PROPRIETĂŢILE TERMICE ŞI MECANICE ALE COMPOZITELOR POLIPROPILENĂ/FILER SILICIOS/STIREN-BUTADIENĂ-STIREN

THERMAL AND MECHANICAL PROPERTIES OF POLYPROPYLENE / SILICEOUS FILLER/STYRENE BUTADIENE STYRENE COMPOSITES

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In this paper, new composites based on polypropylene (PP) filled with 5 ÷15% natural siliceous material and 20% styrene-butadiene –styrene block copolymer (SBS) were prepared. The siliceous fillers used in this work were the volcanic tuff and kaolinite clay. PP composites filled with mineral filler and SBS were prepared using extrusion compounding and injection molding by dynamical melt processing.

The influence of the mineral filler and SBS on the thermal properties (thermal conductivity and thermal stability evaluated by TG/DTA analyses) and mechanical properties (elasticity modulus, yield strength, elongation at break and Izod impact strength) of the PP matrix composites were studied.

The results showed that the elasticity modulus of new composites is enhanced with increasing the filler content. A better behavior is noticed in case of volcanic tuff using. The Izod impact strength is more enhanced (of 2.5 times) by adding of 20% SBS in the PP/mineral filler composites. Also, the thermal behavior of PP matrix composites is improved.

In această lucrare s-au preparat noi compozite pe bază de polipropilenă (PP) aditivate cu 5 ÷15% material silicios natural şi 20% bloc copolimer stiren-butadien-stiren (SBS). Materialele minerale de umplutură utilizate în această lucrare au fost tuful vulcanic şi argila caolinitică. Compozitele pe bază de PP aditivate cu filer mineral şi SBS au fost preparate utilizând procedeul de extrudare şi injecţie prin prelucrare dinamică în topitură. S-a studiat influenţa filerului mineral şi a SBS asupra proprietăţilor termice (conductivitate termică şi stabilitate termică evaluată prin analiză termogravimetrică TG/ATD) şi mecanice (modul de elasticitate, rezistenţă la tracţiune maximă (la curgere), alungirea la curgere şi rezistenţa la şoc Izod) ale compozitelor cu matrice polipropilenică. Rezultatele au arătat că modulul de elasticitate al noilor compozite s-a îmbunătăţit cu creşterea conţinutului de filer. O comportare mai bună s-a observat în cazul utilizării tufului vulcanic. Rezistenţa la şoc Izod este mult îmbunătăţită (de 2,5 ori) prin adăugarea a 20% SBS în compozitele PP-filer mineral. Comportarea termică a compozitelor cu matrice polipropilenică este de asemenea îmbunătăţită.

Keywords: *organo-mineral composites, volcanic tuff, clay, mechanical properties, thermal properties .*

1. Introduction

The polymeric composites are used in many range of application such as in packaging, automotive industry, aerospace and electronics industries or construction materials. For reduction of their cost and environmental footprint the composites with enhanced properties were designed and developed [1-8]. An important role plays the reinforcement materials. The mineral fillers reduce the cost of polymer composites and increase the rigidity of the polymers, but diminishing the ductility and toughness. Also, may appear issues regarding the interaction to the interface between the polymer and reinforcement material and orientation and distribution of reinforcement in the polymer matrix.

The most commonly used fillers for polypropylene (PP) composites are calcium carbonate, talc, glass fibers, mica, silica, wood flour and carbon nanotubes [4-7,9-15]. Though there are studies regarding to the polymer – mineral siliceous composites, very few of them are reported to the use of tuff in thermoplastic composites [16,17]. Besides, the information regarding the properties of the ternary composites in the system PP- mineral filler - styrene-butadiene-styrene block copolymer (SBS) is a few [18,19] and refers only to talc – a clay mineral composed by hydrated magnesium silicate- $Mg_3Si_4O_{10}(OH)_2$). The tuff is a volcanic rock contains hydrated alumina-silicates as main mineral phase and is spreading in many countries in the world compared to talc. It is expected that the presence of aluminosilicates as well as that of magnesium silicates contributes to the increase in fire resistance of polypropylene-based composites. In a previous work [20] we obtained promising preliminary results regarding to the elasticity modulus and thermal conductivity of PP- volcanic tuff and PP-clay (a very abundant phyllosilicate

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resource) composites and their morphology. So, in this work we will study the influence of mineral siliceous filler and of styrene-butadiene-styrene bloc copolymer on thermal and mechanical properties (thermal conductivity, thermal stability, elasticity modulus, yield strength, elongation at break and impact strength) of the new PP based composites in order to obtain thermal insulating composite materials with characteristics similar to usual insulating materials (such as expanded or extruded polystyrene) for building construction and industrial equipments.

2. Experimental

In order to prepare the organo-minerale composites the polypropylene (PP), two natural aluminous-siliceous sources - volcanic tuff (T) and clay (C) respectively, and styrene-butadiene-styrene (SBS) block copolymer are used.

PP homopolymer Moplen HP500N produced by LyondellBasell Polymers with a melt flow index of 12 g/10 min (230 °C/2.16 kg) and a density of 0.90 g/cm^3 was used as matrix for composite preparation.

SBS, a linear styrene-butadiene-styrene block copolymer with 31.8% polystyrene, Mn = 89000, MFI = 6.46 g/10 min (190°C/5 kg) (Europrene Sol T 166) was used as impact modifier.

The volcanic tuff and clay were ground in a laboratory tubular ball mill up to a 90 micron sieve residue of 6.0-6.1%. The SiO_2 content was of 66.65% in tuff and of 66.33% in clay, while the Al_2O_3 content was of 11.37% in tuff and of 20.46% in clay, respectively. The particle size analyses performed with Malvern Mastersizer 2000E laser granulometer revealed that the clay contains smaller particle than the tuff, the median diameter (D_{50}) being of 5.6 μm compared with 21.8 μm. From mineralogical point of view we identified the clinoptilolite, $(Na,K,Ca)_{2\text{-}3}Al_3(Al,Si)_2Si_{13}O_{36}\cdot 12H_2O$ / heulandite, $(Ca,Na)_{2\text{-}3}Al_3(Al,Si)_2Si_{13}O_{36}\cdot 12H_2O$ in tuff and kaolinite, $Al_4[(OH)_8|Si_4O_{10}]$ in clay, respectively, as main minerals [20].

MA-PP Polybond 3200 from Crompton (USA) containing 1.0 wt% grafted maleic anhydride and with a density of 0.91 g/cm^3 and a melting point of 157 °C was used as a compatibilizing agent.

PP based composites with 5 ÷ 15% natural mineral filler and 20% SBS were prepared in laboratory conditions. All compositions contain 5% compatibilizing agent MA-PP. The formulas of mixtures are shown in Table 1.

The composites were obtained on DSE 20 Brabender co-rotating twin screw extruder, at 220 rpm. The components were prior mixed in a rotating mixer for 15 minutes and the obtained mixture was introduced in the hopper feed of extruder. Extruder temperature profile from hopper to die was 180, 185, 190, 200, 210, and 220°C, respectively. The extruded filaments passed through the water cooling bath and were granulated with a Brabender Pelletizer. Pelletized composites were conditioned in an oven for 4 h at 80 °C and injection molded (Engel 23/40) to obtain standard specimens for mechanical characterization. The injection temperature was set to 220 °C, the temperature of the mold being maintained at 50 °C.

The influence of the filler and SBS content on the mechanical properties (namely elasticity modulus, yield strength, elongation of break and Izod strength impact) and the thermal characteristics evaluated by thermal conductivity analyses and TG/DTG analyses of the composites were studied.

Young's modulus, elongation of break and yield strength were determined according to ISO 527 with 50 mm/min for the tensile strength and 2 mm/min for the modulus of elasticity, using an Instron 3382 Universal Testing Machine and five dog-bone shaped specimens from each sample.

Izod impact strength was determined according to ISO 180 using a Zwick HIT5.5 Pendulum Impact Testers and seven specimens for each test. The testing was conducted on notched specimens at 23^{0}C and -30^{0}C.

Measurements of thermal conductivity were done on samples with dimensions of 150x150x5 mm prepared by compression molding at the working temperature of 180 ± 5 ° C, pressure of 300 kgf / cm^2 and preheating and pressing time of 4-8 min. / 2 min. The thermal conductivity was determined using a hot plate apparatus type EP500 manufactured by Lambda – Messtechnik.

The thermal stability was evaluated by TG/DTA analyses performed with a Shimadzu DTA-TG-50H instrument. The thermal analyses

Table 1

The formulas of the polypropylene composites /*Rețetele de materiale compozite polipropilenice*

Mixture symbol/ *Simbol amestec*	PP/ *Polipropilenă*, % wt	Volcanic tuff (T)/ *Tuf vulcanic* , % wt	Clay (C)/ *Argilă*,% wt	SBS/*Stiren-butadienă-stiren*, %wt
Ref.	100	0	0	0
T	85	15	0	0
C	85	0	15	0
T_5	75	5	0	20
T_10	70	10	0	20
T_15	65	15	0	20
C_5	75	0	5	20
C_10	70	0	10	20
C_15	65	0	15	20

were conducted into an air atmosphere by applying a heating rate of 10°C/min., the measurement range of temperature being of 20–1000 °C.

3.Results and discussions

3.1 Influence of the mineral filler and SBS on elasticity modulus

The relationship between the elasticity modulus of neat PP and the amount of the filler and SBS added into PP matrix is shown in Figure 1.

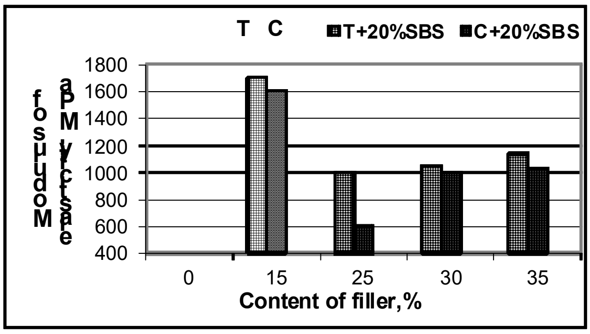


Fig. 1 - Elasticity modulus of the PP based composites depending on type and amount of filler/ *Modulul de elasticitate al compozitelor pe bază de PP în funcţie de tipul şi proporţia umpluturii.*

The mineral filler exerts a positive influence on the elasticity modulus in comparison with the reference sample (only PP); thus, the elasticity modulus increased by 38.5% for the composite filled with 15% tuff and by 31.3% for 15% clay respectively. A positive influence on elasticity modulus was reported in the work [5] in case of PP composites filled with talc, $Mg_3Si_4O_{10}(OH)_2$ - a mineral which belongs to silicate group also. A possible explanation for the favorable effect exerted by both fillers on the Young's modulus of composites based on PP could be their homogenous distribution in PP matrix and a good adhesion between the two phases.

Adding of 20% SBS in PP composites filled with siliceous materials determines decreasing of the elasticity modulus of PP composite with 18.2 - 7.1 % in case of volcanic tuff and with 50.1 - 15.5 % in case of clay, respectively. The maximum value of the elasticity modulus is observed for sample including of 15%tuff +20%SBS, which is quite closed to reference sample (pure PP). The reducing of the elasticity modulus in case of the PP/SBS/talc composites was noticed recent by Liu and colab.[19].

Volcanic tuff exerts a more positive effect on modulus of elasticity than clay both for PP composite filled with mineral filler and PP composite filled with mineral filler and SBS, probably due to its better distribution into matrix and interaction with both polymers.

3.2. Influence of the mineral filler and SBS on yield strength

The results regarding the behavior of the binary PP/mineral filler composites and ternary PP/mineral filler/SBS composites on the yield strength are presented in Table 2.

Table 2

Yield strength of the PP based composites/ *Rezistenţa la tracţiune maximă (la curgere) a compozitelor pe bază de PP*

Mix / Amestec	Yield strength / *Rezistenta la tracţiune maximă (la curgere)*, MPa
Ref.	32.0 ± 0.3
T	33.9 ± 0.8
C	32.6 ±0.2
T_5	25.8±1.4
T_10	24.4±0.3
T_15	23.9 ±0.2
C_5	25.8 ±0.2
C_10	23.7 ±1.4
C_15	25.0 ±0.3

The mineral filler added in proportion of 15% in the PP matrix exerts a slightly positive effect on the yield strength compared to pure PP, the influence of volcanic tuff being more significant than clay.

In the case of the ternary PP/mineral filler/SBS composites have been found a decreasing of the yield strength due to the presence of the SBS elastomer. The reducing of yield strength is quite similarly for both types of aluminous-siliceous filler combined with SBS, but does not exceed 25-26%.

3.3. Influence of the mineral filler and SBS on elongation at break

The elongation at break's variation of the studied organo-mineral composites is shown in Figure 2.

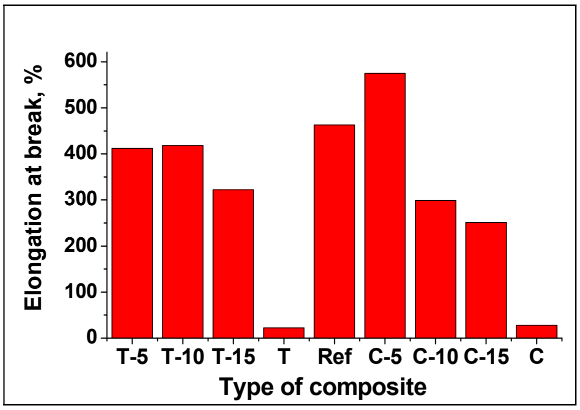


Fig. 2 – Variation of the elongation at break depending on the composite type/ *Variaţia alungirii la rupere în funcţie de tipul compozitului.*

Compared with the neat PP, the presence of 15% mineral filler determined a drastically reducing of the elongation at break (around of twenty times) that confirms the interaction between the two components. Addition of 20%SBS in the PP matrix filled with 15% filler composites leads to diminishing of elongation of break with 30.45% in case of tuff and with 45.79% in case of clay, respectively.

The highest value of elongation at break is obtained for the composition including 5% clay and 20% SBS which exceeds the reference sample (neat PP) with 24.2%, contrary to our expectations.

3.4. Influence of the mineral filler and SBS on impact strength

The siliceous filler added in PP matrix lead to a reducing of notched Izod impact strength determined at 23 ^{0}C, as is shown in Figure 3.

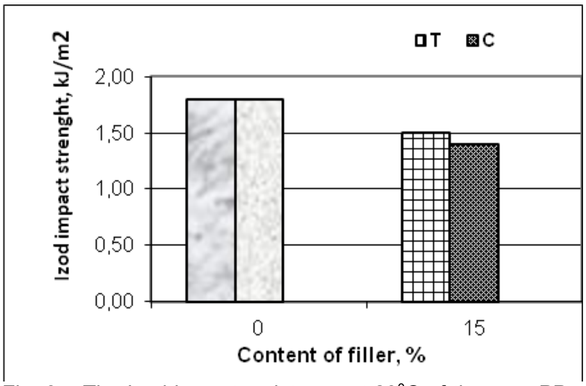


Fig. 3 – The Izod impact rezistence at 23^{0}C of the pure PP and PP filled with 15% mineral filler / *Rezistenţa la şoc Izod la 23ºC a PP şi a matricei polipropilenice care înglobează 15% filer mineral.*

The reducing of impact resistance is slightly lower in the tuff's using case (16.7% compared with 22.2% in case of clay). This behavior suggests a higher compatibility between the tuff and PP and may be a consequence of the better interaction on the interface between the two phases.

In the ternary PP/siliceous filler/SBS blends the impact resistance of composites at 23 ºC is more enhanced compared with the binary PP/siliceous filler blends and pure PP as is revealed in Figure 4.

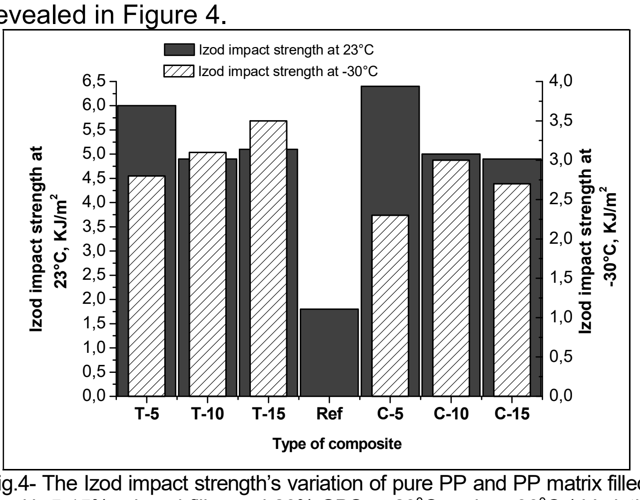


Fig.4- The Izod impact strength's variation of pure PP and PP matrix filled with 5-15% mineral filler and 20% SBS at 23^{0}C and at -30^{0}C / *Variaţia rezistenţei la şoc Izod a PP şi a compozitelor polilpropilenice cu 5-15% filer mineral şi 20%SBS, la 23 ^{0}C şi la -30 ^{0}C.*

The Izod impact strength increases with 233.3 -172.2% for samples containing tuff and 255.6-177.8% for these including clay compared to the neat PP, the maximum values being recorded at the low content of siliceous filler (5%). The adding of 20% SBS into PP composites filled with 15% tuff and 15% clay respectively determined an increasing of the impact resistance with 240% and 250% respectively. A similar trend was reported by Stamhuis [18] for talc filler which can significantly improve the impact resistance of polypropylene if it is physically blended with either an SBS or an EPDM elastomer. Abreu and colab. [21] explain the better impact strength for PP-SBS blends as result of the interactions at the interface between the matrix and the elastomer and the good compatibility with non-crystalline fraction of the polypropylene, which facilitates the diffusion between phases.

At the negative temperature (-30^{0}C), the impact resistance is also improved by the presence of 20%SBS. The values of notched Izod impact strength of PP/mineral filler/SBS blends are still better than of pure PP tested at the room temperature with 55.6-94.4% in samples containing tuff and with 27.8-66.7% in case of blends containing clay. The Izod impact strength at -30 ºC generally increased with the content of the mineral filler in ternary composites, the best resistance being recorded for the composition prepared with 15% tuff.

3.5 Influence of the mineral filler and SBS on thermal conductivity

Thermal conductivity of PP matrix samples (λ_{10}) was calculated based on the thermal resistance of the binary compositions including 15% filler and the ternary composition containing 5÷15% filler and 20%SBS. The relative thermal conductivity of samples is illustrated in Figure 5.

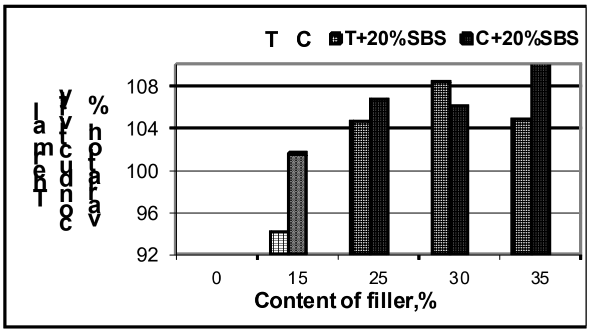


Fig. 5 - Thermal conductivity variation of PP based composites depending on type and amount of filler / *Variaţia conductivităţii termice a compozitelor pe bază de PP în funcţie de tipul şi proporţia adaosurilor.*

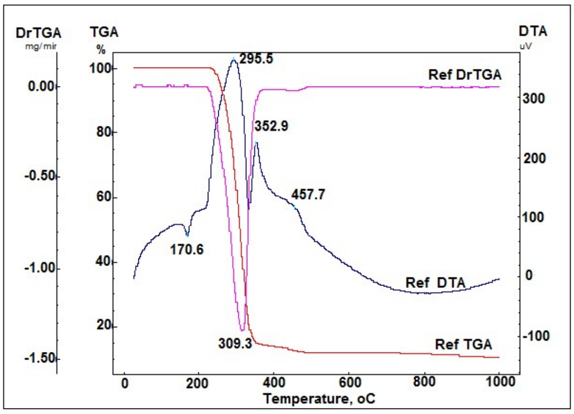
It is evidenced that the thermal conductivity of PP based composites depends on the type and content of the filler. Thus, the clay has a stronger influence on the thermal conductivity; compared to the sample containing only PP (considered as reference; λ=100%), the limits of variation of PP samples filled with clay and 20%SBS ranging between 106.01-110.4% while the PP samples containing 15% clay indicate a value of 101.6%. So, the SBS worsens the thermal insulation capacity of the composites.

The influence of tuff on the thermal conductivity is different. The adding of SBS into the PP matrix filled with tuff has as result an increasing of the thermal conductivity with approximately 4-8% compared with the neat PP. Increase of tuff's amount to 15% determines an improvement of the insulating capacity of the PP-tuff composite, the relative thermal conductivity being of 94.7%. The using of large amount of tuff has beneficial effect on the insulating properties of the composites in comparison with clay. This better behaviour of tuff compared to clay may be explained by its different mineralogical composition and structure which gives it a higher thermal stability as will be seen in section 3.6. Introduction of SBS reduces the insulating properties of PP composites but in a lower extent than of PP-Clay-SBS composites case.

3.6. Influence of mineral filler and SBS on thermal stability

The thermal stability of the ternary 65%PP-15% siliceous filler-20% SBS and binary 85%PP-15% siliceous filler samples were evaluated by thermogravimetric analyses, compared with the pure PP sample. The TG, TDA and DrTGA curves of the samples are presented in Figure 6.

It is noticed on the all TGA derivatogram curves an main endothermic effect attributed to the decomposition of the polypropylene and a small endothermic effect attributed to the SPS's decomposition the latter only in case of the composites containing SBS. According to Klein [22], the phase-transition temperatures of PP



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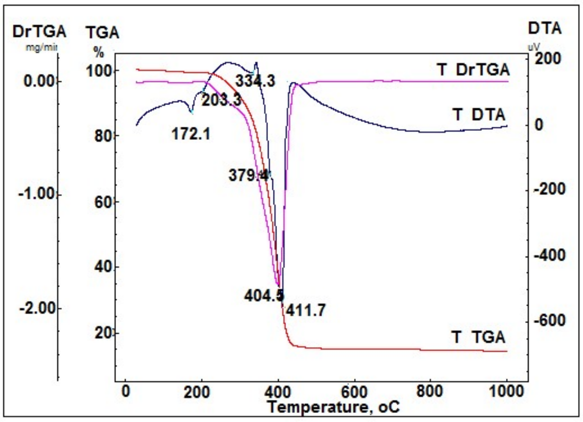

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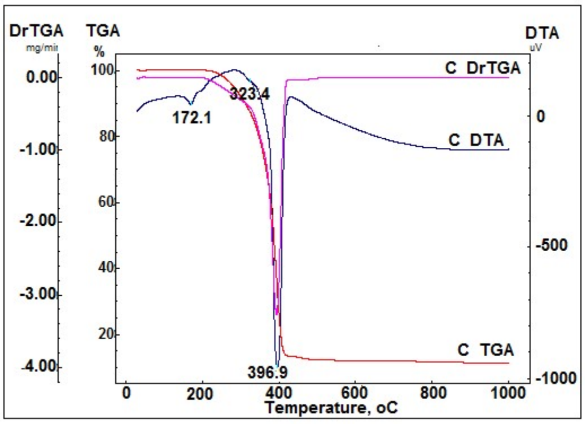

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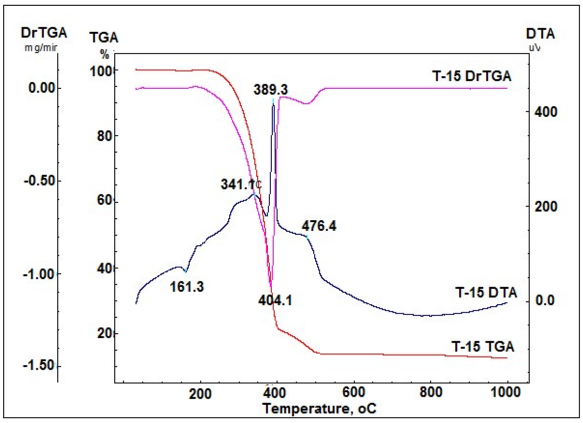

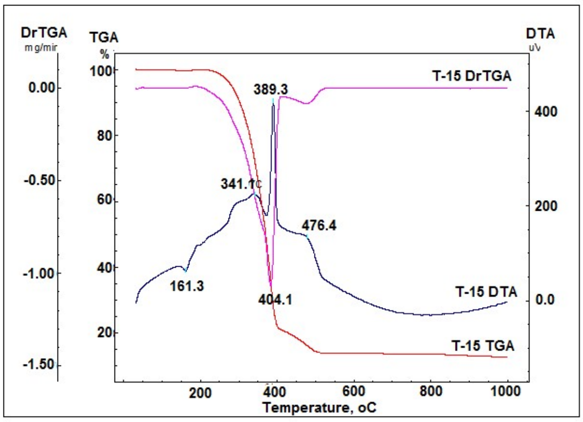

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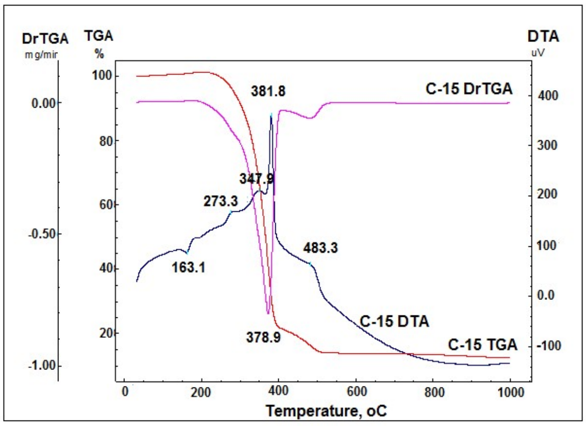

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Fig. 6 – The TGA, TG and Dr TGA curves of the samples/ *Curbele ATD, TG şi DTG ale probelor:* a) PP ; b) PP+15% T, c) PP+15% C, d) PP+15%T+20%SBS; e) PP+15%C+20%SBS.

are ranged between 160-208 °C for crystallite melt temperature (T_m) and between 336-366 °C for decomposition (Td). Thus, it is evidenced the movement of the temperature at which takes place the decomposition of the polypropylene with maximum rate from 309°C (Fig. 6a) to 404°C in case of PP filled with tuff (Fig. 6b) and 397°C for sample including clay, respectively (Fig.6c) and the higher amplitude of the endothermic effect in PP-siliceous filler composites.

Also, by adding of 15% tuff/clay and 20% SBS into the PP matrix, the temperature is shifted to 404°C (Fig.6d) and 379° C (Fig. 6e) respectively, with the modifying of the TG and DTA curves' allure; all these changes prove the improvement of thermal behavior by using of siliceous fillers.

The DTA curves shown that the PP melting temperature corresponding to 170.6°C (Fig.6a) does not practically influenced by the filer addition (172 °C – Fig 6b, c).

The weight loss in the temperature range of 20-400°C which corresponds to decomposition of polypropylene is of 86.01% for pure PP compared with of 78.1% for ternary PP/Clay/SBS composite and 77.87% for ternary PP/Tuff/SBS composite, whereas the weight loss in the temperature range of 400-520°C is only of 1.92% for pure PP compared with of 7.96% for ternary PP/Clay/SBS composite and 8.22% for ternary PP/Tuff/SBS composite. The total weight losses are of 89.36% for pure PP and of 87.62- 87.21% in case of ternary PP-C/T-SBS composites. These results reveal a better thermal stability of PP/C(T)/SBS composites.

4. Conclusions

The following conclusions can be summarized based on the experimental data obtained in this study:

- The elasticity modulus of PP filled with 15% mineral filler composites is increased compared with the neat PP; the volcanic tuff exerts a stronger effect than clay, the growth being of 38.7% compared with 31.3%. Adding of 20%SBS in PP-mineral filler composites leads to a decreasing of Young's modulus, but the sample including of 15%tuff +20%SBS reveals a value quite closed to reference sample (neat PP).
- The yield strain and elongation at break of binary PP- mineral filler composites are better than reference sample (neat PP).
- The impact resistance of ternary PP/siliceous filler/SBS composites is improved compared with the pure PP, whereas the PP matrix filled with 15% mineral filler is relatively close to neat PP sample. A better behavior of composite including tuff was observed compared to clay suggesting a higher compatibility between the tuff and the PP and SBS.
- The influence of filler on the thermal conductivity is quite different. Adding of 5-15% wt. tuff in the PP matrix +20%SBS has as result a slight increasing of the thermal conductivity with approximately 4-8% while the adding of 15% tuff in PP matrix determines a diminish of thermal conductivity, therefore an improvement in insulating capacity of composite. The thermal stability is improved with adding of mineral filer, also.
- It can be modeled the properties of composite materials towards increasing of the elasticity modulus, impact resistance and insulation capacity of the composite materials based on PP matrix by the proper selections of the amount and type of filler and SBS. The use of volcanic tuff in amount of 15% leads to the best thermal and mechanical properties of the PP based composite.

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