THERMO-DEPOLARIZATION ANALYSIS OF POLYMER MIXTURE OF ISOTACT POLYPROPYLENE AND ETHYLENE-PROPYLENE CO-POLYMER

1. Marin GENCHEV

1. TECHNICAL UNIVERSITY SOFIA – BRANCH PLOVDIV, BULGARIA

ABSTRACT: The reliable functioning of the electrical-technical facilities basically depends on their electrical isolation in working conditions. This entails the use of high-quality isolation materials with suitable electrical-physical properties. The evaluation of these properties is conducted by applying certain electrical-physical methods of research. In this work the method of thermo-stimulated electrical current has been employed to study the structural changes of homo-polymer polypropylene, in whose matrix high-molecule additives are inserted in definite quantity ratio. The modification of propylene allows its wider application as electrical isolation.

Keywords: polypropylene, ethylene-propylene, structure modifying, thermo stimulation current

INTRODUCTION

The requirements of modern practice goes ahead of the synthesis of new polymer materials, which demands the application of physical and chemical modification for the implementation of a wide range of modifications. One of the most widely applied methods of physical modification of polymers is their mechanical mixing and the obtaining of polymer mixtures of valuable physical-chemical \[1;2].

The presented work is a continuation of a series of scientific researches \[3;4\] related to the implementation of polymer mixtures of polypropylene (PP) with different types of polymers, with a view to their application as isolation materials in modern electronics and electrical engineering.

The aim of the work is to study the relaxation processes and phase transitions within the temperature interval \([20 - 200]°C\). The physical method of thermo-depolarization analysis has been used.

The used PP homo-polymer, of the BULPEN 6531 make, has been produced by LUKOIL Bulgaria Corp., in the city of Bourgas. The ethylene-propylene co-polymer (EPC) has the trade mark NOVOLEN 1200 NX of the German BASF Company.

To the polymer mixture is added a high-melting additive Hydrotalcite-like Compound DHT-4A, hydrate of magnesium-aluminium hydroxyl carbonate. It is white, non-toxic, finely dispersed powder with size of particles from 0.4 to 41 μm. It does not dissolve in water and oils. It is a product of the Kyowa Chemical Industry Company – Japan. DHT-4A inactivates through chemical reactions the remaining quantities of substances connected with catalysts of acid type, like those of Cigler-Natta, used in the production of polyenes, of PP \[5\] in particular. DHT-4A not only substantially reduces, but even eliminates the corrosion activity of the polymer \[6\].

Scientific literature presents a number of announcements on the influence of DHT-4A on the corrosion activity of PP when processed \[7;8\] but there is no data about the influence of the additive on the electrical-physical properties in mixture with PP.

The obtained specimens are circular disks with a diameter \((50-51)\)mm and thickness \((2,00-2,06)\)mm. From these circular disks specimens of rectangular form \((30\times40)\)mm have been cut out. After being cleaned with spirit and ultra-sound, they are put in a vacuum camera, where through evaporation of aluminum electrodes of rectangular forma and sizes \((20\times30)\)mm are laid out.

TEST RESULTS AND THEIR PHYSICAL INTERPRETATION

The test specimens have been subjected to thermo-depolarization at temperature \(T_p=110°C\) and intensity of constant electric field \(E_p=5kV/cm\). The thermo-depolarization has been conducted at linear heating with velocity \(v=(2.17 \div 2.30)°C/min\) within the interval \((20 \div 200) °C\). Thermo-graphs have resulted, presenting the temperature correlations of density of the thermo-depolarizing electric current \(i=f(T)\).

Figure 1 shows the correlation \(i=f(T)\), while curves \((1 \div 5)\), correspond to polymer specimens given in table 1. For each of the resulting maximums of \(i=f(T)\) for the tested polymer specimens the following are presented: \(T_m\), temperature of the maximum; the maximum figure value of the density of the electric current \((i_m)\); the temperature intervals \((ΔT)\), for which the correlation \(lni=f\left(10^1/T\right)\) is linear; the activating energy \((W_0)\), of the respective process and pre-exploitation co-efficient \((i_0)\). For this purpose we assume that \(i=f(T)\) is of the type \((1)\) \[9\], and \((W_0)\) is defined by \((2)\)
\[ l = l_0 \exp \left[ -\frac{(W_0)}{R_1 T_1} \right] \]  
\[ (W_0) = \frac{R_1 T_1 T_2}{(T_2 - T_1)} \left[ \frac{i(T_1)}{i(T_2)} \right] \]

where: \( i(T_1) \) and \( i(T_2) \) are the figure values of \( i \) at temperatures \( T_1 \) and \( T_2 \), corresponding to the experimental figure values of \( i \) for the interval \( (\Delta T) \); and \( (R_1) \) is the universal gas constant.

Figure 1. Temperature correlation between the density of the thermo-depolarizing current as a function of the temperature for polymer mixtures of PP and EPS

In this case the method of initial slant has been applied, presented in [10], but it is fair to note [11], that this method has been used in earlier scientific research of thermo-activating electroscopy [12;13]. With thermo-stimulating depolarization the method of initial slant is used in order to define \((W_0)\) of electric-active defects [14] at volume-charging polarization and in [15] in case of dipolar polarization. The advantage of the method of initial slant, together with its simplicity, is illustrated in the independence of the result for \(( \Delta_0) \) within the kinetics of the relaxation processes. According to [16;17] the permitted relative deviation in defining \((W_0)\), is not greater than \((2 \pm 3)\% \) and depends on the appliance measuring the electric current \( (i) \) and the parameters of the volume of research. Generally, for both types of semi-crystal polymers and their polymer mixtures it can be assumed that the maximums \( (T_m) \) of \( i=f(T) \), which exist at \( T_m <100°C=373K \) are due to the realization of the low-temperature part of the complex \( \alpha \)-relaxation, which is connected with the thermal “defrosting” of the mobility of the chain segments within sections of the macro-molecular chains, available along the wrinkles of the crystal surface - \((\alpha-C)\) relaxation.

The maximums of \( i=f(T) \) given that \((100°C < T_m < 140°C) \) are presumably due to the high-temperature part of the complex \( \alpha \)-relaxation, connected with “defrosting” of the thermal mobility of the chain segments within sections of the macro-molecular chains, available within the inner volume parts of the crystals - \((\alpha-C)\) relaxation. The maximums of \( i=f(T) \) at \( T_m > 140°C \) are due to the melting of the crystals of PP, phase transition of first type.

The melting temperature of industrial specimens of PP can change within the interval \((166 ÷ 176) °C \) and depends on: surveillance method [18]; stereo-isomer composition – availability of atactic and stereo-block structures [19]; molecule mass [20]; crystallization conditions [21], etc. The existence of different forms of super-molecule structure of crystal-type organization makes it possible to have a process of step-by-step melting of different types of crystallites [22].

PP can crystallize in three modifications: monoclinic \((\alpha)\); hexagonal \((\beta) \) and triclinic \((\gamma)\). Within the interval \( (139 ÷ 156) °C \) thermal activated phase transition is produced from triclinic \((\gamma) \) to monoclinic \((\alpha)\) crystal modification (phase transition of the second type), whose velocity strongly depends on the temperature. In [23] it is defined that the hexagonal \((\beta) \) modification melts at \( 150°C \), while the monocystal \((\alpha) \) at \( 168°C \). The melting temperature of the triclinic \((\gamma) \) modification is defined for \( 152.4°C \) [24] by the method of the differentiating thermal analysis (DTA) at a warming speed of \( b=4°C/min. \). Figure 1, Curve 1 corresponding to \( i=f(T) \) for PP shows that \( (\alpha-C) = 159°C \) and if we correlate this temperature to the melting temperature of the crystallites in PP, it follows that these crystallites are of monoclinic \((\alpha)\) modification. Within the interval \( (140 ÷ 159) °C \) melting of crystallites of hexagonal \((\beta) \) modification can be realized. PP structures are viewed in detail in [25;26], where the respective parameters of \( \alpha \) elementary cell have been defined of the three crystal modifications and their respective density: \( \rho (\alpha) = 936kg/m^3 \) and \( \rho (\beta) = 880kg/m^3 \). The tested PP (in this case) has initial density \( \rho = 900kg/m^3 \) and it can be assumed that it contains crystallites of the three modifications.
The influence of the temperature of crystallization [27;28] on the melting temperature of PP is narrowed down to: spherulites, formed at temperature of 130°C melt at 165°C and those formed at 150°C melt at 173°C, that is, increase in the temperature of crystallization goes together with an increase in melting temperature [29]. Figure 1, Curve 1 for \(i=f(T)\) of PP shows that the melting of different types of crystallites occurs within the interval \((140 \pm 184)\)°C.

Figure 1, Curve 6 shows \(i=f(T)\) for PELD, the maximum of \(i=f(T)\) at temperature of \((\tau_m)=124\)°C is due to the melting of its crystallites (phase transition of the first type). The melting temperature of crystallites in industrially obtained types PE [30] is: a) for PELD from \((105 \pm 108)\) °C; b) for polyethylene of high density (PEHD) from \((120 \pm 125)\)°C; c) for polyethylene of average density (PEAD) from \((125 \pm 132)\)°C; d) for high-molecular polyethylene (HMPE) from \((133 \pm 137)\)°C.

Figure 1, Curve 3 represent an outline of remarkably greater (I), maximum of \(i=f(T)\) at \((\tau_m)=154\)°C, which represents a super-position of the melting of the crystallite of the two components PP and PELD, with additional participation of the maximum of \(i=f(T)\) at \((\tau_m)=120\)°C, corresponding to \((\alpha_c-C)\) relaxation of the chain segments from the inner volume parts of the crystallites of PP.

Figure 1, curves 4 and 5 shows only two well outlined maximums of \(i=f(T)\), respectively with temperatures \((\tau_m)\) and \((\tau_m)\), which are due to \((\alpha_C-C)\) relaxation melting of the crystallites of the two components PP and PELD.

Defining of the co-efficient \((i_o)\) in (1) is conducted through correlation (3)

\[
i_o = \exp \left[ \frac{1}{2} \ln(i_o) \right]
\]

Here there is a compensation mechanism, so that \((i_o)\) represents a compensating co-efficient in (1) and with greater figure values of \((W_a)\) to avoid too great figure values of \((i_o)\) and finally real figure values of \((i)\) be obtained. For this purpose we assume that \(i_o=f(i_o,W_a,T_c)\) of the type (4), while \((i)\) and \((T_c)\) are the respective constants of the density of the electric current \((i)\) and the temperature \((T)\).

\[
i_o = i_o \exp \left[ \frac{(W_a)}{T_c} \right]
\]

We replace (4) in (1) and we obtain (5). Calculation of the constants \((i_o)\) and \((T_c)\) is conducted through correlations (6 and 7).

\[
i_o = \exp \left[ \frac{1}{2} \ln\left[\frac{(i_o)}{(i_0)}\right] \right]
\]

\[
T_c = \ln\left[\frac{(W_a)_{m=1}}{(W_a)_{m=2}}\right] \cdot \ln\left[\frac{(i_o)_{m=1}}{(i_o)_{m=2}}\right]
\]

In correlations (4;5 and 7) \((R)\) is Boltzmann’s constant \(R=1,38.10^{-13} \text{ J/K}=8,625.10^{-5} \text{ eV/K.}\)

The compensation law of the type (8) is applied at testing of temperature correlation between the relaxation time \((\tau)\) of the chain segments of the macro-molecular chains in conducting the respective relaxation processes in different types of polymers [31;33]. The finally obtained correlation for \(\tau=f((W_a)_{m=1},T_c,T)\) is of type (9).

\[
\tau_c = \tau_c \exp \left[ \frac{(W_a)}{RT_c} \right]
\]

\[
\tau = \tau_c \exp \left[ \frac{(W_a)}{R} \right]
\]

For PP the following maximums of \(i=f(T)\) have been obtained:

a. within the interval (50 – 75)°C, which is due to \((\alpha_C-C)\) relaxation;

b. within the interval (75 – 110)°C, which is due to \((\alpha_C-C)\) relaxation;

c. within the interval (110 – 137)°C, which are due to phase transition of the second the in the crystallites;

d. within the interval (137 – 185)°C, which are due to phase transition of the first type – melting of the crystals of PP.

For the semi-crystal polymers the complex \(\alpha_c\) relaxation, with its chemical mechanisms of relaxation appearance, has been studied in detail in [34]. The case of \((\alpha_C-C)\) relaxation is carried out in crystallites with folded macro-molecular chains, while \((\alpha_C-C)\) – relaxation is due to the “defrosting” the
thermal mobility of chain segments of sections of the macro-molecule chains, available along the wrinkles on the surface of the crystallites, and \((\alpha,-\alpha) - \) relaxation, of the chain segments available in the inner volume parts of the crystallites.

At \(i=f(T)\) for polymer mixtures from PP and EPC the superposition is observed for the last maximum of \(i=f(T)\) of pure PP, appearing in a wide maximum within the interval \((110-180)\) °C. It has to be kept in mind that the melting temperature of the crystallites in PP (industrial specimens) within the interval \((166 \div 176)\) °C depends on a number of physical-chemical characteristics of the semi-crystal polymers: molecular mass, crystallizing conditions, etc.

Table 1 presents for each of the obtained maximums of \(i=f(T)\), \(T_m\) – temperature of the maximum, the respective maximum value \((i_m)\) of \(i\); temperature intervals \((\Delta T)\) for which the correlation \(lni=f(10^3/T)\) is linear; activating energy for the tested process \((W_a)_o\); \(\alpha\)-coefficient \((i_\alpha)\) from correlation (1). The definition of \((W_a)_o\) has been obtained from correlation (2). The method of initial slant has been used, of the maximum of \(i=f(T)\) to define \((W_a)_o\) and \((i_\alpha)\). The resulting figure values of \((W_a)_o\) are within the interval: from \((0.29 \div 5.24)\) eV for a phase transition of the first type – melting of the crystallites, from \((0.48 \div 4.59)\) eV for \((\alpha,-\alpha)\) relaxation in the crystallites.

A verification test has been conducted of the compensating role of \((i_\alpha)\) in relation to \((W_a)_o\) the compensation law of the type (4) being allowed, while the defining of \((i_\alpha)\) is obtained from (3), as well. This makes it possible to present \(i_0=f((W_a)_o, T, T_\alpha, i_\alpha)\), the defining of the correcting constants \((T_\alpha)\) and \((i_\alpha)\) is obtained from (6) and (6).

The resulting figure values of the physical characteristics thus defined, obtained by the method of thermo-depolarization analysis, are given in table 1.

![Image](image_url)

**Figure 2. Correlation between the thermo-depolarizing current \(i\) and the temperature for polymer systems polypropylene with 15% and 20% EP.**

**Figure 3.**

**Figure 4 shows the correlation \(ln\tau= f(10^3/T)\), while for the linear sections \((W_a)_o\), and \(\tau_0\) – have been defined \(\tau_0\) \(-\) for pure PP and polymer mixtures of PP and 25 mass% EPC.
Figure 3. Correlation between the density of the thermo-depolarizing current $i$ and the temperature for polymer systems polypropylene with 25% and 30% EP.

Figure 4. Correlation between the relation time $\tau$ and the temperature for PP and polymer system PP with 25% EP.

Figure 5 and Figure 6 show the correlation $\ln i = f[(W_a)]$, respectively for: temperature interval $(330 \div 371)K$, where ($\alpha$ - $\gamma$) – relaxation occurs and from $(384 \div 432)K$ for a phase transition of the first type (melting of crystallites). As it is seen, the points laid down from the tested specimens, lie on a straight line, which proves that the compensation law for the two tested processes is valid.

Figure 5. Correlation between the pre-exponent co-efficient $i_0$ and the activating energy $W_a$ within the temperature interval $(57+98)°C$.

Figure 6. Correlation between the pre-exponent co-efficient $i_0$ and the activating energy $W_a$ within the temperature interval $(111+159)°C$. 

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A second methodology has been applied for the processing of the obtained test information for i=f(T), related to the occurrence of the ($\alpha$-C.) – relaxation within the interval (75 ÷ 110)°C. For this purpose the following have been defined: maximum remaining di-electric polarization ($P_m$) and formula (10) from [4]; the relaxation time ($\tau_n$), (at $T = T_m$) of chain segments from section of macromolecular chains – formula (11); the temperature intervals ($\Delta T_i$) for which $ln f(T/\tau)$ is linear and ($W_m$), could be defined by the formula (13) when reading (12), from [4]; the pre-exponent co-efficient ($\tau_o$) according to the formula (10),

$$\tau_o = \exp \left( \frac{1}{2} \ln \left( \frac{n(T, \tau_o)}{n(T, \tau_2)} \right) \right)$$

(10)

where ($T_o$) and ($T_2$) are the extreme temperatures within the interval ($\Delta T$); the change in the enthalpy ($\Delta H^\alpha$), the entropy ($\Delta S^\alpha$), the free energy ($\Delta G^\alpha$) at $T= ( T_o + T_2 )/2$. The resulting information for the figure values of these physical characteristics is presented in table 1 for tested specimens of PP and PP + 25mass% EPC. To define $\Delta H^\alpha$, $\Delta G^\alpha$ and $\Delta S^\alpha$ the respective formulas (14-16) have been applied, presented in [4].

### Table 2a

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<th>No</th>
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<th>$P_m$ cm²</th>
<th>$\tau_m$ S</th>
<th>$\Delta T$ K</th>
<th>($W_m$) Mj/kmol</th>
<th>($W_m$) eV</th>
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<th>$\Delta H^\alpha$ Mj/kmol</th>
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<td>130</td>
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<td>1,22.10⁹</td>
<td>291</td>
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<td>133</td>
<td>1,38</td>
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### Table 2b

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<th>$\Delta G^\alpha$ Mj/kmol</th>
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<th>$W_m$ (Mj/kmol)</th>
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### Table 2c

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<th>$\delta$</th>
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<th>($W_m$)$_{av}$ eV</th>
<th>($W_m$)$_{av}$τ Mj/kmol</th>
<th>($W_m$)$_{av}$τ eV</th>
<th>$H_{1/2}$ 10¹⁰ cm</th>
<th>$\mu_{ef}$ D</th>
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</table>

It is possible to define ($W_m$) of the ($\alpha$-C.) – relaxation according to other (applied in scientific literature) methods:

a) according to the method of the maximum of $i=f(T)$ - ($W_m$)$_{av}$, by the formula (17) from [4];
b) according to the method Turnhout of [35] - ($W_m$)$_{av}$, by the formula (18) from [4];
c) according to the method of Grossweiner [36] - ($W_m$)$_{av}$, by the formula (19) from [4];
d) according to the method of Chen [37] - ($W_m$)$_{av}$ through defining first of the form-factor ($FF$) – form the formula (20) of [4], to define the kinetics of PP, and then the temperature intervals are defined $\text{min} = \text{max} - \Delta T_i$, with $\text{min} = 1T_m$ and $\text{max} = 1T_{1,2}$, for which ($W_m$) is defined – by the formula (21) from [4]. For this purpose, ($\tau_0$) in (20) changes consecutively the figure values $\text{min} \tau_0 \text{max}$ and the constants ($C_{\alpha}$) and ($B_{\alpha}$) from (21) are defined by the method of the molecular kinetics ($FF = 0.40$ – mono-molecular and $FF = 0.55$ – bi-molecular by use of information (22) and (23), presented in [4]. In this case ($\tau_o$) and ($\tau_{max}$) are the extreme temperatures of the maximum of $i=f(T)$ - ($\tau_i = i_{1,2}$) and ($\tau_{max}$) ($\tau_{max} = i_{max}/2$), while ($\tau_{min}$). In addition, ($W_m$)$_{av}$ is defined by the method of Keating [38] – formula (24) from [4], this method being based on the transition of Reandelle and Willikins [39] for the molecular kinetics.

We compare the figure values of ($W_m$)$_{av}$ ($W_m$)$_{av}$ and ($W_m$)$_{av}$, because in their defining information for ($\tau_m$) is used - a very good coincidence is obtained. We can compare ($W_m$)$_{av}$ ($W_m$)$_{av}$ and ($E_s$)$_{av}$ - ($\tau_m$, $\tau_{max}$) are used – again good coincidence.

The method of additive atom-group mixtures has been used [40] and the following are defined:

a) the volume of monomer unit of PP - $V_{m=un}(PP) = 8.15.10^{-9}$ m³ and the number (n) of the monomer units of PP in a unit of volume (with a correction of the free volume of 2.5%) - n(PP) = $V_{m=un}(PP) = 1.26.10^{-4}$ m³;
b) the volume of the average statistical unit (ASU) in polymer mixtures of (PP + 25mass% EPC) - $V(ASU) = 2.27.10^{-9}$ m³ and respectively n(ASU) = $V(ASU) = 4.30.10^{-10}$ m³. In this case ASU is of the type [(CH$_2$)$_{29.12}$ - (CH$_{29.12}$)] through applying the formula of Debai – Lanjven [41], presented through (26) in [4] the effective dipole moment ($\mu_{ef}$) is defined, respectively a monomer unit as well of PP and ASU for polymer mixtures of PP + 25mass% EPC. The resulting figure values of $\mu_{ef}$ are

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presented in table 1. Defining of \( \mu_{\text{eff}} \). Through the formula of Lanjven-Debai allows for the fact that all (Pm) is defined by the lower component. If we assume that uncompensated is only the dipole moment of the group (C−H) of the methyl group (CH\(_3\)) in ASU, then \( \mu_{\text{theor.(ASU)}} = 27.12 \mu \) (C−H) = 27.12(1.33×10\(^{-3}\)) cm = 36.07. 10\(^{-3}\) cm = 10.28[D]. The result is that for polymer mixtures of (PP+5mass\%EPSC) \( \mu_{\text{eff.(ASU)}}=9.05[D]<\mu_{\text{theor.(ASU)}}=10.28[D] \). For pure PP \( \mu_{\text{eff.(SU)}}=0.53[D] > \mu_{\text{theor.(SU)}}=0.4[D] \) – again under the same assumption.

It is possible to define (I) from the release of electrical carriers localized in different types of catchers which are thermally activated at different temperature intervals within which the respective relaxation processes or phase transitions occur. In this case at deducing the (\( \alpha_cm-Cm \)) – relaxation this mechanism can justify the resulting correlations between the figure values of \( \mu_{\text{eff}} \) and \( \mu_{\text{theor}} \). It is not possible to assume any additional occurrence of non-dipole component, except of such carriers related to the chain segments from the inner volume parts of the crystallites.

**CONCLUSIONS**

The information of the thermo-depolarization analysis shows the occurrence of \( \alpha_cm-Cm \) and \( \alpha_cm-Cm \) – relaxations, as well as phase transitions of 1 and 2 types in the crystallites of PP. The validity of the compensation law is proved, relating \( (i_{\omega}) \) to \( (W_{\omega}) \), and the great sensitivity and segregation ability of the method of thermo-depolarizing analysis. The figure values of important energy thermo-dynamical, dielectrical and geometrical characteristics of the tested specimens have been obtained.

The individual influence of 0.05\% DHT-4A does not show any change of the electrical-physical properties of pure and modified PP. The use of DHT-4A shows the efficiency of its employment as an anti-corrosion modifier of polypropylene.

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