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Synthesis and Characterization Of PVC.$Zn_{(Etx)2}$ Thin Film Fabricated Via The Spin Coating Method

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Abstract:

Pure Polyvinyl chloride (PVC) and Polyvinyl chloride/Diethylzinc (PVC.$Zn_{(etx)2}$) thin films were prepared on glass substrates using the spin coating technique. The effect of $Zn_{(etx)2}$ doping ratio (0, 0.2, 0.4, 0.6 wt%) on the properties of the prepared films was studied. The Field-emission scanning electron microscope (FE-SEM) was used to examine the morphological properties of the thin films, and it was found that all films have a homogeneous surface with very few cracks. The effect of $Zn_{(etx)2}$ on the functional groups of PVC was also studied using FTIR spectroscopy. The optical properties were studied based on the absorption and transmittance spectra of these thin films in the wavelength range (200-900 nm), as well as the electronic transitions of the prepared films. The type of transitions for these films was determined, and it was found that these films have a direct allowed optical energy gap (E_g), which decreases with increasing $Zn_{(etx)2}$ doping ratio, in addition to altering the behavior of the absorption spectrum after doping as the absorption of visible wavelengths increases, which makes it suitable for solar cell applications and applications that require absorption of visible wavelengths.

Keywords: Polyvinyl chloride, spin coting, $Zn_{(etx)2}$.**Note:** The research is based on an M.A thesis or a PhD dissertation (if any). No

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Introduction:

Polyvinyl chloride (PVC) is one of the most widely used plastics on the market, with a production of over 30 million tons per year, being the second thermoplastic in volume, only behind polyethylene (PE). Due to its low cost and versatility in processing, combined with its good physical and chemical properties, it has become a universal plastic with a wide variety of applications, both in its rigid form and in its flexible form, thanks to the incorporation of plasticizers (e.g. pipes, cable insulation, packaging and floor coverings). Flexible PVC is widely used in military applications, construction, biomedicine, children's products and food packaging (Ayodeji et al., 2023; Kudzin et al., 2023). Flexible PVC contains significant amounts of liquid plasticizer to obtain the appropriate physical properties for each application. During the past 50 years, this type of plasticizer has been used mainly in industry because it has excellent properties, including its chemical resistance to aqueous solutions (water, saliva, blood, etc.). This versatility means that it is used mainly not only in products such as flexible hoses but also in products that must take extreme safety and hygiene measures, such as blood bags and tubes, toys, intravenous feeding, and, naturally, medicines, pharmaceuticals, pharmaceutical products, and pharmaceutical products (Sabana et al., 2020). The molecular chains of PVC are slightly branched. The side groups are disordered (atactic) along the macromolecules. PVC is an amorphous thermoplastic (Michael, 2009) . The starting product for the polymerisation of PVC is vinyl chloride, also known as chloroethane (C_2H_3Cl). This compound is a colourless gas that liquefies at -14ºC. Liquefaction can also be carried out at room temperature at a pressure of 2.5 bar. It is synthesised from acetylene by adding hydrochloric acid, although the predominant synthesis industrially is from ethylene and chlorine, passing through 1,2-dichloroethane and the elimination of hydrochloric acid. Vinyl chloride is stored in pressure vessels at low temperatures and is considered carcinogenic from an occupational-safety perspective. PVC can be produced industrially by three different processes, (Emulsion polymerization, microsuspension polymerization, and mass polymerization (Elgharbawy, 2022, Andrady, 1999, Summers, 1997). The addition of modifiers improves the properties of PVC and its

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transformation. In many cases, this addition is of vital importance, given the many applications that PVC has and is more extensively studied than other thermoplastics. It is worth highlighting, due to its importance, those modifiers capable of stabilizing PVC to heat or light (Xianbao, 2022). Diethylzinc ($Zn_{(etx)2}$) is an organometallic compound represented by the chemical formula $C_4H_{10}Zn$, including two ethyl groups (C_2H_5) covalently bound to a zinc (Zn) atoms. Diethylzinc is classified under the category of organozinc compounds, which are a subset of metal-organic compounds. These compounds are typically synthesized via the covalent bonding of one or more organic groups with metal atoms. They possess distinctive physical and chemical properties and are extensively used in organic synthesis and materials research. It is used in semiconductor manufacturing for the MOCVD (metal-organic chemical vapor deposition) method for epitaxial growth, as well as serving as a catalyst for polymerization processes. It may enhance reaction efficiency and augment product characteristics. In organic synthesis, diethylzinc may engage in many reactions, including the addition to aldehydes to yield secondary alcohols and the addition to unsaturated bonds. It may also substitute the more reactive Grignard reagent in certain processes (National Center, 2025, George and Sutton, 2001). Thin films are used in several domains; they constitute sophisticated materials with diverse compositions, including polymers, inorganic and organic molecules, membranes, and more. A thin film is formed when atoms or molecules deposit onto the surface of a substrate (Baker, 2005). The use of thin films in opto-electronic devices has advanced rapidly in recent years. Thin film materials have been extensively used in the conductivity and insulation of electronic circuits, electronic devices, photovoltaics, optical coatings across numerous domains, and in micro- and nano-electronics. These films possess a thickness ranging from 1 to 100 nanometers on the nanometric scale. Thin films may be produced by several deposition techniques based on the substance to be applied (Mainak, 2024, Muhammad et al, 2020). In this work, thin films of PVC doped with a diethylzinc were prepared by the spin-coating method on glass substrates, and the effect of increasing the doping ratio on the structural and optical properties of these films was studied.

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Experimental Part:

PVC $(C_2H_3Cl)_n$ provided by Fluka-A.G, Diethylzinc or $Zn_{(etx)2}$ ($C_4H_{10}Zn$) provided by Aldrich. PVC mixed with different weight ratio of $Zn_{(etx)2}$ (0, 0.2, 0.4, 0.6 wt%), and dissolved in tetrahydrofuran (THF) provided by Riedel-dehen Co. Thin film of PVC and PVC.$Zn_{(etx)2}$ prepared on glass substrates by the spin coating method. The glass plate was cleaned with ultrasonic bath water and then flowed with acetone. The spin coating apparatuses' rotation reaches 3000 rpm for 30 sec. During centrifugal spinning, the evaporated solution leads increased viscosity and the formation of a solid film. The absorbance and transmittance were measured with UV-Vis spectrophotometer (SHIMADZU UV-1650 PC) at the range 200-900 nm. Fourier transform infrared spectroscopy FTIR was used to study the structure bonds of the samples using a FTIR Shimadzu spectrophotometer model 8300, FE-SEM type TESCAN MIRA3 was used to study the morphology of PVC and PVC.$Zn_{(etx)2}$ thin films.

Results and Discussion:

Figure 1 shows the FE-SEM images of the pure PVC and PVC.$Zn_{(etx)2}$ thin films doped with different weight ratios of $Zn_{(etx)2}$. The images show that all samples are very similar and have an even distribution of material particles. Some small clusters of particles are present, but these decreases as the amount of $Zn_{(etx)2}$ increases. It is noted that the morphology of the prepared samples is more regular and homogeneous with an increasing weight ratio of $Zn_{(etx)2}$. The $Zn_{(etx)2}$ helps enhance the structure of PVC by improving how zinc interacts with the polar groups. This leads to better distribution and a more uniform shape in the PVC's morphology.

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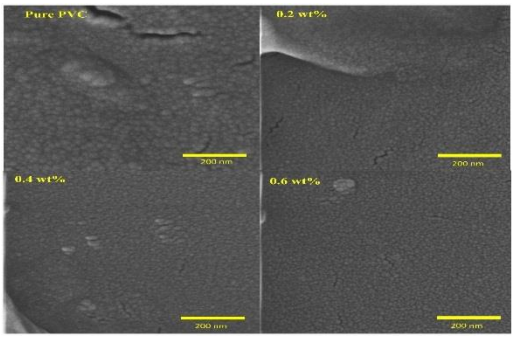

Figure 1. FE-SEM images of pure PVC thin films and doped with $Zn_{(etx)2}$ at different wt%.

Figure 2 shows the FTIR spectra pattern of pure PVC and PVC.$Zn_{(etx)2}$ thin films. In pure PVC thin films, a set of peaks is observed, at wavenumbers 607 and 678 cm^{-1} corresponding to C-Cl bonds, 1250, 1324 and 1421 cm^{-1} refer CH_2 (bending vibrations) groups, 2901 and 2960 cm^{-1} refer to CH_2 (stretching vibrations), 1726 cm^{-1} refer to C=O bonds. which agree with the standard bonds of the PVC polymer (Mayank, 2014 , Roy, 2017). It is noted from the spectrum of the doped samples that there is a clear change in the group of peaks confined in the range 400 to 1450 cm^{-1}. This is due to the effect of $Zn_{(etx)2}$, as the peak at that wavenumber 487 and 533 cm^{-1} indicates to Zn-O and Zn-Cl bonds, 1011 cm^{-1} refer to C-Zn bands, it is also noted that there is a slight shift in some peaks, as well as an increase in the intensity of the peaks with the increase in the percentage of doped $Zn_{(etx)2}$. This is consistent with what was obtained in the literature (Guedri et al, 2020, Amani et al, 2022).

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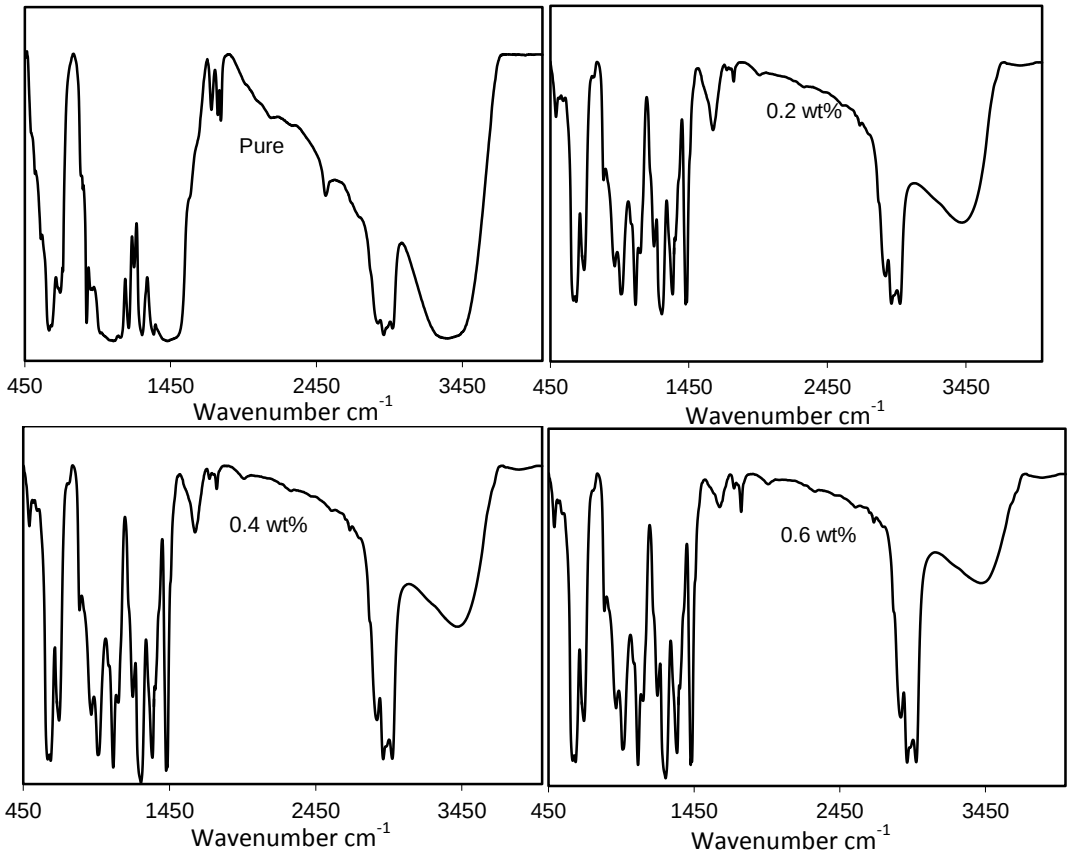

Figure 2. The FTIR spectra samples.

Figure 3 represents the absorbance as a function of wavelength for pure PVC and PVC.$Zn_{(etx)2}$ thin films. From the figure, it is noted that the absorbance of pure PVC thin films decreases with increasing wavelength, with a peak at wavelength 285 nm, attributed to the $\pi - \pi^*$ transition (Mudassir et al, 2015). When doping with $Zn_{(etx)2}$, an increase in absorbance is observed with increasing doping ratio. This effect is attributed to the appearance of a new localized state within the energy gap due to doping, which increases the absorption process. In addition, the behavior of the absorbance of PVC.$Zn_{(etx)2}$ thin films change with wavelength, as the absorbance begins to increase after wavelength 355 nm. The variation is attributed to the appearance of new n- π^* transitions

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after doping (Dyari et al, 2025), which causes the absorption of lower energy photons.

Figure 4 represents the transmittance as a function of wavelength for pure PVC and PVC.$Zn_{(etx)2}$ thin films, and from it the opposite behavior of the absorbance spectrum is observed, and this is attributed to the nature of the relationship between absorbance and transmittance. The absorption coefficient α in the basic absorption region was calculated using the following relationship (Surajudeen et al, 2023, Rana 2024):

$$\alpha = 2.303\ A/\ t$$

Where A represents the thin films absorbance, t represents the thin films thickness.

Figure 5 shows the change in the absorption coefficient as a function of the incident photon energy for pure PVC and PVC.$Zn_{(etx)2}$ thin films, the absorption coefficient values were $\alpha > 10^4$ (Chand et al, 2022), which indicates a high probability of direct electronic transitions, as the allowed direct transition occurs when the momentum is conserved ($\Delta\acute{K}=0$), and the high energies at which these values were calculated are the energy gap. The absorption coefficient is low at low photon energies, in which the probability of electronic transitions is low, and the absorption coefficient values increase at the absorption edge towards high energies. The forbidden optical gap energy E_g for the allowed direct transition is given by the following relation (Chand et al, 2022):

$$\alpha h\nu = A^*(h\nu - E_g)^{1/2}$$

Where: A* is a constant that depends on the probability of transition.

In direct transition, the process of photon absorption is a direct process and does not require the assistance of lattice vibration. When a photon is absorbed, the electron is excited directly in a perpendicular direction from the valence band to the conduction band without changing the vector Ḱ or the momentum of the crystal since the photon momentum is minimal. In some materials, the quantum selection rules prevent direct transitions at $\acute{K}=0$ but allow them at $\acute{K}\neq0$, and the probability of transition increases with $\acute{K}^2$. Figure 6 represents the relationship between $(\alpha h\nu)^2$ and the photon energy ($h\nu$) for pure PVC and PVC.$Zn_{(etx)2}$ thin films, and from it the value of the optical energy gap can be calculated, which is noted to decrease with increasing $Zn_{(etx)2}$ doping ratio (Table 1). This decreased

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value is due to what was previously mentioned about creating localized levels within the energy gap due to doping.

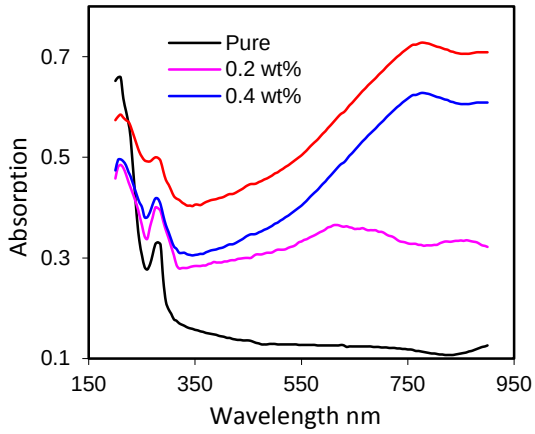

Figure 3. The variation of absorption spectra with wavelength of pure PVC and doped thin films at different $Zn_{(etx)2}$ wt%.

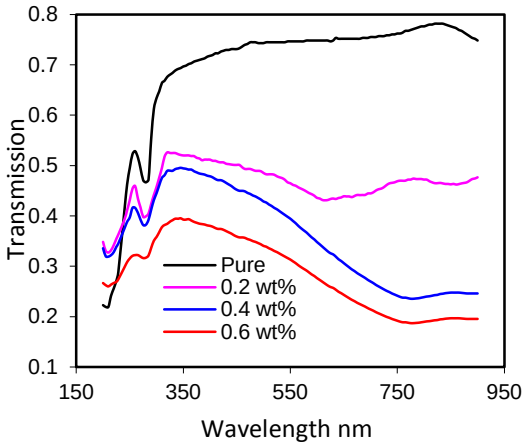

Figure 4. The variation of transmission spectra with wavelength of pure PVC and doped thin films at different $Zn_{(etx)2}$ wt% .

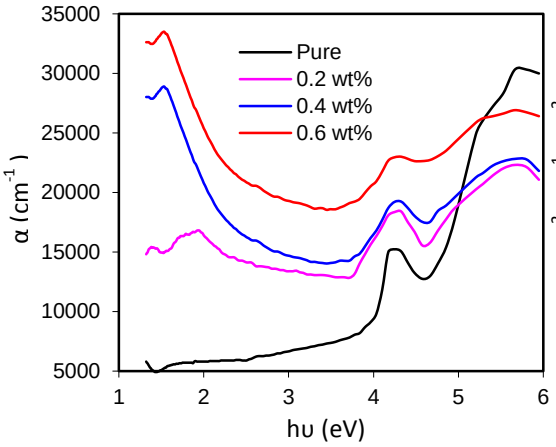

Figure 5. The variation of α with hυ of pure PVC and doped thin films at different $Zn_{(etx)2}$ wt%.

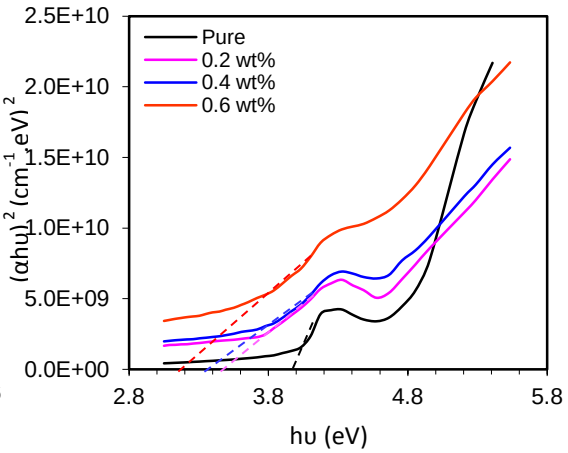

Figure 6. The variation of $(\alpha h\upsilon)^{1/2}$ with hυ pure PVC and doped thin films at different $Zn_{(etx)2}$ wt%.

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Table 1: Effect of $Zn_{(etx)2}$ on E_g PVC and PVC.$Zn_{(etx)2}$ thin films.

$Zn_{(etx)2}$ wt%	E_g eV
0	4
0.2	3.51
0.4	3.41
0.6	3.22

Conclusion

It is concluded from the above results that highly homogeneous pure PVC and PVC.$Zn_{(etx)2}$ thin films can be prepared using the spin coating method. Doping with $Zn_{(etx)2}$ leads to improvement of both morphological and optical properties. The homogeneity increases with increasing $Zn_{(etx)2}$ ratio, and the optical energy gap decreases with it. It is evident from the study that the absorbance behavior changes after doping, especially at 0.6% $Zn_{(etx)2}$, to be highly absorbent for visible wavelengths after the pure samples were semi-transparent for these wavelengths. According to the behavior of absorption and absorption coefficient, the prepared samples are promising in solar cell applications as they have absorption in the visible region or can be used in applications that require light absorption.

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تخليق وتوصيف اغشية PVC.$Zn_{(etx)2}$ الرقيقة المحضرة بطريقة الطلاء الدوراني

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مستخلص البحث:
تم تحضير الاغشية الرقيقة من PVC النقي وPVC.$Zn_{(etx)2}$ على ركائز زجاجية باستخدام تقنية الطلاء الدوراني. دُرس تأثير نسبة التشويب لـ $Zn_{(etx)2}$ (0، 0.2، 0.4، 0.6) wt% على خصائص الأغشية المُحضّرة. استُخدم المجهر الإلكتروني الماسح ذو تاثير المجال `، لتحديد الخصائص المورفولوجية للأغشية الرقيقة، ووُجد أن جميع الأغشية لها سطح متجانس مع شقوق قليلة جدًا. كما دُرس تأثير $Zn_{(etx)2}$ على المجاميع الفعالة لبولي كلوريد الفينيل باستخدام تقنية تحويل فوربيه للأشعة تحت الحمراء. دُرست الخصائص البصرية بناءً على أطياف الامتصاص والنفاذية لهذه الأغشية الرقيقة في نطاق الطول الموجي (200-900 نانومتر)، بالإضافة إلى الانتقالات الإلكترونية للأغشية المُحضّرة. تم تحديد نوع التحولات لهذه الأغشية، ووجد أن هذه الأغشية تمتلك فجوة طاقة بصرية مباشرة مسموحة وأن هذه قيمتها تتناقص مع زيادة نسبة التشويب لـ $Zn_{(etx)2}$، بالإضافة إلى تغير سلوك طيف الامتصاص بعد التشويب حيث زيادة امتصاص للاطوال الموجية المرئية، مما يجعلها مناسبة لتطبيقات الخلايا الشمسية والتطبيقات التي تتطلب امتصاص الأطوال الموجية المرئية.

الكلمات المفتاحية: بولي فينيل كلوريد، $Zn_{(etx)2}$، الطلاء الدوراني.
ملاحظة: هل البحث مستل من رسالة ماجستير او اطروحة دكتوراه؟ كلا