Chitosan-Graft-Sodium Alginate Encapsulated Vanadium Pentoxide (V$_2$O$_5$) Nanocomposite for Adsorption and Antimicrobial Studies

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ABSTRACT

A hybrid vanadium pentoxide (V$_2$O$_5$)/chitosan-graft-sodium alginate nanocomposite was synthesized via the graft co-polymerization technique. The nanocomposite was characterized by X-ray diffraction analysis, Fourier transform infra-red spectroscopy, scanning electron microscopy and transmission electron spectroscopy. Batch studies were employed for the adsorption of model methyl violet onto V$_2$O$_5$/chitosan-graft-sodium alginate from aqueous solution. The adsorption best suited the Langmuir isotherm and pseudo-second order kinetic model. The positive value of the change in enthalpy, $\Delta H^\circ$ (6.62 kJ/mol) established an endothermic process. The negative values of the free Gibbs energy, $\Delta G^\circ$ (-4.19 to -4.87 kJ/mol.K) within the range of temperatures of 300 K to 318 K, indicated that the adsorption process was thermodynamically favourable and spontaneous. The value of the change in entropy, $\Delta S^\circ$ (0.035 kJ/mol) revealed a randomness in the adsorption process. These outcomes show a potential use of V$_2$O$_5$/chitosan-graft-sodium alginate composite for the removal of organic dyes from polluted water.

Keywords

Adsorption, Chitosan, Sodium alginate, Graft copolymerization, V$_2$O$_5$.

Introduction

Safe water for human consumption and other uses is becoming increasingly scarce in recent times. This is due to increasing demand of water resources and contamination by natural and anthropogenic means [1]. Industries such as textile, dyeing, paint and leather tanning are major contributors of the release of wastewater [1,2]. The wastewater released after the dyeing and finishing processes from textile plants is considered the most contaminating of all the industrial sources, taking into account the magnitude and composition of the released effluents [3-6]. As demand for textile products is rising, there is a proportionate increase in effluent output as well. The use of synthetic dyes in the
manufacture of textile products has contributed greatly in recent
times to the source of water contamination. The effects of these
pollutants, even if they are smaller than < 1 mg/L in water, can
seriously affect the consistency and clarity of most water bodies
[7]. Some of these dyes are mutagenic and highly toxic, reduce
light penetrations and subsequent photosynthetic activity. This
affects the oxygen supply necessary for aquatic life survival [8,9].
Dyes are engineered to resist biodegradation in such a way that
they survive for a considerably long time in the climate [10].

Several wastewater remediation methods such as coagulation,
chemical oxidation, membrane separation and adsorption have been
employed over the years to remedy the wastewater contamination
[11]. However, among these methods, adsorption presents itself as
advantageous due to it being effective, economical and ease of use.

Polymeric nanoparticles have generated considerable attention
due of their distinctive properties and ease of surface modification
[12,13]. Incorporation of nanoparticles within the polymer matrix
enhances the surface area and improves the binding capabilities [14].

Vanadium pentoxide (V$_2$O$_5$) is a useful ceramic that has spurred
massive investigation in the use of optical switching devices,
catalysis, sensors, etc. [15–17]. Due to its low isoelectric point (1-1.5)
in aqueous solution, its surface becomes negatively charged
and can be used to attract cationic dyes [18].

Alginate is an environmentally friendly and biocompatible
polymer having numerous applications [19,20]. It is a naturally
polyanionic polysaccharide obtained from brown sea weed and
comprises of alternating units of 1,4-linked β-D-mannuronic
and α-L-guluronic acid [21]. However, alginate has the tendency
to form films whose water resistance is deficient owing to the
presence of hydroxyl and carboxylic groups [21]. To improve the
properties of alginate, it is crosslinked with metal ions such Ca$^{2+}$
or polyamines like chitosan which can network with it to produce
insoluble polymers [21].

Chitosan is a cationic polysaccharide with biodegradability,
biocompatibility and bioabsorbable properties [22]. It is derived
from the deacetylation of chitin [23]. Chitosan and alginate have
been reported as excellent biomaterials for numerous applications
[1,21,24-26]. Literature has shown that no work has been done on
the synthesis of V$_2$O$_5$ impregnated chitosan-graft- sodium alginate
nanocomposite for dye adsorption.

Herein, this work reports a novel V$_2$O$_5$/ chitosan graft sodium
alginate nanocomposite for the adsorption of model dye, methyl
violet, from water and antimicrobial studies. The effects
of adsorption parameters were investigated and analysed by
different adsorption isotherms. Thermodynamic studies as well as
antimicrobial studies were also conducted.

Materials and Methods
Materials
All chemicals utilized were of analytical grade and used without
further treatment. Sodium alginate (SA), chitosan (Ch), acetic acid,
oxalic acid, glycerol, methyl violet (MV) and vanadium pentoxide
(V$_2$O$_5$) were procured from Sigma-Aldrich, Germany.

V$_2$O$_5$ nanoparticle (NP) synthesis
V$_2$O$_5$ (3.6 g) powder was added to 20 ml deionized water with
constant stirring and 2 ml of glycerol was added whilst on a
magnetic hot plate stirrer. Oxalic acid (7.6 g) was then added and
the resulting mixture heated at 80°C for 3 hours. The mixture was
filtered, washed with deionized water and dried at 100°C overnight
and further heated at 200°C for 5 h in an electric oven. The dried
precipitate was ground and stored for further use.

Preparation of V$_2$O$_5$/chitosan-g-sodium alginate (V$_2$O$_5$/Ch-g-
SA) nanocomposite
One (1) ml of (1% v/v) acetic acid was added to 100 ml volumetric
flask and topped to the mark with deionized water. This solution
was added to chitosan (1 g) under continuous stirring. V$_2$O$_5$ (0.5 g)
nanoparticle was added to the solution and stirred for an additional
30 minutes. Sodium alginate (1 g) was mixed with 100 ml of
deionized water and stirred till it dissolved. The sodium alginate
solution was then added dropwise to the chitosan-V$_2$O$_5$ mixture
until a precipitate was formed. The resulting mixture was stirred
for 30 minutes, sonicated and washed with deionized water to
remove homopolymers. The resulting precipitate formed was then
dried at 80°C in a hot air oven and pulverized for subsequent use.

Characterization
V$_2$O$_5$ NP and V$_2$O$_5$/Ch-g-sA nanocomposite were characterised
using various analytical techniques such as fourier transform
infrared spectroscopy (FTIR), x-ray powder diffraction (XRD),
screening electron microscopy (SEM), transmission electron
microscopy (TEM) and ultraviolet-visible (UV-Vis) spectroscopy.

Equilibrium Adsorption
The experiments were carried out at room temperature (300 ±
1 K) except for the analysis of the impact of temperature studies.
Appropriate mass of V$_2$O$_5$/Ch-g-SA nanocomposite was added to
50 ml of methyl violet (MV) solutions and shaken on a mechanical
shaker at 150 rpm. Certain quantities of the dye solution (10
ml) were taken for each sample after a predetermined time. The
residual MV concentration in aqueous solution was measured
using a Shimadzu (UVmini-1240) UV-Visible spectrophotometer
at an absorbance of 583.5 nm. The following equations were
employed to determine the amount of residual dye:

\[
R.E \% = \left( \frac{C_i - C_e}{C_i} \right) \times 100
\]

and

\[
Q_e \ (mg/g) = \frac{C_i - C_e}{M} \times V
\]

where $C_i$ is the initial MV concentration (mg/L), $C_e$ is the
equilibrium concentration (mg/L), M is the mass of the adsorbent
(g) and V is the volume of MV (L).

The impact of varying mass of V$_2$O$_5$/Ch-g-SA was conducted by
varying the mass between 0.05 g and 0.30 g using 50 ml of 100
mg/L of MV solution. The contact time impact on MV adsorption
was investigated using 0.05 g /50 ml of 50 mg/L MV solution. The influence of initial MV concentration was done within a range of 60 to 100 mg/L using 0.05 g adsorbent. The effect of pH was studied by varying pH of MV solution between 2 and 10. The impact of temperature was examined between 300 and 318 K using a MaxQ 8000 incubator shaker with 0.05 g / 50 ml solution.

**Adsorption Isotherms**

The data obtained was fitted to the Freundlich, Langmuir and Dubinin-Radushkevich (D-R) adsorption isotherm models. Langmuir isotherm model assumes a homogenous surface adsorption [27]. The linear form is:

\[
\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{q_m K_L}
\]

where \(C_e\) is the residual dye concentration at equilibrium (mg/L), \(q_e\) is the amount of MV removed at equilibrium per unit mass (mg/g), \(q_m\) is the monolayer adsorption capacity of the adsorbent and \(K_L\) is the Langmuir constant (L/mg).

The Freundlich isotherm assumes a multilayer adsorption on a heterogeneous surface [28]. The linear equation is:

\[
\log q_e = \frac{1}{n} \log C_e + \log K_F
\]

where \(q_e\) is the quantity of dye adsorbed at equilibrium per unit mass (mg/g), \(C_e\) is the residual concentration at equilibrium (mg/L), \(K_F\) is the adsorbent capacity (mg/g)(L/mg) and \(1/n\) is the heterogeneity factor. A linear plot of \(\log q_e\) against \(\log C_e\) determines the value of \(1/n\) and \(K_F\).

Dubinin-Radushkevich (D-R) isotherm is used to ascertain the mean free energy and nature of adsorption as to whether it is physical or chemical in nature [28]. The linear form is:

\[
\ln q_e = \ln q_{DR} - \beta \varepsilon^2
\]

where \(q_{DR}\) is the D-R maximum monolayer adsorption capacity (mg/g), \(\beta\) is the activity coefficient (mol\(^3\)F) related to the mean adsorption energy and \(\varepsilon\) can be estimated using the relation:

\[
\varepsilon = \frac{RT}{C_e} \ln \left(1 + \frac{1}{C_e}\right)
\]

where \(R\) is the molar gas constant (8.314 J/mol/K), \(T\) is the temperature (K) and \(C_e\) is the residual concentration of the adsorbate at equilibrium (mg/L). The mean free energy, \(E\), can be determined using:

\[
E = \frac{1}{\sqrt{2\beta}}
\]

\(B\) and \(q_{DR}\) values can be obtained from a plot of \(\ln q_e\) against \(\varepsilon^2\).

**Adsorption Kinetics**

The pseudo-first order and pseudo-second order kinetics were used in this study. The pseudo-first order kinetic model is applicable at the initial stage of adsorption when the uptake rate is relatively fast [29]. The linear equation is:

\[
\log(q_e - q_t) = \log q_e - k_1 t
\]

where \(q_t\) and \(q_e\) are the amount of dye uptake at time \(t\) and equilibrium respectively (mg/g), \(k_1\) is the pseudo-first order constant.

The pseudo-second order kinetic model is applicable over the entire adsorption process [29,30]. The linear equation is:

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t
\]

where \(q_e, q_t\) and \(t\) are defined earlier, \(k_2\) is the pseudo-second order constant.

**Adsorption Thermodynamics**

Change in enthalpy (\(\Delta H^o\)), change in Gibbs free energy (\(\Delta G^o\)) and change in entropy (\(\Delta S^o\)) were calculated from the following relationship:

\[
\Delta G^o = \Delta H^o - T \Delta S
\]

\[
\Delta G^o = -RT \ln K_d
\]

\[
\ln K_d = \frac{q_e}{C_e}
\]

\[
\ln K_d = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT}
\]

where \(R\) is the molar gas constant (8.314 J/mol/K), \(T\) is the temperature (K), \(q_e\) is the amount of MV adsorbed at equilibrium (mg/g) and \(C_e\) is the amount of MV remaining in solution at equilibrium (mg/L). Equation (11) can be used to compute the values of \(\Delta G^o\) at various temperatures. \(\Delta S^o\) and \(\Delta H^o\) can be estimated from a plot of \(\ln K_d\) against 1/T.

**Antimicrobial studies**

Nutrient agar plates were prepared to determine the susceptibility of cultured microbes (Escherichia coli, Bacillus megaterium and Staphylococcus aureus) to both V\(_2\)O\(_5\) NP and V\(_2\)O\(_5\)/Ch-g-SA nanocomposite. Three tubes of 5 ml microbial suspension were prepared from the microbes grown in the nutrient broth. Wells were created on the nutrient agar plates and filled with 0.05 g of both the NP and V\(_2\)O\(_5\)/Ch-g-SA nanocomposite. The plates were then incubated for 24 hours at 37°C. The diameter of the inhibition by both samples were recorded and the averages taken.

**Results and discussion**

**FTIR Studies**

The FTIR study was carried out in order to establish the formation of vanadium pentoxide nanoparticles (V\(_2\)O\(_5\)) and the impregnation of V\(_2\)O\(_5\) into the graft copolymer. The IR bands of V\(_2\)O\(_5\) nanoparticle (Figure 1b), show a broad band ranging from 3600 to 2000 cm\(^{-1}\) which may be attributed to vibrations from absorbed water molecules and hydroxyl groups. The peak at 1123 cm\(^{-1}\) was attributed to V= O stretching vibrations [31] and V- O bonds were attributed to peaks < 1000 cm\(^{-1}\) [32].
In the IR spectrum of V$_2$O$_5$/Ch-g-SA nanocomposite (Figure 1a), the peak observed at 3246 cm$^{-1}$ suggested stretching vibration from O-H and N-H bonds. At 2880 cm$^{-1}$, a weak peak observed may be due to C-H stretching whereas 1596 cm$^{-1}$ corresponds to N-H bending and C-N stretching [26,33]. Also, peaks situated at 1401 cm$^{-1}$ and 1025.45 cm$^{-1}$ were assigned to carboxylate group of alginates associated with chitosan [33]. Peaks ranging from 1500 to 400 cm$^{-1}$ indicate the presence of V-O bonds hence peaks at 1123 cm$^{-1}$ and 989 cm$^{-1}$ and the shift in V-O peak position show that V$_2$O$_5$ nanoparticle was embedded in the polymer matrix of the chitosan-g-sodium alginate.

**X-Ray diffraction analyses**

The XRD patterns of V$_2$O$_5$ nanoparticles and V$_2$O$_5$/Ch-g-SA nanocomposite are shown in Figure 2. The peaks from the V$_2$O$_5$ diffractogram (Figure 2a) at 2θ=15.22°, 20.22°, 26.28°, 30.83° and 50.26° corresponds to 200, 001, 110, 400, 601 planes of crystalline form of an orthorhombic system (JCPDS 9-387) [34,35]. The sharp peaks observed in the XRD patterns indicate the crystallinity of the sample. However, the XRD pattern of the V$_2$O$_5$/Ch-g-SA nanocomposite showed the amorphous nature of the composite (Figure 2b) which is mainly from the graft biopolymers. This may be ascribed to the disruption in the close packing that arises from the grafting of alginate with chitosan [21,36-38].

Using the Scherrer’s equation, the crystalline size of the samples were calculated:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$  \hspace{1cm} (14)

where the wavelength of the X-ray (nm) is given by $\lambda$, $\beta$ is the full width at half maximum (FWHM) of the diffraction peak (radian), the angle of diffraction at the maximum intensity is given by $\theta$ and $D$ is the average crystalline size of the sample under investigation.

The average crystalline size of the materials were computed to be in the range of 29-48 nm.

**SEM and TEM studies**

The SEM and TEM images of the samples are shown in Figure 3. The SEM and TEM micrographs (Figure 3a,b) of the V$_2$O$_5$ show a rodlike shape. Similar rodlike morphology has been reported by other researchers [15,39]. The TEM image of the V$_2$O$_5$/Ch-g-SA nanocomposite (Figure 3c) showed that the V$_2$O$_5$ was impregnated in the graft copolymer because the nano rods can be seen in the graft copolymer.

**Equilibrium adsorption studies**

**Impact of contact time**

The trend observed for the contact time was identical for both Q$_e$ and R.E (Figure 4). There was a gradual increase in R.E and Q$_e$ within the first 70 minutes. The increase could be attributed to the availability of unoccupied adsorption sites within the first 80 minutes, after which the V$_2$O$_5$/Ch-g-SA surface became saturated and a subsequently no significant adsorption was observed.

**Impact of varying of V$_2$O$_5$/Ch-g-SA dose**

MV adsorption onto V$_2$O$_5$/Ch-g-SA nanocomposite was investigated by varying the adsorbent dose (0.05, 0.1, 0.15, 0.2,
Figure 3: SEM of V_2O_5 nanoparticle (a), TEM of V_2O_5 nanoparticle (b) and TEM of V_2O_5/Ch-o-SA nanocomposite (c).

Figure 4: Impact of contact time on MV adsorption.
0.25, 0.3g/50 ml) in the dye solution while keeping other variables constant (Figure 5). An increase in removal efficiency was observed from 83.75% to 87.91% which may be attributed to the availability of vacant adsorption sites. However, the decrease observed from 85.16% to 81.31% could be as a result of aggregation of the adsorbent molecules leading to the observed decrease. \( Q_e \) declined from 83.75 mg/g to 11.62 mg/g with an addition in adsorbent dose. This may result from the overlapping of the \( V_2O_5/Ch-g-SA \) surface area available to MV.

**Effect of temperature**

Figure 7 describes the effect of temperature on adsorption of MV onto \( V_2O_5/Ch-g-SA \) nanocomposite at different temperatures of the MV test solution. With increasing temperatures, both R.E and \( Q_e \) increased from 83.30% to 86.32% and 83.30 mg/g to 86.32 mg/g respectively. This may be ascribed to the dye molecules acquiring adequate kinetic energy as temperature increases, allowing the adsorbate to adhere to the surface of the nanocomposite. In addition, the observed data could be indicative of an endothermic process.

**Influence of initial concentration**

From Figure 6, it can be seen that the removal efficiency, R.E and quantity of dye removed per unit mass, \( Q_e \) both increased. The increase in R.E may be attributed to vacant available adsorption sites as the concentration increased. The increase in \( Q_e \) may result from the significant mass transfer driving force towards the \( V_2O_5/Ch-g-SA \) surface as concentration increased.

**Effect of pH**

The effect of pH on the removal of MV onto \( V_2O_5/Ch-g-SA \) is presented in Figure 8. The adsorption capacity was low at pH 2. This could be due to the protonation of the amino groups in chitosan in acidic pH [40]. As the pH of the solution increases, interactions between carboxylic and amino groups of the nanocomposite allow for MV uptake. Similar findings have been reported [1,40].

![Figure 5: Effect of \( V_2O_5/Ch-g-SA \) nanocomposite dose on MV adsorption.](image1)

![Figure 6: Effect of initial MV concentration on MV adsorption.](image2)

![Figure 7: Effect of temperature on MV adsorption.](image3)

![Figure 8: Effect of pH on MV adsorption.](image4)
Adsorption Isotherms
Adsorption isotherms are used to analyse the adsorbate-adsorbent interaction in the adsorption process. The data obtained from these models help to design appropriate adsorption systems. For this study, the adsorption data was fitted to the Langmuir, Freundlich and Dubinin-Radushkevich (D-R) isotherm models (Figure 9a-c). The Langmuir isotherm best suited the adsorption process ($R^2=0.9999$) compared to the Freundlich ($R^2=0.9958$) and Dubinin-Radushkevich ($R^2=0.9208$) isotherms. The monolayer adsorption capacity of $V_2O_5/\text{Ch-g-SA}$ was computed to be 77.52 mg/g and the value of the separation factor $R_L$ was 0.00975 indicating a favourable adsorption because the value lies between zero and unity. The mean free energy $E$ was computed to be 312.5 kJ/mol.

Adsorption Kinetics
The correlation coefficients $R^2$ of the pseudo-second order (0.9995) was observed to be larger than the pseudo-first order kinetic (0.8528) (Figure 10a,b). This means that the model was applicable over the entire period of adsorption. Similar findings have been reported [1,41].

Adsorption Thermodynamics
The values of $\Delta G^\circ$, $\Delta S^\circ$ and $\Delta H^\circ$ which are the thermodynamic properties that describe the adsorption process are presented in Table 1. The negative values of $\Delta G^\circ$ within the range of temperatures suggest the spontaneity and thermodynamically feasibility of the adsorption process. The positive value of $\Delta H^\circ$ confirmed the endothermic nature of the process. The value of $\Delta S^\circ$ indicated an affinity of MV species towards the $V_2O_5/\text{Ch-g-SA}$ surface and a decrease in disorderliness during the adsorption [42].

Table 1: Thermodynamic parameters of MV adsorption onto $V_2O_5/\text{Ch-g-SA}$ nanocomposite

<table>
<thead>
<tr>
<th>$\Delta H^\circ$ (kJ/mol)</th>
<th>$\Delta S^\circ$ (kJ/mol)</th>
<th>$\Delta G^\circ$ (kJ/mol.K)</th>
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<td>6.62</td>
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<td>-4.87</td>
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Antimicrobial Activity
Microorganisms such as *Escherichia coli*, *Bacillus megaterium*, *staphylococcus aureus* and *Vibrio cholera* are known to cause water borne diseases. These microorganisms cause typhoid fever, diarrhea, dysentery, gastroenteritis and cholera. In this work, $V_2O_5$ nanoparticle and $V_2O_5/\text{Ch-g-SA}$ nanocomposite were investigated and tested on *Escherichia coli*, *Bacillus megaterium* and *staphylococcus aureus* which are Gram-positive and Gram-negative bacteria. The results (Figure 11) indicated that all the

![Figure 9: Langmuir (a), Freundlich (b) and Dubinin-Radushkevich (c) isotherm plots of MV adsorption onto $V_2O_5/\text{Ch-g-SA}$ nanocomposite.](image-url)
samples had antimicrobial activity against these Gram positive and Gram-negative bacteria with more inhibition against *Staphylococcus aureus*. In all the microbes tested, V$_2$O$_5$ showed higher inhibition zones as compared to V$_2$O$_5$/Ch-g-SA [21].

![Figure 10](image_url) Pseudo-first order (a) and pseudo-second order (b) kinetic plots of MV adsorption onto V$_2$O$_5$/Ch-g-SA nanocomposite.

**Figure 10**: Pseudo-first order (a) and pseudo-second order (b) kinetic plots of MV adsorption onto V$_2$O$_5$/Ch-g-SA nanocomposite.

**Conclusion**

V$_2$O$_5$ NP and V$_2$O$_5$/Ch-g-SA nanocomposite was synthesized successfully. The study revealed that the material could be employed as an adsorbent for methyl violet (MV) dye uptake from wastewater. The FTIR spectra established the formation of V$_2$O$_5$ and its encapsulation into chitosan-graft-sodium alginate matrix. SEM and TEM confirmed the morphology of V$_2$O$_5$ to be a nanorod. The XRD pattern of V$_2$O$_5$ matched the crystalline orthorhombic phase of V$_2$O$_5$. Batch adsorption tests indicated that the adsorption of MV onto V$_2$O$_5$/Ch-g-SA nanocomposite was dependent on adsorption kinetics such as pH, adsorbent dosage, temperature, contact time, initial dye concentration and temperature. The optimum MV removal dose was 0.1 g. The optimum pH and contact time for the effective removal of MV dye in water was in the weak acidic range (pH 6) and 80 minutes respectively. The Langmuir isotherm model and the pseudo-second order model best suited the adsorption data. The thermodynamic parameters inferred that the adsorption of MV onto V$_2$O$_5$-copolymer was a spontaneous and thermodynamically feasible process. Antimicrobial studies showed that the material has an activity against Gram positive and Gram-negative bacteria with more inhibition towards *Staphylococcus aureus*.

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**Data accessibility**

The data collected in this research article is from the experiment. The information used to support the results of this study is included in the article and can also be accessed upon request from the corresponding author.

**References**


