Identification, Sorting and Characterization of Plastics from WEEE

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The plastics components of a random sample of waste electrical and electronic equipment (WEEE) from small household appliances were identified and sorted. Moreover, by taking into account the practical needs for applications demanding medium to less severe restrictions from the standpoint of end-use characteristics, the mechanical and thermal properties are investigated for the four most used plastics in the EU, including acrylonitrile-butadiene-styrene (ABS), styrene-acrylonitrile (SAN), polystyrene (PS) and polypropylene (PP), as well as for a mixture resulting on the basis of corresponding EEE market evaluations. The obtained results show that in the case of these materials, separated prior to processing, even if a minor decrease arises from the standpoint of thermal properties due to inherent degradation induced by processing, the problem is the decline of different mechanical properties. In fact, this appears to be the net result of different morphologies of components – the essentially amorphous-like nature of ABS, SAN and PS, and the semi-crystalline component (PP). Accordingly, the loss in mechanical properties was lower for the elastic modulus, higher in case of impact strength and intermediate for the tensile strength, when the components and the resulting blend properties are compared.

Keywords: WEEE, plastics, recycling, thermal properties, mechanical properties

The importance of WEEE recycling has become more evident over the last decade. Consumption of electrical and electronic equipment (EEE) in Europe has risen to 13,6 million tons in 2000, being estimated that about 6 million tons of waste from EEE is produced annually [1]. Moreover, the amount of post-consumer plastics from WEEE available for plastics recovery (above 1.130.000 t by 2005 [2]) is on the rise due to the EC legislation implementation, and the recognition that there exists viable business opportunities.

Plastics recycling from End-of-Life EEE is technologically possible, and is essential to reach the targets set in the WEEE directive, although plastics recovery from post-consumer EEE is complex, including energy and time consuming.

Although it has been found that there are many plastic resins within electric and electronic equipment plastic streams, there are even more grades of plastics within the same resin family, that may inhibit or worse, to affect recovery yields. Moreover, there can be many grades of plastics with different mechanical and/or melt flow properties, and furthermore, containing plastic grades from various suppliers manufactured over multiple years, with dissimilar types and levels of additives, and that can significantly influence the mechanical properties of the recycled plastic.

Mechanical recycling of plastics, mainly through remelting, is a part of the waste management approach, along with feedstock recycling and energy recovery [3]. As a matter of fact, the mechanical recycling (MR) is a good economical and environmental option, being used for the 21 % of the post-consumer plastic wastes in Europe in the year 2008 [4]. Thus, MR of plastics refers to processes involving the grinding, washing, separating, drying, re-granulating and compounding by melting of waste plastics [5]. This last process implies the blending of immiscible polymers e. g., styrenics and olefinics, the compatibility of these blends being improved by using different compatibilizers.

The first aim of this work consists in the identification and sorting of a random sample of plastics from WEEE of small household appliances using simple techniques as well as their assessment of thermal damage. Then, based on a majority share of the first four types of plastics in the total plastics used in the European Union WEEE, the thermo-mechanical characterization both of each relevant polymer material and of the obtained mixture according to the respective share is desired, as a starting point for improving their properties by the addition of the compatibilizers in a later stage.

Experimental part

Materials

The following plastic materials were supplied by Gremlin Computer Ltd., one of the largest EEE recyclers in Romania, and Stena DTM Ltd for use in this study:

- unrecycled plastics from casings of mixed waste of telephones and small household appliances (e.g., steam irons, food processors, blenders, juice extractors), as such, or chopped into pieces approximately 120 –240 x 80 – 120 mm and 80 - 100 x 60 – 80 mm;
- unrecycled ABS and PS from plastic casings of monitors, and back or front cover of television sets.

Identification process

After preconditioning (i.e., washing with a 0.5 % detergent solution followed by drying) and conditioning (i.e., sorting by type of plastic, for marked items, and by component parts of the same type, for unmarked items, respectively as well as large parts cut in smaller pieces). The unrecycled plastics from casings of various electrical

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equipment were grounded in a two cascading mills system. Then, a 1 kg quantity of the resulting materials was selected for densities based separation (sorting) by sink-float process.

Initially the material mixture (test sample) placed in a flask containing distilled water, was stirred 15 min and then left to stand another 15 min for separation of heavy fraction (i.e., > 1 g/cm^3) from the light one (i.e., < 1 g/cm^3). The process was continued in same way except that a calcium chloride solution of various concentrations was used instead of distilled water in case of the heavy fraction, and an alcohol solution in case of the light fraction.

A Bruker Vertex 70V Fourier transform infrared (FTIR) spectrometer, which used the attenuated total reflection method in medium IR spectral range, was considered to identify the plastic types from the unmarked items as well as the sediment samples taken from the solution having 1.19 g/cc density. The spectrograms from each test were identified by comparing the main peaks from the spectrum of the unknown sample with those from a reference spectra.

To prove the correctness of the assumptions made about the polymers separated by densities two common and fast processes were used : the burning & odour and solvent solubilization tests.

Mechanical recycling process

The grounded materials from the cascading mills system were particles approximately $5 \div 7$ mm^2 nominal surface. These granules were used for reprocessing.

The blends were weighted according to the required proportions and hand mixed. This was followed by thermal processing in an intensive batch mixer Brabender Plastograph, at 180°C, for 10 min and 100 rpm. This part of the process was used to blend four materials - ABS, SAN, PS and PP, process resulting in a matrix. Afterwards the matrix was converted into sheets with a thickness of 0.5 mm by pressing at 15 MPa and 180±5°C.

The blend of the four thermoplastics was obtained by using 31 wt % ABS, 16 wt % SAN, 27 wt % PS, and 26 wt % PP, on the basis of four most used plastics in EEE in Western Europe 2000 [2, 6].

Characterization

Mechanical testing

A Testing Machine (Model FPZ 100) was used to measure the standard tensile properties of the materials at room temperature (T = 23°C). The used crosshead speeds were 5 and 50 mm/min respectively, all measurements being performed for five replicates of dog-bone shaped specimens and averaged to get the final result. The measurements of tensile strength (σ_b) and elongation (ε_b) followed the SR EN ISO 527-2: 1996 protocols at the testing speeds resulting for the relative rate of grips (i.e., 5 and 50 mm/min.); the measurements of density (ρ) and notched impact strength (A_k) followed the SR EN ISO 1133:2003 and SR EN ISO 180:2001 protocols, respectively.

The dynamic-mechanical behavior was studied in the ultrasonic domain by using the UNIPAN-543 Material Tester, at the frequency of 1 MHz. The apparent dynamic elasticity modulus, EM_L, was determined in accordance to ASTM E 494 norm, from the value of longitudinal propagation speed of ultrasonic waves, v_L, and the density (in accordance of SR ISO 1183:1994), using the relationship: $EM_L = \rho v_L^2$. The test specimens were cut from roll-milled and pressed sheets with a punch press.

Thermal testing

The glass transition temperature (Tg) was determined by DSC and the weight losses of analyzed samples were determined by thermal gravimetric analysis (TGA).

The DSC measurements were conducted under a helium atmosphere. The heating rate was fixed at 5°C/min.

The thermal stability of the composites was evaluated on a Netzsch STA 449C operating at room temperature, in air at a rate of 5°C/min.

The dynamic mechanical analysis (DMA) was performed in atmosphere at a heating rate of 3°C/min by recording the temperature dependence of storage modulus (E'), and loss modulus (E''), respectively by using the TA – Q800 system in dual – cantilever mode at 1 Hz frequency in the temperature range from 30 to 150°C.

Results and discussions

Identification analysis

The percent amounts of plastics identified from the 1 kg test sample are shown in figure 1.

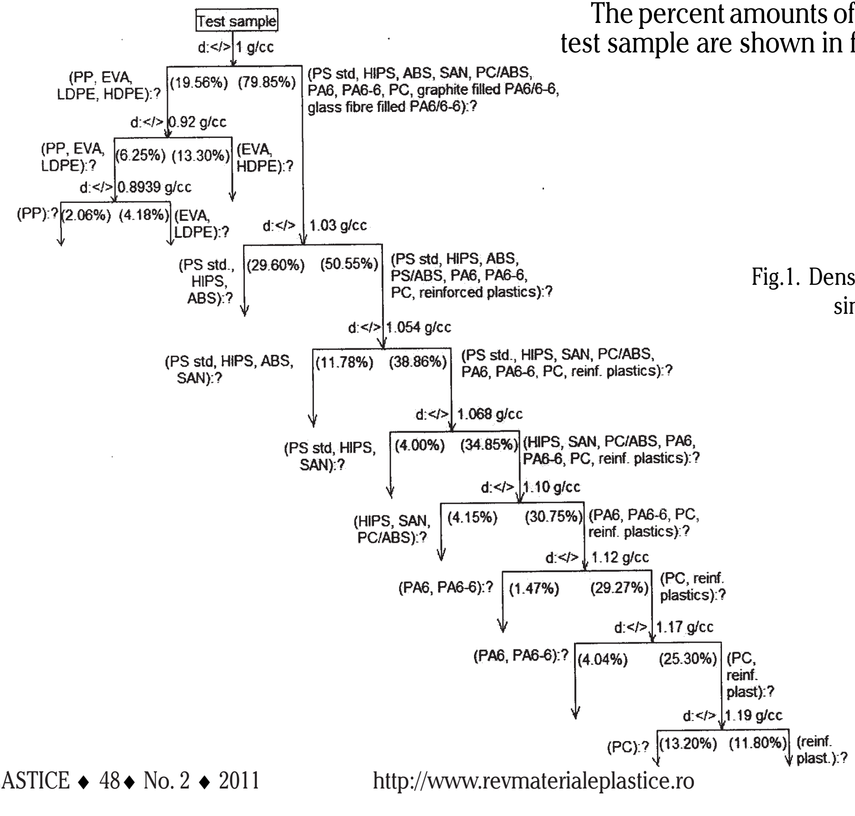


Fig.1. Densities based separation by sink-float process

It can be seen that HIPS and ABS were the two most widely used plastics for casings of steam irons food processors, blenders, telephones and juice extractors comprising about 42 wt % of the casings, followed by PC (13 wt %). Furthermore, moderate proportions of HDPE, LDPE made up about 13 wt %, whereas only about 2 ÷ 4 wt % of casings were made with plastics such as EVA, SAN, or blend such as PC/ABS.

Although in principle, FTIR is an accurate identification method (fig. 2 a, b, c), it would not be suitable as part of a commercial recycling process due to the slow speed of identification.

The results of burning and odour are presented in table 1 being in good agreement with observations of Whelan [7].

The random sample of densities based separation has shown that, in principle, in the tested wastes PS, HIPS and ABS are found mostly, followed by PC and reinforced

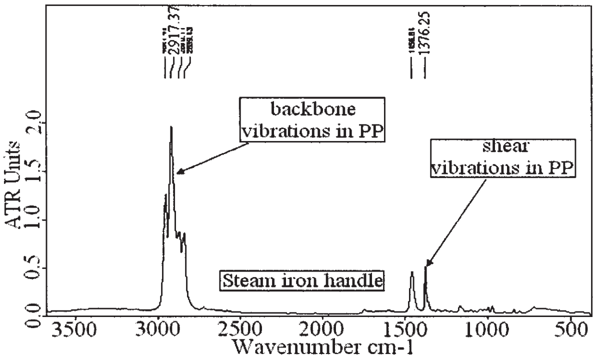


Fig. 2.a. FT-IR spectra of the steam iron handle (PP)

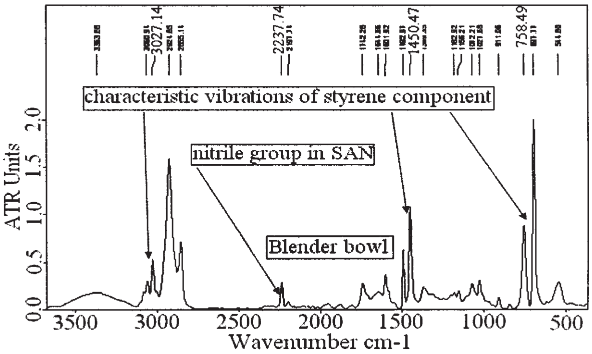


Fig. 2.b. FT-IR spectra of the blender bowl (SAN)

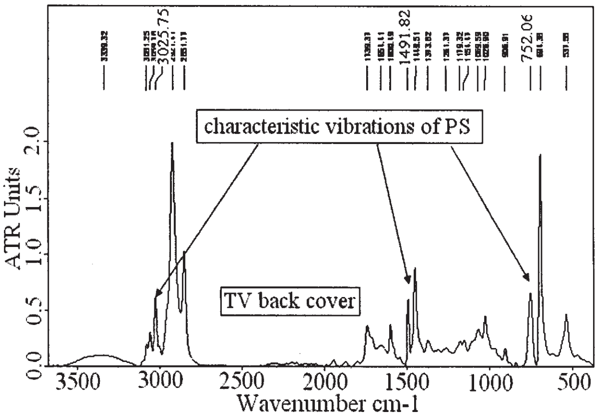


Fig. 2.c. FT-IR spectra of the back cover TV (PS)

Table 1
RESULTS OF BURNING & ODOUR AND SOLVENT SOLUBILIZATION TESTS

Mixed polymers (a-mixed polymers from various WEEE) (b-mixed polymers from steam irons)	Burning & odour test	Solvent		
		Toluene	Carbon tetra-chloride	Formic acid 98 %
(a) 1.00 - 1.03 g/cc density fraction	PS : black smoke with soot, dense, aromatic smell;	PS		
	ABS : black smoke with soot, dropping material, smell of coal gas, yellow flame		ABS	-
(a) 1.03 - 1.054 g/cc density fraction	PS : black smoke with soot, dense, aromatic smell;	PS		
	ABS : black smoke with soot, dropping material, smell of coal gas, yellow flame		ABS	-
(a) 1.054 - 1.068 g/cc density fraction	PS : black smoke with soot, dense, aromatic smell;	PS		
	ABS : black smoke with soot, dropping material, smell of coal gas, yellow flame		ABS	-
(a) 1.068 - 1.10 g/cc density fraction	PS : black smoke with soot, dense, aromatic smell;	PS	-	-
(a) 1.12 - 1.17 g/cc density fraction	PC : gray smoke, material turns black and dripping, phenol smell, yellow flame	-	-	-
(b) 1.17 – 1.19 g/cc density fraction	PA : Blue with yellow top flame, burnt hair smell	-	-	PA

Crt. no.	Polymer	MFI (g/10 min) ASTM D1238	σ_b (MPa) ASTM D638	ε_b (%) ASTM D638	A_k Izod (J/m) ASTM D256	σ_f (MPa) ASTM D790
1	PP [8] - isotactic	-	31 - 41	100 - 600	20 - 53	-
	- atactic	-	21.4	300	763	-
2	PS [8] - standard	-	27 - 69	1.6 – 3.0	19 - 24	90
	- HIPS	-	35 - 100	36 - 50	133	35 - 39
3	ABS [8]	-	41 - 62	20 - 100	105 - 440	28 - 97
4	SAN [9]	7	42 – 72 62 – 82 [10]	1.6 – 3.2	19 – 27 [10] 16 – 32 [11]	96 – 117 [10]

Table 2
PHYSICO-MECHANICAL PROPERTIES OF REFERENCE POLYMERS

Crt. no.	Sample	Supplier	MFI (g/10 min.)	σ_b (MPa)	ε_b (%)	A_k (kJ/m^2)	σ_f (MPa)
1	PP	Stena	11.70^1	31.3*	5.8*	18.20^a	-
2	PS	Stena	4.03^2	23.30**	25.20**	20.84^a	-
3	ABS extruded	Gremlin	-	39.65**	30**	14.05^a	66.65
4	SAN	Stena	3.30^2	66.70**	6**	10.46^b	-
5	PC	Gremlin	-	52.03*	20*	-	86.64

Note : * - testing speed = 50 mm/min; ** - testing speed = 5 mm/min;

a - 2 J hammer ; b - 4 J hammer; 1 -at 230°C/2.16 kg; 2 - at 200°C/5 kg.

Table 3
PHYSICO-MECHANICAL PROPERTIES OF POLYMERS FROM WEEE

Sample	ρ (g/cm^3)	MFIs (g/10 min)	MFI (g/10 min)	σ_b (MPa)	ε_b (%)	EM_L (GPa)	A_k (kJ/m^2)
ABS	1.0428	31.51^1	4.25^2	34.60	21.60	6.74	9.49
SAN	1.0659	30.23^1	3.30^2	66.70	6.00	8.38	10.46
PS	1.0267	3.62^2	3.62^2	23.30	25.20	6.11	20.84
PP	0.9787	18.73^3	38.48^2	31.30	5.8	5.46	18.20
M	1.0243		24.62^2	15.44	5.5	5.915	2.41

Note: 1 – at 220°C/10 kg; 2 – at 200°C/5 kg; 3 – at 230°C/2.16 kg.

Table 4
PHYSICO-MECHANICAL CHARACTERISTICS OF THE RELEVANT POLYMERS AND THEIR MIXTURE.

plastics, then polyolefines, SAN and PA. Also, the accuracy of the method is quite low unless is not accompanied by more appropriate methods of analysis such as FTIR in real time or spectroscopic-like methods.

Mechanical testing

For evaluation purposes of the available properties remaining in case of various polymers sorted from wastes, comparisons were performed concerning the physico-mechanical properties of both considered polymers from the reference (table 2) and WEEE (table 3), respectively. The results showed that, in comparison with the reference polymers, the tensile strength of the polymers sorted/identified from wastes was approx. 30% lower in case of ABS and PP, 50% lower in case of PS, and only 6% lower in case of SAN; the impact strength is also approximately 45% lower, whereas for the flexural modulus comparable values were obtained. The reciprocal values of different physico-mechanical characteristics for relevant polymers are presented in table 4. This summarizes the density, the melt flow index, the tensile strength and elongation at break, the apparent dynamic elasticity modulus and the notched impact strength of the polymers and their mixture.

The effect of mixing of some amorphous polymers (e.g., ABS, SAN, PS) with a semi-crystalline one (PP) resulted in an incompatible blend. Besides, in the recycled blend M there are present multi-contamination effects due to the contribution of ABS, SAN, PS and PP components.

Concerning the presence of PP, it can be seen an increase of MFI, as result of the presence of additives as well as to the possible contribution of the recycled PP (table 3).

Moreover, the MFI of recycled PP was significantly higher than those for ABS, SAN and PS, respectively, a high MFI implying low resistance to flow and in fact a low interaction between polymer chains (table 4).

On the other hand, the density of the blended materials decreased in proportion to the amount of PP, being equal with the weighted average of the materials, ρ_M = 1.0243 g/cm^3 (measured); $\rho_{weighted}$ = 1.0255 g/cm^3 (calculated).

Furthermore, the reduction in mechanical properties due to the incompatibility between 74 % amorphous polymers and 26 % semi-crystalline polymer was less pronounced for the tensile strength and the apparent dynamic elasticity modulus at ultrasonic frequency, but more pronounced for the elongation at break and the impact strength.

As it is well known, in an ABS/PP blend a phase separation appears [12], the phase segregation occurring in the molding phase causing a weakening of the internal structure [13]. It is suggested that the less ductile material, e.g., ABS, forms a skin around the PP particle [14], while the PP rich core has various sized ABS droplets dispersed throughout; this core forms a large part of the fracture surface, the absence of ABS reducing the impact strength from 10-20 to 2.5 kJ/m^2.

Consequently, both the deformation and strength properties are controlled in various manners by the characteristics of components and the resulting morphology.

Thermal testing

Exploratory thermal testing of some sample recovered from components of small household appliances

The samples of SAN (fig. 3) show a thermal behavior similar to that of SAN corresponding to the first processing. The degradation of all samples arises in the range 310 - 500°C, the loss at 200° C being about 0.1 to 0.5%, while the weight loss in the range 200 - 300°C is about 0.6 to 1%.

All samples degrade in one main step in the range 190-240°C, but eliminates 0.2 -0.5% volatile, probably small molecular compounds remaining from the synthesis process. In this temperature range there are not present fragments characteristic for the main chain.

Black polystyrene sample recovered from the back covers TV (fig. 4) suffers more severe degradation, with a total weight loss of about 2.6% at 300°C. This may be due to previous processing cycles. The sample is still usable by reprocessing, with the loss of about 0.5% at 200°C, and 260-500° C the degradation range.

PP sample (fig. 5) preserve the properties of primary polypropylene, with a total weight loss at 300°C of about 0.2%. Degradation range is 350-550°C, more than the usual processing domain.

The materials recovered from household waste appliances have a similar thermal degradation behaviour like polymers on first use. Exceptions are materials that are already after one or more cycles of recycling, as back covers TV.

From the results obtained by TGA-DSC it can be seen that analyzed polymers are stable at their processing temperature (> 200° C).

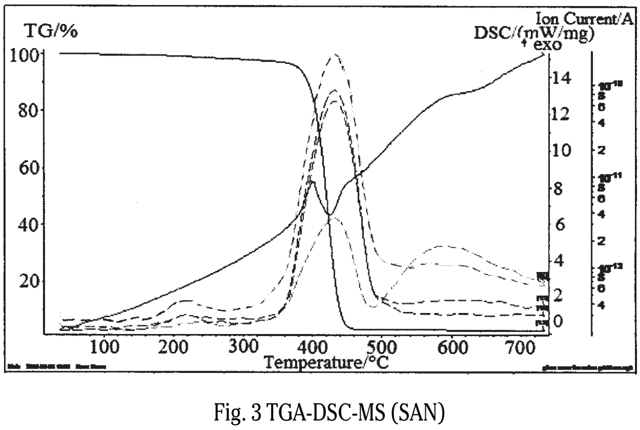


Fig. 3 TGA-DSC-MS (SAN)

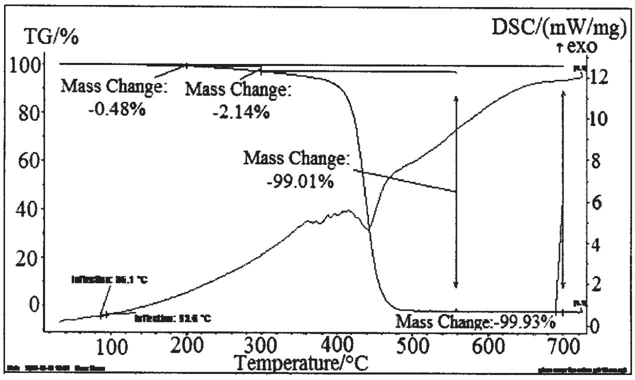


Fig. 4.TGA-DSC TV back cover (PS)

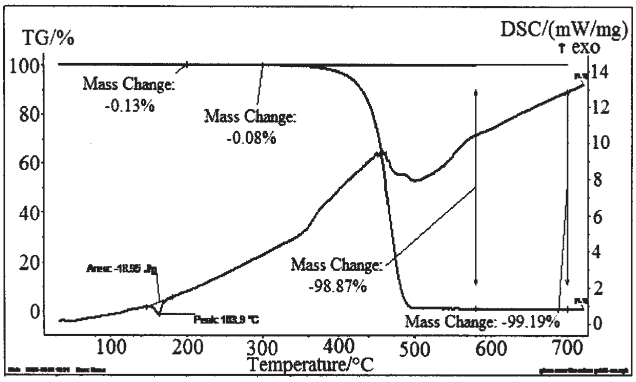


Fig. 5.TGA-DSC iron handle (PP)

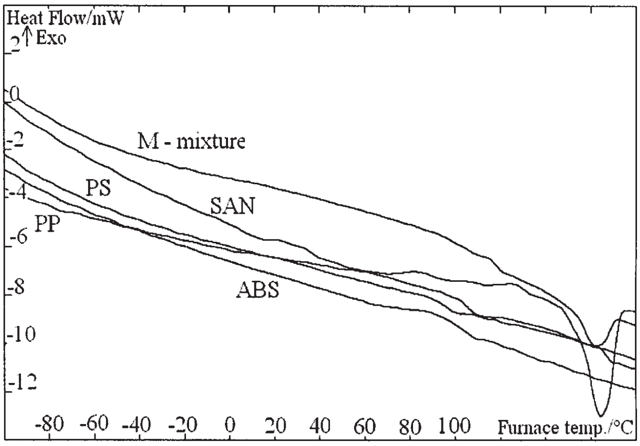


Fig. 6. Heat flow curves taken of the relevant polymers and their mixture

Thermal testing of the relevant polymers and their mixture

DSC data

The heat flow curves taken on polymers and their mixture (fig. 6), as well as the values of characteristic temperatures obtained by DSC measurements (table 5) performed in the atmosphere in the range from –100 to 200°C with a heating rate of 5°C/min are presented.

Taking into account the distinct features of component constitution, well definite situations are present. In the case of PS one obtains Tg = 97.03°C, as well as a second one, at Ts = 167.78°C. On the other hand, for PP arise Tg = -1.35°C and Ts = 89.21°C. Moreover, concerning the ABS, there are four characteristic values, including the PS (105.39°C) and PB – i.e., the rubber-phase glass transition (-63.06°C) contributions, as well as the combination effects at -50.29°C and 126.91°C (an increase in Tg is expected for degraded samples [15]). Furthermore, regarding the SAN, the contribution of PS appears at 106.34°C while a second

Table 5
GLASS TRANSITION TEMPERATURES OF THE RELEVANT POLYMERS AND THEIR MIXTURE

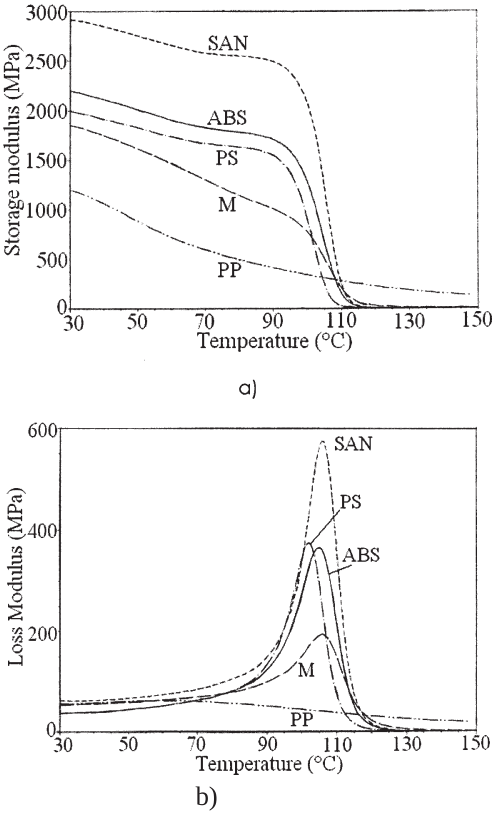
Sample {Tg (°C), [#]}	Tg (°C)
ABS {110, [16,17]}	-63.06; -50.29; 105.39; 126.91
SAN {108±1, [18]}	36.63; 106.34
PS {107±2, [19]}	97.03; 167.78
PP {-10, [20]}	-1.35; 89.21
M	-93.96; -49.61; -24.16; 85.37; 117.76

Note: {Tg (°C), [#]} represents the value of Tg indicated in different references.

distinct characteristic temperature value is present at 36.63°C.

Finally, in case of M mixture there are present a lot of individual and additional contributions in the range from -93.96°C to 117.76°C, including also those at -49.61°C and 85.37°C as well as at 117.76°C (related to the melting of crystalline phase of PP).

The DMA data (fig. 7) obtained on the basis of temperature dependence of matrix components including ABS, SAN, PS and PP, as well as the resulting mixture, point out distinct variation trends. Thus, in the case of storage modulus there is a decrease with the increasing of temperature, the relative positions of experimental curves being in the descending sequence SAN, ABS, PS and PP for the range of temperatures from 25°C to 105°C; on the other hand, opposite effects of PP and polystyrene result in a reciprocal compensation of E' values in case of M mixture.



a)

b)

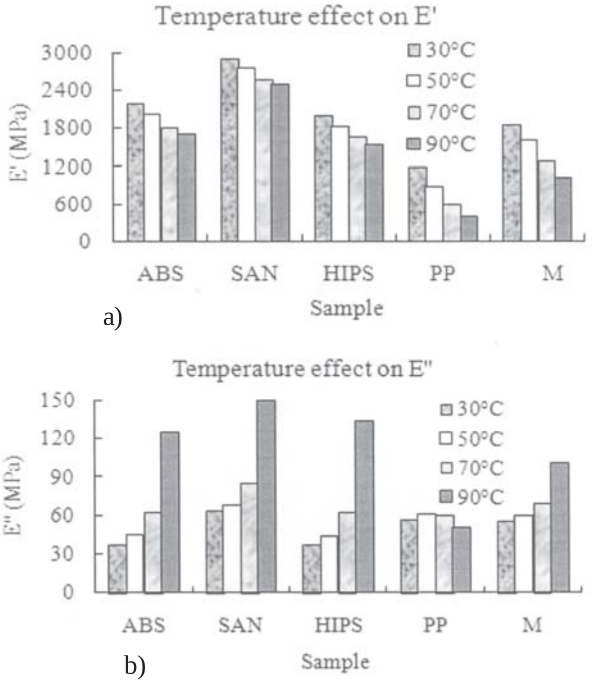
Fig. 7. The storage (a) and loss modulus (b) dependences on temperature for the relevant polymers and their mixture

Moreover, concerning the values of different characteristic temperatures of dynamic mechanical relaxations, as revealed by different criteria-including the “on set” value of storage modulus as well as those of maximum ones for the loss modulus and the loss factor, respectively – well defined deformational instabilities may arise. The corresponding relaxation mechanisms control the mechanical behavior of mixture especially in the range from 90 to 110°C.

Moreover, from the stand point of the loss modulus the lowest value of relaxation transition temperature arises for PP, and in an increasing sequence for PS, ABS, SAN and M, the corresponding values of characteristic dynamic mechanical relaxations being given in table 6.

Table 6
CHARACTERISTIC DYNAMIC MECHANICAL RELAXATION TEMPERATURES OF THE RELEVANT POLYMERS EVALUATED BY USING DIFFERENT CRITERIA

Sample	$T_{E''max}$ (°C)	$T_{Tan\delta}$ (°C)	$T_{E'onset}$ (°C)
ABS	104.87	111.71	98.01
SAN	106.00	115.67	100.27
PS	102.05	115.17	95.89
PP	59.53	-	65
M	105.88	114.98	112.03



a)

b)

Fig. 8 The temperature effect upon the E' (a) and E''(b) for the relevant polymers and their mixture

In order to evidence the comparative effect of temperature upon the dynamic mechanical characteristics, the corresponding trends are presented in figure 8 for storage modulus (a) and loss modulus (b), respectively. The levels of values obtained for the mixture compared to those of components point out, on the other hand the decline of the slope of E' with the temperature increase, as well as, on the other hand, the decrease of the step-like variation in the temperature range where high values are obtained.

Conclusions

In order to establish the representative components resulting in case of different end-users it is necessary to perform the identification and sorting processes.

The composition including ABS, SAN, PS and PP was used to obtain the corresponding mixture taking into account the values typical to EEE polymers application – wt% (ABS – 31, SAN – 16, PS – 27, and PP – 26). The mechanical and thermal characterizations point out that for practical needs in case of medium to less severe restrictions the resulting mixture shows acceptable characteristics.

The (minor) decreases of thermo-mechanical characteristics due to inherent processing degradation as well as to the effect of different morphology of components suggests the need of an appropriate compatibilizer able to ensure both high level end-users as well as the sustainable recycling of different wastes.

Summarizing, the thermal and mechanical properties of the individual plastics were almost retained after first thermal recycling. Mixtures of polymers can be produced economically, without sophisticated sorting, to produce low value products. Incomplete separation of the materials investigated will ultimately lead to plastics with inferior properties that may not be useful in applications that the material was originally used for.

Modifiers need to be used to improve the interaction between polymers within an incompatible blend, thereby improving the materials property.

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