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A study of the biophysical properties of polystyrene films incorporated with clove oil as bio-based plasticizer

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Abstract

Polystyrene based films loaded with different concentrations of clove oil (0, 10, 20, 30 and 40 wt.%) were prepared using solution casting technique. The properties of the novel composites are characterized using dielectric spectroscopy (DS), attenuated total reflection-fourier transform infrared spectroscopy (ATR-FTIR), scanning electron microscope (SEM), X-ray diffraction (XRD),UV absorption spectra, contact angle measurements, mechanical analysis, in addition to test the anti-bacterial effect of the composites against two strains of microorganisms: *Escherichia coli*(gram-negative bacteria) and *Staphylococcus aureus* (gram-positive bacteria). According to dielectric study, by increasing the CLO content both The permittivity ε', dielectric loss ε'' and the electrical conductivity σ increases. The contact angle measurements indicate that, the PS-CLO films demonstrate hydrophilic behavior compared to the pure PS. Tensile strength σ_R values decreased by increasing CLO content and elongation at break ε_R values were found to be increased. The increase in ε_R may be due to the softening effect of CLO. The incorporation of the clove oil improved the light transmittance of the films. PS-CLO 40% sample have antibacterial activity against both *E. coli* and *S. aureus.*

Keywords: polystyrene; clove oil; dielectric; mechanical; optical properties

Introduction

Polystyrene(PS) is an inexpensive, environmentally friendly polymer it is among the most popular materials which has many applications in industry, building and construction, domestic appliances and food packaging. In food packaging, Polystyrene can be used in many shapes like monolayer plastic film, plastic sheet, or injection molded and foamed[1]. Plasticizers usually used in the industry for improving the workability of the polymers by lowering the glass transition temperature (Tg). Polystyrene is compatible with most plasticizers but it is usually plasticized with butadiene rubber. The use of natural biodegradable plasticizers, with good compatibility low migration and low toxicity for substitution of the conventional synthetic plasticizers now has gained much interest. Among these natural plasticizers essential oils which represent a renewable resources that can be used as valuable materials to obtain new polymer products.

Essential oils incorporation in polymers affects the properties of the polymer matrix, the changes are influenced by the oil composition and interaction between the polymer and the oil components. The oil structure usually reveals its efficiency as an active ingredient[2].

Films of poly vinyl chloride loaded with 2%, 10% and 30% (w/w) orange essential oil (OEO) were prepared and studied for food packaging purpose by Da Silva et al., 2018. The results revealed that addition of the (OEO) supports the antimicrobial properties of the films. In addition to provide flexibility to the films and that the 30 wt% OEO films have protective effect for gamma radiation[3]. Díaz-Acosta, Edgar, et al. 2018 fabricated polystyrene (PS) and Low density polyethylene (LDPE) films incorporated with different concentrations (0.5-1.5 % v/v) of oregano essential oil (OEO) for antimicrobial packing applications. Results confirmed that,the antimicrobial activity of the films increased with OEO concentration. OEO addition did not affect the LDPE mechanical properties. [4].

Clove oil is an essential oil extracted from Eugenia caryophyllata it is commonly used as a herbal medicine or food ingredient[5]. It has antiseptic and analgesic properties and is usually applied to cure tooth and gum pain.The main active ingredients of clove oil are (70–95%) eugenol (2-methoxy-4-[prop-

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2-enyl]phenol), (up to 20 %) eugenol acetate and (12–17 %). β-caryophyllene[5].

The aim of the present work is to prepare new composites films based on polystyrene and clove oil with different concentrations. Study the effect of clove oil as a bio-based plasticizer on the dielectric, optical, mechanical and structural properties of the polymer, in addition to microbiological investigation and contact angle measurements.

2. Materials and methods

2.1. Materials

Polystyrene, Mw: 350,000 was purchased from Sigma-Aldrich.

Clove oil from Egyptian market.The strains used in this study are Gram-Negative Bacteria: (*Escherichia coli* (ATCC25922). Gram Positive Bacteria: *Staphylococcus aureus* (ATCC 6538).

2.2. Methods

2.2.1. Preparation of the composites films

Different ratios of Polystyrene -clove oil composites were prepared by solvent casting method using Benzene as a solvent. Either the oil or the polymer was dissolved in benzene solution separately to obtain a total concentration of 5 wt% of each component. Different Polystyrene -Clove oil composites ratios (90/10, 80/20, 70/30, 60/40) wt% were prepared by mixing calculated volume of each component together to obtain films of 0.3 mm thickness and mixed for one hour under magnetic stirring. The mixed solutions were then poured into Petri dishes and the solvent was slowly evaporated under ambient conditions. The resulting films were dried at 37 ^{o} C for 48 hours.

2.2.2. Attenuated Total Reflection- Fourier Transform Infrared Spectroscopy (ATR -FTIR)

The functional groups in the Polystyrene -clove oil composites backbones were identified and recorded using JASCO FT/IR 300 E (Tokyo, Japan). ATR-FTIR spectroscopy with range of measurements of 400–4000 cm^{-1}.

2.2.3. Scanning electron microscope (SEM)

The morphology of the surfaces, bulk, and structural changes of the polymer composites were examined using Scanning Electron Microscope. The electron micrographs were obtained by Scanning Electron Microscope (SEM Philips XL30Japan) model with energy dispersive spectroscopy (EDX) unit.

2.2.4. Mechanical tests

Mechanical properties of the PS-CLO system were determined according to the standard methods using an electronic Zwick tensile testing machine (model Z010, Germany), in accordance with ASTM D412 standard

2.2.5. X-Ray diffraction (XRD)

The amorphous or ordered nature for the prepared samples was examined by diffractometer (Bruker AXS D8 Advance) using a Cu Kα X-ray tube radiation (λ = 1.5406 A) with a scanning mode detector and a Gobel Mirror. Measurements were made over the range 4° to 70° on 2θscale with 0.4 s as a dwell time.

2.2.6. Dielectric measurements

The dielectric measurements were performed between 0.1 Hz and 10 MHz with an impedance analyzer (Schlumberger Solartron1260), an electrometer amplifier and measuring cell. The investigated samples with thicknesses 1–2 mm measured at room temperature with error in temperature measurements about ± 0.5^{o}C. The dielectric permittivity ε' and dielectric loss ε'' was measured with error in ε' and ε'' amounts to ± 1% and ± 3% respectively.

2.2.7. Optical absorption spectra

Transmission spectra of the studied samples were recorded at room temperature using UV/Vis. spectrophotometer (JASCO 630, Japan) where (λ) range from [190–1100] nm through transmission spectrum with a resolution of 2 nm to investigate the variation in the structure of the prepared Polystyrene -clove oil composites.

2.2.8. Contact angle measurement

Water contact angle measurements were carried out at room temperature with sessile drop method films were analyzed by contact angle tester. The testing liquid was distilled water. Nitrogen was used to eliminate dust particles from the surface. Static contact angles were measured by the sessile drop method. A minimum of 5drops were used per sample. Both sides of the film were analyzed. Five measurements were carried out for each sample an average values were calculated.

2.2.9. Antibacterial test

Inhibition of bacterial growth by the clove essential oil and PS-CLO films with different concentrations of the oil was investigated using disk diffusion methodology (BSAC Disk Diffusion Method. plates were inoculated with a standardized culture of *S. aureus* (ATCC 6538) and *E. coli* (ATCC 8739). samples (5 mm diameter and 2 mm thickness) were then placed onto the inoculated plates.Those plates were incubated overnight in air at 37 ^{0}C. The diameters of inhibition zones formed around the disks were measured.

3. Results and discussion

3.1. The appearance and morphology of the composites

Photographic images of the PS-CLO composite films are shown in Fig. 1. From the visual appearance, The PS-CLO films were uniform and smooth demonstrating that the oil components are regularly distributed. The transparency increased by increasing the oil content.

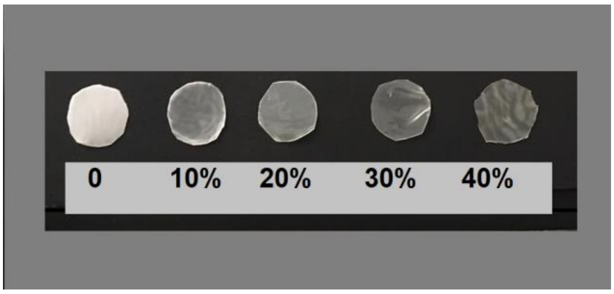


Fig. 1. appearance of PS-CLO films with different concentrations.

3.2. X-Ray diffraction (XRD)

XRD patterns of the prepared PS and PS –CLO composite films are shown in Fig. 2. As seen from the Fig. ure, there is no diffraction sharp peak observed in the spectra, which shows that the samples are indeed amorphous in nature. The Fig. ure presented the main broad diffraction peak for polystyrene at 2Ѳ=20.11º[6]. With increasing clove oil content the peak for the polystyrene become sharper and its width decreases. Markedly increased intensity of diffraction peak at 2Θ=20.11ºup to 20% clove oil, and with increasing oil ≥20 wt% there is certain decrease in intensity as shown in Fig. 3. The appearance of a new band around 41.45 indicating some type of interaction or formation of new phases due to gradual increasing clove oil 10-30 wt%. The increase of intensity of such band upto 20% oil pointing to increase in crystallinty or polymerization effect on polymer. Further, increase in clove oil content may cause disordered in the structure appears as absence of crystalline peak at 2Θ =41.45. By calculating the degree of crystallinity using (Material studio 8) program, the results are presented in Fig. 4 there were sudden increase in degree of crystallinity with increasing clove oil content reaching maximum value at 20 wt%, then it tends to decrease at higher oil content. The decrease of intensity related to disorder in the PS structure.

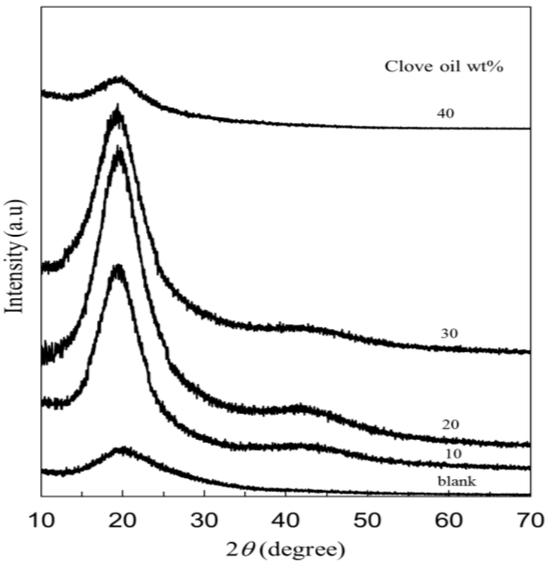


Fig. 2. XRD pattern of the polystyrene and polystyrene –clove oil composite films with different concentrations of clove oil.

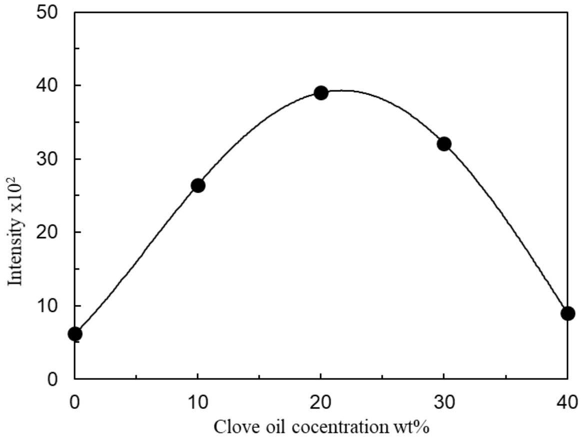


Fig. 3. The change of intensity of XRD peak of polystyrene and polystyrene –clove oil as function of clove oil concentration.

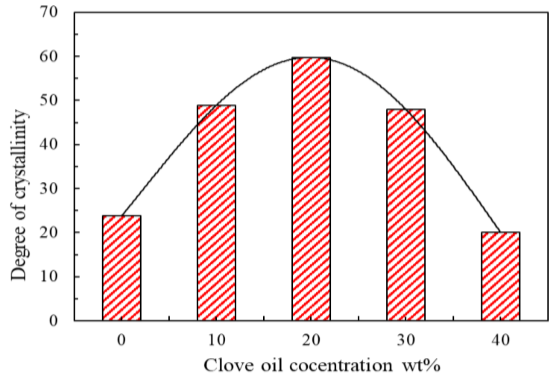


Fig. 4. Degree of crystallinity of PS and PS-CLO as function of clove oil concentration.

3.3. Scanning electron microscope (SEM)

SEM analysis of the fractured surface of the composite films was used to examine the degree of dispersion and miscibility of the clove oil in the polystyrene matrix. Fig. 5 displays the SEM micrographs for pure PS and PS-CLO composite films with 10 and 30wt% for sake of brevity. As seen from the micrographs, Pure PS film had a regular, homogeneous surface. After incorporation of the clove oil, the micrographs indicate that a homogeneous and regular aspect has been obtained. There is not any aggregation on some local area and the polymer composites exhibited layers with surface integrity in a continuous matrix. This can be attributed to a homogenous distribution of clove oil into the polymer matrix which indicates a good miscibility between PS and CLO plasticizer systems. These results also support the enhanced optical and mechanical properties of the composite films.

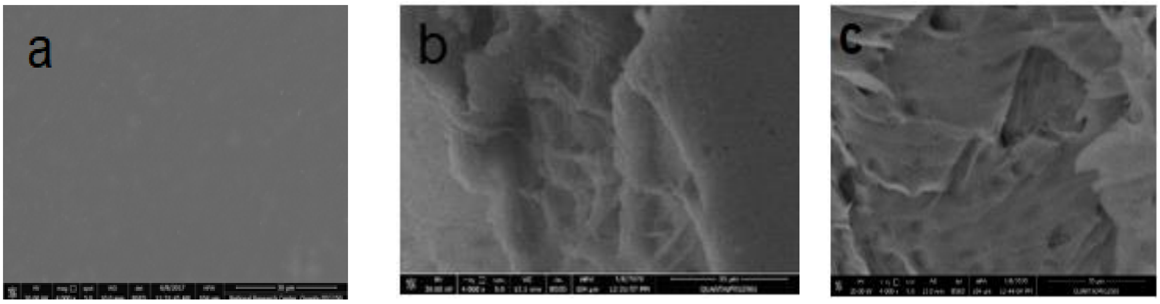


Fig. 5. SEM micrographs of PS-CLO films with different CLO concentrations ‘(a)0, (b)10% and (c) 30%.

3.4. Attenuated total reflection- Fourier transform infrared (ATR-FTIR)

Characterization of PS and PS-CLO composites was carried out by ATR-FTIR in the spectral range between 400 and 4000 cm^{-1}. Fig. 6 represented the spectrum of PS and clove oil. For pure PS, the spectrum shows bands at 686 cm^{-1} that refers to the ring deformation vibration. At 748 cm^{-1}, C-H deformation vibration band of hydrogen’s benzene ring. The two bands at 1451 cm^{-1} and 1600 cm^{-1} are attributed to aromatic C = C stretching. Whereas, the bands at 2970and 2870 cm^{-1} are referred to aliphatic and aromatic C-H stretching respectively. The main bands of PS have been identified in accordance with the literature[7] .

Clove oil's main constituent is Eugenol (2-methoxy-4-[prop-2-enyl]phenol[8].It forms about 75% of its constituent. It also contains secondary compounds like Isoeugenol (2-methoxy-4-(prop-1-enyl)-phenol)and Methyl eugenol (1,2-dimethoxy-4-prop-2-en-1-ylbenzene. the FTIR spectrum of clove oil is displayed in Fig. 6. As seen in Fig. 6 the spectrum shows eugenol characteristic bands,(O—H stretching) at 3452 cm^{-1}, C—O bending at 1226 cm^{-1} and C—C stretching vibrations of the phenyl ring at 1600 cm^{-1}, 1517 cm^{-1}referred to axial deformation of C=C group in the aromatic ring, 1430 cm^{-1}. Sharp peak at 1746 cm^{-1}ascribed to the stretching vibration of the ester carbonyl group of the triglycerides have been detected. Bands in the region between 910 and 740cm^{-1} attributed to deformation outside the plane of C-H group of aromatic rings. The spectrum of CLO in agreement with that previously reported[9]and [10].

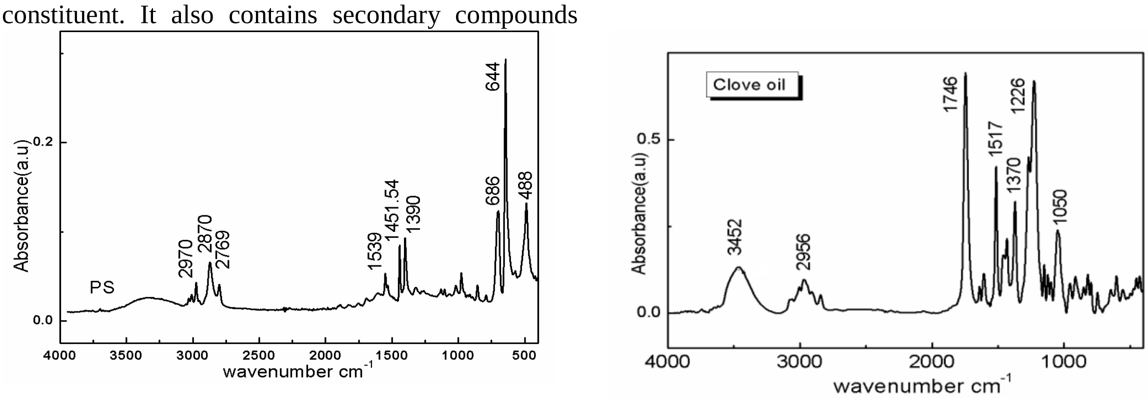


Fig. 6. ATR-FTIR spectra for polystyrene and clove oil

The ATR-FTIR spectra for Clove oil loaded PS films with different concentrations are displayed in Fig. 7. As seen, the spectra showed a new band appeared at 1746cm^{-1} attributed the carbonyl group C=O which present in the structure of CLO. In addition, new bands at 1036, 1216, 1360 cm^{-1}has been observed, these bands increase in intensity with increasing of the oil content. Besides, there were shift in the band at 644and 686$cm^{-1}$to 695 and 740cm^{-1} respectively. From the previous results it is seen that incorporation of the clove oil could alter the structure of the PS matrix.

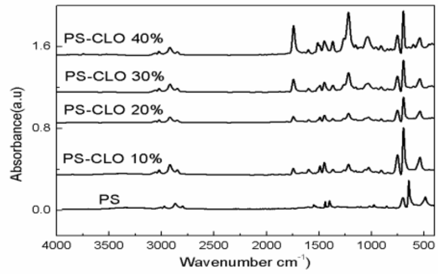


Fig. 7. ATR-FTIR spectra of the polystyrene and polystyrene –clove oil composite films with different concentrations of clove oil.

3.5. Optical absorption spectra

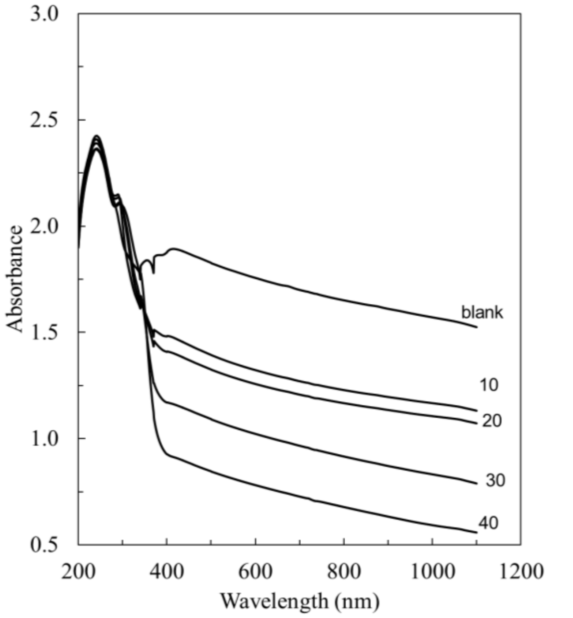


Fig. 8. The UV/Vis. absorption spectra of the PS-CLO films.

The variation of the optical absorbance as a function of photon wavelength in the range of 200-1100 nm is presented in Fig. 8. In general, the obtained spectra have an absorption band at 244 nm that may be attributed to the electronic transition π→π∗ (k-band) due to the presence of C=O group within the structures of PS polymer[11] . The absorption edge is lying in the UV region was shifted towards higher wavelength. This means that the optical band gap values of the PS-CLO system decreases as the oil content increasing in the polymer structure.

The optical behavior of the PS-CLO films, the optical energy band gap (E_g) and refractive index (n) are calculated using the following equations:

$$\alpha = 2.303\,\frac{A}{t} \qquad (1)$$

$$(\alpha h\upsilon)^2 = B(h\upsilon - E_g) \quad \text{For the direct transition (2)}$$

$$\frac{n^2-1}{n^2+1} = 1-\sqrt{\frac{E_g}{20}} \qquad (3)$$

where, αis the absorption coefficient, A is the absorbance, t is the film thickness, $h\nu$ is the photon energy, B is a constant. Fig. 9 shows the $(\alpha h\nu)^2$ versus $h\nu$ for the PS-CLO films. The obtained E_g and n values are shown in Fig. 10 that described the decrease of E_g(3.45-3.1 eV) values and increase n values. The slightly increase of n values between 1.95-2.02 with increasing oil content may be due to the variation in packing density/molecular weight distributions and the improvement of inter-atomic spacing of these samples[12]. As presented by the XRD analysis, the crystallinity regions due to the filling process induces the phenomenon of light interference throughout the matrix resulting in the observed increase in n values.

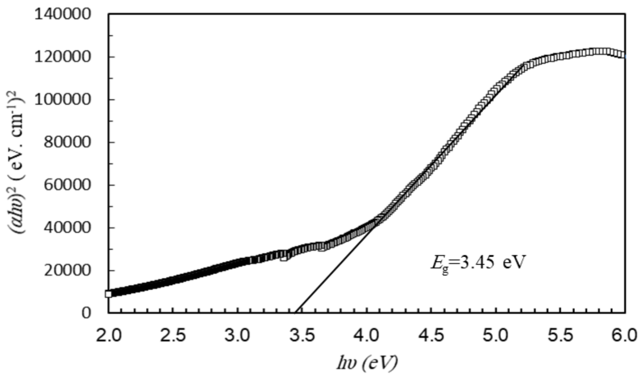


Fig. 9. (αhν)2 versus hνplots of blank sample as an example for the determination of indirect (E_g) energy band gap.

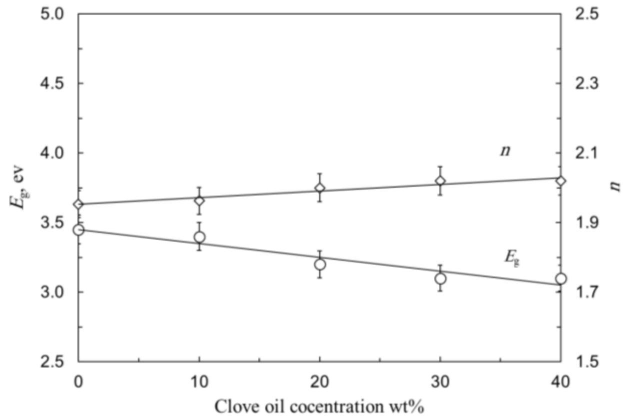


Fig. 10. Refractive index and energy gap of PS-CLO composite films.

3.6. Dielectric Properties

The permittivity ε', and dielectric loss ε", were measured over a wide range of frequency 10^{-2} up to 10^8 Hz and at room temperature 25^0C.

The obtained data were illustrated graphically in Fig.11. From this Fig- ure it is seen that both values increase by increasing CLO content in the sample. The values of ε', for pure PS were found to be comparable with those found in literature which was about 2.5 at 10 kHz[13]. For CLO the literature value of ε' was 2.7 at 50 k Hz.

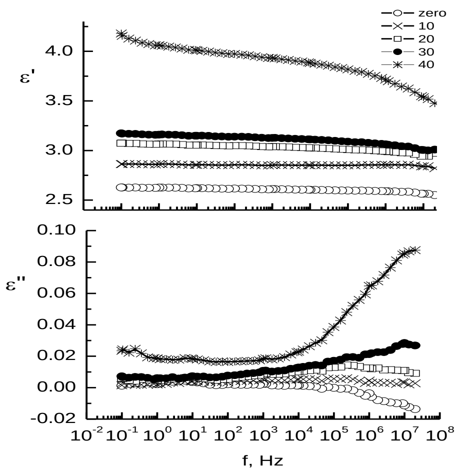


Fig.11.The permittivity ε', and dielectric loss ε', for PS-CLO composites films versus the applied frequency at room temperature 250C.

The permittivity ε' is found to be slightly decrease by increasing the applied frequency showing good frequency independent until 30% CLO. However for 40% CLO ε' shows pronounced decrease by increasing the applied frequency. As it is well known, the dielectric properties are gotten from the polarization of various structure units by increasing the applied frequency, for example, ionic, interfacial, and electronic polarization. The pronounced decrease in ε' was ascribed to the decrease in dipole and electronic polarizations.

The dielectric loss ε'', was found to increase by increasing CLO content. By increasing the applied frequency it is noticed that ε'' values are slightly decrease until 1 kHz while it sharply increase while f is higher than 1 kHz. So, the curves relating ε'' and the applied frequency was found to be more complicated and does not ascribe one absorption process for such reason it was worth to analyze such curves by using dielectric functions[14] in order to understand the molecular dynamics and how could the PS matrix affected the addition of different percentages of CLO.

Example of the analyses which were done by using two Frohlish terms and a Havriliak–Negami function[14]was given in Fig. 12.

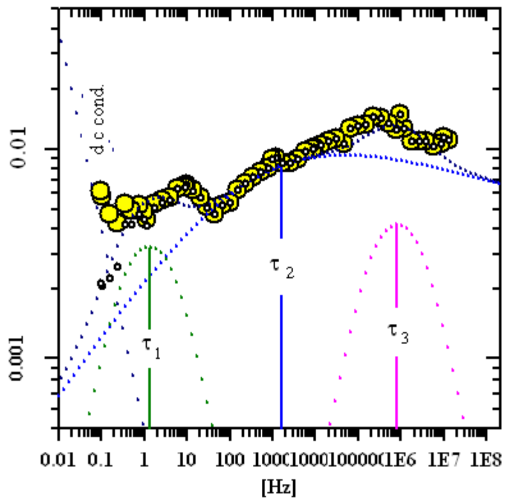


Fig. 12. Example of the analysis for PS-CLO 20%.

From this Figure it is seen that after subtraction of the losses due to the dc conductivity, three relaxation processes were detected. The first one, τ_1 , fitted by Frohlich function[14] was found to be unaffected by the percentage of oil is ascribing the losses due to the electrode polarization which usually appears at such frequency range[15].

The secondτ_2 and third τ_3 ,processes which were fitted by Havriliak–Negami function and Frohlish functions respectively[14], could ascribe the movement of the side chain and the attached groups respectively[14].

To understand how both τ_2 and τ_3 values are affected by the addition of oil, both values are presented graphically versus oil content in Fig. 13.

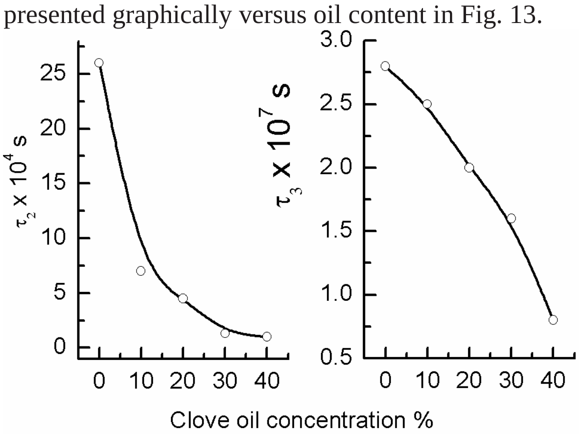


Fig. 13. Second and third relaxation times versus oil content.

The obtained values of the second τ_2 and third τ_3 relaxation times were plotted versus oil content in Fig. 13. From this Figure it is seen that both τ_2 and τ_3 decreased by increasing oil content this decrease is due to the flexibility of the polymeric chain which let the movement is easy under the influence of the electric field and consequently the decrease in relaxation processes was notice.

The dc electrical conductivity was calculated for all the samples under investigation and the obtained data were illustrated graphically versus oil content in Fig. 14. From this Fig. it is seen that the electrical conductivity σ increases by increasing the oil content but still lies in the order of 10^{-16} S cm^{-1} this finding highly recommend such composites to be used in insulation purposes.

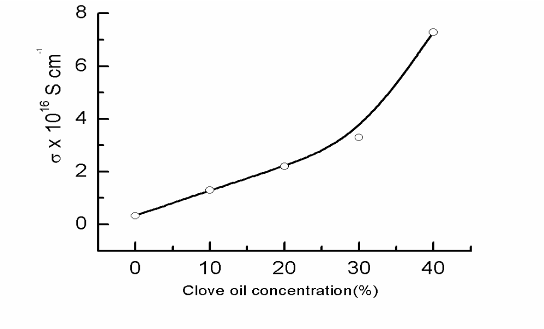


Fig. 14. Electrical conductivity σ versus CLO content

Fig. 15 represent the relation between Optical transmittance (%) and real part of logarithm electrical conductivity for PS-CLO with different concentration of clove oil. A transmittance increases with increasing oil content. The transmittance in the range between (2-18%). Also, the logarithm of the electrical conductivity increases with increasing the oil content. It is well known that, the scattering of light, the linear transmission of light throughout material, crystal in semi crystalline polymer, or additives in polymer composites have a resistance due to the presence of micro-heterogeneities[16]. So, the transmitted light in the direction of incoming light source is much reduced depending mainly on the heterogeneities centration. As a result the transparency is directly proportional to the electrical conductivity depending on the structure and agree with XRD.

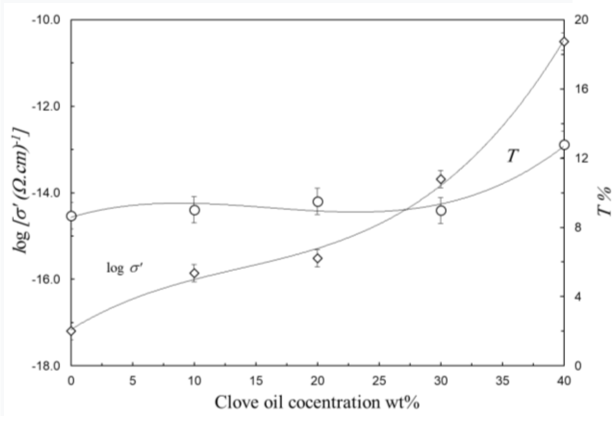


Fig. 15. Optical transmittance (%) and real part of logarithm electrical conductivity for PS-CLO.

3.7. Mechanical properties

Tensile strength σ_R and elongation at break ε_R are parameters characterized the stress distribution within the polymeric matrix and describe the rupture path within it. σ_R and ε_R were measured for PS-CLO composite films with various percentages of CLO and the obtained date illustrated graphically in Fig. 16.

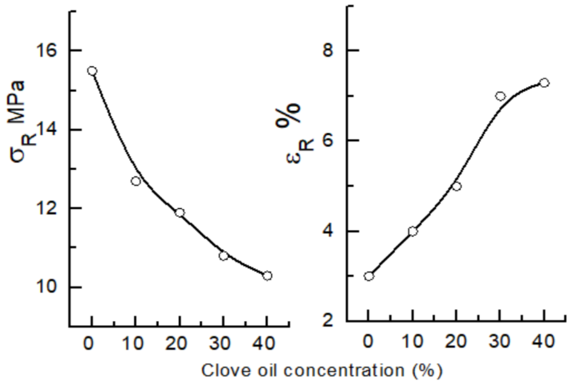


Fig. 16. Tensile strength σ_R and elongation at break ε_R for PS-CLO composite films.

From this Fig. it is seen that σ_R values decrease by increasing CLO content which is logic as PS is considered to be a hard polymer with high σ_R value. The addition of CLO may induce the plasticizing effect of such composites which improve the mechanical integrity of such composites[17]. On the other hand, ε_R values were found to increase by increasing CLO content, the increase in ε_R may be due to the softening effect of CLO. From Fig. 4, it is clear that ε_R increase pronouncedly uptill 30% after this value a slight increase was obtained at 40wt % CLO.

3.8. Contact angle

Biocompatibility and biodegradability are key parameters for polymeric materials that used in medical, agriculture applications or in food packaging. Improvement of these properties could be reached by suitable immobilization of active coatings on the surface of the polymer surface. A major obstacle is often poor adhesion due to the poor wettability[18]. For greenhouse covering, anti-fogging films that have hydrophilic surface are required. These surface scan change the water drops that form from the condensation process to water layer that can flow down easily and doesn't reflect the light[19].

In this study the contact angle of 0, 20 and 40% PS-CLO composite films were measured at room temperature with water as a testing liquid. Contact angle reported is the mean of three measurements (±SD).The data recorded in table1. Fig. 17 is the representative pictures for the measurements.

Table1. Contact angles (degrees) for PS –CLO composite films (0,20 and 40%).

Sample	PS	PS-CLO (20%)	PS-CLO (40%)
Contact angle(°)	87±1	65.9±0.4	22.1±0.6

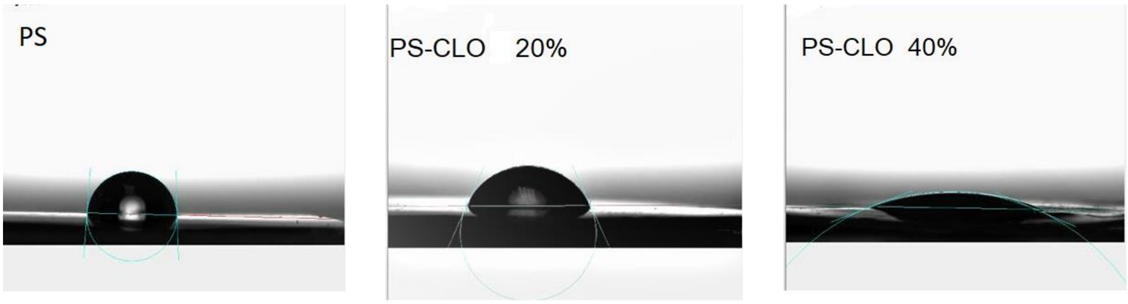


Fig. 17. Appearance of a water droplet for (a) 0, (b) 20% and (c) 40%PS-CLO samples.

As seen in Table1, the contact angle of blank polystyrene was about 87°±1 which is comparable with that previously reported[20].The contact angle for PS-CLO films decreased by increasing the oil content which means increased wettability of the PS. Similar trend observed for other polymers after addition of essential oils[21-23]. This decreasing may be due to introducing polar groups to the surfaceof the films which confirmed by the FTIR measurements. WANG et al.2012 reported that, increase polar groups at the material surface triggers reduction of the contact angle[24].

3.9. Antibacterial activity of the films

Antibacterial activity of the clove oil, PS and PS-CLO composite films was tested against gram positive bacteria: *S.aureus* and gram negative bacteria: *E. coli*. The inhibition zones for pure clove oil and PS-CLO 40% are shown in Fig. 18.The results illustrated that pure clove essential oil has clear effect on both the tested bacteria with Inhibition zone diameter range (14.5- 16.5mm) for *E.coli and S. aureus* respectively.

Ps and PS-CLO samples with concentrations(10-30%) have not any antimicrobial effect on both the tested microorganisms, while the PS-CLO 40% composite film showed antibacterial activity with inhibition zone diameter of 4 and 5.2mm, for *E. coli and S. aureus respectively*. It is well known that the nature and quantity of the essential oil and the ratio of essential oil to polymer in the film and the probable interactions between the active compounds of EO and the polymer play a principal role in the film's antibacterial properties. The disappear of the antibacterial effect in the PS-CLO 10-30% films in spite of the high efficiency of the pure clove oil may be attributed to loss of volatile compounds during the drying of the films therefore high concentrations of clove essential oils are needed to reach significant antibacterial effect.

4. Conclusion

Novel films based on polystyrene and clove oil has been successfully prepared. Characteristics of PS-CLO composites films were examined and the results were analyzed. According to the FT-IR spectra**,** Incorporation of clove oil into polystyrene matrix alters the structure and properties of the pristine polymer. SEM micrographs of the 20 and 40wt% CLO films show homogenous distribution of clove oil into the polymer matrix which indicates a good miscibility between PS and CLO.

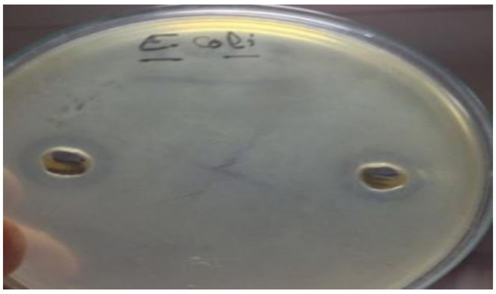
Optical study of the samples pointed to decrease of E_g(3.45-3.1 eV) values and increase n values with increasing oil content. The slightly increase of *n* values between 1.95-2.02 may be due to the variation in packing density/molecular weight distributions and the improvement of inter-atomic spacing of these samples. CLO increased the PS film's transparency. Enhanced the crystalline structure of PS upto20% CLO concentration.

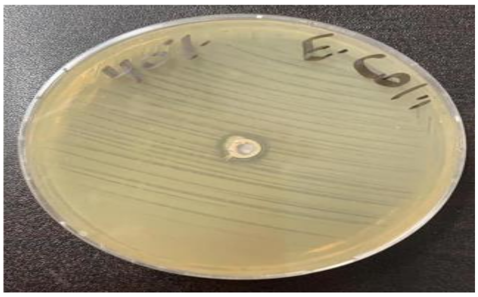
The Dielectric measurements indicated that, by increasing CLO content both the dielectric parameters ε' and ε" were increased, the relaxation times τ_2 and τ_3 were decreased and The electrical conductivity σ increases but still lies in the order of 10^{-16} S cm^{-1}. The decrease in relaxation time reflects flexibility of the polymer chains due to the presence of CLO. Which confirmed by the mechanical analysis that revealed a decrease in the tensile strength σ_R of the films and an increase in the elongation at break ε_R with increasing the oil concentration.

As indicated by the contact angle measurements there were an increase in the wettability of the films by increasing the oil content.

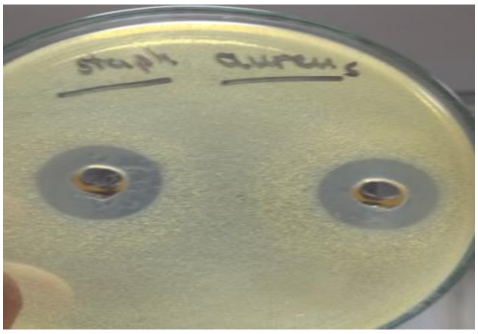
PS films containing CLO 40wt% exhibited antibacterial activity against both tested bacteria (*E-coli and S-aureus*) whereas no activity shown for the lower concentration films. From the previous results it is concluded that, the enhancement of wettability, transparency and flexibility of the PS-CLO films make them interesting material in many applications such as packaging or in agriculture as greenhouse films.

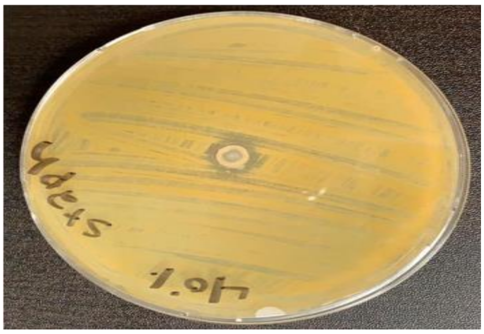
E. coli





S.aureus





Clove oil PS-CO 40%

Conflict of interest statement

The authors state that they have no conflicts of interest.

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