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EPDM-HDPE Blends with Different Cure Systems/Mechanical and Infra-Red Spectrometric Properties

¹Zenovia Moldovan, ²Florica Ionescu, ¹Simona Lițescu, ³Ioana Vasilescu and ³Gabriel Lucian Radu

¹Department of Analytical Chemistry, Faculty of Chemistry, University of Bucharest,
4-12 Regina Elisabeta Blvd, 030018, Sect. 3-Bucharest, Romania

²Research Development National Institute for Textile and Leather-Division Leather and Footwear Research
Institute, 93 Ion Minulescu St., Sect 3, 031215-Bucharest, Romania

³Department of Analytical Chemistry, Faculty of Chemistry, University Politehnica of Bucharest,
1 Gheorghe Polizu St., Sect. 1, Cod 011061-Bucharest, Romania

Abstract: The physico-mechanical properties and the Infra-red (IR) spectra (obtained by applying the Attenuated Total Reflectance (ATR) technique) of high density polyethylene (HDPE)/ethylene propylene diene terpolymer (EPDM) blends were investigated using a Zwick Tensile Testing machine and a Fourier Transform Infra-red (FTIR) spectrometer. Three kinds of cure systems were used: sulfur, tetramethylthiuram disulfide (TH) and 2-mercaptobenzothiazole (M); sulfur, tetramethylthiuram disulfide (TH) and zinc-diethyldithiocarbamate (ZDEC); sulfur, tetramethylthiuram disulfide (TH) and diphenylguanidine (D). The composition of HDPE and EPDM was fixed at 50/50 by weight and the ratio cure accelerators/sulfur was varied from 0.4 to 6.34 by weight. Results indicated that polymer alloys EPDM/HDPE containing as cure agents Sulfur, TH and M, in a ratio cure accelerators/sulfur = 6.34 and 1.85, respectively, showed improved physico-mechanical properties as compared to the other polymer blends. This behavior was confirmed by the recorded ATR-FTIR spectra of the studied samples.

Key words: HDPE-EPDM thermoplastic vulcanizates, attenuated total reflectance, mechanical properties

INTRODUCTION

Blends of different polymers are of considerable importance as blending provides a means of improving physical and mechanical properties. Thermoplastic elastomers (TPEs) represent materials prepared by blending rubber component with polyolefin. Studies on thermoplastic elastomers with various types of rubber and polyolefin have been reported by many researchers (Abdou-Sabet, 1998; Goharpey *et al.*, 2005; Mahajan *et al.*, 1996; Manoj and Unnikrishnan, 2007; Mishra *et al.*, 2004; Naderi *et al.*, 2007; Sadek *et al.*, 2003; Tzong-Ming and Mu-Shun, 2005; Van Duin, 2006; Wang *et al.*, 2003). TPEs are generally categorized into two with one consisting of simple blends of uncured EPDM rubber and polyolefin. This class of TPEs is commonly called thermoplastic elastomeric olefins (TEO) (ASTM D 5593, 1999).

In the other category, the rubber phase is dynamically vulcanized, giving rise to a thermoplastic vulcanizate (TPV) (ASTM D 5046, 1998). TPVs are

characterized by the presence of finely dispersed cross-linked rubber particles distributed in a continuous thermoplastic matrix (Jha and Bhowmick, 1997; Legge *et al.*, 1987; Patel *et al.*, 2005; Patil and van Ooij, 2005). If the rubber particles are sufficiently vulcanized, the physical and chemical properties of the blend are generally improved. This study investigated the preparation and characterization of some thermoplastic vulcanizates based on EPDM-HDPE system. The aim of our studies was to improve the physico-mechanical properties of some micro-structured polymer blends, in order to find the better specimens valuable for industrial applications. For this reason, different cure systems (conventional, semi-efficient and efficient) were used. A tentative explanation is given which tries to help to understand better the relations between structure and properties of the composites containing HDPE, EPDM and polymer additives. Also, a correlation between ATR-FTIR spectra and physico-mechanical properties of the obtained thermoplastic elastomers was proposed as an important step in the quality control of polymer blends in routine analysis.

MATERIALS AND METHODS

Preparation of polymeric blends and the physico-mechanical procedures were made at Research Development National Institute for Textile and Leather-Division Leather and Footwear Research Institute and the proposed spectrometric method was conducted at Department of Analytical Chemistry, University of Bucharest, Faculty of Chemistry, Bucharest, Romania (the experiments were performed in the year 2007, as part of the National Program Matnantech 88/2006).

All composites contain the same two polymers: the EPDM elastomer (Nordell IP 3745P, DuPont) and the high density polyethylene (HDPE B084, Petro-Midia, Romania), in the ratio 1:1 by weight.

The following cure accelerators were used: (tetramethylthiuram disulfide (TH); 2-mercaptobenzothiazole (M); zinc-diethyldithiocarbamate (ZDEC); diphenylguanidine (D)). As cross-linking agent, sulfur was chosen. The activators (zinc oxide and stearic acid) and antioxidants (di-lauril-orto-dipropionate (Irganox 1010) and 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ)) were added to all receipts.

Apparatus for physico-mechanical tests: Tensile tests of the samples were carried out according to ASTM D412-98 using a Zwick Tensile Testing machine 1445, at a constant crosshead speed of $500 \pm 5 \text{ mm min}^{-1}$. Hardness of the samples was measured using a Shore type A Durometer (Zwick Model) according to ASTM D412-98.

Apparatus for FTIR measurements: ATR-FTIR measurements were run with a Bruker instrument (model Tensor 27), in the following conditions: wavenumber range: $600\text{-}4000 \text{ cm}^{-1}$; aperture setting: 6 mm; scanner velocity: 2.2 kHz; background scan time: 32 sec; sample scan time: 32 sec; resolution: 6 cm^{-1} ; beamsplitter: KBr; angle of incident radiation: 45° . After recording, the ATR-FTIR spectra were converted into transmission FTIR spectra. The plate samples ($6 \times 6 \text{ mm}$) were simply posed on the sampling stage, in intimate contact with the optical element, a hemicylindrical prism of SeZn (called Internal Reflection Element (IRE)). The incident radiation arrives onto the sample with a certain angle (usually, between 30 and 60°) to the normal of the sample plane and the reflected beam is collected by a mirror that focuses the reflected radiation onto the detector.

Preparation of polymer blends: The blends were prepared by melt-mixing the ingredients, the two polymers (EPDM and HDPE) and the additives, in a laboratory cam mixer (Brabender Plasti-Corder PLE-60), at 180°C for about 7-8 min. A constant rotor (cam type) speed of 80 rpm was

applied. Finally, once homogenous mixing was assured, the polymer blend was recovered from the mixer and transferred to a two-roll mill at $155 \pm 5^\circ\text{C}$ and 24 rpm. The compound was then compression-molded (using an electrically heated hydraulic press at 190°C under 20 MPa pressure) to achieve a sheet of about 2 mm thick. The sheet was then cooled down to room temperature under same pressure. The specimens were die-cut from the compression molded sheet and used for testing after 24 h of storage at room temperature. All experiments were made at room temperature. Fifteen compositions of EPDM/HDPE blends had been prepared by using various systems of accelerators in different ratio cure accelerators/sulfur.

Aging experiments: In order to investigate the effect of lab aging conditions on the physico-mechanical properties of the prepared thermoplastic vulcanizates, the test specimens were placed in an air-circulating oven at 100°C for 168 h.

RESULTS AND DISCUSSION

It is widely recognized that the formulation has a strong influence on the properties of polymer composites. For this reason, the effect of the type of cure system and of the ratio of cure agents on the physico-mechanical properties of the prepared polymer blends was investigated. The EPDM/HDPE composites were obtained by different chemical methods of vulcanization and in different formula. All polymer samples have the same ratios (parts in weight) EPDM: HDPE: ZnO: Stearic acid: TMQ: Irganox 1010 = 50:50:5:0.4:1:1 and different ratios of cure agents. The cure systems used are shown in Table 1. We mentioned that when the ratio of sulfur to accelerator is less than one, the receipt is known as an Efficient Vulcanization (EV) system.

Shore A durometer hardness: The value of Shore A Durometer is the measure of elastic modulus of the polymer blend, which is often measured indirectly by measuring the elastic indentation of prescribed size and shape, pressed into the surface under specified loading conditions. Higher values of this parameter correspond to harder compounds and vice versa. As it can be shown in Fig. 1, the values of this parameter were not significantly different for all investigated compounds; the loading with cure agents and the presence of HDPE gives a great hardness of the cured compounds.

Stress at 300% (Modulus) and tensile strength (stress-at-break): The stress values at 300% strain for all polymer composites were investigated. The importance of 300%

Table 1: Cure agents in formulation of some micro-structured polymer blends (parts in weight)

Cure system	Symbol of polymer blends	Cure agents				
		Sulfur	TH	M	ZDEC	D
Conventional cure system	S2	2.00	1.0	0.5		
	S4	2.00	1.0		0.5	
	S6	2.00	1.0			0.5
	S8	2.00	0.5	1.0		
	S10	2.00	0.5		1.0	
	S11	2.00	0.5			1.0
	S13	2.00	0.5	0.3		
	S15	2.00	0.5		0.3	
	S16	2.00	0.5			0.3
	S26	2.00	0.5			0.3
Semi-EV system*	S18	1.00	1.2	0.7		
	S20	1.00	1.2		0.7	
	S21	1.00	1.2			0.7
EV system*	S23	0.41	1.8	0.8		
	S25	0.41	1.8		0.8	
	S26	0.41	1.8			0.8
	S26	0.41	1.8			0.8

*: EV system = Efficient vulcanization system. Semi-EV system = Semi-efficient vulcanization system

modulus lies in the fact that it can be taken as an index of the total sum of chemical and physical cross-links, such as chain entanglements (Patil and Van Ooij, 2005). As it is shown in Fig. 2, the value of modulus at 300% is higher for the compounds S18, S20, S23 and S25. The stress-at-break values for all polymer composites are shown in Fig. 3. The highest tensile strength corresponds to the compounds S18, S21 and S23. As the literature data reported, the tensile strength depends on the compatibility of EPDM phase and HDPE phase of TPV (Mishra and Chang-Sik Ha, 2005), which is evident in the case of the mentioned samples. The other compounds i.e., S2, S8, S10, S11, S15, S16 showed lower and nearly equal tensile strength values. This suggests that using conventional cure systems of compounding did not hinder the strain crystallization behavior of these samples. In almost all cases, the cure conditions obtained by using semi-EV and EV systems always results in higher tensile values.

Strain at break (Elongation, %): Strain at maximum elongation i.e., the extensibility of the studied composites is shown in Fig. 4. It is known that the extensibility of the polymer blend decreases with the increases of cross-linking (Hamed and Rattanasom, 2002). In the same time, a high crosslink makes the compound stiff and it loses its extensibility and strength (which is confirmed from its elongation, % value).

The results shows in Fig. 4 seem to confirm this assumption. The effect of crosslink density on the extensibility is evident in the case of the compounds noted S18, S23 and S25, which have the lower values of Elongation. On the other hand, on the basis of data given in Table 1, we observe the following ratios cure accelerators/sulfur: 0.75 for the conventional cure systems S2, S4, S6, S8, S10, S11 and 0.4 for S13, S14, S15; 1.85 for the semi-EV cure systems S18, S20, S21; 6.34 for EV cure systems S23, S25, S26. So, even the conventional systems

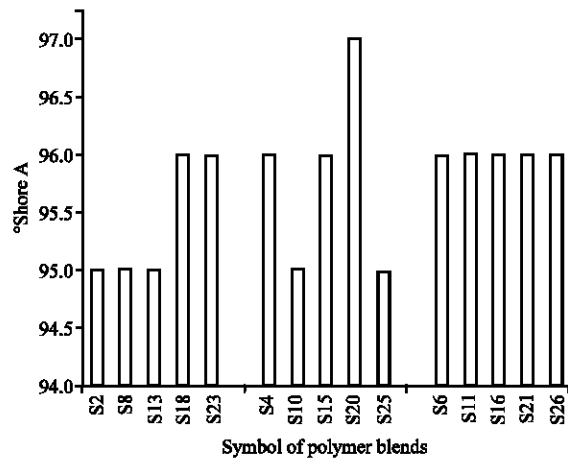


Fig. 1: °Shore A hardness

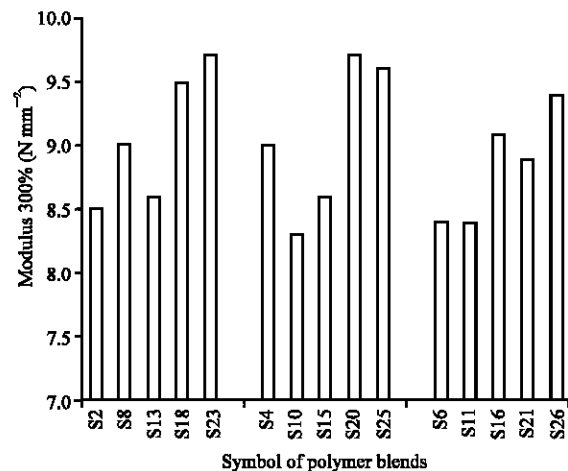


Fig. 2: Stress at 300% strain (modulus)

have higher sulfur loading, more efficient seem to be the semi-EV cure system S18 and EV cure systems S23, S25, which contains lower amounts of sulfur. This behavior

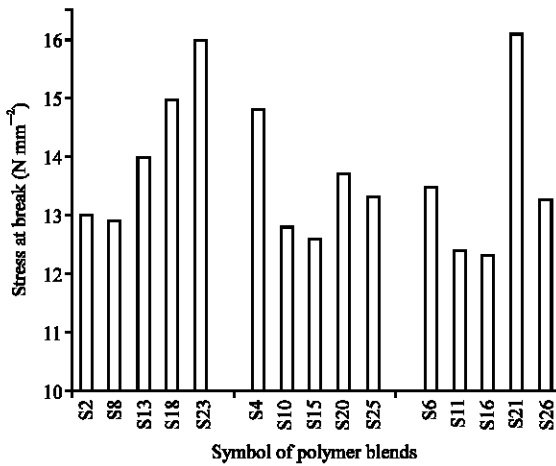


Fig. 3: Stress at break (N mm⁻²)

could be explained as follows: too many crosslink junctions (assured by the polysulfide bonds) will hinder the structural regularity needed for crystallization (it is the case of samples such as S2, S4, S6, S8, S10, S11, S16), which have higher values of strain at break. Also, in high loading of sulfur (it is the case of the last mentioned systems), most of links are dangling and do not contribute towards the strength of the compounds. The highly cross-linked compounds can not crystalline easily because of the immobility of the network structure. In the case of semi-EV system S18 and EV systems S23, S25 the cross-linking obtained is beneficial in terms of providing the mobility to align the chains upon deformation; as a result, the strain at break is lower. On the other hand, the semi-semi EV system (containing the accelerators TH+M, in sample S18) and EV-cure systems (containing the accelerators TH+M, in samples S23, TH+ZDEC in sample S25) seems to be more efficient than those containing TH+D (in samples S21, S26). This could be a consequence of the medium action of CZ (see the value of Elongation for S18, S23, S25, comparatively with S21, S26).

Tear behavior: The tear strength values for the compounded polymer blends are shown in Fig. 5. Results indicated that, the tearing strength values were influenced by the kind of sample. As a confirmation of this assumption, the literature data reported that tear strength values completely depends on the type of sample itself, filler type and test conditions (De and Gent, 1996; Hamed and Rattanasom, 2002). We observed that the compounds S18, S23, S25 have higher tear strength values than the others. This behavior could be explained by the strain crystallization property in the mentioned compounds.

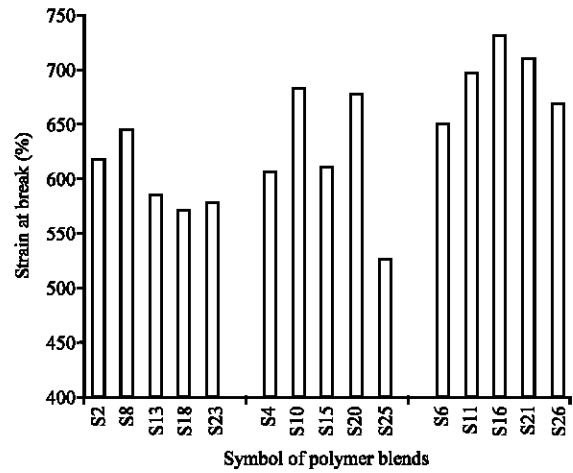


Fig. 4: Strain at break (%)

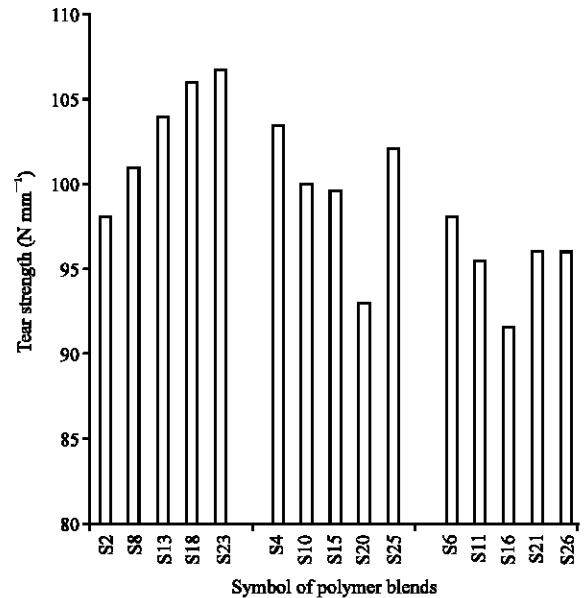


Fig. 5: Tear strength (N mm⁻¹)

Elasticity and wear: As it is shown in Fig. 6, in the almost all cases, a higher value of the elasticity is confirmed by a lower wear. These results were predictable taking into account that a low elasticity could be a result of regularity of the polymer chains and vice versa.

The effect of accelerated aging: Generally, an increase of modulus 300% and stress at break and a decrease of strain at break were observed (Table 2, 3, 4). This represents an improvement of some physico-mechanical properties of the obtained thermoplastic vulcanizates at high temperature. This behavior should be explained as follows: at high temperature, a physical interaction

Table 2: The effect of aging on the physico-mechanical properties of some thermoplastic vulcanizates containing as cure agents S, TH, M

Physico-mechanical parameters	Symbol of polymer blend									
	S2		S8		S13		S18		S23	
	a	b	a	b	a	b	a	b	a	b
°Shore A hardness	95.0	95.0	95.0	96.0	95.0	94.0	96.0	95.0	96.0	96.0
Elasticity (%)	32.0	30.0	30.0	28.0	30.0	29.0	30.0	30.0	28.0	34.0
Modulus 300% (N mm ⁻²)	8.5	10.7	9.0	10.8	8.6	11.8	9.5	10.4	9.7	10.4
Stress at break (N mm ⁻²)	13.0	19.3	9.7	18.7	14.0	19.0	15.0	18.7	16.0	19.0
Strain at break (%)	620.0	553.0	647.0	567.0	587.0	520.0	580.0	560.0	600.0	580.0
Tear strength (N mm ⁻¹)	98.0	96.0	101.0	92.5	104.0	98.5	106.0	96.5	106.5	105.0

a: Normal conditions, b: After accelerated aging

Table 3: The effect of aging on the physico-mechanical properties of some thermoplastic vulcanizates containing as cure agents S, TH, ZDEC

Physico-mechanical parameters	Symbol of polymer blend									
	S4		S10		S15		S20		S25	
	a	b	a	b	a	b	a	b	a	b
°Shore A hardness	96.0	94.0	95.0	96.0	96.0	95.0	97.0	95.0	95.0	96.0
Elasticity (%)	33.0	28.0	26.0	26.0	29.0	30.0	21.0	30.0	28.0	32.0
Modulus 300% (N mm ⁻²)	9.7	12.2	8.3	10.1	8.6	17.2	9.7	9.8	9.6	10.8
Stress at break (N mm ⁻²)	15.1	22.0	12.8	18.9	12.6	19.4	13.7	16.7	13.3	16.3
Strain at break (%)	607.0	573.0	687.0	593.0	613.0	573.0	680.0	600.0	527.0	547.0
Tear strength (N mm ⁻¹)	103.0	111.5	100.0	107.5	99.5	93.5	93.0	84.5	102.0	93.0

a: Normal conditions, b: After accelerated aging

Table 4: The effect of aging on the physico-mechanical properties of some thermoplastic vulcanizates containing as cure agents S, TH, D

Physico-mechanical parameters	Symbol of polymer blend									
	S6		S11		S16		S21		S26	
	a	b	a	b	a	b	a	b	a	b
°Shore A hardness										
Elasticity (%)	32.0	28.0	28.0	26.0	30.0	30.0	30.0	28.0	26.0	26.0
Modulus 300% (N mm ⁻²)	8.4	10.0	8.4	12.3	9.1	10.9	8.9	10.6	9.4	9.9
Stress at break (N mm ⁻²)	13.5	17.5	12.4	18.0	12.3	17.0	16.1	19.0	13.3	18.7
Strain at break (%)	653.0	560.0	700.0	513.0	733.0	527.0	713.0	560.0	673.0	600.0
Tear strength (N mm ⁻¹)	98.0	96.5	95.5	92.0	91.5	85.0	96.0	98.5	96.0	88.0

a: Normal conditions, b After accelerated aging

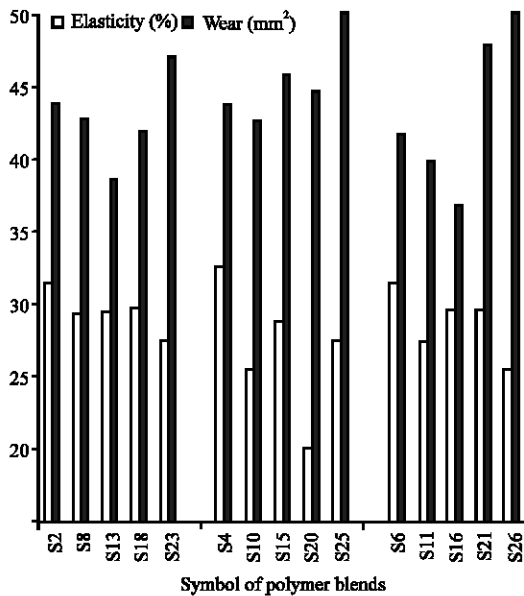


Fig. 6: Elasticity (%) and wear (mm²)

between many of vulcanized rubber particles takes place, this resulting in a network of vulcanized elastomer (Coran and Patel, 1996). Also, the substantial change on most of tensile properties may be an indication of a post-curing process. The effect of accelerated aging shows that the prepared thermoplastic vulcanizates are good candidates for industrial applications to be used at high temperature.

ATR-FTIR spectra: The Attenuated total reflectance Fourier Transform IR (ATR-FTIR) method was chosen for spectral characterization of the prepared polymer blends. This, because the mentioned technique is of considerable value when studying physical properties of materials known as difficult samples such as polymer composites (Harrick, 1987). The ATR-FTIR technique allows the analysis of specimens with minimum sample preparation without solvent casting, grinding or pressing, as in the case in the transmission experiments.

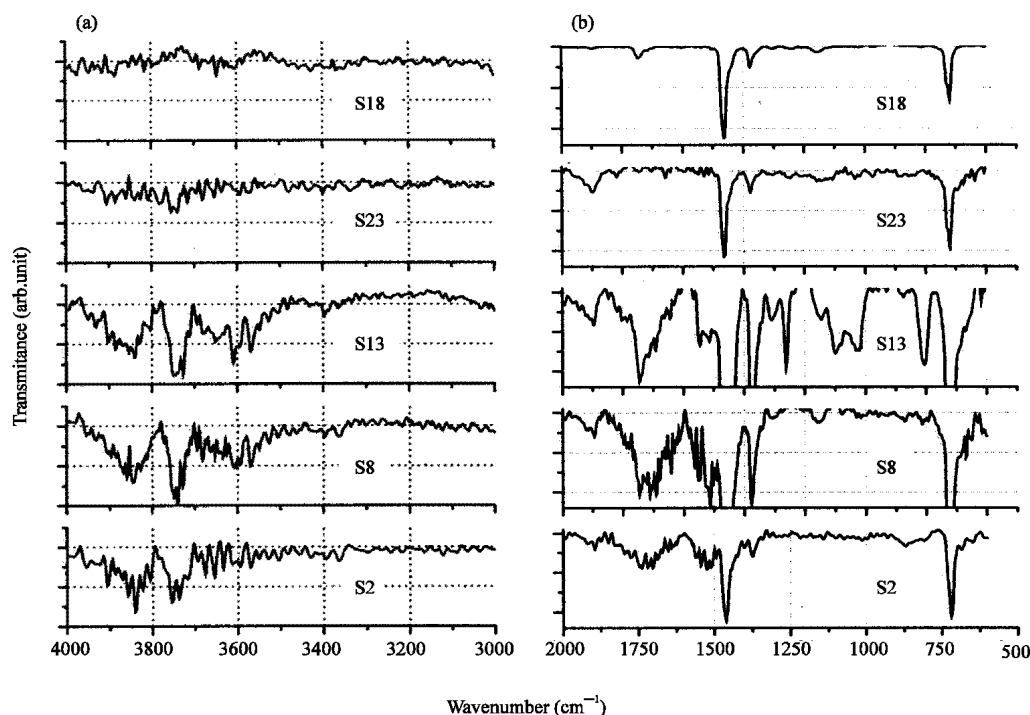


Fig. 7a, b: IR spectra of the polymer blends containing as cure agents S, TH, M

Taking into account that the IR radiation penetrates into the sample a few nanometers depth, it must be precised that the information obtained is characteristic for the surface of the sample and the great advantage of using ATR technique is that no sample preparation was required. The ATR-FTIR spectra recorded for the studied polymer blends gave us the following informations:

- All the investigated samples have the specific strong bands in their IR spectra specific to $-\text{CH}_2-$ bonds in EPDM and HDPE polymers. Thus, the bands near 2925 and 2853 cm^{-1} are due to the asymmetric and symmetric stretching frequency of the C-H group; a medium band located at $1430-1470\text{ cm}^{-1}$ is attributed to deformation frequency of the same methylene groups and a weak peak is located at 720 cm^{-1} (also, for $-\text{CH}_2$).
- No absorption bands were detected in the range $4000-3200\text{ cm}^{-1}$ for the samples S18, S23 and very weak signals for the sample S20 (Fig. 7a, 8a, 9a). The vibrations responsible for bands in this region are O-H and N-H stretching and are characteristic IR signals of primary and secondary amines, organic acids and phenols.
- The C = O group of saturated aliphatic carboxylic acids absorbs very strong in the region

$1740-1700\text{ cm}^{-1}$. These bands are presented in the IR spectra recorded for almost all investigated samples, except the spectrum of sample S23 (Fig. 7b and 8b). In the spectrum of S18, the mentioned absorption band is very weak. On the same context, the N-C = S groups, characteristic for the cure agents as thiazoles (i.e., M) and dithiocarbamates (i.e., ZDEC), give IR absorption bands in the regions $1570-1395$, $1420-1266\text{ cm}^{-1}$ and a weak band at $1140-940\text{ cm}^{-1}$. The two first mentioned bands, with variable intensities are present in almost all spectra, except that of S23 and S18. A weak band is observed in the spectrum of S18 at about 1100 cm^{-1} . So, the absence in the spectra of these samples of the spectral bands attributed to the polymer additives, should be correlated with their good incorporation in the mentioned blends.

- As shown in Fig. 8a, b and 9a, b, many spectral bands occurs. They could be attributed to the polymer additives. For example, in Fig. 8a (spectrum of S25), a large band occurs at $3300-3420\text{ cm}^{-1}$, attributed to tertiary amines (so, it is characteristic for the cure accelerator ZDEC). Also, in Fig. 8b, the spectral bands attributed to ZDEC and located at about 1205 and 1490 cm^{-1} are characteristic for tertiary amines and diethyl amines, respectively. In

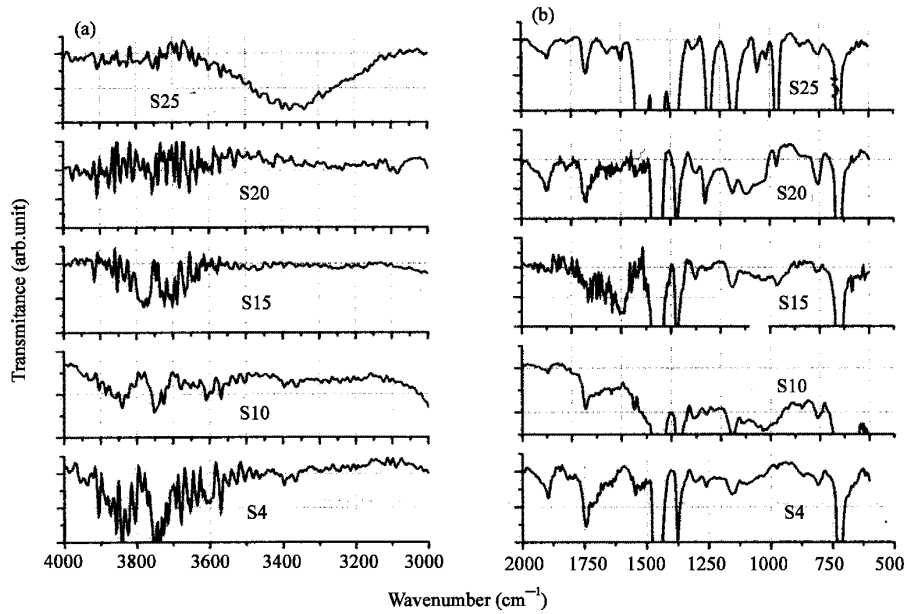


Fig. 8a, b: IR spectra of the polymer blends containing as cure agents S, TH, ZDEC

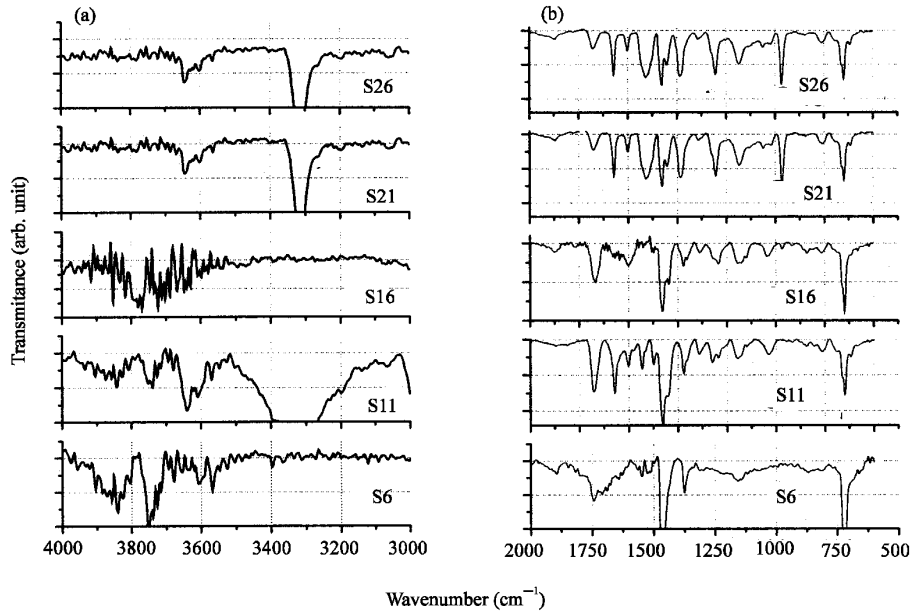


Fig. 9a, b: IR spectra of the polymer blends containing as cure agents S, TH, M, D

Fig. 9a (spectra of samples S11, S21, S26), an N-H stretching band may be observed at 3300-3450 cm^{-1} , attributed to secondary aromatic amines and diamines. This band is characteristic for the cure agent D (Diphenylguanidine).

On the basis of these spectral informations, we assume that the activator (Stearic acid), the antioxidant (Irganox 1010) and the cure accelerators are completely

incorporated in the polymer blends S18 and S23, because in the spectra of these samples do not occur the bands characteristic for these polymer additives.

The spectra in Fig. 7a, b; 8a, b and 9a, b are enlarged twenty times as against the recorded spectra. The assignment of the IR bands was carried out according to literature data (Socrates, 2001) and taking into account the spectra of polymer additives, recorded

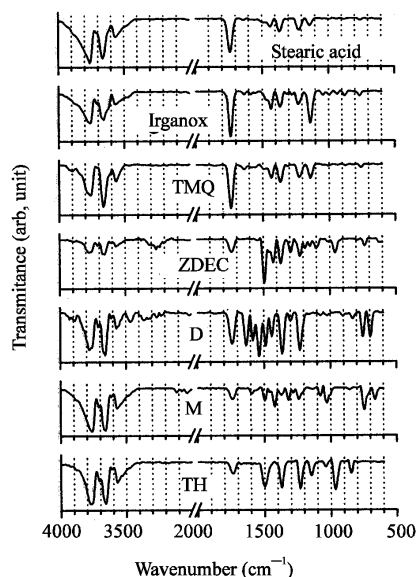


Fig. 10: IR spectra of polymer additives

by applying the ATR-FTIR technique (Fig. 10). We mention that the polymer additives were analyzed in powder state.

CONCLUSIONS

The overall results can be considered important. The optimum values of the main physico-mechanical parameters, characteristic for thermoplastic vulcanizates (high values of Shore A hardness, 300% modulus, stress at break, tear strength and low values of strain at break, elasticity), were obtained in the case of semi-EV and EV systems S18 and S23 respectively. So, by using as cure agents sulfur and the two accelerators M and TH (in the ratio cure accelerators/S = 1.85, in the case of sample S18 and 6.34, in the case of sample S23), we have obtained the best thermoplastic vulcanizates. This was confirmed by the ATR-FTIR analysis and physico-mechanical tests. We can conclude that the results of physico-mechanical tests could be verified by applying the ATR-FTIR. This technique is rapid, simple and required very little sample preparation. Also, one of the major advantages of the ATR technique is that the spectrum obtained is independent of the sample thickness. Typically, the reflected radiation penetrates the sample to a depth of only a few microns. In consequence, the method is particularly useful for surface analysis of many materials, particularly for polymer blends. Thus, it is possible to make the quality control of polymer blends in routine analysis.

In a continuation of the described experiments, many other thermoplastic vulcanizates are in work and they will be analyzed and proposed for industrial applications.

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