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Measuring the Young's modulus of polystyrene-based composites by tensile test and pulse-echo method

Imran Oral · Hatice Guzel · Gulnare Ahmetli

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Abstract In this study, the pure polystyrenes (PS) with different molecular weights $(3.5 \times 10^5 \text{ and } 5.0 \times 10^5)$ have been modified by the chemical modification with succinic anhydride (SA), maleic anhydride (MA), and phthalic anhydride (PhA). The modified polystyrenes (MPS) have been mixed with the pure PS with the molecular weight of 2.3×10^5 in weight % ratio 90:10, 80:20, and 70:30. Young's modulus of obtained composites has been measured mechanically by the tensile test and ultrasonic method at frequency of 5 MHz. Further, the values of Young's modulus measured by both methods have been compared with each other. From the results, a significant difference has not been found between the values of Young's modulus of both methods. As a result it can be stated that measuring the Young's modulus of these materials by the ultrasonic methods is more sensitive and economical than the mechanical methods.

Keywords Polystyrene · Composite · Tensile test · Ultrasound

Introduction

Success in today's marketplace requires improvements in efficiency, quality, and accuracy of testing facilities and testing equipment. Testing machines are used to develop better information on known materials or to develop new materials and maintain the quality of the materials. The elasticity modulus is one of the important

I. Oral (🖂) · H. Guzel

Department of Physics Education, Ahmet Kelesoglu Faculty of Education, Selcuk University, Konya, Turkey e-mail: oralimran@selcuk.edu.tr

G. Ahmetli Department of Chemical Engineering, Faculty of Engineering and Architecture, Selcuk University, Konya, Turkey

parameter that indicate the quality of the materials. The elasticity modulus of material can be measured using destructive methods like tensile and compressive tests and non-destructive approaches like ultrasonic methods. A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material. As you continue to pull on the material until it breaks, you will obtain a good, complete tensile profile. A curve will result showing how it reacted to the forces being applied. For most tensile testing of materials, the relationship between the applied force, or load, and the elongation the specimen exhibits is linear [1]. In this linear region, the line obeys the relationship defined as "Hooke's Law" where the ratio of stress to strain is a constant, or $\sigma/\varepsilon = E$. Tensile strength is defined as a stress, which is measured as force per unit area. In the SI system, the unit is pascal (Pa) or, equivalently, newtons per square meter (N/m^2) . The amount of stretch or elongation the specimen undergoes during tensile testing can be expressed as an absolute measurement in the change in length or as a relative measurement called "strain". It is the ratio of the change in length to the original length, $\varepsilon = L - L_0/L_0 = \Delta L/L_0$. E is the slope of the line in this region where stress (σ) is proportional to strain (ε) and is called the "modulus of elasticity" or "Young's modulus". The modulus of elasticity (E) defines the properties of a material as it undergoes stress, deforms, and then returns to its original shape after the stress is removed. It is a measure of the stiffness of a given material [2-4]. To compute the modulus of elasticity, simply divide the stress by the strain in the material. Modulus of elasticity determines stiffness-resistance of a body to elastic deformation caused by an applied force. A lot of informations can be learned about a substance from tensile testing. But measurement by tensile test is a destructive method and it is not an economical method.

Ultrasonic techniques have been widely used for a number of types of investigation [5]. Ultrasound is finding an increasing number of applications in the modern world. Included amongst these are medical imaging, dentistry, particle sizing, food processing, welding, waste water treatment, and surgical processes [6]. Furthermore, ultrasonic methods have been successfully used to monitor polymer processing [7], chemical reactions [8, 9], film formation from aqueous polymer dispersions [10], glue processes, crystallization in polymers [11, 12], characterization of polymers and also compatibility of polymeric blends [13–17].

Ultrasonic methods have advantages over destructive methods. Ultrasonic measurements can be made on actual components without destroying the samples. In addition, ultrasonic measurements can be performed for different orientations; this means that the number of elastic modulus measured for a single plane can be more than the number of elastic modulus measured using destructive techniques. Ultrasonic techniques are a versatile tool for investigating the changes in microstructure, deformation process and mechanical properties [18]. The various parameters upon which the elastic modulus of polymers depends can be studied by measuring the ultrasonic wave velocities. When propagated in polymeric materials, acoustic waves are influenced by the polymer's structure and by molecular relaxation processes. It is possible to estimate the viscoelastic properties of polymeric materials from the velocity and attenuation of longitudinal or shear waves

[19]. So, recently a lot of attempts have been made to study the sound velocity and attenuation of polymers.

Polystyrene (PS) is a cheap, hard, rigid, transparent thermoplastic [20]. Solid polystyrene is used in disposable cutlery, optical tools, cases, drink cups, food trays, dishes, egg boxes, plastic models, video/audio cassette, CD and DVD cases, toys, light diffusers, beakers, general household appliances, electronic housings, refrigerator liners, and smoke detector housings. The principal limitations of the polystyrene are its brittleness, inability to withstand the temperature of boiling water, its mediocre oil resistance, poor chemical resistance especially to organics and susceptible to UV degradation [21]. These defective properties of PS properties can be improve by bonding various functional groups to the aromatic ring of PS. Modified PS was found to have higher mechanical, thermal and elasticity properties than PS had and they were more durable against impact. But modifying PS is an expensive way. So in this study, it was aimed to obtain composites of PS for improving the pure PS's properties by a cheaper way and to measure the Young's modulus of new materials by ultrasonic velocity method as a non-destructive, economical and very precise method [22, 23] and also by tensile test as a destructive method. After measuring the Young's modulus by two different way, it was aimed to compare the results of both methods with each other.

Experimental

Materials

PS samples of different molecular weights (PS₂₃₀: the pure polystyrene with molecular weight of 2.3×10^5 , PS₃₅₀: the pure polystyrene with molecular weight of 3.5×10^5 and PS₅₀₀: the pure polystyrene with molecular weight of 5.0×10^5), succinic anhydride (SA), maleic anhydride (MA), phthalic anhydride (PhA), chloroform as solvent, methanol as precipitator and cationic catalyst BF₃O(C₂H₅)₂ were purchased from Merck (Darmstadt, Germany).

Chemical modification

A reactor consisting of a mixer, cooler, and thermometer was used in the experiment. For modifiying the PS_{500} , firstly 7.8 g of anhydride (20 wt. % of the polymer amount) was added to the solution of 39 g PS in 300 mL chloroform (CHCl₃) by mixing. After anhydride was dissolved completely, 10 mL $BF_3.O(C_2H_5)_2$ was added drop by drop and was stirred for 3 h at 25 °C to end the reaction. The mixture was poured into a beaker. Modified PS (MPS) was precipitated with methanol (500 mL) from the mixture, filtered and dried under vacuum at 60 °C for 5 h. So by this method the modified PS₅₀₀ with SA, MA and PhA called SAI, MAI and PhAI were obtained, respectively.

The chemical modifications of PS_{350} samples with SA, MA and PhA in the presence of the catalyst $BF_3.O(C_2H_5)_2$ were examined with the same method too. The modified PS_{350} called SAII, MAII, and PhAII were obtained. The binding of the



Scheme 1 The modification reactions of PS with various organic anhydrides: 1 PhA; 2 SA; 3 MA

functional groups to the aromatic ring of pure PS using $BF_3O(C_2H_5)_2$ catalyst was shown in Scheme 1.

Preparation of polystyrene based composites

Firstly 6.5 g of SAI (10% of the pure polystyrene amount) was added to the solution of 65 g PS₂₃₀ (PS₂₃₀: the pure polystyrene with molecular weight of 2.3×10^5) in 300 mL chloroform (CHCl₃) by mixing for 2 h at 25 °C. After MPS (SAI) was dissolved completely. The mixture was poured into a beaker. The composite of PS that obtained was precipitated by methanol (500 mL) from the reaction mixture, filtered and dried under vacuum at 60 °C for 5 h. So the composite of PS₂₃₀/SAI which rate is 90:10 was obtained. By the same way the other composites of PS₂₃₀ with SAI were produced at the 80:20 and 70:30 rates too. In the other hand by the same procedure, the composites of PS₂₃₀/MAI, PS₂₃₀/PhAI, PS₂₃₀/PhAII, PS₂₃₀/PhAII were produced too. The composition content of composites were given in Table 1.

Mechanical testing

All samples of composites were obtained, were melted at the same temperature of 180–200 °C during 3 min in a molding machine, and then all samples of composites melted were poured into a mold of steel which was prepared according to the ASTM D638 standards [24]. The mold is cooled constantly to a temperature that allows the composites to be cool to the touch. So all composites were obtained by this process.

Young's modulus of all samples of composites was measured by tensile test. The tensile test is the most widely used test to determine the mechanical properties of materials. In this test, a piece of material is pulled until it fractures. During the test,

Table 1	The composition rates and amounts of PS230/MPS components

Components	Composition rate	PS ₂₃₀ (g)	MPS (g)
PS:MPS	90:10	65	6.5
PS:MPS	80:20	60	12
PS:MPS	70:30	55	16.5



Fig. 1 The curve of stress-elongation % of tensile test was plotted on the computer automatically

the specimen's elongation and applied load is measured. Strain and stress are calculated from these values, and are used to construct a stress–strain curve (Fig. 1). From the slope of this curve, the elastic modulus is determined.

Density and ultrasonic wave velocities measurements

The density of the samples were measured according to the Archimedes principle using water as the immersion liquid by an analytical balance (Radwag AS220/C/2, capacity 220 g, readability 0.1 mg, Poland). First, the temperature of the room inserted into the balance; next, the mass of the samples were measured in air and in water, and finally, the densities of the samples were measured by the balance automatically. The accuracy of the measurements is about 0.001%.

The ultrasonic wave velocities measurements were done by pulse echo method at room temperature. The ultrasonic pulses are provided by a 5800PR (35 MHz Panametrics Olympus, USA) generator. An electrical impulse with high amplitude and short duration excites the piezoelectrical transducer vibrating on the fundamental mode through the sample, and after reflections on the opposite face returns to the transducer. After propagation in the material, the output signal is displayed on the screen of a numerical oscilloscope (60 MHz GW Instek GDS-2062, Taiwan). 5 MHz (V109-Panametrics Olympus, USA) longitudinal and 5 MHz shear (V155-Panametrics Olympus, USA) contact transducers were used.

As the coupling medium, glycerin (BQ-Panametrics Olympus, USA) was used for the longitudinal wave measurements, and shear wave couplant (SWC) (SWC-Panametrics Olympus, USA) for the shear wave measurements. The knowledge of the transit time through the thickness of the sample allows the determination of the wave velocities by Eq. 1.

$$V = 2d/t \tag{1}$$

where V, d, and t are the velocity of sound, the thickness of the sample, and the time-of-flight between subsequent backwall signals on the oscilloscope, respectively. The measurements were repeated ten times to check the reproducibility of the data. The accuracy of velocity measurements is about 0.04%. The Young's modulus was calculated according to the following formulae [25–27].

$$E = \frac{\rho V_{\rm S}^2 (3V_{\rm L}^2 - 4V_{\rm S}^2)}{(V_{\rm L}^2 - V_{\rm S}^2)} \tag{2}$$

where $V_{\rm L}$, $V_{\rm S}$, E, and ρ are longitudinal ultrasonic wave velocity, shear ultrasonic wave velocity, Young's modulus of elasticity and density of the samples, respectively. The estimated accuracy of Young's modulus is about 0.04%.

Results and discussion

Density and ultrasonic velocity

The variations of density of composites of PS_{230} made with MPSs are shown in Tables 2 and 3. As seen from Tables 2, the density of pure PS_{230} was 1041 kg/m³. The densities ranged between 1038 and 1054 kg/m³ for the composites of PS_{230} / MPS₅₀₀ (SAI, MAI, PhAI) and 1042 and 1052 kg/m³ for the composites of PS_{230} / MPS₃₅₀ (SAII, MAII, PhAII). The binding of the various functional groups to the aromatic ring of pure PS using BF₃O(C₂H₅)₂ catalyst was shown in Scheme 1. As seen from chemical modification schemes, these carboxylic functional groups has aromatic, saturated aliphatic and unsaturated aliphatic parts in case of PhA, SA and MA, respectively. The higher data were obtained with PhA modified PSs. So it can be stated that the reason of this condition is binding functional group contain aromatic ring. Also, it can be stated that the densities of all PS₂₃₀ based composites are higher than the density of pure PS₂₃₀.

 $V_{\rm L}$ and $V_{\rm S}$ data for pure PS₂₃₀ were obtained as 2344 and 1147 m/s, respectively. The variations of $V_{\rm L}$ and $V_{\rm S}$ data of composites with SAI, MAI, and PhAI are illustrated in Table 2. The $V_{\rm L}$ values of all composites were obtained higher than pure PS. The biggest increase for $V_{\rm L}$ is seen for composites of PS₂₃₀/SAI in 70:30 wt% ratio and for composites of PS₂₃₀/MAI and PS₂₃₀/PhAI in 90:10 wt% ratio. For PS₂₃₀/SAI, average $V_{\rm L}$ increased from 2399 to 2409 m/s. The variations of $V_{\rm L}$ and $V_{\rm S}$ with SAII, MAII, and PhAII addition to PS₂₃₀ are illustrated in Table 3. The biggest increase for $V_{\rm L}$ is seen for composite PS₂₃₀/SAII in 70:30 wt% ratio, for other composites in 90:10 wt% ratio, too. For PS₂₃₀/SAII, average $V_{\rm L}$ increased

Table 2 Variation of density (ρ) , longitudinal wave velocity	Composition%	ρ (kg/m ³)	$V_{\rm L}$ (m/s)	$V_{\rm S}~({\rm m/s})$	
$(V_{\rm L})$ and shear wave velocity $(V_{\rm c})$ of PS and PS-based	PS ₂₃₀ :SAI				
composites made with MPS ₅₀₀	100:0	1041	2344 ± 0.04	1147 ± 0.04	
(SAI, MAI, PhAI)	90:10	1052	2399 ± 0.03	1159 ± 0.04	
	80:20	1047	2403 ± 0.04	1167 ± 0.03	
	70:30	1046	$\textbf{2409} \pm 0.03$	1171 ± 0.04	
	PS ₂₃₀ :MAI				
	100:0	1041	2344 ± 0.04	1147 ± 0.04	
	90:10	1038	2404 ± 0.03	$\textbf{1168} \pm 0.04$	
	80:20	1044	2398 ± 0.03	1161 ± 0.03	
	70:30	1053	2393 ± 0.04	1157 ± 0.04	
	PS ₂₃₀ :PAI				
	100:0	1041	2344 ± 0.04	1147 ± 0.04	
	90:10	1049	$\textbf{2399} \pm 0.03$	$\textbf{1173} \pm 0.03$	
	80:20	1054	2394 ± 0.04	1168 ± 0.04	
Significance results were shown in bold	70:30	1046	2390 ± 0.04	1167 ± 0.03	
Table 3 Variation of density (ρ) , longitudinal wave velocity	Composition%	ρ (kg/m ³)	$V_{\rm L}~({\rm m/s})$	$V_{\rm S}~({\rm m/s})$	
$(V_{\rm L})$ and shear wave velocity $(V_{\rm S})$ of PS and composites of PS	PS ₂₃₀ :SAII				
made with MPS ₃₅₀ (SAII, MAII,	100:0	1041	2344 ± 0.04	1147 ± 0.04	
PhAII)	90:10	1043	2398 ± 0.03	1171 ± 0.03	
	80:20	1048	2404 ± 0.04	1176 ± 0.04	

PS230:PhAII 100:0 1041 2344 ± 0.04 1147 ± 0.04 90:10 1049 2405 ± 0.03 1171 ± 0.03 80:20 1052 2396 ± 0.04 1164 ± 0.04 Significance results were shown 1163 ± 0.03 70:30 1046 2388 ± 0.03 in bold

1044

1041

1050

1042

1051

 $\textbf{2407} \pm 0.03$

 2344 ± 0.04

 $\mathbf{2406} \pm 0.05$

 2395 ± 0.04

 2387 ± 0.03

70:30

90:10

80:20

70:30

PS230:MAII 100:0

from 2398 to 2407 m/s, for PS_{230} /MAII from 2387 to 2406 m/s and for PS_{230} /PhAII from 2388 to 2405 m/s.

As seen from Tables 2 and 3, the $V_{\rm S}$ values of all the composites were higher than pure PS, too. The biggest increase for V_S was seen for PS_{230}/SA composites in the 70:30 wt%, for other composites in 90:10 wt% ratio. For PS230/SAI composites, average $V_{\rm S}$ increased from 1159 to 1171 m/s and for PS₂₃₀/SAII from 1171 to 1185 m/s.

 $\textbf{1185} \pm 0.04$

 1147 ± 0.04

 1168 ± 0.05

 1160 ± 0.03

 1157 ± 0.04

Methods of determining the degree of compatibility have been reported, both theoretically and experimentally [13, 28, 29]. Many researchers [30–37] have reported that ultrasonic velocity measurements might show the extent of compatibility in highly viscous or solid forms of polymer blends. Singh et al. [31–34] studied the ultrasonic velocity for compatible, semicompatible, and incompatible polymeric blends, and they found that in compatible blends, the ultrasonic velocity varied linearly with composition. The important result for velocity measurements of the components of a composite. This behavior confirms the good miscibility of the two component based on PS forming one single phase. So the bigger velocity shows the bigger compatibility with the components of a composite. As seen from V_L and V_S data, the most appropriate wt% ratios for composites with SA, MA and PhA-modified PSs were determined as 70:30, 90:10 and 90:10, respectively.

Young's modulus

The mechanical properties of polymers are influenced by molecular weight, crosslinking, branching, segmental motion, morphology, and external conditions such as temperature, pressure, loading rate, environmental condition, extent of compound, etc. [2, 38–41]. The effect of side group structure on the compressive strength of novel biodegradable polyphosphazene based polymers was investigated by Sethuraman et al. Results of mechanical testing studies demonstrated that the nature and the ratio of the pendent groups attached to the polymer backbone play a significant role in determining the mechanical properties of the resulting polymer. The compressive strength of polymer with aliphatic alanine side group was significantly higher than polymers with aromatic alanine groups [42].

Different functional groups may affect Young's modulus of composites in different ways. To understand the effect of molecular structure, three various carboxylic (–COOH) functional groups with aromatic, saturated aliphatic and unsaturated aliphatic parts in case of PhA, SA, and MA, respectively, are specifically studied. Young's modulus of PS-based composites has been measured mechanically by the tensile test. Furthermore, the Young's modulus of these composites have been measured by velocities of propagation of longitudinal and shear ultrasonic waves and the densities of the composites using Eq. 2. The values of Young's modulus that measured by the tensile test and ultrasonic method have been compared with each other. The comparative results are given in Table 4.

As seen from Table 4, the Young's modulus of PS_{230} obtained by ultrasonic method and tensile test were 3.68 and 3.73 GPa, respectively, and tend to increase with all type MPSs addition.

The Young's modulus of PS_{230} was increased with MAI and PhAI addition at 90:10 wt% ratio compositions. According to the results of ultrasonic method, the Young's modulus ranged from 3.81 to 3.86 GPa, 3.79 to 3.81 GPa, and 3.83 to 3.88 GPa for PS_{230}/SAI , PS_{230}/MAI , and $PS_{230}/PhAI$ composites, respectively. According to the results of tensile test, the Young's modulus ranged from 3.61 to 4.06 GPa for PS_{230}/SAI composites, from 3.56 to 4.17 GPa for PS_{230}/MAI composites and

Table 4The comperativeresults of pure PS and PS basedcomposites' Young's modulus(E) values measured by tensile	Composition%	Young's modulus values measured by Ultrasonic method E (GPa)	Young's modulus values measured by Tensile Test E (GPa)
test and ultrasonic method	PS ₂₃₀ /SAI		
	100:0	3.68	3.73
	90:10	3.81	3.61
	80:20	3.84	4.03
	70:30	3.86	4.06
	PS ₂₃₀ /MAI		
	100:0	3.68	3.73
	90:10	3.81	4.17
	80:20	3.79	3.88
	70:30	3.80	3.56
	PS230/PhAI		
	100:0	3.68	3.73
	90:10	3.88	3.95
	80:20	3.86	4.04
	70:30	3.83	3.67
	PS ₂₃₀ /SAII		
	100:0	3.68	3.73
	90:10	3.84	3.70
	80:20	3.89	3.98
	70:30	3.93	3.91
	PS ₂₃₀ /MAII		
	100:0	3.68	3.73
	90:10	3.86	4.19
	80:20	3.78	3.94
	70:30	3.79	3.77
	PS230/PhAII		
	100:0	3.68	3.73
	90:10	3.87	4.39
	80:20	3.83	4.30
Significance results were shown in bold	70:30	3.81	4.00

from 3.67 to 4.04 GPa for PS_{230} /PhAI composites. Figures 2, 3, and 4 illustrate the Young's modulus values as a function of the weight percent of MPSs.

As it is seen from these results and Figs. 2, 3, and 4 it can be stated that the values of Young's modulus measured with ultrasonic method and with tensile test are similar. The Young's modulus of composites with MPS₅₀₀ were higher than pure PS.

The results given in Table 4 and Figs. 2, 3, 4 indicate that Young's modulus of composites with MPS₃₅₀ are higher than pure PS, too. According to the results of ultrasonic method, the Young's modulus ranged from 3.84 to 3.93 GPa for PS₂₃₀/SAII composites, from 3.79 to 3.86 GPa for PS₂₃₀:MAII composites and from 3.81 to 3.87 GPa for PS₂₃₀/PhAII composites. According to the results of tensile test, the



Fig. 2 Variation of the Young's modulus values with 10% of MPS in composites of PS/MPS



Fig. 3 Variation of the Young's modulus values with 20% of MPS in composites of PS/MPS

Young's modulus ranged from 3.70 to 3.98 GPa, from 3.77 to 4.19 GPa and from 4.00 to 4.39 GPa for $PS_{230}/SAII$, $PS_{230}/MAII$, and $PS_{230}/PhAII$ composites, respectively. It can be stated that the values of Young's modulus measured with ultrasonic method and with tensile test are similar too.



Fig. 4 Variation of the Young's modulus values with 30% of MPS in composites of PS/MPS

As it is seen from these results and Figs. 2, 3, 4, according to obtained Young's modulus data, the order of effect of MPSs obtained from both PS with different anhydrides as follow: MPS with PhA > MPS with SA > MPS with MA. The structure of the side-chain substituents on the polymer backbone is a major compositional factor impacting polymer functionality. Important aspects of substitution are the chemical structure of the substituents, the extent of backbone substitution, and the uniformity of substitution. The nature of the side chain substituent type also significantly impacts mechanical properties. Increasing the amount of highly polar, ionic side-chains tends to result in an increased tensile strength [43]. Ahmetli et al. [44] reported that acid number of modified PSs with maleic anhydride were 30 and 35 mg KOH/g sample for 5×10^5 and 3.5×10^5 molecular weight of pure PS, respectively. Deveci [45] investigated the coating and adsorption properties of modified different molecular weight PSs (5 \times 10⁵ and 3.5 \times 10⁵) with succinic (SA) and phthalic anhydrides (PhA) and determined acid numbers were 80-86 mg KOH/g sample for PhA and 75–76 mg KOH/g sample for SA modified PSs. The result, according to the literature as follow: in chemical modification with phthalic anhydride more polar carboxylic functional groups bound to the aromatic ring of PS. Therefore, the highest Young's modulus results were determined for PS230/PhA composites. The most appropriate weight % ratios for composites with SA, MA and PhA modified PSs were determined as 70:30, 90:10 and 90:10, respectively.

As a result, it can be stated that a significant difference has not been found between the values of Young's modulus of both methods. But the results of ultrasonic method are more close to the literature than the results of tensile test. The differences of the Young's modulus values between ultrasonic method and tensile test are effected by composition of material and also by microstucture (porosity, phases, imperfections, etc.) of the samples, too. The porosity, halls and imperfections of the materials are more important while working with tensile testing because while working with ultrasonic method, you can select a place on the face of the sample that does not contain any porosity, hall, and imperfections. But while working with tensile testing, you cannot select any place on the sample as you can at ultrasonic working. So the measurement results of ultrasonic methods are more convenient than the tensile test measurements.

Conclusions

From the results above it, can be stated that material characterization by ultrasonic methods are more convenient than the destructive methods as tensile test measurements. The Young's modulus values show the degree of compatibility between components of any composites. According to the results, the best compatibility was seen at 70:30 wt% composition ratio for PS-based composites with SAI and SAII addition (PS₂₃₀/SAI and PS₂₃₀/SAII). The most appropriate weight % ratios for composites with MA and PhA modified PSs were determined as 90:10. The order of effect of MPSs obtained from both PS with different anhydrides on Young's modulus of PS₂₃₀-based composites as follow: MPS with PhA > MPS with SA > MPS with MA. Finally, it can be concluded that the pulse-echo method has the ability to evaluate the mechanical properties of polymer blends. The measurement of Young's modulus by ultrasonic methods is cheaper, easier and more economical than by the destructive methods. Therefore, measurement of Young's modulus or other mechanical properties of materials by ultrasonic methods can be recommended to all researchers.

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