

ENHANCEMENT OF THE SURFACE DIELECTRIC AND PHOTODIELECTRIC PROPERTIES OF LOW DENSITY POLYETHYLENE BY ADDING EMODIN

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Abstract: In this paper, we report surface dielectric and photodielectric properties of low density polyethylene (LDPE)/Emodin composite. Emodin was extracted from *Fallopia japonica* plant. The composite was obtained from the xylene solution. Dielectric measurements were performed in the frequency range from 240 Hz to 4 kHz and the temperature range from 20 °C – 70 °C. Irradiations of the sample were applied before and during measurement with selected wavelengths from UV to the visible range. Surface dielectric conductance and susceptance of the LDPE/Emodin composite is higher than value of conductance and susceptance of pure LDPE. We found an increase, by one order of magnitude, in conductance and susceptance of the LDPE/Emodin composite with increase of measurement frequency. Furthermore, the surface conductance and susceptance increase due to UV radiation in mentioned temperature range, but during visible radiation at frequency 1 kHz (except in the case green LED lamp) we observed the enhance of conductance only at temperature range up to 50 °C.

Keywords: dielectric properties, photodielectric properties; LDPE, emodin, composite.

1. INTRODUCTION

Fallopia japonica is a Polygonaceae plant, which is widespread across Asia and North America. In recent years, this plant has received increasing global attention due to its high natural phenol content. *F. japonica*, also known as Japanese knotweed, is used as Chinese herbal medicine. Emodin is the most extensively studied anthraquinone which displayed a vast number of pharmacological properties such as antitumor, immunosuppressive, antimicrobial, anti-inflammatory and antidiabetic [1–5].

Low density polyethylene (LDPE) is a one of largest used polymer owing to good electrical insulation and chemical properties with good combination of properties such as flexibility, solvent resistance and its low cost. Significant improvements in the properties of LDPE are achieved by incorporation different fillers into the polymer matrix. Attention of scientists was focused mainly on the mechanical,

thermal and electrical properties of LDPE composites [6-12].

LDPE is non - polar polymer and to enhance LDPE conductivity in matrix, conductive fillers were added such as carbon black, carbon nanotubes, metal particles and so on [6, 13]. In the paper [6] Kazimi and coworkers have found conductivity of LDPE/PANI nano fibers blend about 10^{-4} S/cm. Sabet and coworkers [13] have shown that electrical conductivity of LDPE increases by about nine orders of magnitude at nanotube loadings from 0-10 wt%. Materials which are not photoactive could be stimulated by presence of the external field [14] or by adding some photoactive molecules.

Emodin is organic molecule which has delocalized π -electrons and can absorb light, create photogenerated charge carriers and transport these charges [15]. Based on the literature reviewed, emodin has not been used as a filler in LDPE. In order to improve photodielectric properties of LDPE, we

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have had an idea to make composite LDPE/Emodin because emodin structure, its polarity due to the hydroxyl group and presence of π - electrons trough double bonds in the ring.

In this paper, the emodin was used as a filler in LDPE. Dielectric and photodielectric properties of the LDPE/Emodin composite were studied because it could be promising material for solar cell and photo-sensing application.

2. EXPERIMENTAL

2.1. Materials

Low density polyetilene (LDPE) with a density of 910 kgm^{-3} was received as pellet and prepared in the shape of sheet. Xylene was supplied by Sinex laboratory and used as received without any further treatment. Method for Emodin extraction was described in following paragraph.

2.2. Extraction of Emodin

An Fallopia japonica sample (20 g) was ground and extracted with methanol ($200 \text{ ml} \times 3$) to yield the crude extract [16-18]. The crude extract of Fallopia japonica, which has two active anthra-noid compounds, emodin and physcion, was used. Emodin was extracted from F. japonica in the following way: the crude extract was reconstituted with water and with chloroform (CHl) and after

reconstitution, emodin was purified from the active chloroform fraction. Chemical structure of emodin was shown in the Figure 1..

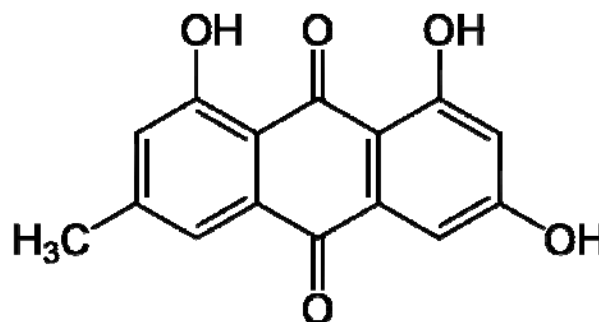


Figure 1. Chemical structure of emodin $C_{15}H_{10}O_5$ (2-methyl-4, 5, 7-trihydroxyanthraquinone)

2.3. Preparation of LDPE/Emodin composite

Emodin was dissolved in 50 ml of Xylene by magnetic stirring at $140 \text{ }^\circ\text{C}$ for 15 min and treated under ultrasonication for 10 min to place the Emodin homogenously. LDPE was also dissolved in 150 ml Xylene at $180 \text{ }^\circ\text{C}$, (Fig.2.). LDPE/Xylene and Emodin/Xylene were mixed together and stirred for 15 min at $180 \text{ }^\circ\text{C}$. The mixture was poured into glass mold and air dried at room temperature for 10 days and vacuum dried for another 10 days. After that, sample was hot pressed at $180 \text{ }^\circ\text{C}$ for 8 min under a pressure of 50 bar (5 MPa).

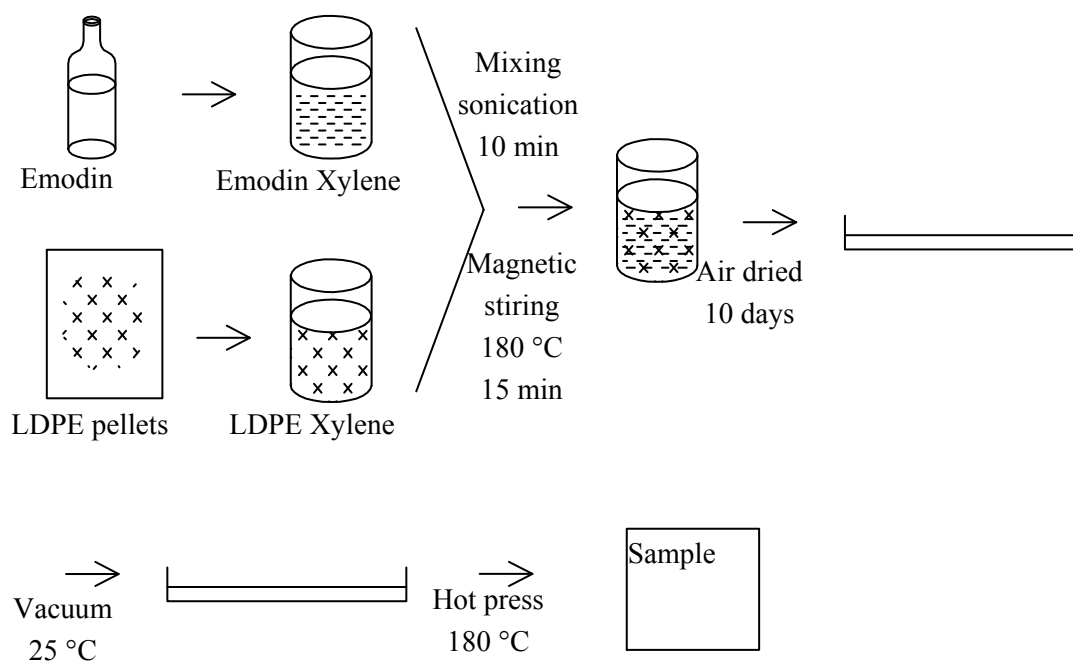


Figure 2. Schemes of the process for the fabrication of LDPE/Emodin composite

2.4. Method

Equipment used for FTIR analysing was Bruker Tensor 27. FTIR Bruker Tenson is a spectrometer that determines the material structure by applying IR beam on the sample and measure the ration between the incident to transmittance wave. The wave range used was between 500 - 4500 cm^{-1} at a resolution 0.01 cm^{-1} . As a holder, Attenuated Total Reflectance (ATR) Platinum modul was used. LDPE and LDPE/Emodin were used as the samples during the analysis.

Dielectric and photodielectric measurements of the samples were performed on a Hameg 8118 instrument in the frequency range between 240 Hz and 4 kHz ($U_0 = 1 \text{ V}$) at temperature range from 20 °C do 70 °C and pressure 100 kPa. Conductance (G) and susceptance (B) were measured using the C_p mode of the instrument ($Y = G + iB$). Copper electrodes were glued to the surface of the sample in a comb shape. We report the specific values of the real part, G , and the imaginary part, B , of surface AC conductivity, obtained using the expression G (or B) = G_{measured} (or B_{measured}) $\times d/L$, where $L = 143 \text{ cm}$ is the length of the electrodes and $d = 1.5 \text{ mm}$ the distance between the electrodes. All measurements were performed in a laboratory made, shielded chamber. Dielectric characterization of the prepared samples was carried out using free sample setup. The sample in the chamber was leaned on a thin pla-

stic frame in order to maintain position in the chamber and uniform illumination.

For irradiation of the samples, we used a selected UV and LED lamps. UV lamps used in measurements have had wavelengths 256 nm and 365 nm and $P = 1 \text{ W}\cdot\text{m}^{-2}$. LED lamps selected wavelength: 620 nm; 520 nm; 440 nm and 395 nm and power $P = 17 \text{ W}\cdot\text{m}^{-2}$ are used too.

3. RESULTS AND DISCUSSION

3.1. FTIR Spectroscopy

LDPE commercial sample was analyzed under normal atmospheric conditions. Fig. 3. shows the transmittance spectra of LDPE and LDPE/Emodin composite. FTIR results for pure LDPE are marked with blue line. The transmittance peaks are located in a wide range of spectra scale. The peaks at 3395 cm^{-1} and 3184 cm^{-1} refer to the presence of hydroxyl group, i.e. for O - H stretching and/or H - bounding because of the presence of moisture on the sample [19]. Peaks at 2916 and 2848 cm^{-1} correspond to CH_2 asymmetric and symmetric stretching band respectively. The peaks between 1500 cm^{-1} - 700 cm^{-1} correspond to CH bending deformation, CH_3 symmetric deformation, twisting deformation and rocking deformation [19-22].

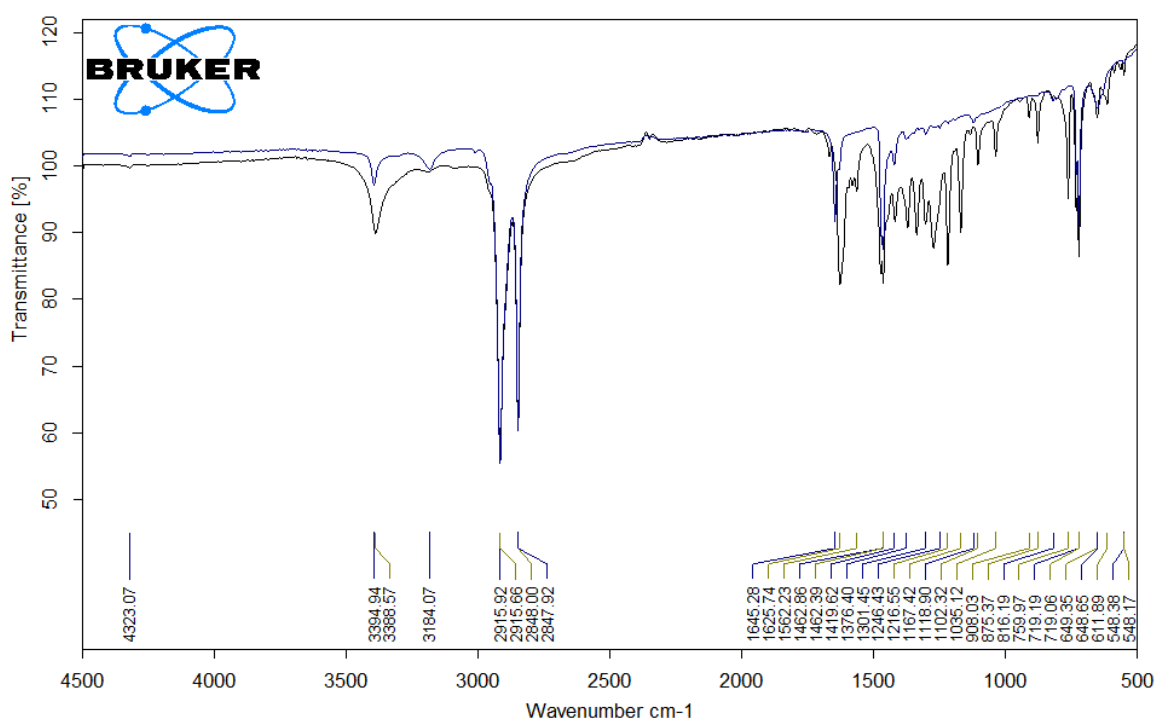


Figure 3. FTIR spectra: LDPE (blue line) and LDPE/Emodin composite (black line)

FTIR results for LDPE/Emodin in Fig 3. are marked with black line. As one could see from the Fig 3., besides LDPE characteristic peaks at FTIR spectrum, emodin peaks appear: O-H stretching at 3388.57 cm^{-1} , C=O stretching at 1625.74 cm^{-1} , C=C vibrations in the benzene ring near at 1562.23 cm^{-1} , C-C stretching in the ring at $875.37, 1035.12\text{ cm}^{-1}$, and O-H bending at 1369.62 cm^{-1} which is also confirmed by literature [22]. The FTIR of the composite show the significant change in the spectrum. The absorption peak of hydroxyl stretching has been shifted to lower wave number in composite (3392.54 to 3389 cm^{-1}) and an increase of its intensity showing the presence of absorbed water due to the presence of emodin. Appearance of strong peak at 1301.45 cm^{-1} corresponds to π -electron delocalization due to emodin planar structure [22, 23]. At composite spectrum, we have not found xylene peaks.

3.2. Surface dielectric spectroscopy

Specific surface conductance and susceptance versus temperature at 1 kHz for neat LDPE and

LDPE/Emodin composite are presented in Fig. 4. Other frequencies have shown similar behaviour and have not been presented in this paper. Specific surface conductance of the composite is significantly higher than conductance of neat LDPE at temperatures higher than $35\text{ }^\circ\text{C}$. Conductivity changes in LDPE by adding emodin were expressed in percentages relative to appropriate values of pure LDPE, according to following relations:

$$\delta G_{composite} = 100 \cdot (G_{composite} - G_{LDPE}) / G_{LDPE},$$

$$\delta B_{composite} = 100 \cdot (B_{composite} - B_{LDPE}) / B_{LDPE}.$$

At $58\text{ }^\circ\text{C}$, value of the specific surface conductance of the composite sample at 1 kHz is 340% higher than value of conductance of neat LDPE. At $50\text{ }^\circ\text{C}$, this increase is about 70%. At frequency 1 kHz, there is an increase in susceptance with the addition of emodin over the entire temperature range. At $25\text{ }^\circ\text{C}$, the increase in susceptance of the composite sample is about 60%, while in the range from $40\text{ }^\circ\text{C}$ to $50\text{ }^\circ\text{C}$ this increase in susceptance is about 110% for the composite sample, and at $70\text{ }^\circ\text{C}$ this increase is reduced to 19%.

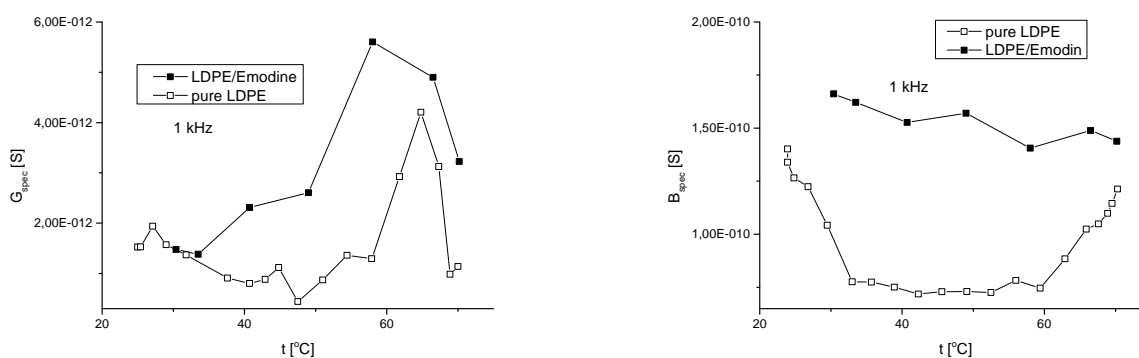


Figure 4. Specific surface conductance and susceptance versus temperature for neat LDPE and LDPE/Emodin composites

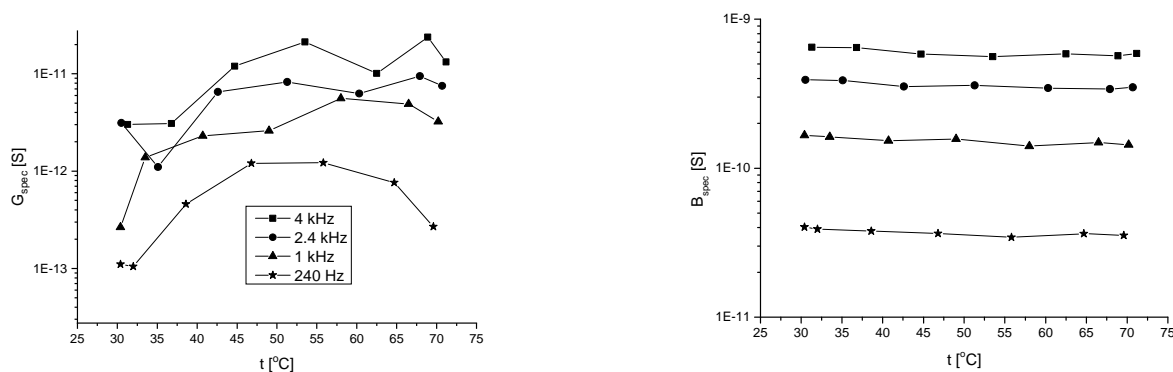


Figure 5. Specific surface conductance and susceptance versus temperature for LDPE/Emodin composites at selected frequencies

Figure 5. shows an increase in specific surface conductance and susceptance with increase in frequency in the whole temperature range. The increase in conductance is one order of magnitude at lower temperatures, while this increase reaches two orders of magnitude at higher temperatures. It was observed a maximum of surface conductance at 50 °C and 240 Hz. These maximums are also observed at higher frequencies but at elevated temperatures, about 68 °C.

3.3. Surface photodielectric spectroscopy

There are several papers that confirmed changes in conductance and susceptance of composites during irradiation [15, 24-28]. L. Csoka et al. have shown an increase of the surface AC conductivity of the paper samples by irradiation with white visible light, $\lambda \geq 405$ nm [24]. Mariya et al. [28] have shown an increase of the conductivity of indium-tin oxide film deposited on polyethyleneterephthalate substrate under UV irradiation ($\lambda = 365$ nm). They have established that the optimum time of illumination is 10 minutes and further UV treatment leads to polymer substrates degradation.

Figure 6 shows specific surface conductance and susceptance of the LDPE/Emodin composite irradiated with UV lamps (256 nm and 365 nm) for 30 min. Samples are kept in the dark for 5 min before measurements. Measurements of conductance and susceptance are performed in the dark in the tempe-

perature range from 20 °C - 70 °C. We observed an increase in specific surface conductance and susceptance due to UV irradiation. This increase is more pronounced for the sample irradiated with UV lamp at 256 nm. Specific surface conductance for sample irradiated with 365 nm at 40 °C is 40% higher compared to non-irradiated sample. Similar behavior is observed at 50 °C, while at 70 °C this increase is 200%. Specific surface conductance for sample irradiated with 256 nm has shown an increase compared with non-irradiated sample, too. These increases are 67% at 40 °C, 100% at 50 °C and 210% at 70 °C. Specific surface susceptance for the irradiated composite sample also shows an increase. This increase is also more pronounced for sample irradiated with 256 nm UV-lamp.

Changes in specific surface conductance and susceptance after irradiation with Vis-light are shown in Fig.7. Changes versus temperature are presented at 1 kHz for LDPE/Emodin composite. Measurements are performed in dark after irradiation with LED lamps selected wavelengths: 620 nm; 520 nm; 440 nm and 395 nm. With black line, presented is the sample before irradiation, irradiated sample changes are marked with appropriate colors. One could see that at elevated temperature, composite show a negative photo-induced changes in conductance, but positive photo-induced changes in susceptance. At temperatures up to 50 °C sample shows opposite behavior.

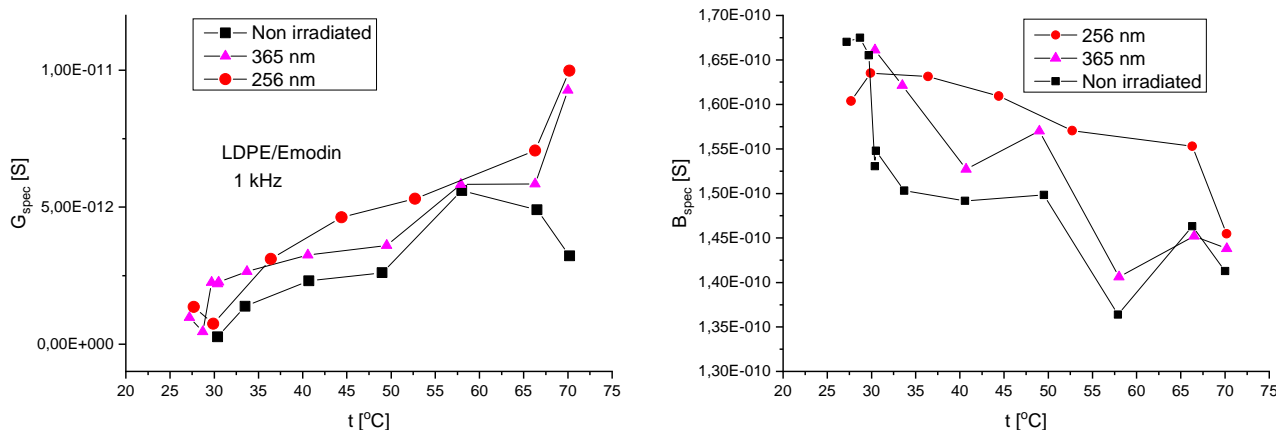


Figure 6. Specific surface conductance and susceptance depending of temperature at frequency 1 kHz for LDPE/Emodin composite measured in dark after irradiation 30 min with UV lamps: 256 nm and 365 nm

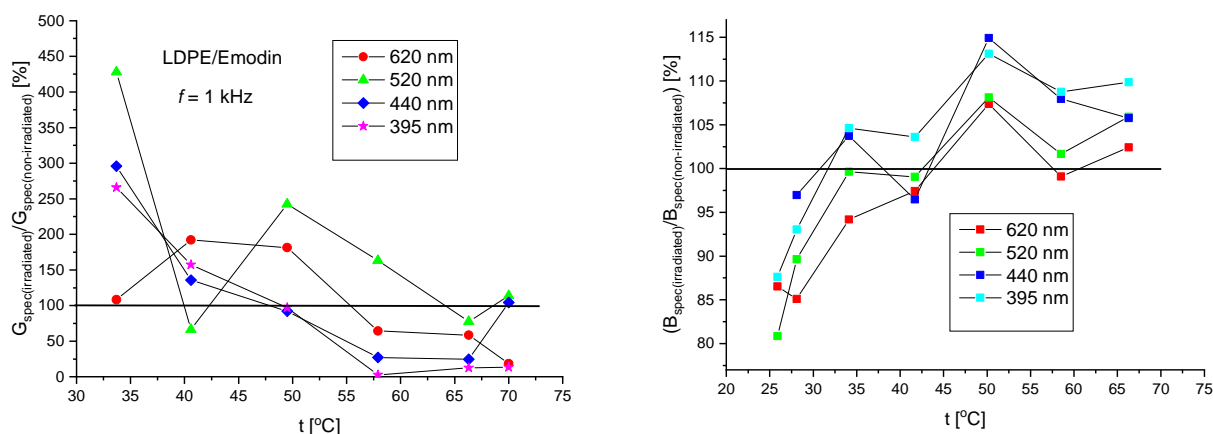


Figure 7. Changes in specific surface conductance and susceptance after irradiation versus temperature at 1 kHz for LDPE/Emodin composite (measurements were performed in dark after irradiation with LED lamps selected wavelengths: 620 nm; 520 nm; 440 nm and 395 nm)

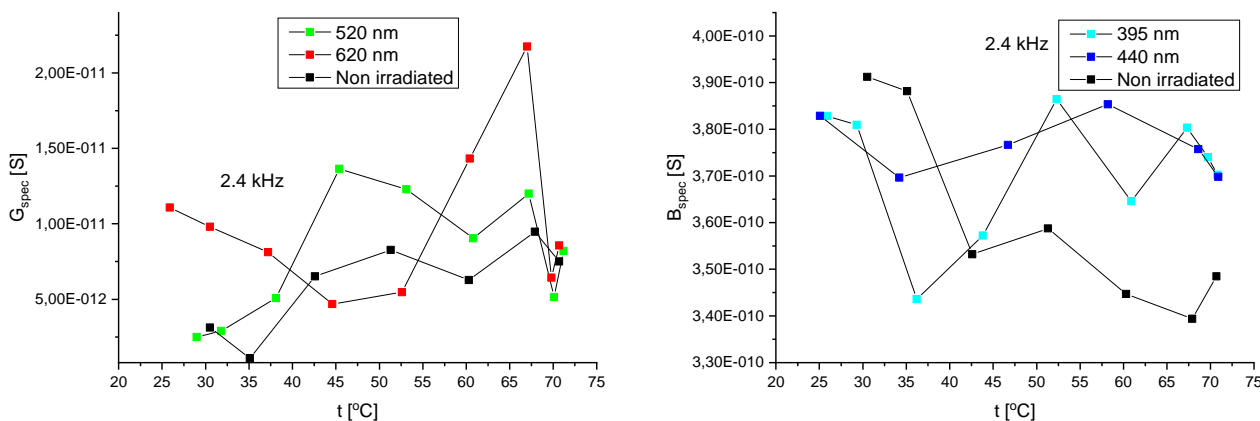


Figure 8. Specific surface conductance and susceptance versus temperature at 2.4 kHz for LDPE/Emodin composite measured in dark after irradiation with LED lamps selected wavelength: 620 nm and 520 nm for G ; 440 nm and 395 nm for B

Figure 8 shows specific surface conductance at 2.4 kHz after irradiation with green (520 nm) and red (620 nm) LED lamps in temperature range from 25 °C - 70 °C. We have observed an increase in conductance after irradiation with green LED lamp in temperature range up to 68 °C. At the same temperature, there is a maximum in conductance for sample irradiated with red LED lamp. At the figure on the right, we have showed specific surface susceptance after irradiation with LED lamps: blue 440 nm and light blue 395 nm. We have observed an increase in susceptance at temperatures above 40 °C. An increase is not noticed for samples irradiated with other LED lamps.

Percentage changes in specific surface conductance and susceptance versus temperature at 4 kHz after irradiating with a blue LED lamp; wavelength 440 nm and five times higher radiation

power is shown in Fig. 9. Changes in conductance (susceptance) after irradiation with blue LED lamp (440 nm; power 85 W/m²) were expressed in percentages relative to values of conductance (susceptance) irradiated with blue lamp (440 nm; power 17 W/m²), according to following relations:

$$G [\%] = 100 \cdot G_{spec1} / G_{spec2}, \rightarrow B [\%] = 100 \cdot B_{spec1} / B_{spec2}$$

G_{spec2} and B_{spec2} are values when sample was irradiated with a blue LED lamp, power 17 W/m², while G_{spec1} and B_{spec1} are values when sample was irradiated with a blue LED lamp, power 85 W/m². After sample irradiation with LED lamp 5 times higher power, we observed an increase in conductivity up to 290% at 65 °C, while the susceptance decrease by up to 20% over the all temperature range. Similar behavior is observed at other frequencies.

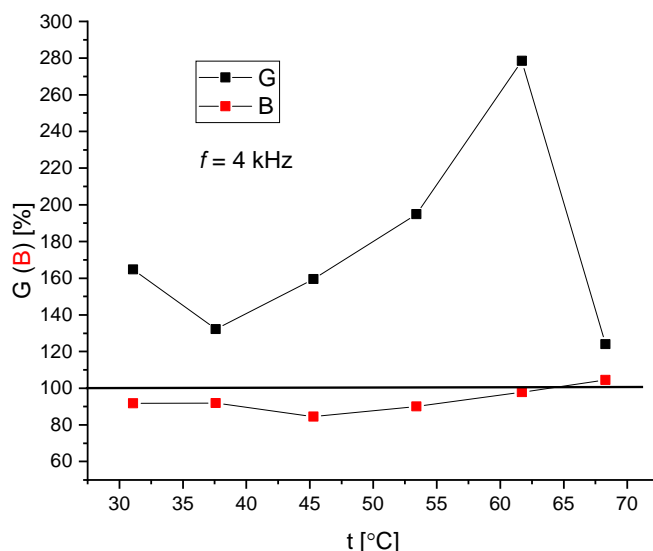


Figure 9. Percentage changes in specific surface conductance and susceptance versus temperature at 4 kHz after irradiating with a blue LED lamp; wavelength 440 nm with power 17 W/m² and power 85 W/m²

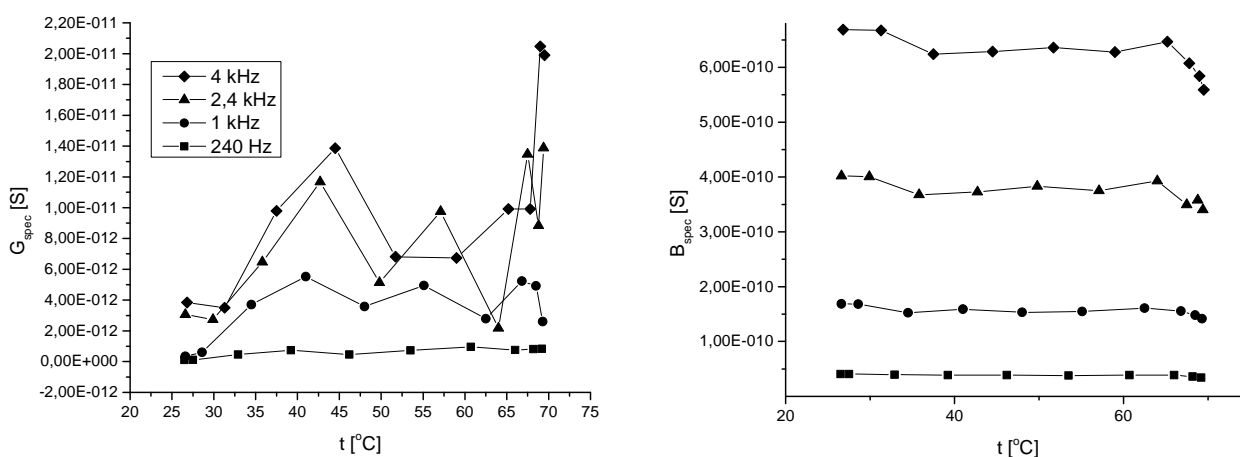


Figure 10. Specific surface conductance and susceptance for LDPE/Emodin composite during irradiation with LED lamp wavelength 440 nm versus temperature at selected frequencies: 240 Hz; 1 kHz; 2.4 kHz i 4 kHz

We observed a few maximums of the surface specific conductance during irradiation and warming (Fig. 10). The first maximum is placed in temperature range from 40 °C - 45 °C and we noticed a right shift with increase in frequency. The second maximum is placed at 55 °C and third one at 68 °C. At 2.4 kHz, second maximum is at 58 °C and third one appears at 70 °C. At 4 kHz, first maximum is at 45 °C and second one appears at 70 °C. Surface specific susceptance shows a slight decrease at the beginning of warming at selected frequencies.

Specific surface conductances for LDPE/Emodin composite during irradiation with 440 nm LED lamp versus temperature at frequencies 1 kHz and 2.4 kHz (blue) are presented in Fig. 11. With black line, we marked the conductance measured without irradiation. For 1 kHz, changes of conductance are: at 35 °C, 655% and at 40 °C, 140%. For 2.4 kHz, changes of conductance are: at 35 °C, 488%, at 42 °C, 156%, but at 60 °C increase was 250%.

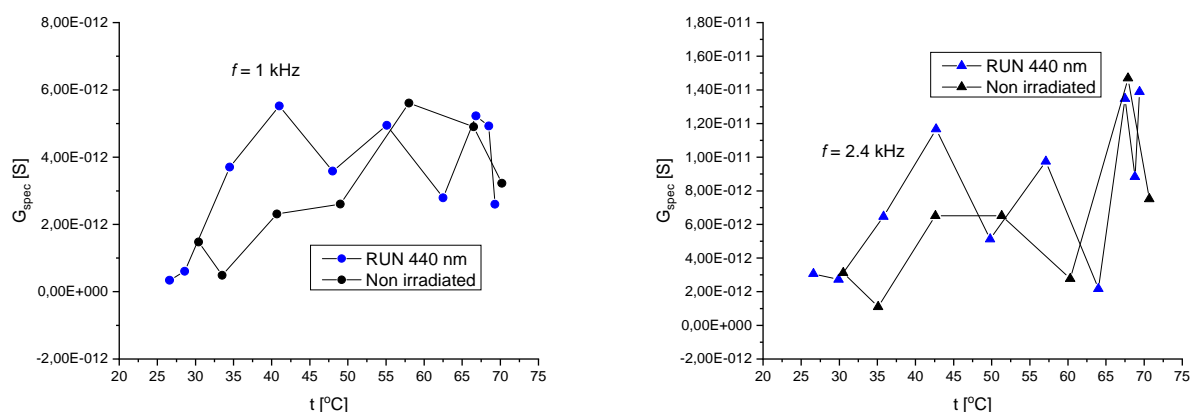


Figure 11. Specific surface conductance for LDPE/Emodin composite during irradiation with 440 nm LED lamp (blue) and without irradiation (black) versus temperature at frequencies 1 kHz and 2.4 kHz

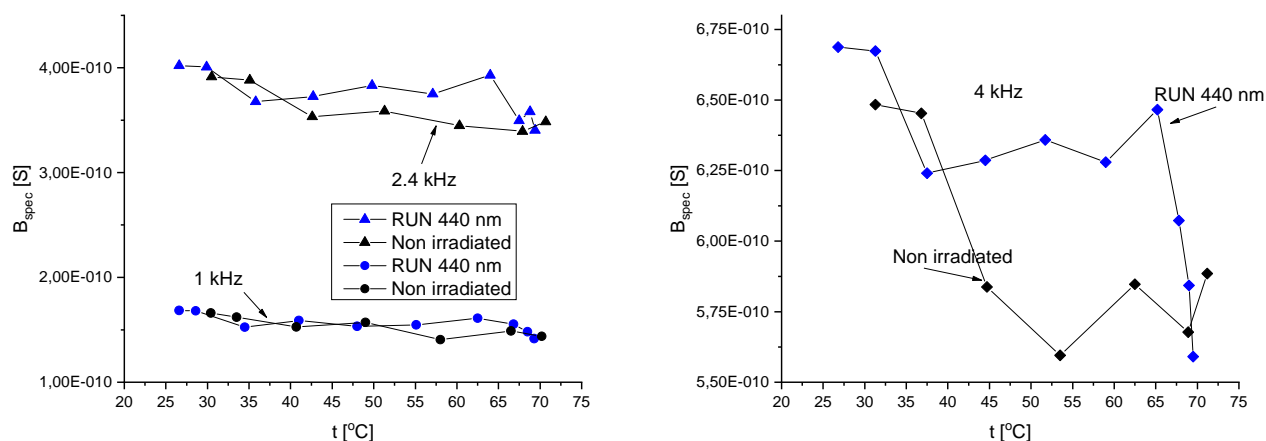


Figure 12. Specific surface susceptance for LDPE/Emodin composite during irradiation with 440 nm LED lamp versus temperature at frequencies 1 kHz, 2.4 kHz and 4 kHz (blue) and without irradiation (black)

Fig 12. presents specific surface susceptance for LDPE/Emodin composite during irradiation with 440 nm LED lamp versus temperature at frequencies: 1 kHz, 2.4 kHz and 4 kHz (blue) and without irradiation (black). One can see that during irradiation, there is an increase in susceptance but less pronounced than an increase in conductance. For example, an increase in susceptance at elevated temperature is by one order of magnitude smaller than increase in conductance.

4. CONCLUSION

The effect of emodin on the dielectric and photodielectric properties of LDPE has been studied. It was obtained an increase of specific surface conductance of LDPE by adding emodin, this increase was 340% at 58 °C and 1 kHz. Maximum change in susceptance was about 110%. Due to emodin struc-

ture, its polarity and presence of π – electrons, emodin could improve photodielectric properties of the LDPE. As we expected, LDPE/Emodin has shown an increase in conductivity during irradiation. The increase in conductance was about 350% at 35 °C by irradiation with green LED lamp (520 nm), while increases of the susceptances (in range from 5% to 15%) were observed at elevated temperature. By adding emodin in LDPE polymer matrix, we get environmentally friendly material with promising properties in solar cell and photo-sensing application.

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УНАПРЕЂИВАЊЕ ПОВРШИНСКИХ ДИЕЛЕКТРИЧНИХ И ФОТОДИЕЛЕКТРИЧНИХ ОСОБИНА ПОЛИЕТИЛЕНА НИСКЕ ГУСТИНЕ ДОДАВАЊЕМ ЕМОДИНА

Сажетак: У раду су истраживане површинске диелектричне и фотодиелектричне особине композита полиетилена ниске густине (ПЕНГ) и емодина. Емодин је добијен екстраховањем из биљке *F. japonica*. Композит је добијен из раствора ксилола. Диелектрична мјерења су вршена у опсегу фреквенција од 240 Hz до 4 kHz и у опсегу температура од 20 °C – 70 °C. Узорак је освјетљаван UV и VIS LED лампама одређених таласних дужина, прије и у току диелектричних мјерења. Додавањем емодина у ПЕНГ повећавају се и површинска диелектрична кондуктанса и сусцептанса. Уочава се и повећање кондуктансе и сусцептансе композита са повећањем фреквенције за један ред величине. Најзначајнији резултат рада је повећање површинске кондуктансе и сусцептансе приликом зрачења узорка UV лампама. Пораст се уочава у цијелом температурском мјерном опсегу. Узорци освјетљавани VIS LED лампама (осим у случају зелене LED лампе) на фреквенцији од 1 kHz показују повећање кондуктансе само при температурама нижим од 50 °C.

Кључне ријечи: диелектричне особине, фотодиелектричне особине, ПЕНГ, емодин, композит.

