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Terephthalaldehyde Cross-Linked Chitosan – Graphene Oxide Nanocomposite: Efficient Adsorbent for the Removal of Cationic and Anionic Dyes from Aqueous Solutions

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ABSTRACT

Terephthalaldehyde cross-linked chitosan-graphene oxide nanocomposite (TCS@GO) has been prepared and employed as an adsorbent to eliminate a set of cationic and anionic organic dyes (Congo Red, Nigrosin, Cresol Violet, Malachite Green, Methylene Blue and Methyl Violet) in aqueous solutions in the present work. Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Brunauer-Emmett-Teller (BET) surface area analysis were used to determine the cross-linking, incorporation of the GO nanosheets, porosity and surface area value of the new adsorbent (TCS@GO). Also, the impact of solution pH on the adsorption behaviour was examined, and it was found that the dye removal efficiency depends on the nature of the dye and solution pH. The initial adsorption experiments indicated that TCS@GO is an effective adsorbent capable of eliminating both dyes tested in aqueous solution, with removal efficiency values of 66-90% depending on the structure of the dye and its initial concentration. The achieved efficiency could be attributed to the nature of the chemical structure of the cross-linked chitosan, GO functionalities, and surface morphology of TCS@GO.

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INTRODUCTION

The continued growth of industrial production capacities in such industries as food, rubber, printing and textiles has led to more complex pollution problems because of large amounts of wastewater flowing into the environment. Organic dyes are also a major environmental hazard, as they are widely released into industrial activities and contaminate water bodies [1-4].

The high chemical stability of dye molecules is extremely hazardous even in small amounts because most of them are not biodegradable [5, 6]. Consequently, it is important to remove these pollutants to ensure that the water is of the right quality to be consumed by humans. Several treatment technologies have been put in place, such as filtration, chemical oxidation, ozonation, microbiological treatment, reverse osmosis, electrochemical oxidation, membrane filtering, adsorption, and coagulation [7-9]. Adsorption is one of these strategies that are deemed as the most effective and practical due to the simplicity

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of its operation, cost-effectiveness, and high removal efficiency [10, 11]. The use of reusable and environmentally friendly adsorbent materials is also an added advantage, as it reduces the secondary pollution and increases sustainability in the water treatment processes [12-14].

Adsorption frequently occurs through intermolecular forces, such as electrostatic forces and/or valence bond forces. Adsorption can be categorised as either physical or chemical based on the type of interaction between the adsorbent and the adsorbate molecules [15]. Synthetic dyes used in the textile industry, namely, anionic and cationic dyes, are major causes of pollution to the environment due to their complex chemical structure, high stability, and non-biodegradation properties. The incessant release of these dyes into aquatic areas results in significant water pollution, negatively impacting aquatic ecosystems and presenting possible hazards to human health [16, 17].

Chitosan is a natural biopolymer that is obtained by the deacetylation of chitin. It is distinguished by numerous advantageous features, such as non-toxicity, biodegradability, and renewability. These properties make chitosan an attractive substance to use in various applications, particularly in water treatment. The amino ($–NH_2$) groups in the structure of chitosan allow chemical modification, which leads to the derivatives with better properties. Chitosan can be easily crosslinked with dialdehydes and transformed into chitosan Schiff bases via the creation of imine (C=N) linkages [18-20].

Chitosan-based adsorbents show the ability to remove both anionic and cationic dyes from water, which highlights their usefulness in dealing with various dye pollutants [21]. Despite its favourable properties, raw chitosan as an adsorbent is associated with limitations such as low specific surface area, poor mechanical and structural stability, and reduced performance in certain aqueous environments. The above limitations have led to a lot of research on chemical modification and development of composite materials to enhance the adsorption properties of chitosan. Graphene oxide (GO) has become a promising carbon-based adsorbent to use in water treatment due to its special structural and chemical properties [22]. Graphene oxide (GO) has become a promising carbon-based adsorbent to be used in water treatment due to its special structural and chemical properties. Graphene oxide (GO) consists of two-dimensional nanosheets of carbon with a large number of oxygen functional groups such as hydroxyl (C-OH), epoxy (C-OC), carboxyl (COOH) and carbonyl (C=O) groups [23, 24]. On its basal planes and edges, which enhance interactions with a variety of aqueous pollutants. The large specific surface area of GO increases the contact between the adsorbent and the contaminants [25]. The oxygen functional groups promote numerous adsorption processes, such as hydrogen bonding, π- π interactions, and electrostatic forces, which together increase the adsorption efficacy. Recent assessments and studies have shown that GO-based materials have significant adsorption capacity of harmful pollutants and that they can be further functionalised or incorporated into composite matrices to enhance their effectiveness in removing toxins in water [26,27].

In this study, the preparation of a biopolymer-based composite adsorbent to remove selected cationic and anionic dyes in aqueous solutions is reported. The synthesised substance has good adsorption properties against both types of dyes, and this could be explained by the combined effect of surface chemistry, functional groups, and structural characteristics as opposed to the use of electrostatic interactions only. The composite was made by chemically combining chitosan and graphene oxide in the presence of terephthalaldehyde as a crosslinking reagent to form a stable network structure. The availability of aromatic regions, π-electron structures, and functional groups that include heteroatoms offers several possible adsorption sites. The adsorption activity of the synthesised TCS@GO was utilized in the adsorption of a sample set of cationic and anionic dyes, and the influence of solution pH was examined using one cationic and one anionic dye, demonstrating its possible use in wastewater treatment.

MATERIALS AND METHODS

General

In this study, chitosan with a molecular weight of between 100,000 and 300,000 and a deacetylation level of over 90% was used. Graphene oxide (98% purity) and terephthalaldehyde as a crosslinking agent were used. All the compounds were taken as they were received via commercial suppliers without any additional purification. The functional groups of the composite produced were determined using Fourier transform infrared (FTIR) spectra obtained with the help of a Bruker spectrometer. Scanning electron microscopy (SEM) was used to study the morphology of the surface with a TESCAN instrument. The specific surface area was calculated by the Brunauer-Emmett-Teller (BET) technique, which was based on the nitrogen adsorption-desorption values with the help of a Quantachrome Quadrasorb SIMP analyser. A Shimadzu UV-1800 spectrophotometer was used to measure UV-vis absorbance of dye solutions before and after adsorption.

Synthesis of TCS@GO Composite

The composite was synthesised based on a previously reported method with appropriate modifications [28]. The TCS@GO was synthesised in the following manner. Firstly, 1.0 g of chitosan was added to 75 mL of 1% (v/v) acetic acid solution and stirred at 40 o C to obtain a homogenous solution. Graphene oxide (5 wt%) was dispersed in 20 mL of 1% (v/v) acetic acid solution and stirred for 1 hour to obtain maximum dispersion. This GO loading was selected as a moderate composition to enhance the surface functionality and adsorption performance of the composite while minimising possible agglomeration at higher GO contents. Graphene oxide dispersion was then introduced into the chitosan solution drop by drop, and the mixture was stirred for a further 2 hours at 40 C0 to obtain a homogeneous suspension of CS/GO.

Subsequently, the temperature was raised to 60 o C and terephthalaldehyde (0.15 g in 25 mL of tetrahydrofuran) was added in drops, and agitation was continued. After 10 minutes, the formation of a thick gel was noted; the reaction mixture was then left to cool spontaneously to ambient temperature. The product was dried and crushed using a mortar and carefully rinsed with tetrahydrofuran to remove any unreacted compounds, then washed using ammonium hydroxide and deionised distilled water and dried.

Adsorption Experiments

The adsorption experiments were carried out using TCS@GO which contained 5 wt.% graphene oxide (GO). The batch adsorption tests were carried out by adding a predetermined amount of the adsorbent (5 wt%) to 25 mL of aqueous dye solutions in 100 ml conical flasks at a constant temperature of 25 C0. The adsorption capacity of the composite was measured using three cationic dyes and three anionic dyes at the same experimental conditions.After reaching equilibrium, filtration was used to isolate the adsorbent, and the concentration of the dye in the solution was measured with a UV-Vis spectrophotometer. Dye concentrations were determined using calibration curves constructed at the maximum absorption wavelength (λ_{max}) of each dye. The corresponding λmax values are listed in Table 1. The percentage of dye removal (R%) and the equilibrium adsorption capacity q_e (mg g^{-1}) were obtained using the following

Table 1. λ_{max}values of the dyes used in the adsorption experiments.

Dye	Type	λ_{max} nm
Congo Red	Anionic	492 nm
Nigrosin	Anionic	570 nm
Cresol Violet	Anionic	580 nm
Malachite Green	Cationic	617 nm
Methylene Blue	Cationic	664 nm
Methyl Violet	Cationic	584 nm

equations [29]

$$R\% = \frac{(C_0 - C_e)}{C_0} \times 100 \tag{1}$$

$$q_e = \frac{(C_0 - C_e)\,V}{W} \tag{2}$$

where C_0 and C_e (mg L^{-1}) represent the initial and equilibrium dye concentrations, respectively; V (L) is the volume of the dye solution; and W (g) is the mass of the adsorbent.

RESULTS AND DISCUSSION

A composite adsorbent was developed using chitosan as the base and graphene oxide nanosheets (TCS@GO) nanocomposite adsorbent and terephthalaldehyde as the crosslinking reagent to develop a chemically crosslinked adsorbent. It is hoped that the addition of graphene oxide will improve the structural and adsorption properties of the crosslinked polymer by providing a range of surface functionalities that can react with cationic and anionic organic dyes. The synthesised TCS@GO composite has more versatile adsorption characteristics than the traditional chitosan-based adsorbents, which majorly depend on the electrostatic interactions. This enhancement can be ascribed primarily to the existence of aromatic domains of graphene oxide and various functional groups that facilitate π- π interactions and hydrogen bonding, with electrostatic effects playing a minor role, resulting in a higher density of active sites and high adsorption capacity.

Synthesis and identification of TCS@GO

The chemical combination of chitosan and graphene oxide to form a chitosan-based composite was done using terephthalaldehyde as a bifunctional crosslinking reagent. The amino groups of chitosan are reacted with terephthalaldehyde through the formation of Schiff

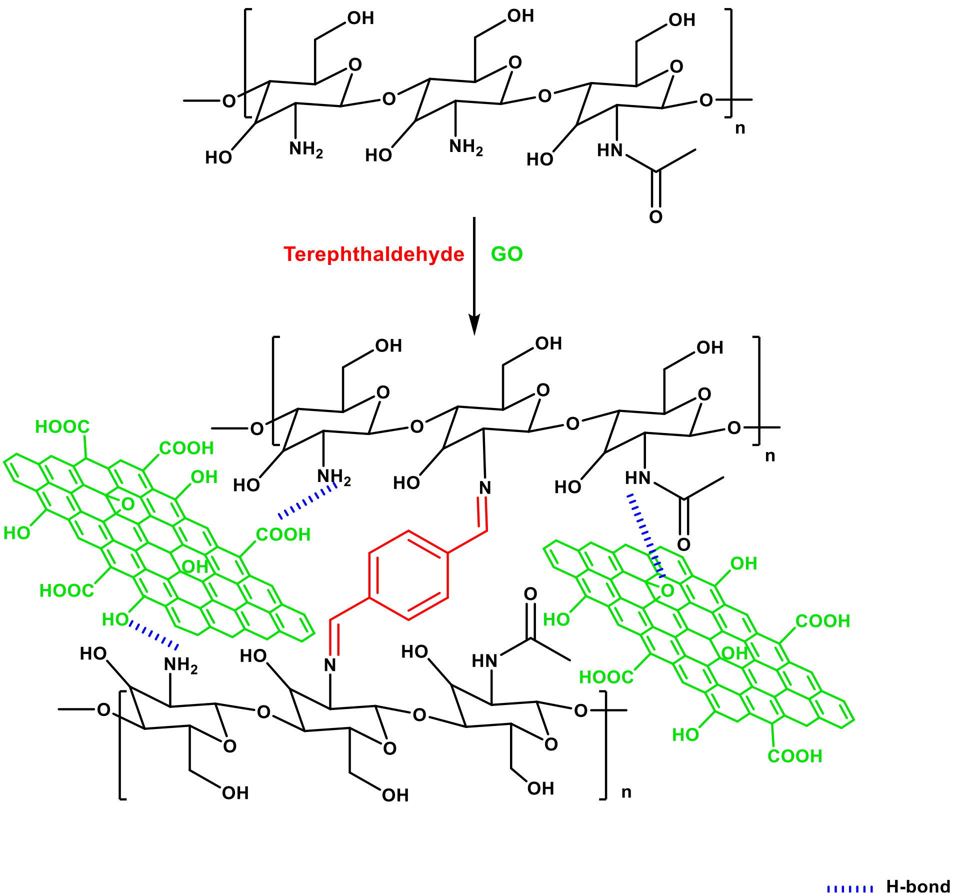

Fig. 1. Synthesis of TCS@GO.

bases, which form imine (C=N)-based bonds and a crosslinked polymer network. The incorporation of graphene oxide in the chitosan network enhances structural integrity and provides new functional sites [30]. Fig. 1 shows the synthesis and structure of TCS@GO.

Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of chitosan (CS) and TCS@GO are shown in Fig. 2. The two spectra show a wide absorption band at 3400 cm^{-1}, which can be explained by the overlapping of the stretching vibrations of O-H and N-H bonds of the chitosan backbone. The presence of graphene oxide can be inferred from the broadening and overlapping of the absorption bands in the O–H and N–H stretching region, which is consistent with the high density of oxygen-containing functional groups on the GO

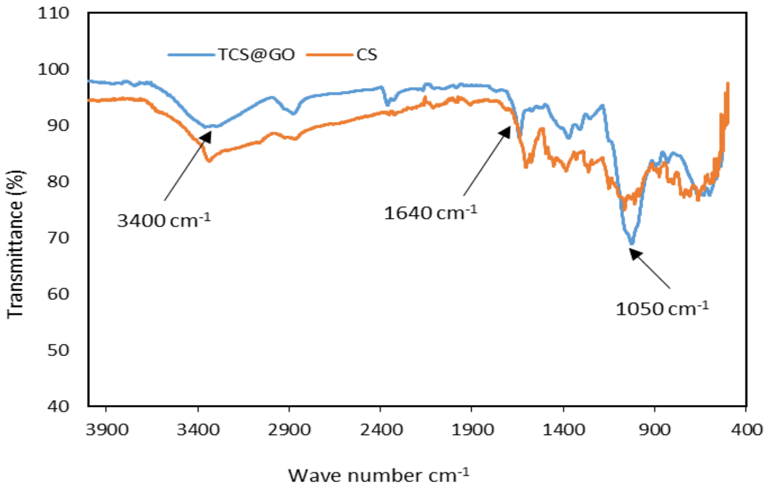

Fig. 2. The FT-IR spectra of both chitosan and TCS@GO.

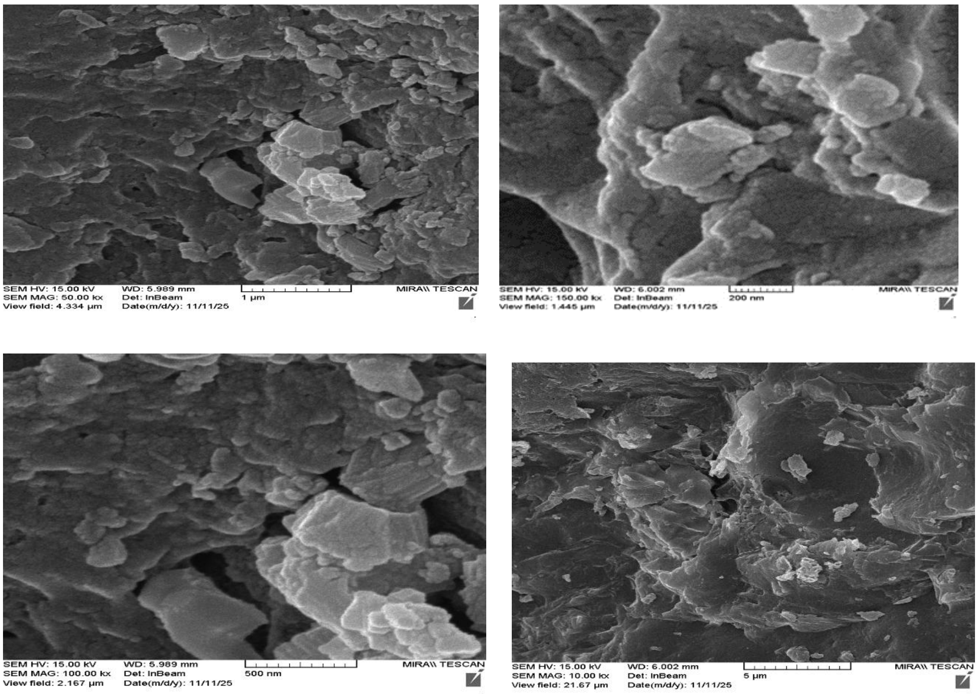

Fig. 3. FE-SEM micrograph of the TCS@GO composite.3.4.

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surface and their interactions with the functional groups of the chitosan matrix. The distinctive band detected at around 1050 cm^{-1} is attributed to the C–O stretching vibration of the polysaccharide structure, affirming that the chitosan framework stays preserved following composite production. The C=N stretching vibration at approximately 1640 cm^{-1} is associated with the synthesis of the Schiff base between the amino groups of the chitosan and terephthalaldehyde and therefore confirms the effective chemical crosslinking in the composite [31, 32]. The appearance and intensification of the bands in the area associated with aromatic C=C stretching vibrations also support the incorporation of graphene oxide into the polymeric matrix. The changes in the intensity and morphology of the observed bands are evidence of the successful integration of graphene oxide and the formation of the TCS@GO composite structure [33].

Surface Morphology Analysis

The surface morphology of the TCS@GO was assessed by using SEM. Fig. 3 shows that the micrographs have a wrinkled, heterogeneous and rough surface morphology characteristic of TCS@GO. The appearance of the irregular surface voids indicates the integration of the GO nanosheets into the cross-linked chitosan. Such a textured surface makes it more accessible and offers a large number of active sites for adsorption, which is in line with a low BET surface area (0.77 $m^2\ g^{-1}$) and trace mesoporous characteristics that prefer surface-mediated uptake of dyes [34].

Porosity Analysis (BET)

The textural characteristics of TCS@GO were analysed by using N_2 adsorption-desorption isotherms at 77 K. Fig. 4 shows the generated isotherm; it is a Type II shape with a small hysteresis loop at a higher pressure value ($p/p_0 > 0.8$). The

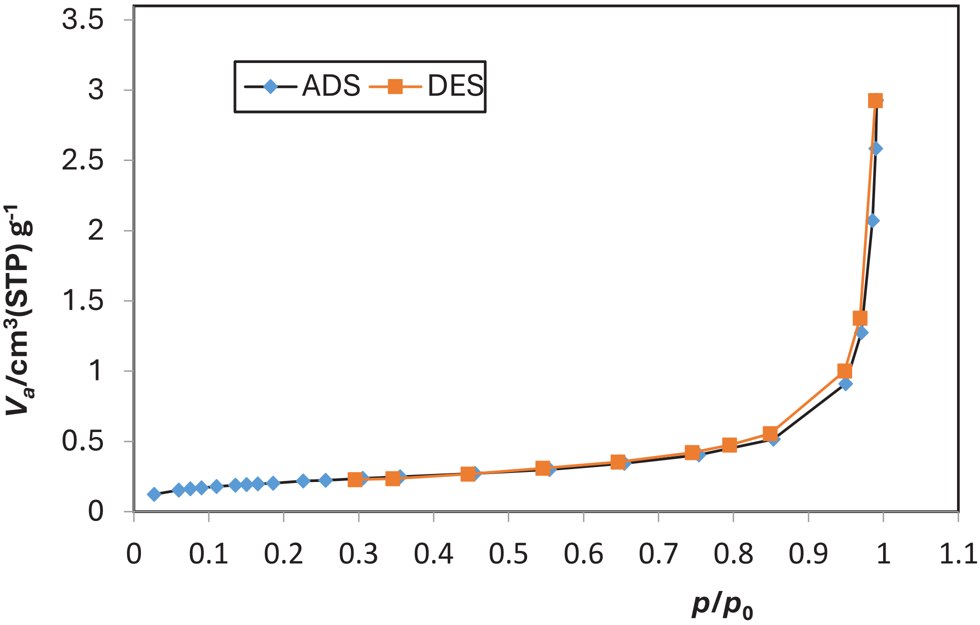

Fig. 4. The sorption isotherms of N_2 for CS/GO.

Table 2. Nitrogen adsorption-desorption analysis data for TCS@GO.

Property	Value
BET surface area	0.77 $m^2\ g^{-1}$
t-plot external area	0.65 $m^2\ g^{-1}$
Total pore volume (p/p_0 = 0.99)	0.0041 $cm^3\ g^{-1}$
BJH mesopore volume	0.004 $cm^3\ g^{-1}$
Mean pore diameter	21.38 nm
BJH peak pore radius	~8 nm

results of BET analysis showed that TCS@GO has a low specific surface area of 0.77 m² g⁻¹. The overall pore volume was determined to be 0.0041 cm³ g⁻¹, with an average pore diameter of 21.38 nm, which is a small mesoporous nanostructure. The t-plot analysis also confirmed the minor microporosity, as the external surface area of 0.65 m² g⁻¹ was obtained, as well as the BET area. These values show that the majority of the accessible surface is external as opposed to internal. The textural properties, as well as the porous morphology determined by SEM, indicate that the adsorption efficiency is not only dependent on the surface area but also on the pore structure and the existence of functional groups in the TCS@GO composite. The adsorption branch was analysed using BJH methods and indicated a narrow distribution of mesopores with a radius of about 8 nm centred but with a very low mesopore volume (V_8 = 0.004 cm³). These findings indicate that TCS@GO has a trace level of mesoporosity, and its adsorption performance is dominated by surface chemistry and rough morphology evident in SEM (Fig. 3) and not large internal pore networks [31].

Adsorption Study of the TCS@GO Composite

Adsorption Performance toward Cationic and Anionic Dyes

TCS@GO was tested in the removal of various anionic and cationic organic dyes in aqueous solutions at 25 o C with an adsorbent dosage of 0.05 g and a solution volume of 25 ml. In the adsorption studies, the selected initial dye concentrations were chosen within a moderate concentration range commonly employed in adsorption studies to evaluate the adsorption performance of the prepared composite under different pollutant levels. Table 3 summarises the TCS@GO results and shows that all the dyes studied were well adsorbed by the composite surface with a removal efficiency of between 60 and 90 per cent, indicating that the composite surface is effective and that there are active adsorption sites. Congo Red was the most effective anionic dye, with a removal efficiency of 89.66% at an initial concentration of 80 ppm, and Nigrosin and Cresol Violet had removal efficiencies of 84.84% and 66.41%, respectively.. In the case of cationic dyes, Malachite Green had the best removal efficiency, 92.39%, then Methyl Violet, 88.75%, and Methylene Blue, 70.10%. The differences in the adsorption performances of various dyes could be explained by the differences in molecular size, structure, and nature of functional groups that influence the interactions of the dyes with the TCS@GO surface.

The new composite (TCS@GO) can efficiently adsorb both cationic and anionic dyes because of different adsorption mechanisms. Electrostatic interactions are relevant under appropriate conditions, although non-electrostatic interactions substantially influence the overall adsorption behaviour. The interactions encompass π–π interactions between the aromatic structures of the dyes and the graphene oxide sheets, along with hydrogen bonding between dye molecules and the hydroxyl and amino functional groups on the composite surface. All of that suggests the presence of a variety of adsorption centres, each

Table 3. Adsorption performance of TCS@GO toward different dyes.

Dye	Type	Initial concentration (ppm)	Removal efficiency (%)	q_e (mg/g)
Congo Red	Anionic	80	89.66	35.86
Nigrosin	Anionic	20	84.84	8.48
Cresol Violet	Anionic	10	66.41	3.32
Malachite Green	Cationic	15	92.39	6.63
Methylene Blue	Cationic	80	70.10	28.04
Methyl Violet	Cationic	15	88.75	6.66

of which can be incorporated in the interaction with certain dyes depending on their structural properties (availability of electron pairs, orbitals, and charged atoms).

Visual Evidence of the Adsorption Process

To complement the spectroscopic absorption results, the adsorption process was visually observed by taking pictures of the dye solutions before and after adsorption in cuvettes. The loss of colour intensity after being in contact with the TCS@GO composite was observed to be significant and provided clear visual evidence of the dye extraction of the aqueous solutions and supported

Table 4. Visual evidence of cationic and anionic dye adsorption by TCS@GO.

Cationic Days		Anionic Days	
Dye	Solution color before (left) and after (right) treatment with TCS@GO.	Dye	Solution color before (left) and after (right) treatment with TCS@GO.
Congo Red		Malachite Green	
Nigrosin		Methylene Blue	
Cresol Violet		Methyl Violet	

Table 5. The effect of pH on the removal percentage of Nigrosin and Malachite Green by TCS@GO at 298 K.

	Removal efficiency (R%)	
pH	Nigrosin	Malachite Green
3	97	73.17
5	90.24	93.12
7	89.06	94.23
10	80.10	96.38

the UV-Vis spectrophotometric results.

Effect of pH on adsorption

The adsorption performance of the synthesised TCS@GO composite with respect to solution pH was measured using one cationic dye (Nigrosin) and one anionic dye (Malachite Green) at a pH of 3-10 as shown in Table 5 and Fig. 5. In the case of Malachite Green, the removal efficiency rose with pH, with the highest removal efficiency of 96.38% at pH 10 and 73.17% at pH 3. This behaviour may be explained by the fact that increasing the pH causes a reduction in the surface protonation, thereby decreasing competition with the hydrogen ions and increasing the interaction of the cationic dye molecules with the available adsorption sites on the composite surface. Conversely, Nigrosin was more efficient in its removal under acidic conditions, with an optimum removal of 97% at pH 3, then a slow decline to 80.10% at pH 10. This trend may be associated with changes in dye ionisation and decreased electrostatic attraction under alkaline conditions. The zero charge point (pHzpc) of the TCS@GO composite was found to be about 7.2 both with Nigrosin and Malachite Green (Fig. 6A and B). At lower pH values, the surface of the composite is dominated by positive charges, but at higher pH values, it is dominated by negative charges. Even though the adsorption process is aided by electrostatic interactions

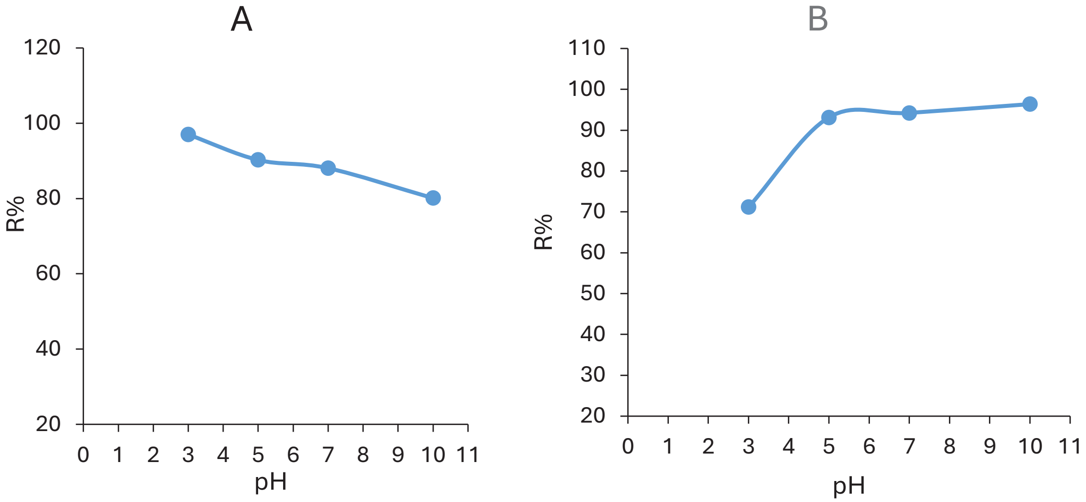

Fig. 5. The impact of pH on the adsorption of Nigrosin (A) and Malachite Green (B).

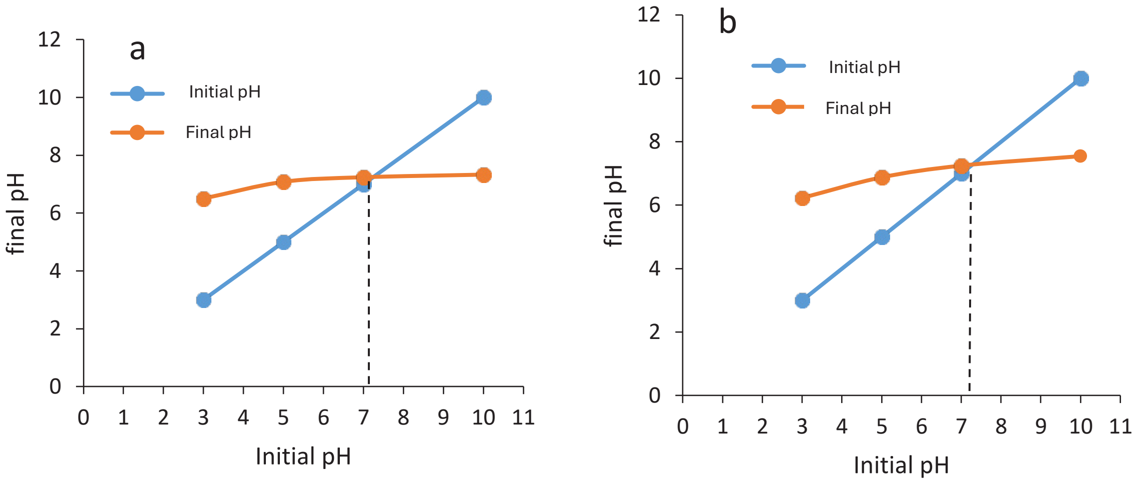

Fig. 6. The pHzpc calculated from the adsorption of Nigrosin (A) and Malachite Green (B).

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between the composite surface and the dye molecules, the fact that the removal efficiencies of the two dyes are relatively high at all pH values indicates that non-electrostatic forces like π-π interactions and hydrogen bonding are also involved in the adsorption mechanism of the two dyes [35, 36].

CONCLUSION

Terephthalaldehyde-cross-linked chitosan/graphene oxide nanocomposite (TCS@GO) was prepared and tested effectively as an organic dye adsorbent for the cationic and anionic organic dyes of aqueous solutions. Structural and surface characterisations established a successful creation of an unchanging cross-linked network and integration of graphene oxide into the chitosan framework. Although the specific surface area was rather low, the composite demonstrated excellent dye removal efficiencies, with a maximum of about 90%, which underscored the strong involvement of surface functional groups and non-electrostatic interactions, such as π–π stacking and hydrogen bonding. The findings indicate that surface chemistry is more of a determinant of adsorption performance as compared to surface area. By and large, the synthesised TCS@GO composite has great potential in wastewater treatment processes using various dye contaminants as a versatile and sustainable adsorbent.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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