Enhancing the optical properties of chitosan, carboxymethyl cellulose, sodium alginate modified with nano metal oxide and graphene oxide

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Abstract
The solution casting method was utilized to synthesize nanocomposite films of chitosan (Cs)/CuO, Cs/graphene oxide (GO), carboxymethyl cellulose (CMC)/TiO2, CMC/GO, sodium alginate (Na-Alg)/TiO2, and Na-Alg/GO owing to their various applications. The influence of CuO, TiO2 and GO concentration on the optical properties of Cs, CMC and Na-Alg films was studied by UV-Vis Spectroscopy. The absorbance of Cs, CMC and Na-Alg increased with increasing the filler’s content, thus reflecting the dependence of Cs, CMC, and Na-Alg increased with increasing the filler’s content, thus reflecting the dependence of Cs, CMC, and Na-Alg properties on the nanofiller’s content, and confirming the interactions between individual polymers and CuO, TiO2 and GO nanoparticles. The obtained absorbance values were then used to calculate the absorption coefficient and, hence, the optical band gap values. The characteristic absorption bands of CuO and TiO2 underwent a red-shift by increasing the filler’s content. The results showed that the optical band gap of Cs, CMC, and Na-Alg decreased with filler’s content, and they possessed 1, 2 and 2 band gaps respectively. The obtained results recommended that Cs, CMC, and Na-Alg nanocomposites can be used in optoelectronic devices.

Keywords Chitosan · CMC · Sodium alginate · TiO2 · Graphene oxide · Optical properties

1 Introduction
The need to fabricate new safe materials is growing due to steady increase in environmental pollution. Therefore, the development of new environmentally friendly materials has become popular (Guido et al. 2003; Xu 2005; Vilela et al. 2018). Using natural composite materials, not only means utilizing environmentally friendly and safe materials, but also allowing the best use of the characteristics of the raw materials by compensating for defects in individual parts and increasing its practical value. The negative influences of utilizing plastic bags, for example, can be diminished by utilizing composite materials in food packaging (Kunz 2013; Qiu et al. and Tang et al. 2016).
Careful selection of the raw materials makes it possible to produce composite films with specific characteristics such as biodegradability and antibacterial properties, which is effective for almost all applications (Tharanathan 2003; Li et al. 2015). Over the past few decades, there has been a growing interest in the invention and development of metal ion-polysaccharide complexes to be used in many popular applications such as metal collection, water purification, formation of new catalysts, and many other industrial applications (Khalil et al. 2020).

Chitosan (Cs), a cationic polysaccharide composed of (1, 4) linked 2-aminodeoxy-βD-glucans, is the deacetylated form of chitin. Cs is characterized by its antibacterial activity, non-toxicity, biocompatibility, biodegradability, antioxidant activity, and film-forming ability. The protonable amino group present along the D-glucosamine residues of Cs plays a key role in explaining most of its favorable properties especially in the field of biomaterials such as mucoadhesion, haemostatic activity, permeation-enhancing property, and analgesic effect (Croisier F. and Jérôme 2013). Cs films are selectively permeable to sense toxic gases such as CO₂ and CO (Othan 2019; Xu et al. 2021). Hussein et al. (2021) prepared nanocomposite films based on Cs and titanium dioxide (TiO₂) nanoparticles utilizing the solution casting method.

Cellulose and its derivatives such as cellulose acetate and carboxymethyl cellulose (CMC) have a wide range of applications including food packaging, thickener, plasticizer, pharmaceuticals, cosmetics, and optoelectronic devices. CMC is a plant-derived cellulose derivative, a polyanionic polymer and a water-soluble polysaccharide (Yu et al. 2021). CMC possesses low conductivity and must be salt doped to increase its conductivity for use in energy storage devices. El-Naggar M.E. et al. (2021) fabricated a nanocomposite membrane material based on silicon oxide nanoparticles and poly (3,4-ethylene dioxythiophene) incorporated into cellulose acetate matrix. Ezzat et al. 14 studied the dielectric properties of cellulose acetate-molybdenum trioxide nanocomposites. Badry R. et al 2020 studied the electronic properties of CMC blended with polyethylene oxide and acetic acid theoretically.

Sodium alginate (Na-Alg) is a natural polysaccharide extracted from brown algae. From a molecular point of view, Na-Alg is a linear, water-soluble, biodegradable, and biocompatible polysaccharide composed of (1–4) linked β-d mannonate (M) and α −l guluronate (G) units. The presence of carboxyl A group in the Na-Alg structure provides electronegative properties to other polymers in aqueous solution (Essifi et al. and Zhao 2021). As Na-Alg is biocompatible, biodegradable, and non-toxic, with its ability to undergo facile synthesis of fine particles, as well as its gelling properties when cross-linked with divalent ions such as calcium and iron cations, it is widely applied in food and pharmaceutical industries (Zhao et al and Yang 2021; Berraouan et al 2017).

It must be noted that polymer membranes are characterized by their electrical insulation properties as well as electrical conductivity, hence, playing important roles in hole/electron transport in optoelectronic applications, lithium-ion conductivity in lithium battery membranes, proton exchange membranes in fuel cell applications, and thermal insulation (Robeson 2012). Additionally, polymer-based solar cells make use of an electron-donating polymer together with an acceptor that may be another polymer or a small molecule which could be a fullerene derivative or an inorganic nanoparticle such as in hybrid solar cells (Grimsdale and Jacob 2012). Polymer membranes with good electronic and optical properties can also provide an effective protection to the semiconductor materials used in optoelectronic devices which are sensitive to environmental conditions (Hamui et al 2018).

Transition metal oxides are the most interesting semiconductors because they can be applied in storage devices, electronics, and catalysis (Rashad 2020; El-Shamy et al. 2017).
Incorporation of metal oxide nanoparticles into polar polymers alters many properties of the polymer. In addition, they can also be utilized in electronics, optics, and catalysis (Jamróz 2019; Taghizadeh 2020; Badry 2021-c).

Cs, CMC, and Na-Alg were selected for this study because of their favorable properties and capability of forming membranes. In this study, copper oxide (CuO), TiO$_2$ and graphene oxide (GO) were infused into the natural polymer matrix as dopants for enhancing the optical properties of Cs, CMC, and Na-Alg. Ultraviolet-visible (UV-Vis) spectroscopy has been utilized to report the influence of dopants on the optical properties of the polymers. Therefore, the major purpose of this work is to develop active films based on a mixture of natural polymer, and metal oxides and GO nanoparticles.

2 Materials and methods

2.1 Materials

Cs with a medium molecular weight and copper chloride were purchased from Sigma Aldrich. CMC with 2.5 x 10$^5$ g/mol molecular weight was purchased from K. Patel Chemopharma PVT, India. Na-Alg was purchased from Qualikems company, India. Sodium hydroxide (NaOH) (≥ 97%), potassium permanganate (KMnO$_4$) (99%), phosphoric acid (85%), and graphite powder were purchased from Fisher Chemical. Hydrogen peroxide (H$_2$O$_2$) (30%) was purchased from Piochem and sulfuric acid (96%) was acquired from Scharlau.

2.1.1 CuO nanoparticles preparation

CuO nanoparticles were produced using the co-precipitation method. A solution of 1M copper chloride in 100 ml deionized water was prepared at 70 °C. 2M NaOH was dissolved in 100 ml of deionized water. NaOH was then added drop wise to the copper chloride solution with strong stirring for 1 hr. The black precipitate was separated by centrifugation at 1000 rpm and washing with deionized water three times. The resulting precipitate was dried in a dryer at 80 °C for 2 hrs and calcinated at 500 °C for additional 2 hrs.

2.1.2 TiO$_2$ nanoparticles preparation

TiO$_2$ nanoparticles were synthesized using a sol-gel technique. Titanium tetra isopropoxide (Alfa, Aesar, Germany) and ethanol were mixed at 60 °C for one hour with continual stirring. The deionized water was then added a drop by drop until the milky colored precipitate appeared. The organic solvent must be completely removed and filtered. Distilled water and ethanol was used to clear the precipitate numerous times. This precipitate was used to make pure TiO$_2$ nanoparticles and left in an oven for half an hour at 100°C, then annealed for two hours at 500 °C.

2.1.3 GO preparation

GO nanoparticles were synthesized using Hummer’s method. H$_2$SO$_4$ and H$_3$PO$_4$ were mixed in a beaker in a 9:1 ratio. In another beaker, a mixture of 6 g KMnO$_4$ and 1 g graphite powder was prepared in an ice bath. The two solutions were then mixed and stirred at 50 °C for 12 hrs.
After cooling the mixture, it was poured into 1 ml of ice containing 30% H₂O₂. The precipitate was collected by centrifugation at 10,000 rpm, first washed with 200 ml of 30% HCl, then washed several times with deionized water, and finally dried overnight in a room temperature vacuum oven.

2.1.4 Cs/CuO nanocomposite preparation

0.5 g of Cs was dissolved in 50 ml of deionized water under vigorous stirring at 60 °C for 1 hr until Cs was completely dissolved and a viscous solution was obtained. CuO nanoparticles were added to Cs solution with different weight percentages (2, 4, 6 and 8 wt%) as tabulated in Table 1. The Cs/CuO solution was stirred for 2 hrs until CuO nanoparticles were dispersed completely. The solution was cast in plastic petri dishes and left to dry in air for 5 days at 40 °C.

2.1.5 CMC/TiO₂ nanocomposite preparation

0.5 g of CMC was dissolved in 50 ml of deionized water under vigorous stirring at 50 °C for 1 hr until CMC was completely dissolved and a viscous solution was obtained. TiO₂ nanoparticles were added to CMC solution with different weight percentages (2, 4, 6 and 8 wt%) as presented in Table 2. The CMC/TiO₂ solution was stirred for 1 hr, then the solution was sonicated for 1 hr for more dispersion of TiO₂ nanoparticles. The solution was cast in plastic petri dishes and left to dry in air for 5 days.

2.1.6 Na-Alg/TiO₂ nanocomposite preparation

Na-Alg solution was prepared by dissolving 0.5 g of Na-Alg in 50 ml of deionized water and stirred vigorously for 1 hr at 50 °C. After a clear viscous solution of Na-Alg is obtained, TiO₂ nanoparticles were added with different concentrations (2, 4, 6 and 8 wt%).

### Table 1 Composition of Cs/CuO nanocomposite films

| Sample | Cs (g) | CuO (g) | GO (g) |
|--------|--------|---------|--------|
| S1     | 0.50   | 0.00    | 0.00   |
| S2     | 0.49   | 0.01    | 0.00   |
| S3     | 0.48   | 0.02    | 0.00   |
| S4     | 0.47   | 0.03    | 0.00   |
| S5     | 0.46   | 0.04    | 0.00   |
| S6     | 0.49   | 0.00    | 0.01   |

### Table 2 Composition of CMC/TiO₂ nanocomposites

| Sample | CMC (g) | TiO₂ (g) | GO(g) |
|--------|---------|----------|-------|
| C1     | 0.50    | 0.00     | 0.00  |
| C2     | 0.49    | 0.01     | 0.00  |
| C3     | 0.48    | 0.02     | 0.00  |
| C4     | 0.47    | 0.03     | 0.00  |
| C5     | 0.46    | 0.04     | 0.00  |
| C6     | 0.49    | 0.00     | 0.01  |
wt%). Table 3 shows the different compositions of Na-Alg/TiO2 films. The last solutions of Na-Alg/TiO2 were stirred additionally for 1 hr. To achieve a complete dispersion of TiO2 nanoparticles, the solutions were sonicated for additionally 30 min at 60 °C. The resulting solutions were then cast in plastic petri dishes and left to dry in air for 5 days.

### 2.2 UV–vis spectroscopy

UV-Vis spectra for all polymer nanocomposites were collected at room temperature in the wavelength range of 200–1000 nm, using Jasco Spectrophotometer V-630, Tokyo, Japan at Spectroscopy Department, National Research Centre, Cairo, Egypt.

### 3 Results and discussion

#### 3.1 UV-vis absorption

Determination of the optical characteristics of polymers and metal oxides is of a great interest to investigate their different applications. Fig. 1 shows the absorbance spectra of pure Cs, Cs/CuO films and Cs/GO film versus wavelength collected in the optical range from 200 to 1000 nm. In certain cases, the absorption spectrum measurements of CuO nanoparticles produce an optimum absorption band at 242 nm and another band in the range of 400–600 nm (Rashad et al. 2013) as illustrated in Fig. 1. There is another band at nearly 670 nm and its intensity increased with increasing CuO content. As presented in the figure, the characteristic absorption band of Cs appeared at nearly 214 nm which is attributed to the n-π* and π-π* transition of C=C and C=O, and refers to the presence of acetate group residuals. The absorption shoulder beyond 300 nm is attributed to the CuO nanoparticles.

The absorption spectra of CMC and CMC/TiO2 nanocomposite films that contain different amounts of TiO2 (2, 4, 6 and 8 wt%), and CMC/GO film, recorded in the range of 200–800 nm, are displayed in Fig. 2. As displayed in the figure, the absorbance of pristine CMC was strongly influenced by the addition of TiO2 and GO separately. As the CMC absorption edge undergoes a shift towards the higher wavelength region, the figure shows that the intensity of absorption increases with increasing TiO2 content in the samples which refers to the strong complexation between CMC, and TiO2 and GO separately. The characteristic absorption band of TiO2 nanoparticles undergoes a slight blue shift with decreasing the CMC content in the prepared nanocomposites. The characteristic absorption band observed at 207 nm was ascribed to n-π* transitions and that at 345 nm was attributed to π-π* which is characteristic for the TiO2 nanoparticles, and similarly for CMC/GO film.

| Sample | Na-Alg (g) | TiO2 (g) | GO (g) |
|--------|------------|----------|--------|
| N1     | 0.50       | 0.00     | 0.00   |
| N2     | 0.49       | 0.01     | 0.00   |
| N3     | 0.48       | 0.02     | 0.00   |
| N4     | 0.47       | 0.03     | 0.00   |
| N5     | 0.46       | 0.04     | 0.00   |
| N6     | 0.49       | 0.00     | 0.01   |
This means that the CMC/TiO\textsubscript{2} nanocomposites have lower particle size.

Figure 3 shows the UV-Vis absorption spectra of pure Na-Alg, and Na-Alg/TiO\textsubscript{2} and Na-Alg/GO nanocomposites. The characteristic absorption peak of TiO\textsubscript{2} nanoparticles is clearly obvious in the Na-Alg/TiO\textsubscript{2} nanocomposites absorption spectra. The redshift of the absorption peaks from 346 to 360 nm can be attributed to the particle size variation during the reaction. Size and morphology of the prepared nanoparticles control the electronic properties of the nanocomposites (Farea et al. 2020; Flores-Hernández et al. 2021). At low concentrations of TiO\textsubscript{2}, very broad absorption peak is observed with very low intensity.
which shows that the number of particles is small with larger particle size. As the concentration increases, the absorption peak becomes narrower with increment in the intensity together with a redshift in the wavelength, indicating that larger particles are formed.

### 3.2 Optical bandgap

The optical absorption of polymer nanocomposites is the most important parameter through which the understanding of the band structure of all solids becomes possible. The Beer–Lambert law can be utilized to determine the absorption coefficient ($\alpha$) of the prepared nanocomposites. The absorption coefficient can be determined directly using the following equation:

$$\alpha = \frac{(2.303 \times A)}{d}$$

(1)

Where, $A$ is the absorbance and $d$ is the sample thickness. It is known that the optical bandgap of the different materials can be determined from the electron transition between valance and conduction bands (the fundamental absorption). As reported by Tauc and Davis - Mott, the bandgap values, and the type of electron transition between the valence and conduction bands can be determined using the following equation:

$$(\alpha h\nu)^r = B(h\nu - E_g)$$

(2)

Where $h\nu$ is the energy of the incident photons, $B$ is a constant and $r$ is a constant that depends on the type of transition. The constant $r$ takes values of 2, 1/2, 2/3 and 1/3 for direct allowed, direct forbidden, indirect allowed or indirect forbidden transition respectively (Badry R. et al. 2021-a and b).

Based on previous work (Kocyigit et al. 2020), the type of electronic transition in Cs is direct allowed transition. The direct bandgap energy of pristine Cs, Cs/CuO nanocomposites and Cs/GO nanocomposites is plotted as a function of photon energy in Fig 4 a, b,
and c respectively. Based on the intercept of the linear part of the curves with the photon energy axis, the values of the direct bandgaps were calculated from the last three figures and recorded in Table 4.

From the figure, the direct bandgap energy value of pure Cs is determined and is equal to 2.66 eV, while those of Cs/CuO nanocomposites, as tabulated in Table 4, are 1.66, 1.73, 1.62 and 1.47 eV for 2, 4, 6 and 8 wt% respectively. On the other hand, for Cs/GO nanocomposites, two absorption shoulders were observed corresponding to two bandgaps. The direct allowed bandgaps of Cs/GO film were estimated as 1.22 and 5.68 eV. The reduction of the Cs bandgap can be attributed to the formation of chemical bonds between Cs and CuO nanoparticles that causes the localized states to generate energy between the bands of the highest occupied molecular orbital (HOMO) and the lowest unoccupied one (LUMO), thus making the lower transitions feasible (Harun M.H. et al. 2009). Therefore, decreasing the direct allowed optical bandgap of pure Cs was achieved with the incorporation of CuO and GO. This result reflects that with increasing CuO content, the direct allowed energy bandgaps decrease in the Cs matrix by increasing the conjugation between Cs unsaturated bonds and CuO nanoparticles, aiming to reduce the energy bandgap. Previous work confirmed that organic polymers and their nanocomposites with reasonable optical bandgaps

![Graphs](image_url)

**Fig. 4** Relation between (αhυ)² and photon energy (hυ) for a pure Cs; b Cs/CuO nanocomposite with different concentrations of CuO (2, 4, 6 and 8 wt%); and c Cs/GO nanocomposite.
are imperative for photonics, and optoelectronic applications. It can then be concluded that Cs/CuO and Cs/GO nanocomposites can be used in optoelectronic devices.

Regarding the CMC/TiO$_2$ nanocomposite films, it was found that CMC has more than one direct bandgap due to its amorphousity, as presented in Fig. 5a, which are equal to 3.93 and 5.04 eV. Table 5 presents the different values of the optical bandgap for pristine CMC, CMC/TiO$_2$ and CMC/GO. Fig. 5b demonstrates that the addition of different weight percentages of TiO$_2$ (2, 4, 6 and 8 wt%) to CMC reduces the optical bandgap of CMC. Based on the obtained results, it was found that all the prepared samples have two direct bandgaps and one indirect bandgap due to the formation of localized states within the forbidden band of CMC (Elkomy et al. 2016). For CMC/TiO$_2$ samples, the first bandgap initially decreased until reaching 2.11 eV, corresponding to 6 wt% of TiO$_2$, and then increased again. The reason for this decrease in the bandgap may be attributed to the decrease in the particle size due to the quantum effects. However, increasing the direct optical bandgap beyond 6 wt% of TiO$_2$ refers to the formation of ion clusters which decreases the mobility, hence increasing the bandgap. These values are determined as presented in Figs. 5 and 6. Therefore, it can be concluded that the sample containing 6 wt% TiO$_2$ is the preferred one as it has the lowest bandgap energy. On the other hand, for CMC/GO nanocomposite film, the first bandgap of CMC decreased to 2.04 eV. This decrease in the CMC optical bandgap is attributed to the presence of new energy levels within the optical bandgap, which facilitates the electrons movement from the valence band to these localized states within the conduction band, consequently increasing conductivity and decreasing the optical bandgap (Murri et al. 1992). However, the second bandgap corresponding to the second absorption edge in the low wavelength region changes irregularly due to the addition of TiO$_2$ and GO to CMC (Table 5).

Figure 7 shows the plot of $(\alpha h\nu)^2$ versus the photon energy ($h\nu$) for pure Na-Alg, Na-Alg/TiO$_2$ and Na-Alg/GO film. It is established that, for direct optical bandgap materials, the top of the valence band and the bottom of the conduction band occur at the same $k$-value (Sweeney and Mukherjee 2017). All of the prepared samples possess two optical bandgaps; the onset bandgap equals 3.90 eV and the HOMO/LUMO gap equal 5.57 eV for pure Na-Alg. It is critical to show that the optical bandgap increased from 2.25 eV for pure Na-Alg to 2.44 eV for Na-Alg doped with 4 wt% of TiO$_2$ nanoparticles and then started to decrease until reaching 2.21 eV corresponding to 6 wt% of TiO$_2$ as presented in Table 6. Additionally, the HOMO/LUMO bandgap of pure Na-Alg (second bandgap) was decreased to 3.57 eV corresponding to 6 wt% of TiO$_2$. However, the optical bandgap for all samples, except those of 2 wt% and 4 wt% of TiO$_2$, is still smaller than that of pure Na-Alg. Additionally, Na-Alg/GO nanocomposites have

| Sample | $E_g^1$ (eV) | $E_g^2$ (eV) |
|--------|--------------|--------------|
| S1     | –            | 2.66         |
| S2     | –            | 1.66         |
| S3     | –            | 1.73         |
| S4     | –            | 1.62         |
| S5     | –            | 1.47         |
| S6     | 1.22         | 5.68         |

Table 4 Direct allowed bandgap energy values for pure Cs and Cs substituted with 2, 4, 6 and 8 wt% CuO, and 2 wt% GO at room temperature.
Fig. 5  Relation between $(\alpha h\nu)^2$ and photon energy ($h\nu$) for (a) pure CMC; (b) CMC/TiO$_2$ nanocomposite with different concentrations of TiO$_2$ (2, 4, 6 and 8 wt%) and (c) CMC/GO nanocomposite

### Table 5

| Sample | $E_{d1}$ (eV) | $E_{d2}$ (eV) | $E_{i}$ (eV) |
|--------|---------------|---------------|--------------|
| C1     | 3.93          | 5.04          | 5.26         |
| C2     | 2.50          | 5.11          | 4.24         |
| C3     | 2.30          | 5.34          | 4.33         |
| C4     | 2.11          | 4.91          | 3.05         |
| C5     | 2.25          | 4.76          | 1.86         |
| C6     | 2.04          | 3.31          | –            |
two optical bandgaps. However, the onset bandgap decreased strongly with the addition of GO when compared with that of both pure Na-Alg and Na-Alg/TiO$_2$ samples. The small optical bandgap of the Na-Alg/TiO$_2$ and Na-Alg/GO nanocomposites confirms its suitability for optoelectronics, photonics, and solar cell applications (Murri et al. 1992; Elkomy et al. 2016).

4 Conclusion

The traditional casting method was utilized to prepare Cs/CuO, Cs/GO, CMC/TiO$_2$, CMC/GO, Na-Alg/TiO$_2$ and Na-Alg/GO nanocomposite films by incorporating CuO, TiO$_2$ and GO nanoparticles within the polymer matrix. The optical properties of the synthesized nanocomposite films were investigated. The absorbance of Cs/CuO, Cs/GO, CMC/TiO$_2$, CMC/GO, Na-Alg/TiO$_2$ and Na-Alg/GO films were increased and the UV protection for Cs, CMC and Na-Alg was improved upon the addition of nanoparticles. Furthermore, the prepared nanocomposites of Cs/CuO, Cs/GO, CMC/TiO$_2$, CMC/GO, Na-Alg/TiO$_2$ and Na-Alg/GO can be utilized in many optical applications since the bandgap energy value is reduced due to the incorporation of different nanoparticles, with increase in the wavelength of the electronic transmission. The obtained results of the current study indicated that Cs, CMC, and Na-Alg nanocomposite films can be utilized as a new membrane material in optoelectronic devices, solar cells, and capacitors.
Fig. 7 Relation between $(\alpha h \nu)^2$ and photon energy ($h\nu$) for a pure Na-Alg; b Na-Alg/2 wt% TiO$_2$; c Na-Alg/4 wt% TiO$_2$; d Na-Alg/6 wt% TiO$_2$; e Na-Alg/8 wt% TiO$_2$; f Na-Alg/2 wt% GO
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Declarations

Conflict of interest There are no conflicts to declare.

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