

Studying the effect of concentration on spectral and optical properties of chitosan polymer

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ABSTRACT

Due to its noteworthy physico-chemical behaviors, the chitosan (CS) biopolymer presents a compelling alternative to conventional biomaterials. By measuring the emission spectra at a spectrofluorophotometer in different concentrations (1×10^{-4} , 1×10^{-5} , and 1×10^{-6} M), the spectral characteristics of the CS polymer were obtained. The crystallinity of the CS polymer was determined by X-ray diffraction (XRD) analysis, which shows only one dominant peak indexed to the orthorhombic structure. With a drop in solution concentration, there was a shift toward the shorter wavelength and a decline in quantum yield. Using a UV-Vis spectrophotometer, the optical properties of the CS polymer, including its refractive index, absorption coefficient, energy band gap, extinction coefficient, and dielectric constant, have been studied. It was observed through the results that the optical parameters are linearly dependent with concentration. In contrast to the optical energy gap, which decreases with increasing concentration.

Keywords: Chitosan polymer, optical properties, quantum yield.

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1. Introduction

Recently, researchers have concentrated on porous hydrogel materials with a natural origin, namely CS [1]. Natural polymers are safe, non-toxic, and extensively employed in a number of applications. CS is a biodegradable polysaccharide that is produced naturally from chitin in marine crusts and is employed in a variety of medicinal sectors because it is non-toxic [2]. The wide range of biological and technical features demonstrated by chitin and chitosan are what generate interest in these polymers. But these characteristics are closely linked to the physical characteristics of the polymers. Therefore, a thorough and accurate polymer characterization is required when working with chitin and chitosan. The only polycation found in nature is CS, and the media's pH and level of acetylation affect its charge density. The polymer's solubility is influenced by its molecular weight and level of acetylation [3]. We zeroed in on CS material because of its wide range of recent uses across a variety of sectors, including manufacturing, agriculture, and medicine. CS has a wide range of compounds with which it interacts well and improves their optical properties. This polymer has recently garnered a lot of interest [4] due to its many appealing properties, such as its film-forming capacity, good stability, high water permeability, strong electrode surface adhesion, biocompatibility, lack of toxicity, high mechanical resistance, affordable price, and susceptibility to chemical modifications due to the presence of reactive amino and hydroxyl functional groups.

X-ray diffraction (XRD) is a technique for investigating atomic-scale periodic structures (single crystal or polycrystalline materials). The method is commonly employed for diffraction-based chemical characterization and identification [5,6]. The wavelength of X-rays and the distance between lattice planes in crystalline materials are on the same scale of magnitude. The X-rays will be scattered as they enter the substance by the electron clouds around the atoms. The X-rays' intensity is displayed as a function of angle 2θ because of the periodicity of the lattice planes, which causes constructive interference. If peaks are exhibited, then the

lattice distance can be determined using Bragg's law. [7,8]. It is the purpose of this research to demonstrate that chitosan's structural, spectral, and optical properties change with increasing concentration. Quantum yield enhancement by decreased concentration is another target.

2. Materials and methods

A linear polysaccharide formed by 2-amino-2-deoxy d-glucan is chitosan, which is produced when chitin is deacetylated. These amine groups are functional and can be chemically altered to change their characteristics, enable various biological processes, and apply modifying reactions. CS is non-toxic, biocompatible, and can be employed in a variety of medical applications, including antibacterial and biomaterials for wound healing. CS has a molecular weight of 400000 and the chemical formula $(C_6H_{11}NO_4)_n$. Figure 1 depicts a schematic depiction of CS's molecular structure. It was given out in the form of a white to off-white powder by MACIAN [9].

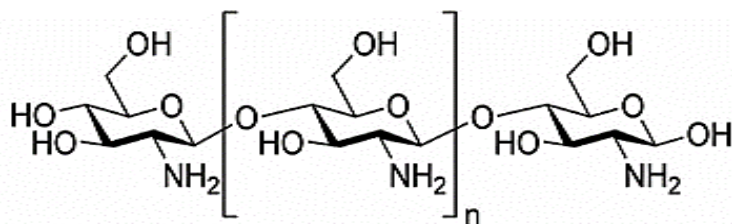


Figure1. Molecular Structure of CS

By means of primary amine protonation, CS can dissolve in aqueous acidic solutions [3]. 2 g of CS was added to two identical containers, each holding 100 mL of a 0.1 M acetic acid solution, and both were magnetically agitated for 4 hours at room temperature to produce a clear, uniform solution. In an ethanol solvent, solutions of CS at concentrations of 10^{-3} , 10^{-4} , and 10^{-5} M were created. The powder was weighed using a German electronic balance of the BL 210 S type with a four-digit sensitivity. The following equation was used to prepare various concentrations [10]:

$$c = \frac{W \times 1000}{M_w \times V} \quad (1)$$

Where W is the weight of the liquid crystals (LC) dissolved in the ethanol solvent (g), Mw is the LC's molecular weight (g/mol), V is the volume of the ethanol solvent (mL), and C is the LC concentration (M). The following equation was used to dilute the produced solutions [11].

$$C_1V_1 = C_2V_2 \quad (2)$$

Where: C_2 : New concentration, C_1 : Primary concentration. Volumes V_1 and V_2 refer to the initial and diluted volumes, respectively.

3. Consequences and discussions

3.1. Structural properties

By using an XRD study (SHIMADZU 6000 Japanese) with a Cu-K radiation source at an angle 2θ ranging from 20 to 70° , the crystallinity of the materials was assessed. It was done using an emission current of 20 mA and an accelerating voltage of 30 kV. The XRD pattern for the CS polymer only showed one dominating peak at 20.397° , which can be attributed to the orthorhombic structure, this is as reported by other studies in [12,13].

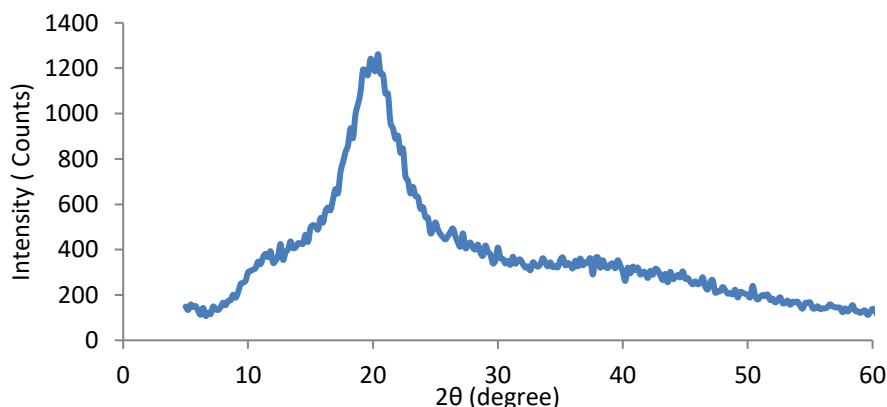


Figure 2. XRD patterns of CS polymer

From XRD readings, the Debye-Scherrer method was applied to determine the size of the grains [14]:

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (3)$$

Here, D stands for the grain's size diameter, β stands for the diffraction line widening at half-maximum intensity, λ stands for the wavelength of (Cu K), θ is the Bragg angle for a certain diffraction, and k is a constant, which is typically equal to 0.9 for powders. In table 1 we see the outcomes for the various grain sizes.

Table 1. XRD results of CS polymer

$2(\theta)$	d (nm)	BG	FWHM	D (nm)
20.397	0.43505	921	1.317	6

The size of CS, which was measured to be nm, suggests that the size of the synthesized chitosan was crystalline. The wide peaks imply that the particles may be nanometric in size. By contrasting the observed XRD pattern to a standard created by the International Center for Diffraction Data (ICDD) utilizing chitosan, several phases were distinguished. Every pattern revealed that CS was the sole phase, this is as reported by other authors [15]. The emission spectra of CS was performed at spectro fluorophotometer type of (RF-5301pc Shimadzu). The results of the measurements as shown in Figure 3

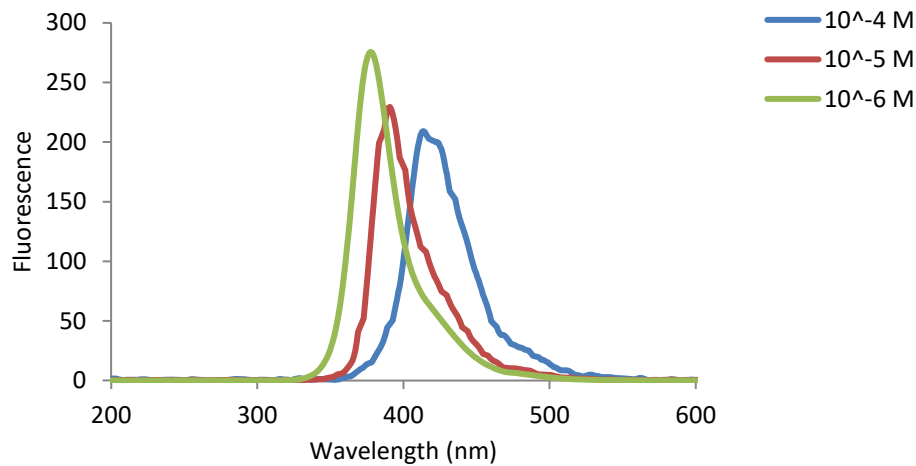


Figure 3. Fluorescence spectra as a function of wavelength of CS polymer solutions

The emission spectra of the CS polymer showed a red shifted emission maximum because of the electron-rich polymer main chain [16]. The results show that an increase in CS concentration causes the peak of fluorescence to shift toward long wavelengths (red shift) and low-lying energy. The increase is explained by the solution's concentration and local electric field, which are both located in the solution. These factors will promote and, as a result, cause shipments to be reordered because of electronic partial transfer, which turns the situation into a bipolar one that is more agitated than the state of the ground. The solution's increasing polarity will reduce the amount of energy and, as a result, the displacement of the spectrum summit fluorescence (red shift) [17]. The relative quantum yield of CS can be calculated through the following formula [18]:

$$\Phi_F = \frac{\text{number of photons emitted}}{\text{number of photons absorbed}} \quad (4)$$

Table 2. Quantum efficiency of CS solutions

C (M)	λ max (nm)	Relative Intensity	Q_F
10^{-4}	413	209	0.54
10^{-5}	390	229	0.61
10^{-6}	377	275	0.68

According to the comment in Table 2, the change in CS concentration had an impact on the lifespan and quantum yield of fluorescence. As a result, the effect was that the lifetime of the fluorescence increased with concentration, and this may be attributed to polarity. The solvent decreases the area of the CS's molecular diffusion and speeds up energy transfers, which boosts the fluoridation's quantitative output, this value agree with the works of the researcher [19,20].

3.2. Optical properties

The generated samples' optical spectroscopic examinations were performed using UV-visible spectroscopy (UV-1800 SHIMADZU, Japan) in the wavelength range of (200–800 nm). The spectra of absorbance, transmittance of CS polymer with different concentration were illustrated in Figures 4 and 5, respectively.

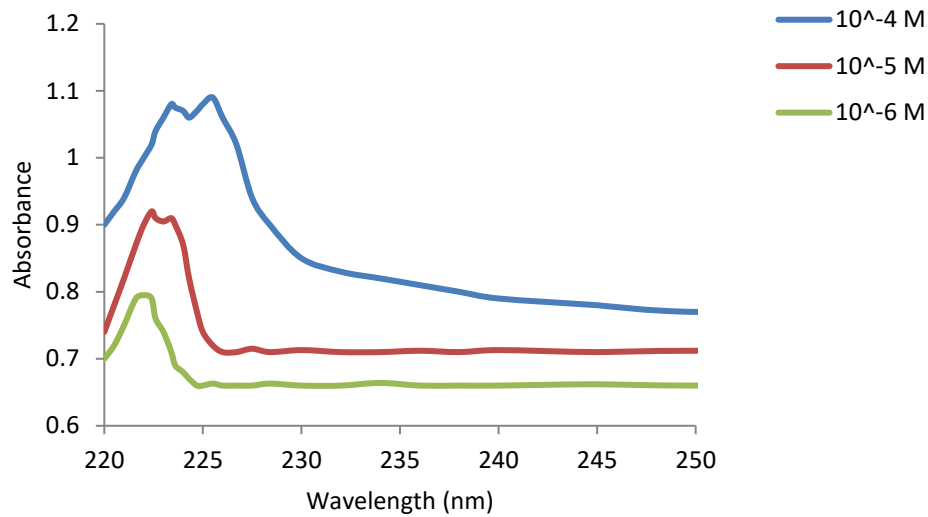


Figure 4. Absorbance spectra as a function of wavelength of CS polymer solutions

The UV-Visible spectrum that was seen had an absorption peak between (220 and 230) nm that was attributed to the (π - π^*) transitions [21,22,23]. These transitions connected to chitosan's carbonyl groups [24]. As the concentration of these transitions increases, the wavelength shifts toward the red, indicating a narrowing of the optical energy gap [25,26].

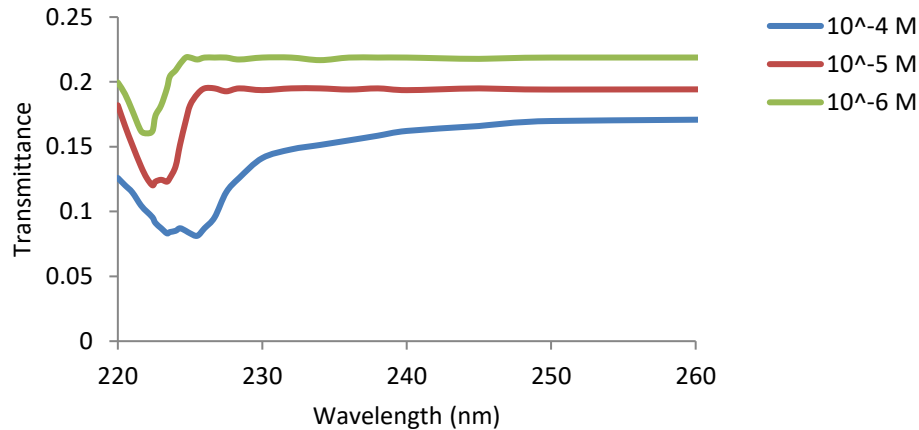


Figure 5. Transmittance spectra as a function of wavelength of CS polymer solutions

Additionally, Figure 5 displays how the transmission spectra of the employed CS polymer change with wavelength. It has been demonstrated that CS exhibits a minor red shift and becomes very transparent at wavelengths longer than 260 nm. This transmission of CS may result from the sample's reported rise in crystallization at lower concentrations [27].

The optical energy band gap of the materials can be ascertained from the absorption coefficient (α), which can be computed using [28, 29]:

$$\alpha_0 = (2.303) \times \frac{A}{t} \quad (5)$$

Where A is the absorbance and t is the sample thickness used.

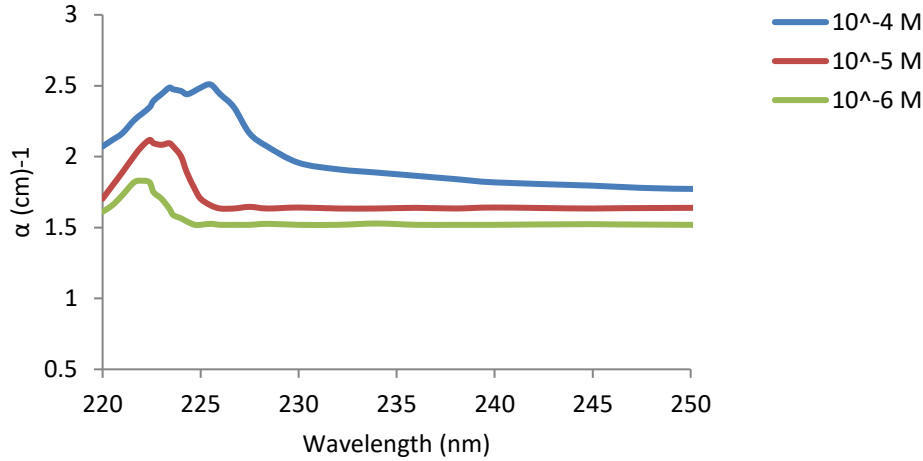


Figure 6. Absorption coefficient as a function of wavelength of Chitosan polymer solution at different concentrations

Because of this concentration-dependent uptick in absorption, linear absorption coefficients also increased [30].

The optical band gap (the fundamental absorption) of a material can be determined by studying its electron transition between the valence and conduction bands [31]. Using Tauc's formula [32], we can determine the values of the band gaps and the kind of electron transfer between the valence and conduction bands:

$$\alpha h\nu = (h\nu - E_g)^r \tag{6}$$

Where A is a constant, $h\nu$ is the photon energy, and r is a constant that varies with the transition. For a permissible indirect transition, r is set to 2.

By graphing the relationship between $(\alpha h\nu)^2$ and photon energy ($h\nu$), as illustrated in Figure 7, it is possible to determine the optical energy gap E_g of CS solutions. The energy band gaps for the solutions are obtained by extrapolating the energy axis' straight line section ($h\nu$).

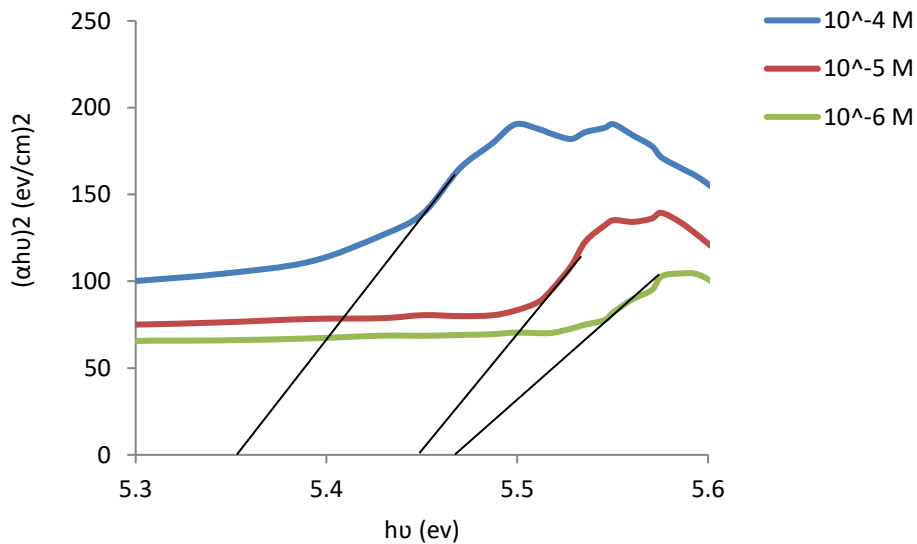


Figure 7. The variation of the $(\alpha h\nu)^2$ with $(h\nu)$ for CS polymer solutions

Refractive index plays a critical role in the investigation of materials and several optical technologies. Chitosan polymer's high refractive index values will enable its usage in a variety of optical applications, such as wave guides and light emitting diodes [33]. Refractive index (n) can be determined from the transmittance spectrum using the equations below [34]:

$$n_o = \frac{1}{T} + \left[\left(\frac{1}{T} - 1 \right) \right]^{1/2}$$

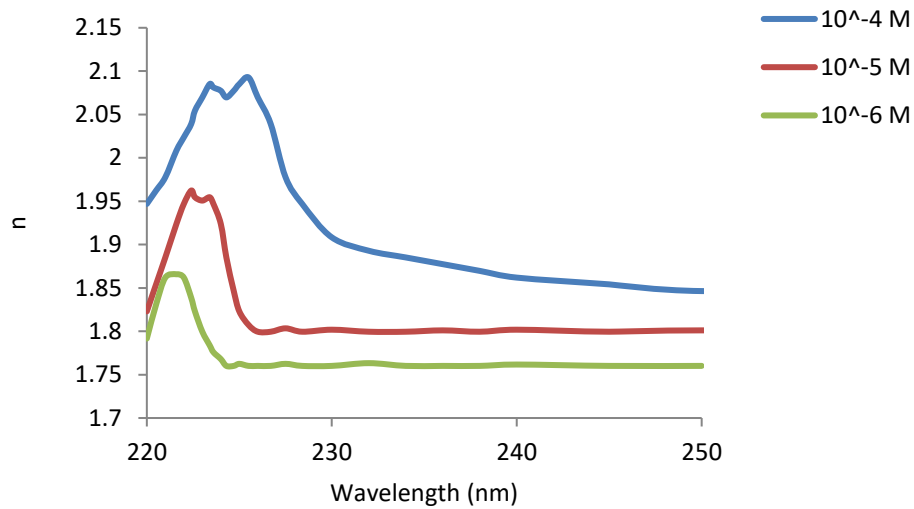


Figure 8. Refractive index as a function of wavelength of CS polymer solutions

It has been noticed that the refractive index varies with wavelength, and that as concentration is raised, the refractive index spectrum peak rises. This is because a higher concentration of dye results in a denser sample [35].

Extinction coefficient Using the values of the wavelengths and the connection presented in the following equation [2,36], we can calculate the coefficient K, which is a measurement of how much light is lost due to dispersion and absorption in the unit of distance from the center penetration.

$$k = \frac{\alpha_0 \lambda}{4\pi} \tag{7}$$

As seen in Figure 9, the coefficient of inactivity has very modest values, indicating that the quantity of energy lost is negligible.

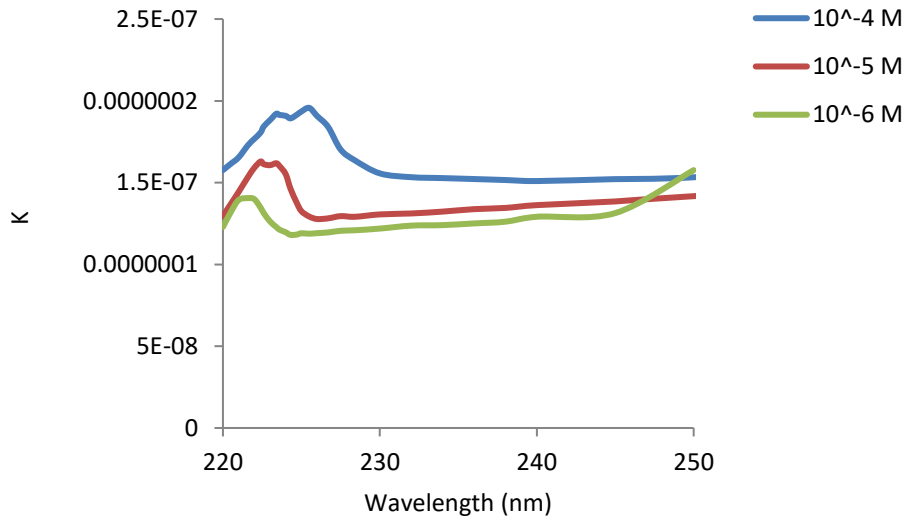


Figure 9. Extinction coefficient as a function of wavelength of CS polymer solutions

The extinction coefficient depends on the absorption coefficient, thus as the concentration rises, the absorption coefficient also rises, increasing the peak of the extinction coefficient. The degree of cross-linking, chain geometry, matrix and filler polarity, free volume, as well as electronic, atomic, and orientational polarization, all have an impact on the dielectric constant of polymeric materials. Below is a formula that can be used to represent the real and imaginary components of the dielectric constant [37,38].

$$\epsilon_{\text{real}} = n^2 - K^2 \tag{8}$$

$$\epsilon_{\text{im}} = 2nK \tag{9}$$

Both the real and imaginary parts exhibit the same pattern, and it is evident from Figures 10 and 11 that the real part has higher values than the imaginary part.

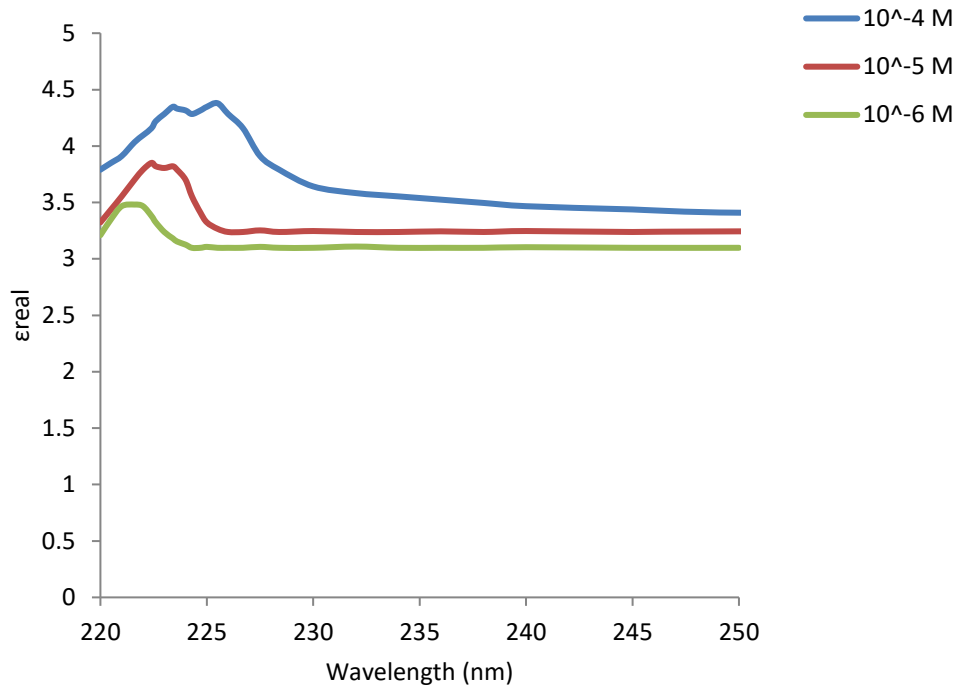


Figure 10. Real dielectric constant as a function of wavelength of CS polymer solutions

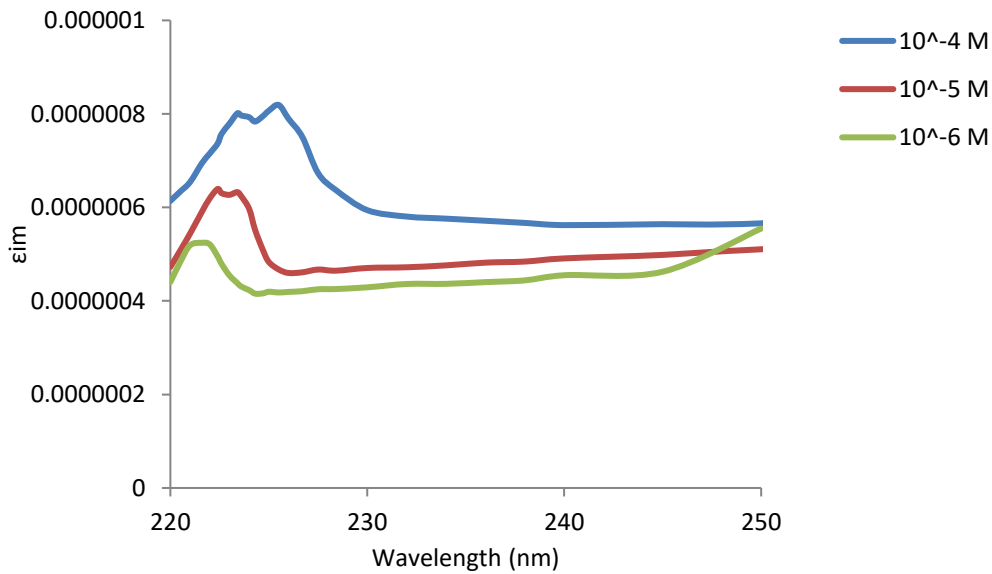


Figure 11. Imaginary dielectric constant as a function of wavelength of CS polymer solution

Table 2. Linear optical parameters of CS solutions

C (M)	A	T	α_0 (cm) ⁻¹	n_0	$K \times 10^{-7}$	ϵ_{real}	$\epsilon_{im} \times 10^{-7}$	Eg
10 ⁻⁴	1.09	0.081	2.509	2.092	1.96	4.379	8.19	5.35
10 ⁻⁵	0.92	0.120	2.117	1.962	1.63	3.850	6.39	5.45
10 ⁻⁶	0.795	0.160	1.830	1.861	1.4	3.466	5.21	5.47

From this table we show that the values of linear parameter for (α , n) are increased with increasing the concentrations of CS solution, this is due to decreasing number of molecules per volume unit at low concentrations. It can see from the value of absorption coefficient ($\alpha \leq 10^4$) cm^{-1} that the transition is indirect allowed of all solutions, this behavior is reported in the literature [32]. The refractive index values are greater than 1.5, indicating that the material is opaque, this value agree with the study in reference [2]. ϵ_{real} variation is dependent on n values since extinction is less variable than refractive index ($\epsilon_{\text{real}}=n$), whereas ϵ_{im} is primarily dependent on extinction values that are related to absorption coefficient variation [39]. It is also consistent with mentioned in the study [2].

Since the optical band gap decreases as the refractive index rises, the two phenomena are connected. Some research suggests that a narrowing of the optical band gap is related to an increase in the optical dielectric constant. Consistent with the research cited in references [9], the density of states increases in the CS as the number of charge carriers delivered into the CS increases with increasing optical dielectric constant [39].

4. Conclusions

In this present research, chitosan polymer were characterized and studied for possible device applications. XRD characterization results proved that CS have orthorhombic structure. The spectral displacement of the fluorescence towards the short wavelength (blue shift) and increase in the fluorescence intensity with a decrease in concentration. Using a UV-VIS spectrophotometer, the optical characteristics of CS solutions at various concentrations had been proven. As concentrations increases, transmittance declined and absorption intensity increased, which is consistent with Beer-Lambert law. The optical parameters (α , n , k , ϵ_{real} , ϵ_{im}) of all solutions were increased when the concentrations were increased, in addition to a decreasing in the optical energy gap for the allowed indirect transitions. The obtained data revealed that the optical band gap can be used in many applications such as solar energy and organic photovoltaic.

Declaration of competing interest

The authors declare that they have no any known financial or non-financial competing interests in any material discussed in this paper.

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