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Original article

Removal of Methylene Blue Using Graphene Oxide Based Silica Coated Magnetic Nanoparticles

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ABSTRACT

Aims*. This article reported the present synthesis, characterization and application of new graphene-oxide based silica coated magnetic nanoparticles (Fe3O4@SiO2–GO) for the removal of methylene blue dye from the waste water.* ***Methods****. The newly synthesized adsorbent was characterized using FT-IR spectroscopy, field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS) and Methylene blue removal efficiency of the Fe3O4@SiO2–GO was evaluated using the external magnet to collect the sorbent and the supernatant was determined for the presence of methylene blue using UV-Vis spectroscopic analysis.* ***Results****. Optimum experimental parameters were reported to be comprises the optimum extraction time (10 minutes), initial sample concentration (5 ppm), adsorbent dosage (20 mg) and solution pH (7).* ***Conclusion****. The results showed that the newly synthesized Fe3O4@SiO2–GO is an efficient adsorbent with good potential for the removal of methylene blue from the aqueous media.*

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INTRODUCTION

Dye polluted wastewaters are becoming a massive trouble challenging the worldwide water environment. Globally, water environment has a large pollution record and is disproportionately dangerous compared to other aspects of environment [1]. Several studies have been carried out to identify and analyses how far pollution has affected water environment. For instance, a study was conducted by Hua et al., and revealed that 17 to 20% of industrial water contamination comes from textile dyeing and treatment procedures [2].

The chemicals industrial production which generates water comprising phenolic compounds of various dyes. These effluents are intensely colored and are contaminated with high concentration of organic compounds such as suspended and dissolved salts and many other recalcitrant compounds. Even small concentration of these compounds presents in effluent causes toxicity and foul odors to water [3]. For the above reasons, it is essential and urgent need for well-organized treatment of these pollutants which may cause adverse effect after their release into water surfaces. More recently, advanced oxidation procedures (AOPs) have been measured as a healthy and effective alternative for treating the polluted wastewater from dye negative impact. Dye is one of the most vital contaminants in the effluents of textile, paper, plastic, food and cosmetic productions [4]. Numerous of the manufacturing dyes are poisonous, carcinogenic, mutagenic and teratogenic [5]. It is deliberated that, over one million existing dyes of dyestuff are manufactured every year. Hence, there is a massive amount of colored waste water from these productions [6].

Methylene blue is a compound consisting of dark green crystals or crystalline powder, having a bronze-like luster. It is soluble in water or alcohol and has a deep blue color. It is a cationic compound and has a molecular weight of 319.851 g/mol and a melting point of 100 to 110 °C. It is generally used as dye in textile and other industries. The major concern regarding this dye material is that it is found to be non-biodegradable and it is essential to have an efficient technique for the treatment of wastewater contaminated with methylene blue dye [7]. The elimination of dyes from water has given much attention in monitoring water contamination. However, among a variety of methods obtainable for dye removal from water, such as adsorption, flocculation, oxidation and electrolysis, adsorption turns to be uncomplicated, effective and economical technique applied for wastewater treatment [8].

Sorption is one of the processes, being widely used for dye removal and has wide applicability in wastewater treatment. The term sorption refers to a process wherein a material is centered at a solid surface from its liquid or gaseous environment. In universal, two classes of sorption processes are available, if the draw between the firm surface and the adsorbed molecules is physical in nature, the adsorption is referred to as physical adsorption (physisorption) and if the attraction forces are due to chemical bonding, the adsorption process is called chemisorption. Carbon materials are known for their high adsorption capacity for organic compounds, and some of them, such as activated carbon, carbon nanotubes, have already been used as sorbents [9]. An innovative technology that has gained much attention is the use of magnetic materials for some separations. Magnetic separation has advantages such as its easy phase separation with aqueous solutions and its capability of treating large amount of wastewater within a short time. Magnetic nanoparticles have been demonstrated to shows some excellent applications in the removal of organic and inorganic pollutants from waste water and for the preconcentration and subsequent assay of low levels of analytes in different samples. In this regard graphene due to its honeycomb-like structure deserves particular attention. In particular, the high surface area, significant adsorption capacity, variety of benzene rings and especially rich π-π electron arrangement makes graphene a suitable candidate for dye removal in aqueous media.

Based on the literature, it was revealed that, graphene-based materials are mostly used for pesticide decontamination but their application can be extended to dye removal [10]. However, graphene-based extraction reagents suffer from some drawbacks such as hydrophobicity as well as dispersive nature in aqueous media which makes the extraction process tedious and time consuming. These drawbacks can be abridged through the functionalization of graphene with appropriate molecular frameworks. In this respect, magnetic Fe_3O_4 nanoparticles have provided important avenues to prepare new, stable and efficient extraction reagents as well as green sample preparation. SiO_2 is cheap, environmentally friendly, chemically stable and highly dispersive in liquid due to it rich O–H groups. So, generally Fe_3O_4 nanoparticles are coated with different silane derivatives via the sol–gel method to increase the surface area porosity and effective binding sites. Thus, the aim of this study was to explore the synthesis, characterization and application of graphene-based silica-coated magnetic nanoparticles (Fe_3O_4@-SiO_2–GO) for the removal of methylene blue dye, the optimum synthesis parameters and subsequently determine the efficacy of the dye removal using UV-Vis spectroscopic analysis

MATERIALS AND METHODS

Chemicals

Methylene Blue MB with Molecular formula $C_{16}H_{18}N_3SCl$, NH_3, NaF deionized water, $(NH_2)_2Fe(SO_4)_2$, $FeCl_3$, HNO_3, H_2SO_4, $KMNO_4$, H_2O_2, tetraethoxysilane (TEOS), graphite, CH_3CH_2OH and NaOH

Equipment and Measurement Parameters

Fourier transform infrared (FTIR) spectroscopy (Spectrum 400 Perkin Elmer, Waltham, MA, USA) using ATR method was recorded, Field Emission Scanning Electron Microcopy (FESEM), pH meter (HANNA instrument HI 2213), magnetic stirrer and Ultraviolet-Visible (UV-Vis) was used as the reflectance standard.

Synthesis of Graphene Oxide (GO)

Graphene oxide was synthesized from graphite using a simple oxidation technique. The natural graphite was powdered, and about 2.0 g was dispersed in a mixture of 20 mL HNO_3 (65 %) and 30 mL H_2SO_4 (97 %) for 24 h. Then, $KMnO_4$ (3.0 g) was added slowly to the mixture with continuous magnetic stirring at 50 °C for 20 h. The mixture was poured into ice (300 g) followed with the addition of 3.0 mL H_2O_2 (30 %) to produce a totally yellow product. The yellow product was diluted with deionized water (800 mL) and left overnight at room temperature until precipitate form. The supernatant was decanted, and the precipitate was washed using water until a neutral pH was obtained.

Synthesis of magnetic nanoparticles

Fe_3O_4 MNPs were prepared by using 1.7 g of $(NH_4)_2Fe\ (SO_4)_2\cdot\ H_2O$ and 3.4 g of $FeCl_3\cdot H_2O$ and mixed in 20 mL of deionized water followed by sonication and degasification for 15 min. The mixture was heated at 50 °C with vigorous stirring, followed by dropwise addition of 5.0 mL of ammonia solution (32 % v/v) until black precipitate was formed. The heat was turned off, and the mixture was continuously and vigorously stirred for 3 h. The product obtained was then washed four times with excess deionized water (200 mL) and oven dried at 80 °C for 24 h.

Preparation of Fe_3O_4@SiO_2

The most popular sol–gel synthesis method was used for coating Fe_3O_4 using silica, SiO_2. Briefly, 500 mg of the synthesized Fe_3O_4 MNPs was dispersed in 100 mL of water/ethanol mixture (1:1, v/v), followed by the addition of 5.0 mL of ammonia solution (32 % v/v) and finally 0.5 mL of tetraethoxysilane (TEOS). The mixture was shakened for 30 min and then left at room temperature for 24 h. The solid obtained was washed with 100 mL of a mixture of deionized water/ethanol (1:1, v/v) and dried to produce slightly dark brown Fe_3O_4@SiO_2.

Preparation of Fe_3O_4@SiO_2/GO

The new adsorbent (Fe_3O_4@SiO_2/GO) was synthesized using the following simple procedure. About 50.0 mL of the synthesized GO (yellow solution, ~100 mg) was transferred to a beaker, and the pH was adjusted to ~10 using NaOH (1M) and sonicated for 30 min. Next, 10 mg of the synthesized Fe_3O_4@SiO_2/GO was dispersed into the GO yellow solution with vigorous stirring for 5 h at room temperature. Then, the supernatant was decanted after the black precipitate was separated from solution using an external magnet. The black precipitate was washed two times with deionized water (300 mL) and dried at 80 °C for 24 h. Finally, the dark black powder (Fe_3O_4@SiO_2/GO) was obtained as a new product.

Dye Removal Process

About 20 mg adsorbents added in 20 mL water, and subsequently 20 mg/L methylene blue was added, pH7 (no adjustment required). Then, it was shaken for 10 min; accordingly, magnetic adsorbent was separated by using an external magnet. Lastly, supernatant was analyzed by UV is spectrophotometer at a wavelength of 664 nm.

Following the successful synthesis of the Graphene oxide-based silica-coated magnetic nanoparticles the dye removal process was explored to obtain an optimum parameter to maximized the dye removal. These parameters include the effect of pH, initial concentration, adsorbent dosage and minimum time required. In each of these parameters explored, the optimum amount was obtained and lastly in each case, the concentrations in an aqueous solution were quantified by the determination of the absorbance at a wavelength of 664 nm using a double beam UV-Vis spectrophotometer.

Adsorption Procedure

Effect of pH

The effect of pH was studied in the range of pH (3-9) at room temperature. Moreover, the pH was adjusted with HCl 0.01 M and NaOH 0.01 M using pH meter. The MB initial concentration was also fixed at 10 mg/L with dosage of 20 mg. Then, the solutions were mixed slowly with a rotary shaker at a rate of 150 rpm for 10 minutes at room temperature. Later on, the magnet was used in order to collect the material that adsorbed the MB dye.

Effect of Dosage

The effect of dosage was studied in the range of (5-60mg) at room temperature. The MB initial concentration was also fixed at 20 mg/L. Then, the solutions were mixed slowly with a rotary shaker at a rate of 150 rpm for 10 minutes at room temperature. Later on, the magnetic was used in order to collect the material that adsorbed the dye (MB).

Effect of Time

The effect of contact time was studied from the range of 1 to 40 minutes at room temperature. Moreover, the pH was adjusted at 7 using a pH meter. The MB initial concentration was also fixed at 20 mg/L with dosage of 10mg. Then, the solutions were mixed slowly with a rotary shaker at a rate of 150 rpm for 10 minutes at room temperature. Later on, the magnet was used in order to collect the material that adsorbed the dye (MB).

Effect of Initial Concentration

The effect of concentration was studied at different initial concentration of MB 5-50 mg/L at room temperature. Moreover, the pH was adjusted to 7 using pH meter. The MB initial concentration was also with dosage of 10 mg. Then, the solutions were mixed slowly with a rotary shaker at a rate of 150 rpm for 10 minutes at room temperature. Later on, the magnet was used in order to collect the material that adsorbed the dye (MB).

Analytical Methods

The concentrations of dye solutions were measured by monitoring the absorbance changes at a wavelength of the maximum absorbance. However, the amount of the dye sorbed at any time, q_t, was calculated from:

$q_t = \frac{v(c_0 - c_t)}{w}$ Equation (3.1)

At equilibrium, $q_{t=}\ q_e\ and\ c_0 = c_t$; therefore, the amount of sorbed dye q_e was calculated from:

$q_e = \frac{v(c_0 - c_e)}{w}$ Equation (3.2)

Where, c_0, c_t and c_e are the initial concentration at any time and equilibrium concentrations of the dye solution (mg/L), respectively, v is the volume of the solution (L), and w is the mass of adsorbent (g).

All in all, the dye removal percentage can be calculated as follows:

$removal\ \% = \frac{(c_0 - c_e)}{c_0} \times 100$ (3.3)

RESULT AND DISCUSSION

Synthesis Mechanism of Fe_3O_4@SiO_2/GO

Schematic diagram for the mechanism of synthesis of (A) Silica-coated magnetic nanoparticles (B) Graphene Oxide and (C) Graphene oxide-based silica-coated magnetic nanoparticles was presented in figure 1. Here, the exact mechanism, in basic condition, the OH group in silica get deporotonated and then O- can attach the epoxy group on graphene while epoxy ring is open during the mechanism

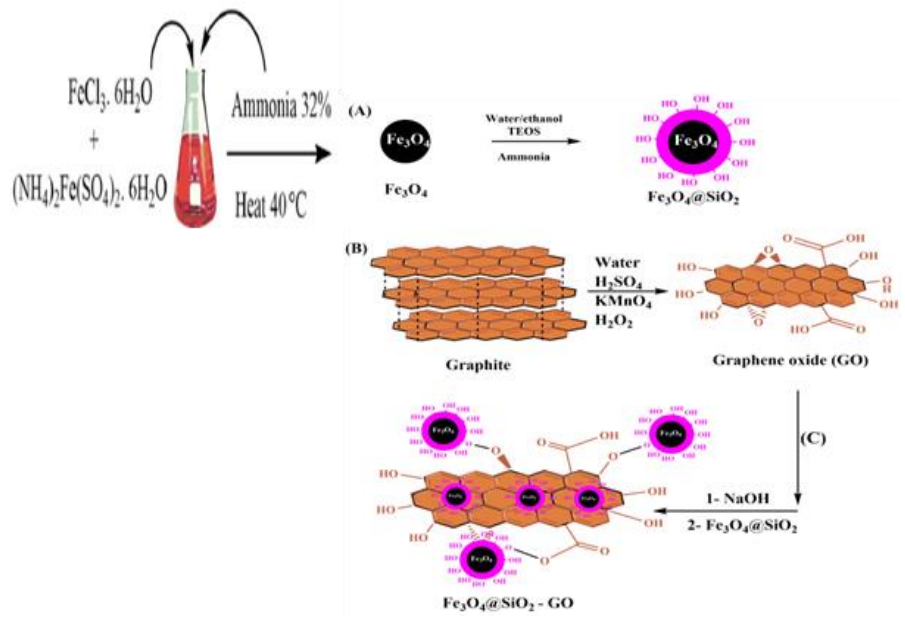

Figure 1. Schematic diagram for the mechanism of synthesis of (A) Silica-coated magnetic nanoparticles (B) Graphene Oxide and (C) Graphene oxide-based silica-coated magnetic nanoparticles

The synthesized graphene oxide-based silica-coated magnetic nanoparticles have a large amount of OH groups that can form H-bonding with MB dye and also, adsorbent have graphene with large pi-stacking that can form π-π interactions

with MB dye and hence these interactions increased the adsorption capacity of the adsorbent.

Characterization of Fe3O4@SiO2/GO

FTIR Spectral Analysis

Presence of functional groups can be confirmed by FT-IR spectroscopic analysis, as such the functionalization of graphene and immobilization of functionalized graphene onto the $Fe_3O_4@SiO_2$ were confirmed by FT-IR spectral analysis (Fig 2). Natural graphite does not contain any functional groups. However, following the functionalization process the resultant GO (Fig 2A) shows some additional bands at 3372, 1720, 1625, 1436, 1168 and 1050 cm^{-1} for O–H, C=O, C=C, C–C, epoxy groups and C–O group stretching, respectively.

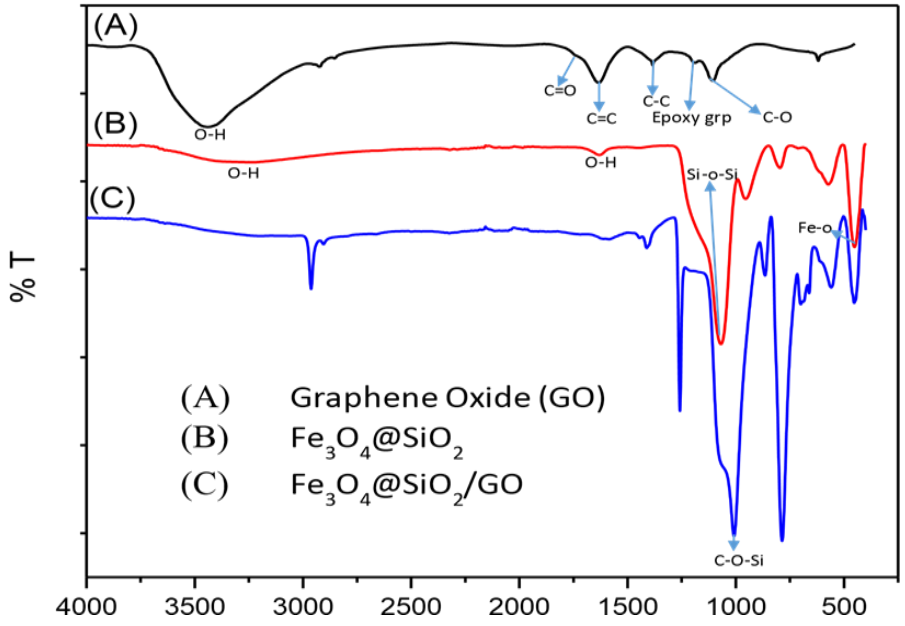

Figure 2. FTIR of (A) Graphene Oxide (B) $Fe_3O_4@SiO_2$ and $Fe_3O_4@SiO_2$-GO. Wave number (cm^{-1})

The appearance of sharp bands (Fig 2B) at 582 and 1100 cm^{-1} corresponds to Fe– O and Si–O symmetric stretching, respectively. The formation of $Fe_3O_4@SiO_2$/GO MNPs was confirmed by the appearance as well as disappearance of characteristic bands. The IR spectra of GO (Fig 2A) does not show a Si–O stretching band, however, following the immobilization with $Fe_3O_4@SiO_2$ the resultant material $Fe_3O_4@SiO_2$/GO MNPs shows a band at 1100 cm^{-1}. Additionally, during the immobilization the Si–O group intensity also reduced and disappearance of characteristic bands at 1720 and 1168 cm^{-1} for C=O and epoxy groups (Fig 2C) respectively also offer proof for the formation of $Fe_3O_4@SiO_2$/GO MNPs. Consequently, appearance and disappearance of some characteristic peaks provide qualitative evidence which confirms the immobilization of GO onto $Fe_3O_4@SiO_2$as well as formation of $Fe_3O_4@SiO_2$/GO MNPs.

Scanning Electron Microcopy

The scanning electron microscopic (SEM) is the primary tool uses for characterization of the surface morphology and fundamental physical properties. The figure below (Figure 3) illustrates the SEM micrographs of synthesized magnetic $Fe_3O_4@Si_2$ and Fe_3O_4

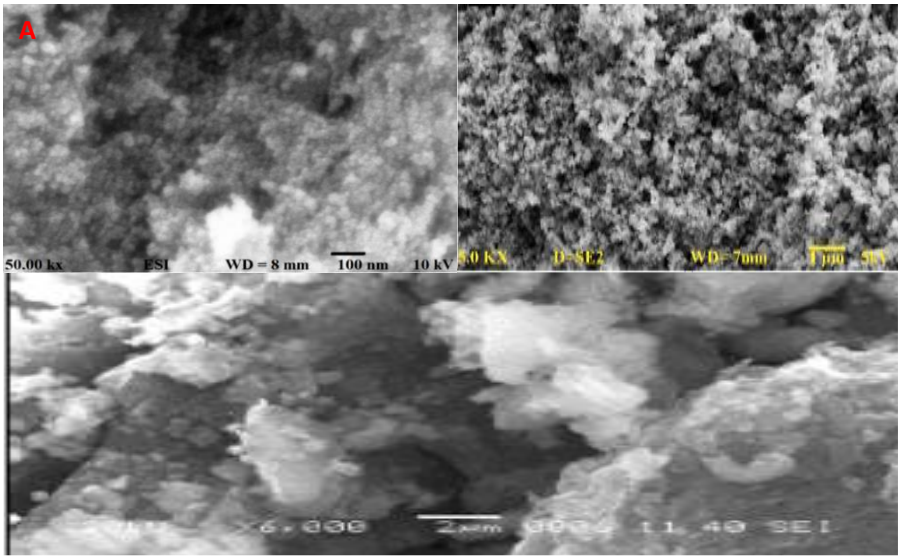

Figure 3. FESEM microscopic picture of A (Fe_3O_4) B (Fe_3O_4@SiO_2) and C (Fe_3O_4@SiO_2/GO)

The morphology of the newly synthesized magnetic graphene-based adsorbent (Fe_3O_4@SiO_2/GO) was analyzed using field emission scanning microscopy (FESEM). The white cloud-like appearance (Fig 3C) clearly shows that nanosize visible graphene sheets have been successfully immobilized with silica coated MNPs of Fe_3O_4@SiO_2. Furthermore, in order to examine the purity and elemental composition of the silica coated MNP immobilized material (Fe_3O_4@SiO_2/GO). It is clear the MNPs are smaller particle size and also, they aggregated however, when the MNPs are coated with SiO_2, the size increased and also particles separated well in the final compound, the graphene sheets appeared in bulky form. Thus, the Fe_3O_4@SiO_2/GO was synthesized successfully.

Effect of Various Parameters on Removal of Methylene Blue Dye

Effect of pH

The adsorption of dye on any specific or nonspecific adsorbent is pH dependent which is a result of the chemistry of dye in the solution and the ionization state of the functional groups of the sorbent. The pH of the solution is the most important factor, which controls the sorption of dye on the sorbent materials.

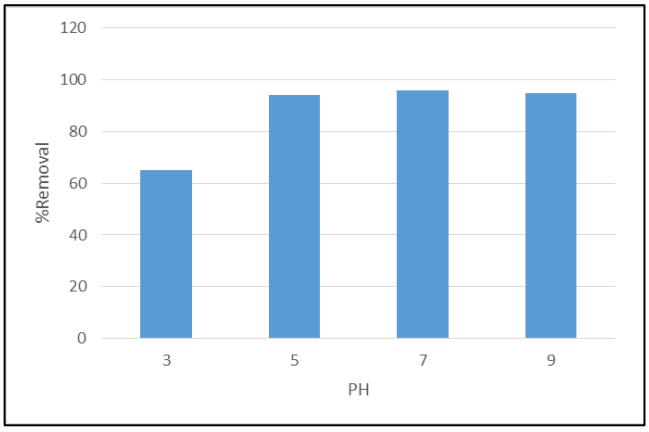

Figure 4. The impact of solution pH on the elimination of MB

In the current experimentation, the impact of solution pH on the elimination of MB was examined at diverse pH rate ranged from 3 to 9. Hence, as indicated in the figure 4, the removal of MB increases in the range of 5-9. However, above and below pH (7) there was a clear decrease in pH of the MB dye. This is because, in acidic pH the adsorbent found as

positively charge and the methylene blue dye also protonated in acidic pH So, repulsion appear between dye and adsorbent, thus efficiency decreased in pH 4-6, adsorbent and dye have both positive and negative nature thus electrostatic interaction is appeared between dye and adsorbent followed an increasing efficiency, hydrogen bonding also can form between hydrogen and electronegative atoms such as O, N, S. In basic medium pH 7 to 9, the adsorbent found as negatively charge but MB dye still have one positive charge on nitrogen atom.

Effect of Dosage

The amount of adsorbent required to treat per-unit volume of a solution is defined as a function of the extent of its reactive groups and the aspect ratio of adsorbent which directly affects the cost of adsorption.

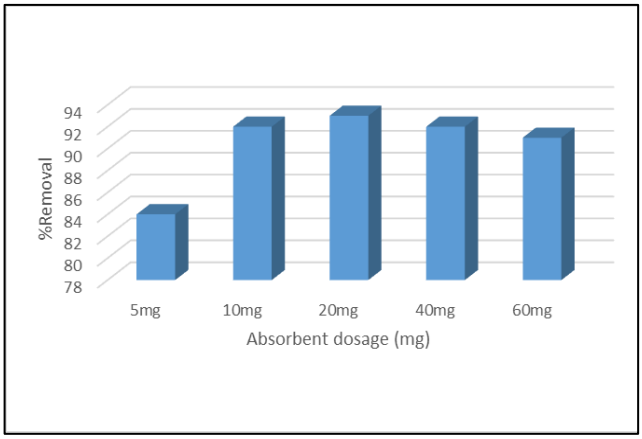

Figure 5. The impact of the adsorbent dosage

The adsorbent dosage is an imperative factor towards acquiring the measurable elimination of the MB. In the current case, as demonstrated in the above Figure 5, the best dosage is 20 mg. By increasing adsorbent dosage, the efficiency also increased sharply because adsorption at the active cites also increased rapidly, after 20 mg the efficiency increased slightly until 20 mg because there is not enough dye to saturate all the active sites.

Effect of Initial Concentration

To evaluate the effect of the amount of initial dye concentration on the adsorption efficiency of the GO for the removal of methylene blue (MB). The MB initial concentration was also fixed at (5-50) ppm with dosage of 10mg. Then, the solutions were mixed slowly with a rotary shaker at a rate of 150 rpm for 10 minutes at room temperature.

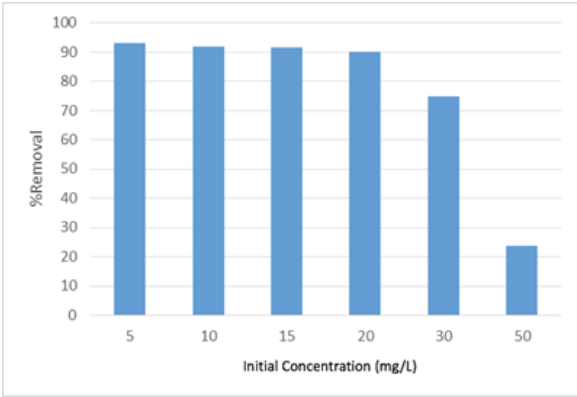

Figure 6. The effect of the amount of initial MB dye concentration

Based on the result shown in figure 6, it is possible to conclude that percentage removal of methylene blue was found to increases with decrease of the initial dye concentration. The best result regarding initial dye concentration was scored

at 5 ppm. This is because by increasing the MB dye concentration the adsorption efficiency is decreased. Because at low concentration all the MB dyes can adsorb by adsorbent, but in high concentration the adsorption active sites are not enough to adsorb all MB dyes. Thus, efficiency is decreased.

Effect of Time

In this case, the solutions were mixed slowly with a rotary shaker at a rate of 150 for 10 minutes at room temperature. Later on, the magnet was used in order to collect the material that adsorbed the dye (MB).

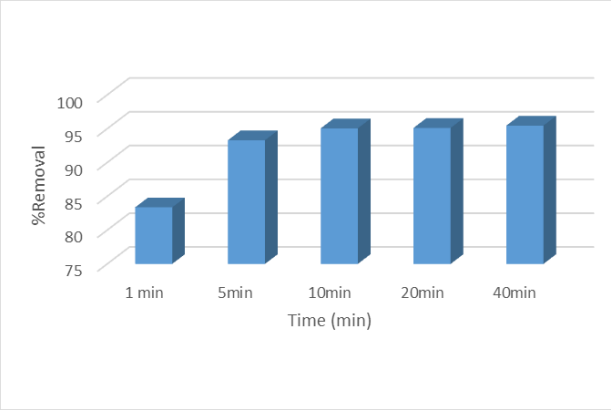

Figure 7. Effect of studied time on the removal of MB

Similarly, the adsorption efficiency was then determined by spectrophotometer. As it was illustrated in figure 7 above, the percentage adsorption was calculated and the result revealed that 10 minutes were enough to eliminate MB with 91.98%. This is because by increasing time the adsorption efficiency increased due to adsorption of more dyes onto the active sites of adsorbent. However, after the adsorption of all dyes by adsorbent the system reached the equilibrium in 10 minutes' time.

CONCLUSION

A new graphene-based silica coated magnetic adsorbent (Fe_3O_4@SiO_2/GO) was successfully synthesized and characterized using FT-IR and FESEM. The newly synthesized Fe_3O_4@SiO_2/GO was successfully applied for the removal of methylene blue from aqueous media at pH 7. The adsorption process is very fast and can reach the adsorption equilibrium within 10 min. The results clearly shows that the graphene-based silica coated magnetic adsorbent (Fe_3O_4@SiO_2/GO) can be used as an effective adsorbent for the simple and rapid removal of methylene blue from water sample

Recommendation for future research

Water curing and treatment is a field which needs a serious consideration all over the global. However, the results of every experiment associated with adsorption of methylene blue (MB) yet not clear to decide the most suitable approach to be adopted for water curing and treatment in activities linked to dye contamination. Therefore, the proposed recommendations as specified below can be used as potential areas for further research: 1). The kinetics and thermodynamics of methylene blue (MB) absorption technique must be analytically examined. 2). The current results definitely encourage more interests and efforts on the graphene based environmental tools.

Conflict of interest. Nil

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إزالة أزرق الميثيلين باستخدام الجسيمات النانوية المغناطيسية المغلفة بأكسيد الجرافين

أميمة عطية

كلية العلوم، جامعة الزاوية، ليبيا

المستخلص

الأهداف. تناولت هذه المقالة التوليف والتوصيف والتطبيق الحالي للجسيمات النانوية المغناطيسية المغلفة بأكسيد الجرافين القائمة على أكسيد الجرافين (Fe3O4@SiO2–GO) لإزالة صبغة الميثيلين الزرقاء من مياه الصرف الصحي. **طُرق الدراسة**. تم تشخيص المادة المازة المصنعة حديثًا باستخدام التحليل الطيفيFT-IR ، والمجهر الإلكتروني لمسح الانبعاث الميداني(FESEM) ، والتحليل الطيفي المشتت للطاقة(EDS) ، وتم تقييم كفاءة إزالة أزرق الميثيلين لـ–Fe3O4@SiO2 GOباستخدام المغناطيس الخارجي لتجميع المادة الماصة و تم تحديد الطاف لوجود أزرق الميثيلين باستخدام التحليل الطيفي للأشعة فوق البنفسجية. **النتائج**. تم الإبلاغ عن أن المعلمات التجريبية المثلى تشتمل على وقت الاستخراج الأمثل (10 دقائق)، وتركيز العينة الأولي (5 جزء في المليون)، وجرعة المادة المازة (20 مجم) ودرجة حموضة المحلول (7). **الخاتمة**. أظهرت النتائج أن Fe3O4@SiO2–GO المُصنَّع حديثًا هو مادة ماصة فعالة ذات إمكانات جيدة لإزالة أزرق الميثيلين من الوسائط المائية.

الكلمات الدالة. أكسيد الجرافين، أزرق الميثيلين، الجسيمات النانوية.