

# Injection molding of transparent polymeric materials with 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide for enhanced fire retardancy

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**Abstract:** Selected transparent polymers [*i.e.*, polystyrene (PS) and poly(methyl methacrylate) (PMMA)] were extruded with 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO). The obtained compositions were formed into testing samples by injection molding process. The obtained materials (containing 1–10 wt parts/100 wt parts of the polymer) exhibited higher melt flow rate and thermal resistance, as well as reduced flammability (while maintaining their mechanical properties). Additionally, Fourier transform infrared spectroscopy (FT-IR) analysis of modified polymers was conducted. Studies confirmed that – during the extrusion and/or injection molding – DOPO reacts with PS and PMMA.

**Keywords:** 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide, polystyrene, poly(methyl methacrylate), mechanical properties, thermal properties.

## Formowane wtryskowo transparentne materiały polimerowe uniepalnione 10-tlenkiem 9,10-dihydro-9-oksa-10-fosfafenantrenu

**Streszczenie:** Wybrane transparentne polimery termoplastyczne [tj. polistyren (PS) i poli(metakrylan metylu) (PMMA)] wytłaczano z 10-tlenkiem 9,10-dihydro-9-oksa-10-fosfafenantrenu (DOPO), a z uzyskanych kompozycji metodą wtrysku formowano próbki do badań. Otrzymane materiały (zawierające 1–10 cz. mas. DOPO/100 cz. mas. polimeru) charakteryzowały się większym wskaźnikiem szybkości płynięcia, większą odpornością termiczną oraz mniejszą zapalnością (przy zachowaniu właściwości mechanicznych) niż polimery niemodyfikowane. Dodatkowo kompozycje z DOPO oceniano metodą spektroskopii w podczerwieni z transformacją Fouriera (FT-IR). Badania potwierdziły, iż w trakcie wytłaczania i/lub formowania wtryskowego DOPO reaguje z PS oraz PMMA.

**Słowa kluczowe:** 10-tlenek 9,10-dihydro-9-oksa-10-fosfafenantrenu, polistyren, poli(metakrylan metylu), właściwości mechaniczne, właściwości termiczne.

The market for transparent plastics (with higher than 80 % visible light transparency) is expanding due to the growth of construction industry. The main types of the transparent plastics are polystyrene (PS) and poly(methyl methacrylate) (PMMA). The contributing factors are: easy molding and processing, low density, ability to damp mechanical vibrations, lack of maintenance, economic and aesthetic reasons. One of the limitations associated with their use is the lack of resistance to fire [1].

9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) is an attractive phosphorus-containing fire re-

tardant due to its high thermal stability, good oxidation and water resistance [2–4]. Nevertheless, low molecular weight DOPO presents disadvantages such as leaching and poor compatibility with polymer matrices [5]. The excellent flame retardant properties of DOPO and its derivatives induce their usage in epoxy systems [6, 7]. Phosphorous-based compounds occupy an important place as flame retardants; their position is strengthened due to the environmental regulations forcing the withdrawal of halogen additives reducing polymer flammability (being source of dioxins and dibenzofuranes during the burning process). The use of phosphorus compounds as flame retardant additives in polymers leads to the production of an intumescent carbonaceous char during the combustion process. The outer layers of a polymeric mixture containing the flame retardant act as a physical and thermal barrier to further combustion by impeding heat transfer to the underlying layers of a virgin polymer and thus releasing further flammable volatiles [8, 9].

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The aim of this work was to investigate the influence of DOPO addition on thermal, optical and mechanical properties of injection moldable transparent polymers (*i.e.*, PS and PMMA). The main purpose of the modifier's application was flammability reduction of the polymeric materials.

## EXPERIMENTAL PART

### Materials

Polystyrene OWISPOL 525 was purchased from Synthos Dwory (Oswiecim, Poland), poly(methyl methacrylate) Plexpol SC was purchased from Heko (Oswiecim, Poland) while 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) (white powder with density 1.39 g/cm<sup>3</sup>, melting point 116 °C and phosphorus content 14.3 wt %) was delivered by Struktol GmbH, Germany. All materials were used as received without further purification.

### Modification of polymers

Before extrusion, the polymer granulates and DOPO powder were dried in an oven at 70 °C for 8 h. An extrusion technique was applied with a conventional co-rotating twin-screw extruder (Thermo Electron Corporation Prism EuroLab 16) equipped with a 2 mm hole die. The temperature profile of the extruder from the components inlet into nozzle was (i) 160/230/230/230/230/230/230/230/230 °C at a screw speed of 100 rpm for PS/DOPO composites and (ii) 220/230/240/250/260/260/260/260/260 °C at a screw speed of 200 rpm for PMMA/DOPO composites. The extruded polymer compositions were cooled down with water and cut into 3 mm pellets. Next, the pellets were dried at 70 °C for 8 h. Subsequently, the pellets were injection molded into 4 mm thick plates for further tests by using an injection-molding machine (Boy 35A, dr. BOY GmbH, Germany). Injection parameters for PS/DOPO compositions were as follows: the barrel temperature ranged from 235 to 245 °C and the mold temperature was kept constantly at 55 °C; the injection pressure, holding pressure and speed were 40 MPa, 40 MPa and 100 mm/s, respectively. For PMMA/DOPO compositions, the injection parameters were as follows: the barrel temperature ranged from 230 to 260 °C and the mold temperature was kept constantly at 60 °C; the injection pressure, holding pressure and speed were 50 MPa, 45 MPa and 75 mm/s, respectively [10].

The DOPO content in the molded mixture was 1, 5, 7.5 and 10 wt parts/100 wt parts of polymer (samples abbreviated as PS-1, PS-5, PS-7.5 and PS-10 for PS composites and PMMA-1, PMMA-5, PMMA-7.5 and PMMA-10 for PMMA composites, respectively). Polymers have been also molded without a DOPO addition as reference samples (PS-0 and PMMA-0).

### Methods of testing

– Fourier transform infrared spectroscopy with attenuated total reflectance (ATR) accessories (Nexus FT-IR;

Thermo Nicolet, USA) was used to study a molecule structure of neat polymers as well as modified PS and PMMA samples.

– Tensile and flexural experiments were performed by means of Instron 4026 machine (Instron Co., USA). Tensile strength, Young's modulus and elongation at break were determined according to PN-EN ISO 527-1 while flexural strength, modulus and strain at yield were tested in relation to PN-EN ISO 178. Eight specimens of each sample were analyzed and mean values and standard deviations were calculated.

– The Charpy impact strength was determined with a Zwick D-7900 impact tester (Zwick, Germany) according to PN-EN ISO 179 standard (notch A-type).

– Hardness of PS-based materials was measured with a Barcol hardness tester (23 °C, PN-EN 59).

– The volume flow rate index (*VFR*) was determined with a melt flow tester (CEAST 6841/048, Ceast, Italy) according to PN-EN ISO 1133. The die diameters were 2.09 mm while the test temperature and the load was 200 °C and 5 kg, respectively. The mean *VFR* value for ten samples of each material was presented.

– Thermal and thermochemical properties of PS/DOPO materials have been investigated as follows: (i) Vicat softening temperature (heating rate 50 °C/h, EN ISO 306, Vicat FWV 633.10, Fritz Heckert GmbH, Germany), (ii) glass transition temperature as the temperature of loss modulus peak (dynamic mechanical analyzer Q800 with a dual cantilever clamp, heating range 45–130 °C, heating rate 3 °C/min, 1 Hz frequency, amplitude 25 μm, TA Instruments, USA), (iii) thermostability, *i.e.*, the temperature of 10 and 50 % mass loss (thermogravimetric analyzer Q500, 25–600 °C, 10 °C/min, air atmosphere, TA Instruments, USA) and (iv) oxygen index (*OI*) according to ISO 4589-2 (100 mm × 10 mm × 4 mm samples). The *OI* value was defined as a minimum oxygen concentration to support 180 s or 50 mm length burning. Polymeric samples were conditioned at 23 °C and 50 % relative humidity for three days before testing.

– The transparency of samples was evaluated using UV-Vis spectrophotometer (Jasco 630, Jasco, Japan).

## RESULTS AND DISCUSSION

Figure 1 shows a comparison of FT-IR spectra for DOPO, unmodified PS (PS-0), PS containing 10 wt % of DOPO (PS-10) as well as for burn residues of these polymeric materials (PS-0B and PS-10B, respectively). The spectra of PS-10 and PS-10B include slightly higher peaks or new ones characteristic for functional groups of DOPO molecule, *i.e.*, P-O-Ar (absorbance at *ca.* 1232 cm<sup>-1</sup> and 865–989 cm<sup>-1</sup>) and P-O-C (*ca.* 1050 cm<sup>-1</sup>). Nevertheless, these spectra exhibited a new (small) peak at *ca.* 785 cm<sup>-1</sup> representing P-C groups (not detected for DOPO). Probably, DOPO reacts with polystyrene during their processing (extrusion and/or injection molding) and the above mentioned bonds are created. P-C groups are present in a

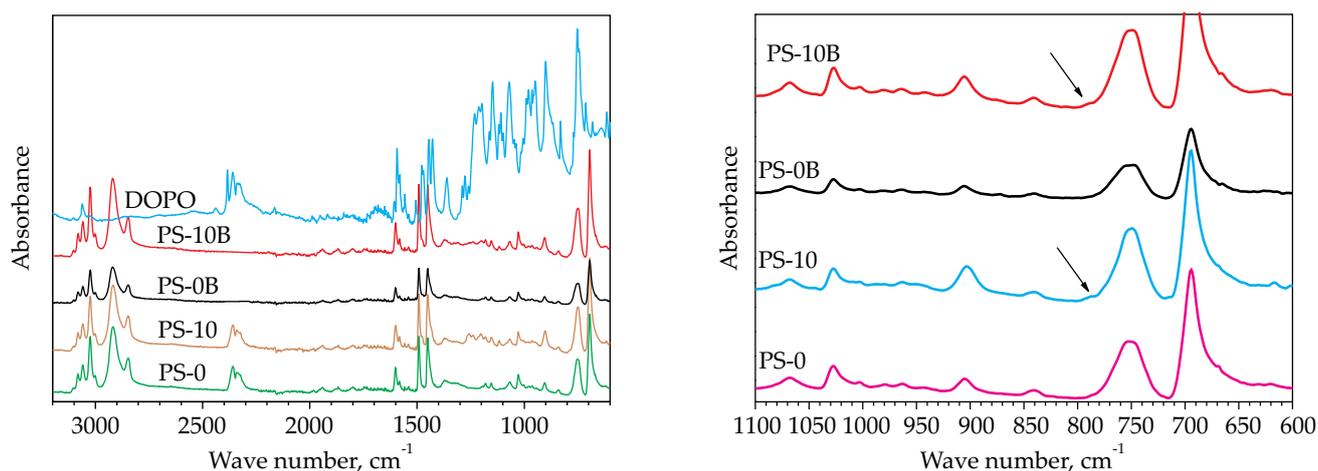


Fig. 1. FT-IR spectra for DOPO, PS-0, PS-10 compositions and burn residues of these materials (PS-0B and PS-10B)

burn residue of PS-10 sample (PS-10B) thus DOPO is firmly embedded in the polymeric matrix. Taking into consideration the results of FT-IR analysis and the references [10, 11] (describing PMMA and unsaturated imide-based materials modified with DOPO) it could be claimed that DOPO reacts with unsaturated bonds located at the end of PS chains. FT-IR spectra for PMMA-0 and PMMA-10 compositions were described in previous publication [10].

Table 1 presents mechanical properties of PS, PMMA [10] and its compositions with DOPO. As one can observe, PMMA-based materials are almost twice as strong as PS. Tensile and flexural strength values for PS and PMMA compositions with DOPO do not differ significantly from those for the neat polymers. Effect of the DOPO addition to the polymer matrix is notable by addition of DOPO to the polymer matrix by 10 wt parts (per 100 wt parts of polymer). The values of tensile strength are almost 10 % lower for PS-10 and 20 % for PMMA-10 in comparison with unmodified polymers. The highest decrement of flexural strength was registered for PS-10 (13 % in comparison with PS-0). In the case of PMMA the flexural strength value didn't decrease with the increase of the loading level of DOPO. The lowest value has been regis-

tered for PMMA-1 (112.3 MPa), whereas PMMA-0 sample exhibited higher flexural strength (117.9 MPa). The maximum addition of DOPO to PMMA matrix didn't affect this parameter. In contrast, DOPO caused an increase in Young's modulus for both the PS and PMMA. Also the modulus values were higher for composites with DOPO. In the case of PS composites, the greatest growth in rigidity was observed for PS-10 composition (Young's modulus went up to 2486 MPa and flexural modulus to 3172 MPa). In comparison, neat polystyrene reached 2236 MPa and 3051 MPa, respectively. For the poly(methyl methacrylate) compositions the improvement of these properties was even greater (Young's modulus went up to 2718 MPa and flexural modulus to 3176 MPa for PMMA-10).

Both properties elongation at break as well as strain at yield have slightly worsened with the increase of DOPO content. The decrease of those parameters has been significant for maximal concentration of the modifier (10 wt parts of DOPO per 100 wt parts of polymer) especially in case of PMMA composites [10]. The modification of polymer matrix caused decrease of impact strength only in the case of PS-based materials (up to 22 % for PS-10 in comparison with neat polymer PS-0). The addition of

Table 1. Mechanical properties for PS or PMMA-based materials modified with DOPO

Composition acronym	Tensile strength MPa	Flexural strength MPa	Young's modulus MPa	Flexural modulus MPa	Elongation at break %	Strain at yield mm	Impact strength kJ/m <sup>2</sup>	Hardness HBa
PS-0	36.7 ± 1.5	67.2 ± 1.5	2236 ± 34	3051 ± 80	2.3 ± 0.2	2.4 ± 0.1	10.6 ± 0.7	52 ± 2
PS-1	36.1 ± 1.4	63.6 ± 2.3	2283 ± 27	3148 ± 110	2.2 ± 0.16	2.1 ± 0.1	9.3 ± 0.5	53 ± 1
PS-5	35.1 ± 1.1	62.3 ± 1.9	2339 ± 59	3140 ± 37	2.0 ± 0.2	2.1 ± 0.1	9.2 ± 0.4	53 ± 2
PS-7.5	35.0 ± 1.2	61.7 ± 0.5	2367 ± 42	3143 ± 20	2.0 ± 0.14	2.1 ± 0.02	8.5 ± 0.8	53 ± 2
PS-10	33.2 ± 2.4	58.5 ± 1.3	2486 ± 25	3172 ± 25	1.6 ± 0.12	2.0 ± 0.1	8.3 ± 0.4	54 ± 2
PMMA-0	64.8 ± 2.3	117.9 ± 1.6	2340 ± 100	2431 ± 30	5.5 ± 0.3	7.3 ± 0.7	13.7 ± 1.2	68 ± 2
PMMA-1	63.8 ± 2.4	112.3 ± 3.1	2341 ± 58	2442 ± 51	5.4 ± 0.9	6.7 ± 0.8	14.0 ± 0.9	68 ± 2
PMMA-5	65.2 ± 3.1	116.6 ± 3.0	2540 ± 22	2807 ± 77	4.5 ± 0.6	4.9 ± 0.5	13.3 ± 1.3	69 ± 1
PMMA-7.5	65.9 ± 3.9	115.9 ± 9.5	2597 ± 72	3047 ± 81	4.2 ± 0.5	4.3 ± 0.7	13.6 ± 1.2	72 ± 1
PMMA-10	52.6 ± 6.6	117.9 ± 8.7	2718 ± 43	3176 ± 74	2.8 ± 0.4	4.0 ± 0.5	13.4 ± 1.2	73 ± 2

**Table 2.** Rheological, thermal and optical properties for PS or PMMA-based materials modified with DOPO

Composition acronym	VFR <sup>a</sup> cm <sup>3</sup> /10 min	Mass loss in air atmosphere		Oxygen index (OI) % O <sub>2</sub>	Softening temperature °C	Glass transition temperature °C	Transparency %
		T <sub>10</sub> <sup>b</sup> °C	T <sub>50</sub> <sup>c</sup> °C				
PS-0	13.1	316	367	17.9	97 ± 2	112	28 <sup>d</sup> / 59 <sup>e</sup>
PS-1	18.8	313	365	19.5	93 ± 1	107	21 / 56
PS-5	27.0	312	366	21.1	87 ± 2	103	16 / 53
PS-7.5	32.8	311	368	21.5	84 ± 2	101	12 / 51
PS-10	48.4	305	365	21.5	82 ± 2	99	3 / 40
PMMA-0	1.6	315	351	17.9	109 ± 3	111	85 / 90
PMMA-1	2.2	293	353	18.0	99 ± 4	102	52 / 72
PMMA-5	3.0	283	352	19.4	96 ± 4	98	47 / 74
PMMA-7.5	4.1	281	351	19.8	96 ± 2	97	37 / 74
PMMA-10	5.3	284	355	20.4	94 ± 3	93	25 / 75

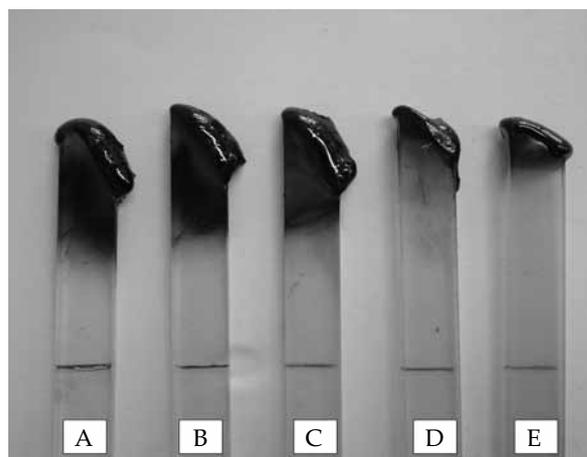
<sup>a</sup> – volume flow rate; <sup>b</sup> – temperature at 10 % mass loss; <sup>c</sup> – temperature at 50 % mass loss; <sup>d</sup> – at 400 nm; <sup>e</sup> – at 600 nm.

DOPO resulted in slight increase of composite's Barcol hardness.

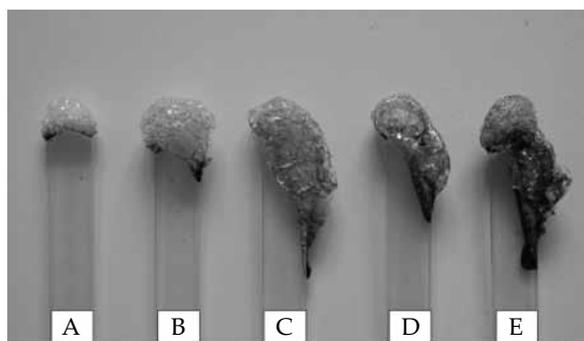
Rheological and thermal properties of PS/DOPO and PMMA/DOPO [10] systems are presented in Table 2. The most important parameter for thermoplastic polymers from the processing and the properties of the final product is the volume flow rate (VFR). The addition of DOPO has great influence on VFR values for both modified polymers. After addition of only 1 wt part of DOPO per 100 wt parts of polymer VFR value increased by 40 % both for polystyrene as well as poly(methyl methacrylate). A further increase in the share of DOPO resulted in approximately 350 % increase in the value compared with virgin thermoplastic (48.4 cm<sup>3</sup>/10 min for PS-10 instead of 13.1 cm<sup>3</sup>/10 min for PS-0 and 5.3 cm<sup>3</sup>/10 min for PMMA-10 instead of 1.6 cm<sup>3</sup>/10 min for PMMA-0). The results of research on thermogravimetric analysis in the form of temperature values at which a 10 wt % (T<sub>10</sub><sup>o</sup>, onset temperature of degradation) and 50 wt % (T<sub>50</sub><sup>o</sup>, the mid-point of degradation) showed low impact of the incorporation of DOPO into polymer matrix, especially in the case of polystyrene-based compositions. Significant changes have been noted for PMMA: decrease of T<sub>10</sub> value from 315 °C for unmodified PMMA to 281 °C for PS-7.5. Softening temperature as well as glass transition temperatures values decreased with the increasing of DOPO content.

Oxygen concentration in atmosphere is crucial parameter for continuous burning of materials. Therefore, its determination is useful in the flammability rating of polymers. If oxygen index (Table 2) is lower than 21 %, the material burns easily in the air after removing an ignition source. Neat PS and PMMA are easily flammable polymeric materials and both burnt in the atmosphere containing 17.9 vol % of oxygen. The presence of DOPO in the polymers matrix affects the increase of flame retardant property. In case of polystyrene-based compositions oxygen index was increased to 19.5 % for PS-1 and 21.5 % for both PS-7.5 and PS-10, whereas for poly(methyl methacrylate) compositions increase was lower (from

18 % for PMMA-1 up to 20.4 % for PMMA-10). Literature data show that it is possible to increase the value of the OI even to 35.4 % by the introduction of halogen atoms in a benzene ring [12]. The use of environmentally safe fire retardants does not provide such significant effects (e.g., 24 % by the incorporation of silica gel-potassium carbonate in the PS matrix [13]).



**Fig. 2.** Photographs of PS-based samples after flammability tests: A – PS-0, B – PS-1, C – PS-5, D – PS-7.5, E – PS-10



**Fig. 3.** Photographs of PMMA-based samples after flammability tests: A – PMMA-0, B – PMMA-1, C – PMMA-5, D – PMMA-7.5, E – PMMA-10 [10]

Based on photographs of samples after a flammability test (Fig. 2) it can be deduced that the addition of DOPO to polystyrene slows down the combustion process and reduces molten drips. It should be noted that the described phenomenon as well as  $OI$  for PS/DOPO compositions appear to correlate with their thermal stability (Table 2). It may be possible that DOPO facilitates PS thermal degradation (observed lower  $T_{10}$  values for PS/DOPO materials in comparison with an unmodified polymer) and reacts with reaction products only at a high temperature (similar or higher  $T_{50}$  values and higher oxygen index values for PS/DOPO samples).

The addition of DOPO into PMMA caused char formation on the surface of the burning material. The higher the DOPO content the greater amount of char has been observed (Fig. 3). Nevertheless, it is noteworthy that the mentioned phenomenon as well as  $OI$  values for a PMMA/DOPO material do not correlate with their thermal stability observed under the air or nitrogen atmosphere (Table 2). Most probably DOPO facilitates PMMA depolymerization/degradation (observed as lower  $T_{10}$  values for PMMA/DOPO materials in comparison with a neat polymer) and reacts with the mentioned reaction products only at a high temperature (similar or higher  $T_{50}$  values as well as higher oxygen index values for PMMA/DOPO samples) [11].

For some applications, an important parameter is the transparency of the product. Transparency was measured at 400 nm and 600 nm wavelength. The study of received material showed significantly greater transparency of PMMA-0 (85 % at 400 nm, 90 % at 600 nm) than PS-0 (28 % at 400 nm, 59 % at 600 nm; Table 2). As can be observed the polymers modification by DOPO caused reduction of their transparency. Figure 4 presents the influence of DOPO share on material transparency. The percentage decrease in the value of transparency is observed with increasing DOPO especially at a wavelength of 400 nm. The transparency for PS-10 decreases by 89 % compared to the unmodified PS and 71 % for PMMA-10. The lowest

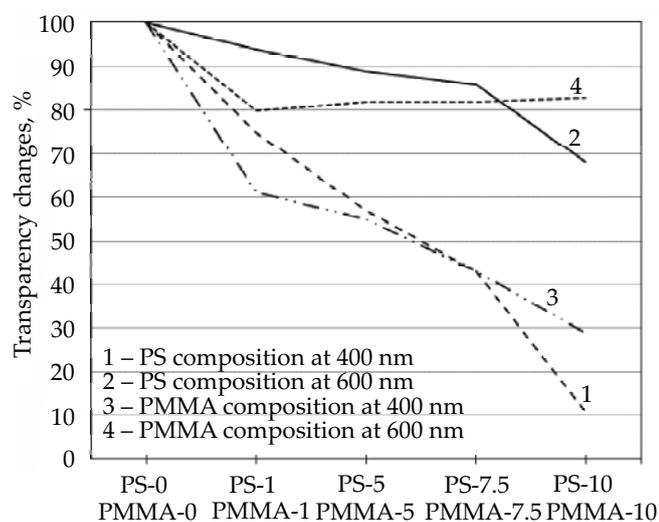


Fig. 4. Influence of DOPO addition on transparency of polystyrene (PS) and poly(methyl methacrylate) (PMMA)

decrease was noted for PMMA at a wavelength of 600 nm (only 20 % in comparison with PMMA-0). In this case the increase of DOPO content didn't cause further decrease of transparency.

## CONCLUSIONS

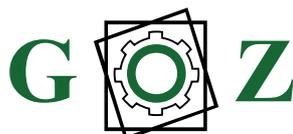
PS as well as PMMA modified with DOPO (9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide) were prepared with a convenient melt blending technique using a co-rotating twin-screw extruder and an injection molding machine. Research has shown that the addition of 1–10 wt parts of DOPO per 100 wt parts of polymer significantly increases the volume flow rate index as well as hardness of polymer composites. The strength properties for modified polymers do not differ significantly from neat polymers, especially if the DOPO content does not exceed 7.5 wt parts. The phosphorous modifier causes the reduction of softening and glass transition temperatures, but more importantly, it upgrades the oxygen index value from 17.9 % (neat PS and PMMA) to 21.5 % for compositions with 7.5 and 10 wt parts of DOPO per 100 wt parts of PS and 20.4 % – for composition with 10 wt parts of DOPO per 100 wt parts of PMMA. The presence of the modifier significantly influenced the transparency of products. Decrement of transparency was observed with increasing DOPO content, especially at 400 nm wavelength. It should be noted that transparency for PMMA-based samples containing 1–10 wt parts of DOPO (per 100 wt parts of the polymer) was similar (72–75 %, 600 nm) and markedly higher in comparison with PS-0 and PS/DOPO systems (45–40 %).

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