \left( \frac{\text{mg}}{\text{g}} \right) = \frac{(C_0 - C_t)V}{m}.
\]

where \(m = \text{mass of adsorbent in g}\), \(V = \text{dye solution volume (L)}\), \(C_0 = \text{initial dye concentration (mg l}^{-1}\)), \(C_e\) or \(C_t = \text{residual dye concentration (at equilibrium or time ‘t’) [30, 31]}\).

2.5. Statistical analysis

Average values of triplicate experiments were considered for adsorption modelling. Solver tool of Microsoft Excel® 2019 was used for non-linear regression analysis of models. Coefficient of determination \(R^2\), residual sum square (RSS) and average relative error (ARE) were evaluated using the equations given below to determine the best models.

\[
R^2 = \frac{\sum(q_{e,\text{pre},i} - q_{e,\text{exp},\text{avg}})^2}{\sum(q_{e,\text{pre},i} - q_{e,\text{exp},\text{avg}})^2 - \sum(q_{e,\text{pre},i} - q_{e,\text{exp},i})^2}.
\]

\[
\text{RSS} = \sum_{i=1}^{N}(q_{e,\text{exp},i} - q_{e,\text{pre},i})^2.
\]

\[
\text{ARE} = 100 \sum_{i=1}^{N} \left| \frac{q_{e,\text{exp},i} - q_{e,\text{pre},i}}{q_{e,\text{exp},i}} \right|
\]

**Figure 1.** X-ray diffraction pattern of synthesised nMgO.
where $q_{e,\text{exp},i}$ and $q_{e,\text{pre},i}$ are experimentally measured and model-predicted adsorbate solid-phase concentration on adsorbent at any observation $i$, respectively. $q_{e,\text{exp,avg}}$ is the average of experimentally measured observations and $N$ is the number of observations [31–33].

### 3. Results and discussion

#### 3.1. Characterisation of nMgO

3.1.1. Powder x-ray diffraction analysis

X-ray diffraction pattern of MgO powders synthesised with different Fuel to Oxidizer (F/O) ratios are depicted in figure 1. All the diffraction planes can be readily indexed to the cubic phase of MgO and match with the standard JCPDS card 43-1022 with space group Fm3m [34, 35]. The diffraction peak positions observed are at $2\theta = 36.6, 42.8, 61.9, 74.5$ and 78.4° and these peaks can be indexed to the planes of (111), (200), (220), (311) and (222) respectively. It is interesting to note that the peak width (FWHM) of the diffraction peaks increases with increasing fuel ratio from 0.5 to 2.0. This can be attributed to the increasing number of moles of gaseous by-products in fuel-rich synthesis. As the number of moles of gaseous products increases, it dissipates the heat generated during combustion synthesis and results in reduced flame temperature [36]. Therefore, the particle growth is suppressed in fuel-rich synthesis and is reflected in XRD pattern (increased FWHM). Further, it is also observed from the XRD pattern that except for the diffracted peaks corresponding to MgO, no other peaks are identified. This suggests that the MgO formed is pure and is devoid of any other impurity crystalline phases. In order to calculate the crystallite size of the synthesised MgO, Scherrer’s equation [37–39] is used and the crystallite size is found in the range of 10–17 nm.

3.1.2. Scanning electron microscopy

Figure 2 shows the scanning electron micrographs of MgO prepared with F/O = 2.0. It can be seen that the surface morphology of the MgO is dominated by the agglomerated particles forming clusters of different
dimensions. The agglomerated particles are found to be in varying size with different sizes of voids on them. The agglomeration of particles is due to the fact that fine particles are characterised by high surface energy and agglomeration is the natural way of reducing the surface energy of the particles. Variation in the size of the agglomerated clusters is due to the uneven distribution of the heat during combustion of the redox mixture. The voids and fine pore-like structures seen are due to the evolution of the gases during combustion. The porous, agglomerated particle clusters with voids are typical of combustion of synthesis [27].

### 3.1.3. Transmission electron microscopy

Figures 3(a) and (b) depict the TEM images of MgO nanoparticles prepared with F/O = 2. TEM images clearly showed that the obtained MgO nanoparticles were crystalline in nature, with a size range of 20–40 nm. It is clear from the TEM images that MgO nanoparticles are highly agglomerated and of porous nature. This result is in reliable with the SEM results presented in the previous section. SAED patterns (figure 3(c)) shows the diffused rings with a few bright spots indicating the polycrystalline and nano size of the particles. The diffraction patterns

![Figure 4. FT-IR spectrum of nMgO, IC adsorbed nMgO and OG adsorbed nMgO.](image)

![Figure 5. Effect of nMgO dosage on adsorption of IC and OG.](image)
are indexed readily to the cubic MgO phase from the d spacing calculated. The lattice spacing \( d \) was calculated using the equation, \( r(\text{hkl}) \times d(\text{hkl}) = L\lambda \), where \( r \) represents the radius of the electron diffraction ring and \( L\lambda \) (camera constant of the transmission electron microscope) is considered 1. The d-values determined from the SAED patterns readily matches with cubic MgO and are indexed with respective (hkl) values.

3.1.4. Fourier-transform infrared spectroscopy

The infrared spectrum that determines surface functional groups and bonds can be used as supplementary data to powder x-ray diffraction data. The FTIR spectrum is given in figure 4. The band at (400–600 cm\(^{-1}\)) is attributed to stretching vibrations of Mg–O. It is seen in the samples before and after adsorption suggesting stability of MgO even after adsorption of the dyes. When spectra for unadsorbed MgO was observed, a peak at (3625–3750 cm\(^{-1}\)) was seen which is attributed to the stretching vibration of interlayer water molecules. While comparing IR spectra of unadsorbed MgO and IC adsorbed MgO, the difference in spectra was seen. Bands in the range (3000–3750 cm\(^{-1}\)) are due to \(-\text{OH}\) stretching modes of water molecules or hydroxyl groups. The sharp peak at 3687 cm\(^{-1}\) is attributed to \(-\text{C}=\text{O}\) group and peak might be due to \(-\text{C}=\text{O}\) group of IC. Thus, one may interpret the above seen due to adsorption of IC [40–43]. Comparing IR spectra of unadsorbed MgO and OG adsorbed MgO, the major difference was seen at 3687 cm\(^{-1}\). Sharp peak seen at 3687 cm\(^{-1}\) is attributed to the presence of \(-\text{OH}\) group. This peak might be due to \(-\text{OH}\) group of OG [44]. Thus, one might interpret that OG is adsorbed on MgO. The presence of the functional groups corresponding to the dyes IC and OG along with the adsorbent vibrational frequency confirms the successful adsorption of the pollutants on MgO.

3.2. Effect of adsorbent dosage

The effect of adsorbent dosage on dye removal was assessed at an initial dye concentration of 50 mg L\(^{-1}\) and by varying nMgO concentration from 0.2 to 2 g L\(^{-1}\). The effect of adsorbent dosage on dye removal efficiency for both IC and OG is shown in figure 5. As adsorbent dosage increases, removal efficiency first increases and reaches a maximum. The increased dye removal efficiency of IC and OG dye with an increase in nMgO is typically due to the availability of a large number of adsorption sites [26, 45]. Further increase in adsorbent dosage, reduced the dye uptake. Increase in the number of active sites with an increase in adsorbent concentration results in initial higher adsorption observed. However, at a higher adsorbent concentration agglomeration of adsorbent particles can exist, resulting in decreased uptake efficiency of the adsorbent [46].

3.3. Effect of pH

Influence of solution pH on the ionisation degree of dye molecules, adsorbent surface charge and surface functional groups makes it a critical parameter of analysis [47, 48]. The effect of pH on IC and OG adsorption on MgO nanoparticles was studied at different pH values ranging from 2–10 for 2 h. The influence of pH on the removal efficiency of IC and OG is as shown in figure 6(a). It is evident from the figure 6(a) that the pH has an insignificant influence on the dye removal efficiency of nMgO. This could be attributed to the formation of Mg(OH)\(_2\) during the reaction process, which can result in moderating the effect of pH [49]. The independency of pH also suggests the existence of different adsorption mechanisms such as hydrogen bonding other than electrostatic interaction [26]. The point of zero charge of the nMgO is depicted in figure 6(b) and PZC was found to be 9.3.
3.4. Adsorption isotherm

Adsorption isotherms play an essential role in the analysis of adsorption process as it explains the interaction between the adsorbate and adsorbent. Furthermore, it also provides an idea about the adsorption capacity of the adsorbent. Adsorption isotherm defines the performance of adsorbents under equilibrium conditions at a constant temperature. It depends on various physical properties (pH, temperature, and ionic strength) of the solution along with adsorbate and adsorbent [50, 51].

In the present study, adsorption equilibrium data was fitted to two-parameter and three-parameter isotherm models. The non-linear equations and description of each model are given as follows:

**Two parameter models:**

- **Langmuir isotherm:**
  \[ q_e = \frac{q_L K_L C_e}{1 + K_L C_e}, \]

- **Eundlich isotherm:**
  \[ q_e = K_F C_e^{1/n}. \]

**Three parameter models:**

- **Sips isotherm:**
  \[ q_e = \frac{q_S K_S C_e^{n_s}}{1 + K_S C_e^{n_s}}, \]

- **Redlich-Peterson isotherm:**
  \[ q_e = \frac{K_{RP} C_e}{1 + \alpha C_e^{1/\beta}}, \]

where \( q_L \) (mg g\(^{-1}\)) is saturated monolayer adsorption capacity, \( K_L \) (L mg\(^{-1}\)) is Langmuir constant, \( K_F \) (L mg\(^{-1}\)) is Freundlich constant, \( n \) is Freundlich intensity parameter, \( q_S \) (mg g\(^{-1}\)) is specific adsorption capacity at saturation, \( K_S \) (L mg\(^{-1}\)) is Sips equilibrium constant, \( n_s \) is index of heterogeneity, \( K_{RP} \) (L g\(^{-1}\)), and \( \alpha \) (L mg\(^{-1}\)) is Redlich-Peterson isotherm constants, \( \beta \) is exponent which ranges between 1 and 0 [31, 50, 52].

The values of model constants and statistical parameters are tabulated in table 1. As observed from table 1, Sips isotherm model with the highest \( R^2 \) value fitted best with experimental equilibrium data obtained for both

### Table 1. Isotherm model parameters for adsorption of IC and OG onto nMgO.

|                   | Indigo carmine (IC) | Orange G (OG) |
|-------------------|---------------------|---------------|
| **Isotherm**      | **Model parameters**| **Statistical parameters** | **Model parameters** | **Statistical parameters** |
| Langmuir         | KL, qL              | \( R^2 \), RSS, ARE | KL, qL              | \( R^2 \), RSS, ARE |
| Freundlich       | K_F, n              | 0.96, 539.65, 10.19 | K_F, n              | 0.97, 84.33, 6.79 |
| Sips             | K_S, q_S, n_s       | 0.97, 312.86, 5.67 | K_S, q_S, n_s       | 0.96, 121.6, 8.03 |
| Redlich Peterson | K_{RP}, \alpha, \beta | 0.98, 166.21, 3.49 | K_{RP}, \alpha, \beta | 0.97, 61.34, 4.4 |

Figure 7. (a) Plot of \( q_e \) versus \( C_e \) for IC. (b) plot of \( q_e \) versus \( C_e \) for OG. In both cases, a smooth line depicts model predicted values.
IC and OG. Further, the calculated theoretical adsorption capacity based on Sips model is in close agreement with experimental adsorption capacity for both IG and OG (figures 7(a) and (b)). This equilibrium information indicates that the dye adsorption process follows the Sips model indicating monolayer coverage of the dyes on the surface of nMgO. As evident from table 2, nMgO is a promising adsorbent for amputation of IC and OG from wastewater compared to the other reported nanomaterial-based adsorbents.

3.5. Adsorption kinetics
Analysis of adsorption kinetics is crucial in understanding the rate of dye uptake, thus aids in estimating the equilibrium time of adsorption. It is also vital to assess the mass transfer limitation during the adsorption process [68, 69].

The experimental kinetic data obtained at various concentration was fitted to two reaction-based viz, pseudo-first-order (PFO), and pseudo-second-order (PSO) kinetic models. The non-linear forms of these models are presented below:

\[ q_t = q_e (1 - e^{-kt}) \]  

### Table 2. Comparison of the adsorbent capacities of various adsorbents.

| Sl. No. | Adsorbent                                                                 | \( q_e, \text{mg} \cdot \text{g}^{-1} \) | References |
|---------|--------------------------------------------------------------------------|------------------------------------------|------------|
| 1.      | Indigo carmine (IC) Polyampholyte nanocomposite superabsorbent hydrogels | 320                                      | [53]       |
| 2.      | Copper–aluminium–layered double hydroxide/single-walled carbon nanotubes | 294                                      | [54]       |
| 3.      | MgO Nanoparticles                                                        | 158                                      | [55]       |
| 4.      | APAN/Fe3O4–MPA                                                           | 152                                      | [56]       |
| 5.      | Organo-palygorskite-Fe3O4 nanomaterial                                    | 135                                      | [57]       |
| 6.      | CTAB-modified TiO2 nanoparticles                                          | 106                                      | [58]       |
| 7.      | Graphene oxide/polyaniline/manganese oxide ternary nanocomposites        | 76                                       | [59]       |
| 8.      | Mg/Fe layered double hydroxide nanoparticles                             | 63                                       | [60]       |
| 9.      | Cobalt hydroxide nanoparticles                                           | 62                                       | [61]       |
| 10.     | nMgO                                                                     | 262                                      | Present Study |
| 11.     | Orange G (OG) Alumina nanoparticle                                        | 93                                       | [62]       |
| 12.     | Nano zirconia                                                            | 54                                       | [63]       |
| 13.     | CoFe2O4 Nanoparticles                                                    | 53                                       | [64]       |
| 14.     | Nanoclay                                                                  | 39                                       | [65]       |
| 15.     | Magnetic Biochar                                                          | 32                                       | [66]       |
| 16.     | Magnetic graphene oxide                                                  | 21                                       | [67]       |
| 17.     | nMgO                                                                     | 126                                      | Present Study |

Figure 8. (a) Plot of \( q_e \) versus time for different concentration of IC (b) plot of \( q_e \) versus time for different concentration of OG. In both cases, smooth line depicts model predicted values.
where $q_t$ and $q_e$ (mg g$^{-1}$) are the amount of dye adsorbed at time $t$ (min) and at equilibrium per unit weight of the adsorbent. $k_1$ (min$^{-1}$) is the PFO rate constant and $k_2$ (g mg$^{-1}$ min$^{-1}$) is PSO rate constant [11, 70].

Kinetic parameters with respect to different adsorbate concentration of IC and OG from non-linear regression are given in table 3, along with the statistical values for each model. For PFO model, $R^2 \approx 1$, while other statistical parameters were smaller. This indicates that adsorption of IC and OG onto MgO nanoparticles can be well described by PFO mechanism. The non-linear plot of PFO model for IC and OG is provided in figures 8 (a) and (b) respectively.

Further analysis of adsorption mechanism and determination of rate in influential step of IC and OG adsorption onto nMgO was carried out using Weber-Morris intraparticle diffusion model. The model equation is given in equation (14):

$$q_t = k_{id}\sqrt{t} + C.$$ 

where $k_{id}$ (mg g$^{-1}$ min$^{0.5}$) is intraparticle diffusion rate constant and $C$ (mg g$^{-1}$) is the parameter which represents the interfacial film resistance or a boundary layer effect [70–72].

The values of $q_e$ and $C$ are estimated from slope and intercept of plots between $q_e$ versus $t_{0.5}$ (figures 9 (a) and (b)) and are presented in table 4. From the model, the following observation can be arrived at: two distinct stages of adsorption are evident, initial linear phase (stage I) followed by a curved phase (Stage II) plateau. The initial linear phase witnesses $\sim 50\%$ of dye uptake and this could be attributed to the readily available adsorption sites on the surface of the adsorbent. Stage II represents slow diffusion of the adsorbed dye molecules from the surface site to the inner pores of the adsorbate Thus, while surface diffusion process is the controlling step during stage I, pore diffusion is the rate-controlling step of stage II. Similar adsorption behaviour with an initial linear phase followed by plateau has been reported in the literature. The intraparticle diffusion renders straight lines at stage I for both IC and OG. However, the intercept of these lines fails to pass through the origin, which could be attributed to different mass transfer rate in the initial and final stages of adsorption, indicating some degree of
boundary layer control which implies that intraparticle diffusion is not only the rate-controlling step. This data may be further used to learn about the slow step occurring in the present adsorption system using pore diffusion model \([73, 74]\).

3.6. Regeneration of adsorbent
The reusability of adsorbent is one of the critical parameters for practical application in terms of economic and cost-effectiveness. The reusability also is indicative of the stability of the adsorbent. After batch adsorption, MgO nanoparticles were regenerated by calcinating at 500 °C for 30 min \([75, 76]\). The regenerated MgO nanoparticles were evaluated for their dye uptake capability for 5 repeated cycles. Results obtained are shown in figure 10, and it is observed that the uptake capacity of the regenerated adsorbent for both IC and OG dyes remained higher up to 2 cycles, beyond which it is slightly reduced. The decrease in dye uptake could be attributed to the loss of the surface-active sites during adsorption-desorption processes and also due to repeated calcination. Nevertheless, these findings indicate the potential of regeneration and reuse of nMgO nanoparticles for amputation of anionic dyes.

4. Conclusion
Nano magnesia was synthesised by solution combustion method using glycerol as fuel and successfully employed as an adsorbent for the removal of IC and OG dye. The equilibrium data regressed well with Sips isotherm with a maximum adsorption capacity of 262 mg g\(^{-1}\) for IC and 126 mg g\(^{-1}\) for OC. The PFO model fitted well with kinetic data for both the dyes. The intra-particle model described that both film and pore diffusion is the rate-controlling step in the process of adsorption of IC and OG on nMgO. Regeneration studies demonstrated that the nMgO can be efficiently recycled for at least five cycles without significant loss in adsorption capacity. These results support that nMgO can be considered as a reliable, cost-effective and promising adsorbent for the removal of dye pollutants from wastewater.

### Table 4. Intraparticle diffusion model constants for adsorption of IC and OG on nMgO.

| Dye          | \(C_{0p}\) mg L\(^{-1}\) | \(K_{sl}\) | \(C\) | \(R^2\) | \(K_{sl}\) | \(C\) | \(R^2\) |
|--------------|-------------------------|-----------|------|--------|-----------|------|--------|
| **Step-1**   |                         |           |      |        |           |      |        |
| Indigo Carmine | 50                      | 9.26      | 2.96 | 0.99   | 0.60      | 43.29| 0.95   |
|              | 150                     | 22.51     | 1.03 | 0.97   | 0.74      | 122.47| 0.77   |
|              | 250                     | 32.03     | 3.77 | 0.99   | 1.93      | 161.25| 0.83   |
| Orange G     | 50                      | 3.70      | 6.62 | 0.97   | 0.49      | 20.35| 0.97   |
|              | 150                     | 9.51      | 12.99| 0.99   | 0.73      | 51.84| 0.94   |
|              | 250                     | 14.26     | 15.92| 0.98   | 0.74      | 78.39| 0.92   |

![Figure 10. Recycle stability of nMgO towards adsorption of IC and OG.](image)
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Conflict of interest

The authors declare that they have no conflict of interest.

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