

## DIELECTRIC PROPERTIES OF PP AND PP+DK<sub>1</sub> NANO-COMPOSITES IN DIFFERENT PERCENTAGE

**A.A. KHADIYEVA, H.S. IBRAGIMOVA, V.A. ALEKPEROVA, A.R. SADIGOVA**

*Institute of Physics Academy of Sciences of Azerbaijan,*

*H.Javid ave. 33, AZ-1143 Baku*

*hicran90@rambler.ru*

The dielectric properties of pure PP and PP+DK<sub>1</sub> nano-composites researched in alternating electric field are presented in the given paper. It is revealed that dielectric properties change in the dependence on field frequency and temperature and also on filler concentration. This is explained by relaxation losses which take place because of composite structural elements with mobility different degree and also with appearance of low-molecular impurities, for example with revealing of CO polar groups.

**Keywords:** nano-composite, polypropylene, nanogel, rest degree

**Pacs:** 79.60.Dp ; 78.66.Li; 78.30.Am

### INTRODUCTION

The obtaining and investigation of nano-composites is of interest in nano-technology field in the latest works. The obtaining of new materials consisting of disperse fillers with size 1-100nm uniting the photoluminescent, magnetic and catalytic characteristics are perspective ones. The investigations in the field of obtaining and properties of nano-composite materials strongly increase. Such dielectric characteristics of PP+DK<sub>1</sub> nano-composites as electrical conduction, processes of relaxation and polarization in alternating currents aren't studied enough and that's why the study of these properties in high-temperature and frequency interval is of great interest.

### THE EXPERIMENT TECHNIQUES AND TEST PREPARATION

The dielectric properties of PP+DK<sub>1</sub> nano-composites present in the given paper. The samples with thickness 140-170μm are used for measurement of dielectric properties in alternating electric field. The mixtures of PP and DK<sub>1</sub> powders are prepared in different component ratio and further the nano-composites in film form with further cooling are prepared from these mixtures by hot pressing at polymer matrix melting point under pressure 15MPa during 10 min.

The samples are obtained in different temperature-time crystallization regimes and especially by rapid cooling (RC) method in mixture ice-water with velocity 30 grad/min. RC samples are investigated by us. The samples for measurement of dielectric characteristics in alternating current are prepared in the disc form by diameter 20mm. The stable electric contact is supplied for electrodes from stainless steel by diameter 15mm applying the extruded electrodes from aluminum foil by thickness 7μm. The samples are put into cell with brake electrodes. Knowing the electrocapacity C and sample geometric sizes one can define the complex value of dielectric constant with the help of formula

$$C = \frac{\epsilon \epsilon_0 S}{d}$$

where C is electrocapacity of the samples, d is thickness, S is electrode thickness being on the surface,  $\epsilon_0 = 8,85 \cdot 10^{-12}$  is electric constant. The electric constant frequency changes from 25Hz up to  $10^6$  Hz and temperature changes from 20 up to 150°C. The obtained results are presented in the form of real and imaginary parts of dielectric constant ( $\epsilon, \epsilon'$ ),  $\text{tg}\delta$  dielectric loss on electric field frequency and temperature. The temperature dependences of  $\epsilon$  on  $\text{tg}\delta$  in obtained nano-composites at frequency 1 Hz are shown in fig.2. From the figure it is seen that  $\epsilon$  increase in nano-composite PP+10%DK<sub>1</sub> (where  $\epsilon = 3,1$ ) on 40% relatively pure PP ( $\epsilon = 2,0$ ) is observed,  $\epsilon$  for DK<sub>1</sub> filler is  $\epsilon = 2 - 8$  and  $\epsilon = 2$  in matrix [107]. Such dependence of  $\epsilon$  on T in nano-composite is defined by Maxwell-Wagner polarization. The values of surface energy, system conduction and other parameters of dielectric characteristics are close to percolation threshold in dependence on filler concentration.  $\epsilon$  decreases relatively pure PP in small concentrations. The clusters form the instability of phase interactions and particles in nano-gels in 2% filler content because of the difference in component surface energy. The surface of these clusters is less than sum surface of their component particles. The cluster number increases and dielectric layer between particles decreases with increase of filler volume content and this leads to increase of electric capacity (i.e. increase of  $\epsilon$ ). As it is seen from the figure 1 for composites PP+2%DK<sub>1</sub>  $\epsilon$  doesn't change at temperature increasing 290-330K. But,  $\epsilon = f(T)$  dependence character changes at following addition increase in PP+10%DK<sub>1</sub> composite. Firstly, the dielectric constant increases with temperature increasing and the strong  $\epsilon$  increase takes place in temperature interval of crystallite melting ( $\sim 393K$ ), further  $\epsilon$  doesn't change with temperature increasing. The temperature decrease on 15K in PP+10%DK<sub>1</sub> composites shifts to low-temperature region in comparison with pure polymer PP and PP+2%DK<sub>1</sub> composite. The dielectric losses significantly increase at consideration of  $\text{tg}\delta$  change in dependence on temperature in PP+10%DK<sub>1</sub> composite (fig.2).

At temperature 293K for pure PP  $\text{tg}\delta$  is  $0,007 \pm 0,001$  and  $0,025 \pm 0,001$  is for PP+10%DK<sub>1</sub>. The significant value of dielectric loss is connected with electric conduction.

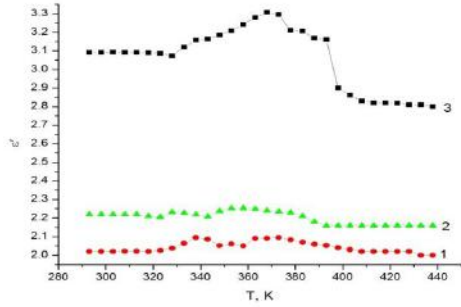


Fig.1. The dependence of real part of dielectric constant ( $\epsilon'$ ) in frequency 1kHz on temperature. 1 is pure PP, 2 is PP+2%DK<sub>1</sub>, 3 is PP+10%DK<sub>1</sub>.

From the dependence on addition volume content at frequency 1 kHz one can see the increase of electric conduction in PP+10%DK<sub>1</sub> composition in comparison with pure PP. From the fig. 2 it is seen the difference of temperature dependences of dielectric losses of pure PP and nano-composites. For pure PP the changes aren't observed and for nano-composites the character of dependence change  $tg\delta = f(T)$  differs, i.e.  $tg\delta$  increases with temperature increasing. The dependence has the maximum value at temperature of matrix smoothing, further decreasing it passes through minimum near crystallite melting point and it increases again with temperature. In this case, the melting point moves to the side of low-temperature region on 10-15K. Such change of dielectric losses in nano-composites PP+ DK<sub>1</sub> is totally correlated in the dependence on electric conduction on temperature in alternating field.

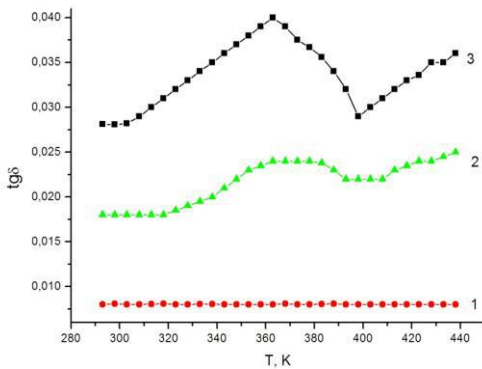


Fig.2. Temperature dependence of  $tg\delta$  in obtained nano-composites at frequency 1Hz. 1 is pure PP, 2 is PP+2%DK<sub>1</sub>, 3 is PP+10%DK<sub>1</sub>.

The frequency dependences of  $\epsilon$  and  $tg\delta$  at temperature 20°C are obtained. From the figures 3 and 4 it is seen that in pure PP and nano-composites at low volume content DK<sub>1</sub> additions aren't revealed with the frequency increase for  $\epsilon$  change.  $\epsilon$  decreases at big concentrations of addition in obtained nano-composites with frequency increasing. Such characteristic of dielectric loss at low frequencies one can explain by the kinetics of separate structural elements of nano-

composite. These structural elements take participation in relaxation processes with frequency increase. As it is mentioned in PP+DK<sub>1</sub> composite the accumulation of DK<sub>1</sub> particles take place in matrix separate parts. These accumulated particles are clusters distribute spontaneously in PP matrix. The cluster number increases with the increase of DK<sub>1</sub> volume content. The clusters closed with each other by sample thickness one can consider as active resistance. As DK<sub>1</sub> has the high conduction with the comparison with PP one so one can consider that composite resistance mainly will be defined by contacts between nano-gel particles.

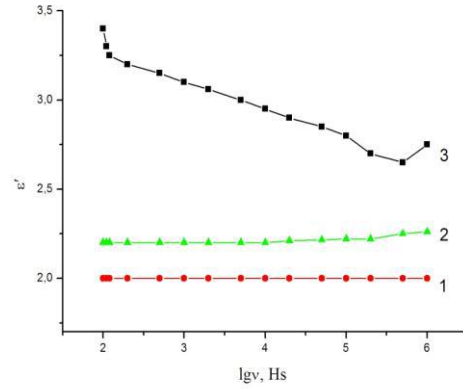


Fig.3. The dependences of ( $\epsilon'$ ) real part on ( $lgv$ ) frequency at temperature  $T=273$  K, 1 is pure PP, 2 is PP+2%DK<sub>1</sub>, 3 is PP+10%DK<sub>1</sub>.

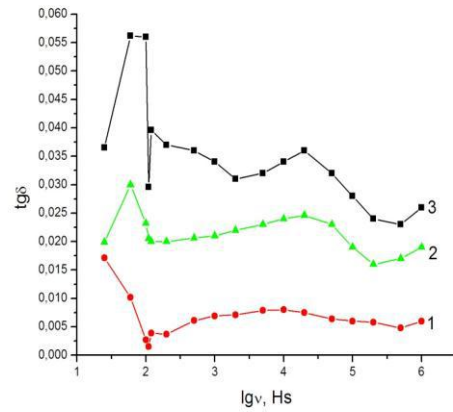


Fig.4. The dependences of dielectric losses at temperature  $T=293$  K on frequency ( $lgv$ ) 1 is pure PP, 2 is PP+2%DK<sub>1</sub>, 3 is PP+10%DK<sub>1</sub>.

The accumulation and redistribution of free electric charges (Maxwell-Wagner volume polarization) take place on boundaries of clusters in alternating field. By this fact one can explain the decrease of dielectric strength on DK<sub>1</sub> volume content. Such volume polarization distorts the initial internal electric field at absence of external electric field. It is known that at low frequencies the internal electric field distributes to conduction correspondingly at low frequencies and to dielectric conduction at high frequencies correspondingly.

Consequently,  $\varepsilon$  decrease with frequency increase can be explained by the appearance of comparably strong internal field in nano-gel –clusters.  $tg\delta$  dependence on frequency shows that for pure PP and for PP+2%DK<sub>1</sub> nano-composite the maximums at  $\nu=1.5 \cdot 10^4$  are observed, and also the positive dielectric effect at  $\nu=10^2-1.5 \cdot 10^4$  Hz frequency because of  $tg\delta$  increase in nano-composites is revealed. The inertia of space charge formation in nano-composite structure is observed with frequency increase and it leads to  $tg\delta$  decrease. At low frequencies the surface charge follows the field and dielectric losses decrease. The polarization in the field of high frequencies doesn't establish for field half-period and dielectric losses decrease. The maximum observable in average frequency region ( $\nu=1.5 \cdot 10^4$  Hz) of full scale is defined by relaxation losses. The relaxation losses can take place because of low-molecular compounds which have the mobility different degrees. In this case it is carried out at appearance of CO polarization groups. PP partial oxidation takes place in preparation process.  $tg\delta$  increase in the dependence on frequency depends on polarization process. It is established that the relaxation time in the investigated films at frequencies of less than

ones of  $\omega=2\pi/T$  variable signal is bigger than Maxwell dielectric relaxation ( $\tau_M = \varepsilon \varepsilon_0 \rho$  where  $\rho$  is sample specific resistance in  $\text{Ohm}\cdot\text{m}$ ) and at high frequencies is less than  $\tau_M$ . The external action time is approximately  $100\mu\text{sec}$  for our films at frequency  $\nu=1.5 \cdot 10^4$  Hz. The least Maxwell relaxation time is  $200\text{msec}$ , i.e. satisfies the condition  $\tau < \tau_M$ .  $tg\delta$  increasing in PP+10%DK<sub>1</sub> nano-composite in  $\nu \leq 2 \cdot 10^3$  Hz regions shows that dielectric loss in given composites takes place because of electric conduction.

## CONCLUSION

Taking into consideration the above mentioned one can propose that polymer stabilizing in the volume, leads to appearance of polar groups on the matrix-nanoparticle boundary. The frequency and temperature dependences in PP polymer and PP+DK<sub>1</sub> and their dielectric properties show that relaxation losses take place because of composite structural elements with mobility different degree and appearance of low-molecular impurities, for example, with appearance of CO polar groups.

---

[1] L. Chazeau, J. Cavaille, G. Canova et al. J. Appl. Polym. Sci., 1999, №71, p. 1797-1808.

[2] Yu.A. Fedetov, N.N. Smirnova. Plasticskie massi, 2008, №9, c. 42-43. (In Russian).

Received:06.09.2016