

AC Electrical Properties, Thermal Analysis and Emission Spectrum of Chitosan Polymer

Hajer Abusnina^{*1,2}, N. A. El-Ghamaz¹ and E. A. Gaml¹

¹Department of Physics, Faculty of Science, Damietta University 34517, Egypt.

²High and Intermediate Institute of Agricultural Technology-Gheran. Tripoli, Libya.

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*Corresponding author's E-mail: hajer.gege2014@gmail.com

Abstract

The physical properties of Chitosan in as received form were investigated by different techniques. XRD analysis of the powder form confirmed the amorphous structure. Thermal stability was evaluated through Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). DSC measurements identified an endothermic peak associated with water loss at 57 °C and an exothermic peak at 308 °C attributed to the decomposition of the Chitosan backbone. The dielectric properties, including dielectric constant and AC conduction mechanisms, were investigated within a temperature range of 305 – 395 K. Both the real and imaginary parts of the complex dielectric constant exhibited a decreasing trend with increasing frequency at a fixed temperature. Furthermore, the AC conduction mechanism was analyzed. The Bulk Modulus and conduction mechanism were also investigated. Finally, emission spectra were examined.

Keywords: Chitosan; Thermal, Dielectric constants, emission properties.

Introduction

Recently, a great attention has been focused on fabrication and characterization of the organic polymers for using in multi-applications (Lin, 2016; Zhang et al., 2018). Chitosan, a naturally occurring and promising biodegradable polymer, ranks second after to cellulose in abundance among amino polysaccharides (Jangid et al., 2019; M. N. V. R. Kumar et al., 2004a). Its diverse physicochemical and biological properties

make it a strong contender for applications in ion devices and various biomedical fields, including artificial organs (kidney, liver), tissue regeneration (bone, tendon, nerve, blood vessels), anticoagulation therapy, and burn treatment (Pandey et al., 2017). Chitosan has several important features, including film-forming ability, bioactivity, non-toxicity to humans, biodegradability, and application in manufacturing of biomaterials. Therefore, Chitosan finds wide use in pharmaceutical, biomedical, and cosmetic applications (2015). A lot of studies have investigated the use of Chitosan blends with inorganic composites ,

demonstrating significant enhancements in the electrical properties of these materials (Aziz & Abidin, 2015).

Experimental techniques

Material and sample preparation

Chitosan is a natural biopolymer derived from Chitin (G. Kumar et al., 2000; M. N. V. R. Kumar et al., 2004b) Chitin is the structural component of the exoskeleton of insects and crustaceans (especially shrimp and crabs) and the cell walls of fungi, and is the second most abundant natural polysaccharide after cellulose (Dickey, 2007). In this work, Chitosan was used as purchased, the molecular structure of Chitosan is shown in Fig. 1. For emission spectra, Chitosan was prepared in the form of thin film. Chitosan solution is prepared by dissolving 0.1 g Chitosan in 4 mL acetic acid and 2 mL deionized water. This solution was stirred for 24 h at room temperature. Finally Chitosan thin film is prepared by spin coating technique onto quartz and glass substrates using VTC-50A Spin Coater at 1500 rpm for coating and 2500 rpm for drying for 30 s.

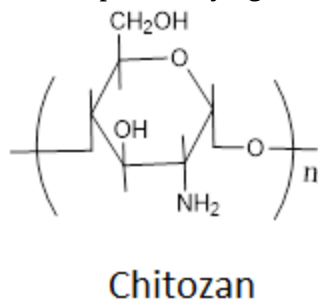


Fig.1: Scheme of the chemical structure of Chitosan

Measurements and Characterization

ATI Mattson (Infinity Series FTIR) spectrometer was utilized for Fourier Transform Infrared spectroscopy (FTIR) analysis of the powdered polymer using (Shimadzu XRD 6000) in the range $4 - 8^\circ$, with step of 0.01 degrees per second with $\text{Cu K}\alpha$ ($\lambda = 0.15406 \text{ \AA}$). The Photoluminescence spectra of thin films on glass substrate are recorded by Fluorimeter spectrophotometer (Model 6285, for Excitation and Emission spectra, 200nm – 700nm) with the excitation light with wavelength 217 nm. Thermal gravimetric analysis (TGA) was performed using a Shimadzu TG-50 instrument to assess

the thermal stability of the samples. The samples were heated from room temperature to 500°C at a rate of $10^\circ\text{C}/\text{min}$ under a nitrogen atmosphere with a flow rate of approximately 20 mL/min. Differential scanning calorimetry (DSC) measurements were carried out using a Shimadzu DSC-50 instrument in the temperature range $30 - 500^\circ\text{C}$ in nitrogen atmosphere and temperature increment rate of $10^\circ\text{C}/\text{min}$ using an aluminum cell. For AC measurements, the powder of Chitosan compressed by a (hydraulic press) under a pressure of 10 Tons/ cm^2 at room temperature to form pellets of thickness about 0.7 mm and cross section surface area 0.90 cm^2 . The AC electrical measurements were performed by LCR, bridge (model 3522-50 HIOKI). The measurements were performed in the frequency range (100Hz – 5 MHz) and in the temperature range (305 – 395 K).

Result and discussion:

Structural analysis

Fourier transforms infrared (FTIR) spectroscopy

Fig 2. displays the FTIR spectra of Chitosan, The FTIR spectrum of Chitosan shows a broad band at 3290 cm^{-1} due to OH and NH_2 groups, The peak at 2215 cm^{-1} in Chitosan polymer corresponds to C=N, The bands at 1668 and 1322 cm^{-1} are assigned to the amino group (Osman & Arof, 2003), while the bands at 1420 and 1371 cm^{-1} are assigned to O-C-O group. The sharp peak around 1590 cm^{-1} and the peak around 1319 cm^{-1} are indicative of N-H bending vibrations and C-N stretching vibrations, respectively, associated with the amide group in Chitosan (Țucureanu et al., 2016).

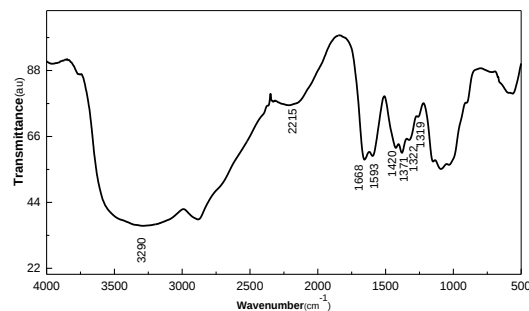


Fig 2. Fourier transform infrared spectra of Chitosan

X-ray diffraction

Fig 3 shows the XRD pattern of Chitosan, it's clear that the X-ray diffraction pattern (XRD) for pristine Chitosan demonstrate the standard behavior of amorphous (S. Kumar & Koh, 2012). with a main broad peak at around 2θ of 19.3° in addition to a low intense peak at 2θ of 10.3° .

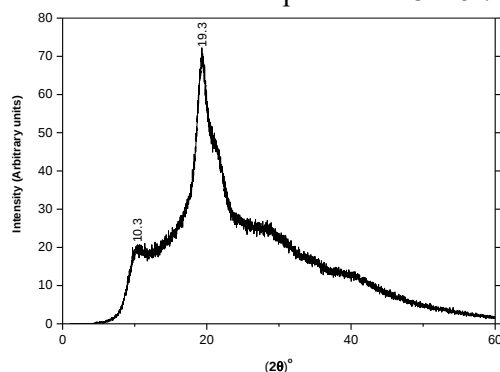


Fig 3: XRD pattern of Chitosan

Thermal Analysis

Fig 4 presents the TGA, DTA and DSC charts for Chitosan in the temperature range 28-490 °C. Regarding the TGA curve, in the first stage of heating up to 80°C, it is noticed that the polymer losses about 8% to 15% from its weight. Also, it shows good thermal stability in the range 110- 210 °C. At higher temperature range (210 to 330 °C), a fast degradation is observed via fast loss of weight. The start temperature of degradation process T_s is 240°C and the maximum degradation rate T_m is 297°C (see DTG). DSC analysis revealed an endothermic peak at 54 °C which may be attributed to water desorption and an exothermic event at the temperature range 309-332 °C associated with the thermal degradation of the backbone the Chitosan matrix.

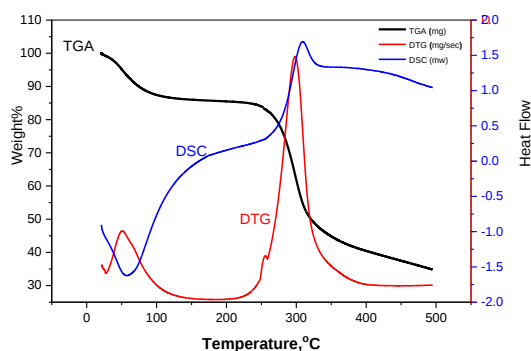


Fig 4 : Thermal Analysis (TGA , DTA& DSC) of Chitosan

Dielectric constants and AC conductivity

The dielectric and conductive characteristics of materials are crucial for technological applications like capacitors and batteries (Li et al., 2018; Tan et al., 2006). To examine these properties, researchers analyze the measured values of capacitance (C_p) and loss tangent ($\tan\delta$). Fig 5 illustrates the values of C_p for Chitosan over the frequency range of 100Hz to 5 MHz and at temperatures between 305 and 395 K. the observed behavior is characteristic of many organic compounds (Winie et al., 2011a) and polymers (Diab et al., 2011) (Gami et al., 2022), where C_p generally decreases as the applied frequency increases (Siekierski et al., 1998). Fig 6 presents the frequency and temperature dependence of ($\tan\delta$) for Chitosan It is clear that $\tan\delta$ has nearly the same behavior of C_p . The dielectric constant ϵ^* is a complex function expressed as (Lee et al., 2003):

$$\epsilon^* = \epsilon_r + i \epsilon_i, \quad (1)$$

in which the real and imaginary parts, (ϵ_r , ϵ_i), are related to the capacitance and loss tangent according to the following relations (Hao et al., 2008; Sankarappa et al., 2008):

$$\epsilon_r = \frac{C_p d}{\epsilon_o A} \quad (2)$$

$$\epsilon_i = \epsilon_r \tan \delta, \quad (3)$$

where ϵ_o , d , and A represent the permittivity of free space, the thickness of the material, and its cross-sectional area, respectively. The calculated values of the real part (ϵ_r) and imaginary part (ϵ_i) of the permittivity are illustrated in Figures 7 and 8, respectively. Both ϵ_r and ϵ_i exhibit a decreasing trend with increasing frequency at a specific temperature. This observed behavior aligns with the expected dielectric relaxation phenomenon within the polymer (Winie et al., 2011a). The AC conductivity (σ_{ac}) of the investigated polymer at a constant temperature can be determined using equation (4):

$$\sigma_{ac} = \omega \epsilon_i \epsilon_o, \quad (4)$$

On the other hand, the AC conductivity σ_{ac} can be expressed as:

$$\sigma_{ac} = A^* \omega^S, \quad (5)$$

where A^* is a constant, $\omega = 2\pi f$, and S is the power exponent. The conduction mechanism

within the material is linked to the temperature dependence of S . To determine S , $\log(\sigma_{ac})$ versus $\log(\omega)$ curves were plotted for Chitosan at various temperatures, as shown in Figure 9. Charge carrier hopping is the dominant conduction mechanism in these samples, with σ_{ac} exhibiting a power-law dependence on ω , as reported by (Siekierski et al., 1998). As expected for many polymers (Diab et al., 2017), σ_{ac} increases with increasing frequency. This increase can be attributed to the enhanced oscillation of the electric field at higher frequencies, leading to increased polarization and, consequently, an apparent rise in conductivity (Aziz & Mamand, 2018; Winie et al., 2011b). The values of S were directly extracted from the slopes of the $\log(\sigma_{ac})$ versus $\log(\omega)$ curves in the high-frequency region for each temperature. Figure 10 illustrates the temperature dependence of the exponent S , revealing a decreasing trend with increasing temperature. According to (Elliott, 1987) and (Long, 1982) this behavior of S is characteristic of the Correlated Barrier Hopping (CBH) conduction mechanism. The frequency exponent S for the CBH model can be expressed as (Zeyada et al., 2017):

$$S = 1 - \frac{6k_B T}{w_m - k_B T \ln(1/\omega\tau_0)}, \quad (6)$$

where w_m stands for the potential barrier height, k_B is Boltzmann's constant, T , is the absolute temperature, τ_0 is the characteristic relaxation time and ω is the angular frequency. The value of maximum barrier height w_m can be calculated according to equation (6). The values of w_m for Chitosan is found 0.212 eV.

Jonscher's relation may suggest a potential relationship between the complex bulk modulus (M^*) and the complex dielectric constant, according to the relation (Ambrus et al., 1972; Jonscher, 1999; Zangina et al., 2017):

$$M^* = \frac{1}{\mathcal{E}^*} = M_r + iM_i \quad (7)$$

where M_r and M_i are the real and imaginary parts of the bulk modulus, respectively. The values of M_r and M_i can be calculated as follows (Ambrus et al., 1972; Jonscher, 1999; Zangina et al., 2017):

$$M_r = \frac{\mathcal{E}_r}{(\mathcal{E}_r)^2 + (\mathcal{E}_i)^2} \quad (8)$$

$$M_i = \frac{\mathcal{E}_i}{(\mathcal{E}_r)^2 + (\mathcal{E}_i)^2} \quad (9)$$

Figure 11 illustrates the variations in the behaviors of M_r and M_i with respect to angular frequency across different selected temperatures. Observations reveal that M_r exhibits an increasing trend with rising frequency, particularly enhancing its efficiency in depletion and aggregation zones. Conversely, M_i demonstrates a decreasing trend as the angular frequency increases

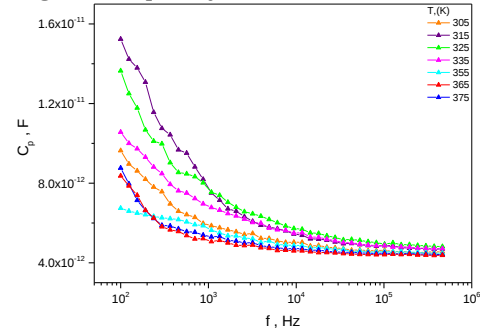


Fig 5: The relation between the capacitance (C_p) and both of frequency (f) of Chitosan

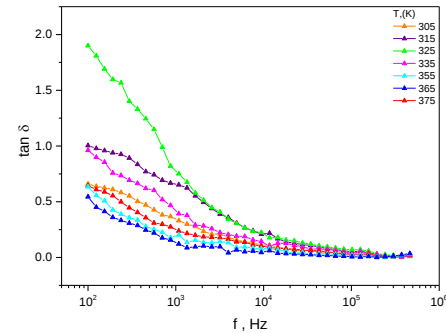


Fig 6 : The loss tangent ($\tan \sigma$) versus frequency (f) of Chitosan

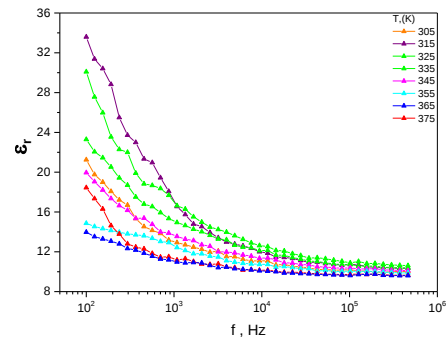


Fig 7 : the real dielectric constant (ϵ_r) versus frequency (f) of Chitosan

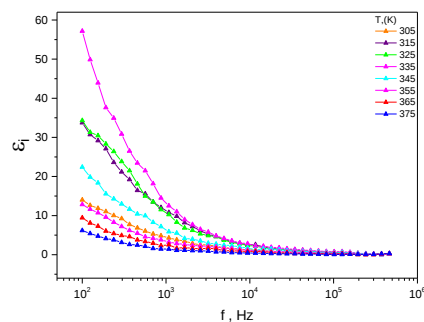


Fig 8: the imaginary dielectric constant (ϵ_i) versus frequency (f) of Chitosan

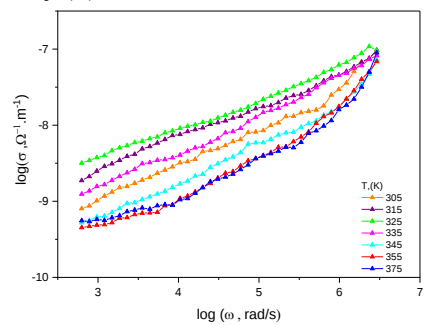


Fig 9: $\log \sigma_{ac}$ versus $\log \omega$ for Chitosan

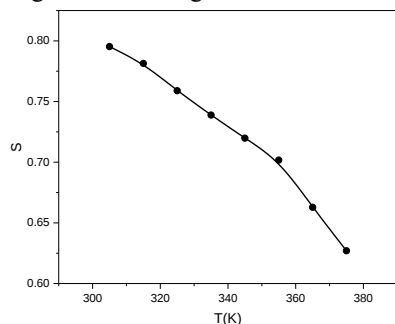


Fig 10: The dependence of S on temperature for Chitosan

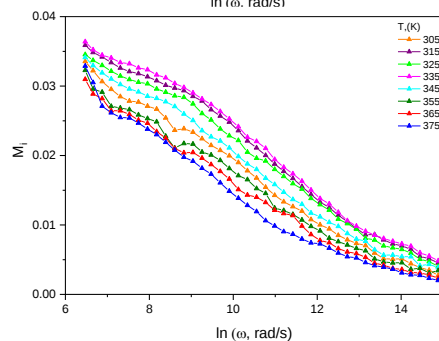
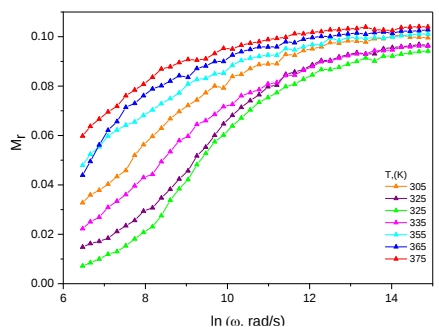


Fig 11: M_r & M_i as a function of frequency for Chitosan

Emission properties of Chitosan.

The emission spectrum for Chitosan thin film is illustrated in Fig 12. The emission spectrum has one peak at 280 nm and a shoulder at 351 nm. These emission peaks can be attributed to the formation of tautomer and dimer by intra molecular and inter molecular hydrogen bond (A. Alkathiri et al., 2022) The color coordinates of Chitosan thin film is calculated using color calculator software (Dang et al., 2012). Fig 13 shows the CIE diagram of Chitosan. The thin film emits white light making it promising candidates for OLED applications

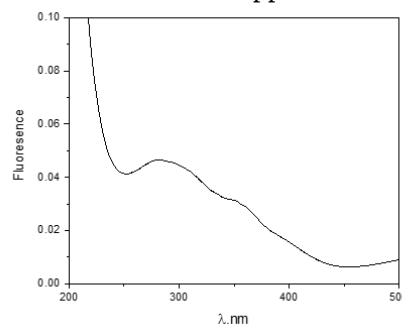


Fig 12: Fluorescence emission spectra for Chitosan

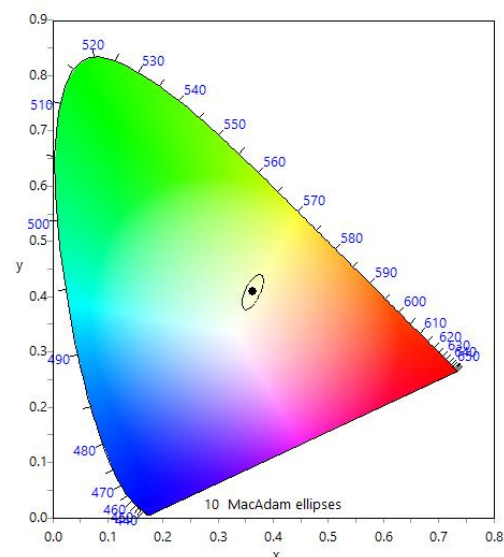


Fig 13: CIE diagram of Chitosan

Conclusion

Structural, thermal, electrical and emission spectrum investigations were performed on Chitosan. The amorphous phases of Chitosan is confirmed by XRD results. Thermal analysis revealed that degradation process started at temperature 230 °C. ϵ_r and ϵ_i showed high values at low frequency due to electrode polarization. Correlated barrier

hopping (CBH) model was the best model to interpret the data. The value of maximum barrier height w_m was found 0.212 eV. The emission spectrum for Chitosan thin film was measured. The CIE diagram showed light white color emission. These results suggested that Chitosan can be used in OLEDs.

Competing Interest

The authors declare that there are no competing interests.

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الملخص العربي

عنوان البحث: الخواص الكهربائية للتيار المتردد والتحليل الحراري وطيف الانبعاث للكيوتوزان

هاجر عاشور محمد أبوسنينة^{1*}، ناصر عبده عبدالرازق الغماز¹، إيمان أسعد جمل¹

¹قسم الفيزياء – كلية العلوم – جامعة دمياط – مصر.

في هذا البحث تم التحقيق في الخصائص الفيزيائية للكينوزان في باستخدام العديد من التقنيات. أكد تحليل الأشعة السينية (XRD) على البنية غير المتبلورة لمسحوق الكينوزان. تم تقييم الاستقرار الحراري من خلال التحليل الحراري الوزني (TGA) والمسح الحراري التفاضلي (DSC). حددت قياسات DSC ذروة مرتبطة بفقدان الماء عند ٥٧ درجة مئوية وذروة خارجية عند ٣٠٨ درجة مئوية تعزى إلى تحلل سلسلة الكينوزان الأساسية. تم التحقيق من خصائص العزل الكهربائي، بما في ذلك ثابت العزل الكهربائي وآليات التوصيل للتيار المتردد ضمن نطاق درجة حرارة يتراوح بين ٣٠٥ و ٣٩٥ كلفن. أظهر كل من الجزء الحقيقي والجزء التخيلي لثابت العزل الكهربائي اتجاهًا تناقصيًا مع زيادة التردد عند درجة حرارة ثابتة. وكذلك أظهرت ϵ_r و ϵ_i قيمًا عالية عند ترددات منخفضة بسبب استقطاب الأقطاب الكهربائية. وجد أن نموذج القفز المترابط (CBH) هو أفضل نموذج لتفسير البيانات حيث كانت أعلى قيمة لحاجز الجهد (wm) 0.21 eV. تم قياس طيف الانبعاث لفيلم الكينوزان الرقيق حيث أظهر مخطط CIE انبعاث ضوء أبيض. تشير هذه النتائج إلى أن الكينوزان يمكن استخدامه في الثنائيات العضوية الباعثة للضوء (OLEDs).