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Article

The Influence of Compatibilizer Addition and Gamma Irradiation on Mechanical and Rheological Properties of a Recycled WEEE Plastics Blend

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Abstract: Waste electrical and electronic equipment (WEEE) is growing rapidly, and the plastics within WEEE have an important role in fulfilling the recovery and recycling targets defined in the European WEEE Directive. This study considers recycling of WEEE plastics by making a blend of the different plastics instead of separating them. The mechanical and thermal properties can be enhanced by adding a compatibilizer. It was found that one compatibilizer, a styrene-*b*(ethylene-co-butylene)-*b*-styrene (SEBS) copolymer named Kraton[®] G1652 E, had a large impact on the ductility of the recycled WEEE plastics blend. By adding 2.5 weight % (wt%) of this copolymer, the elongation at break increased by more than five times compared with the non-compatibilized samples, with only a small decrease in stiffness and strength. The storage modulus (G') decreased slightly with increasing compatibilizer amounts while the impact strength increased with increasing amounts of compatibilizer, from 2.1 kJ/m² (reference material) to 3.6 kJ/m² (5 weight % (wt%) compatibilizer). It was found that Kraton[®] FG1901 E (styrene-*b*(ethylene-co-butylene)-*b*-styrene (SEBS) grafted with maleic anhydride (MAH)), Royaltuf[®] 372P20 (styrene acrylonitrile (SAN) modified with ethylene-propylene-diene elastomers (EPDM)) and Fusabond[®] P353 (polypropylene (PP) with a high degree of grafted MAH) were ineffective as compatibilizers to the blend. Gamma irradiation (50 kGy) did not improve the mechanical properties however: the impact strength of the gamma-irradiated samples was lower than that of the non-irradiated samples.

Keywords: WEEE; recycling; compatibilizer; SEBS; plastics blend

1. Introduction

Electronic waste is one of the fastest growing waste streams today, making it important to find new recycling strategies for the different materials included, for environmental, legal and economic reasons. The reported annual amount of globally-generated WEEE in 2014 was 42 million tons [1,2], and considering that the plastic fraction of WEEE was 10 wt%–30 wt% [3–5], that is 4.2 to 12.6 million tons to reuse or recycle. The WEEE plastics contain up to 15 different types which makes it difficult and costly to separate the plastics from each other, which is how plastics material recycling mainly is done today. Plastic fillers and overlapping plastics densities makes the separation even more troublesome. While near infrared spectroscopy (NIR) might be able to distinguish between polymers it is often hindered by the pigments present in “black plastics” [6], while the electrostatic separation methods will be impeded by the thin layer of moisture which exists on all surfaces when the humidity is high [7].

Additionally, washing and de-dusting of the plastics are common. Previous composition studies on WEEE plastics made by the authors have revealed that it consists mainly of high impact polystyrene (HIPS, 42 wt%), acrylonitrile-butadiene-styrene copolymer (ABS, 38 wt%), and polypropylene (PP, 10 wt%) [8], but there are also other plastics, such as polyethylene (PE), polyurethane (PUR), and polycarbonate (PC), depending on the waste stream, reported by others [8–12]. The conventional way of recycling plastics is by means of different sorting and separation steps followed by melt-blending of the different types [9]. Such a recycling method may leave large rest fractions that are excluded from recycling. In this study, another approach is taken. We have investigated the use of a compatibilizer to enhance the mechanical properties of a blend material of all of the different thermoplastics within WEEE, with the exclusion of thermosets and other contaminants that could impact the processing equipment or recycled blend negatively [13]. This process would simplify recycling by reducing many of the sorting, separation and washing steps. A composition control of the blended material may though appear. The influence of gamma irradiation on the plastic properties was also investigated since it is known that it can, firstly, cause free radicals and with that either create crosslinks or chain breakage of the polymer chains [14], and, secondly, it can be used to graft one polymer onto another [15].

2. Experimental Section

2.1. Materials

The material used was a melt-blended and melt-filtered WEEE plastics blend of recycled material (WEEEER) determined by combustion analysis to be C, 83.46; H, 8.77; N, 2.20; O, 1.27; Cl, 0.13; Br, <0.10% from post-consumer flame-retardant free waste. This collected post-consumer waste was obtained from Stena Technoworld in Halmstad (5 July 2011) and has previously been analyzed with respect to its composition [8]. The material should be compliant with the European Directive on the Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment (RoHS) [16]. Before the reprocessing in our laboratory, the material had undergone dust and surface cleaning, melt blending, melt-filtration, and hot die granulation at Next Generation Recycling Maschinen in Feldkirchen, Austria. The recycling equipment used for melt-blending, filtration, and granulation was an S:GRAN 85 in conjunction with an Ettlinger rotation drum melt-filter and hot die granulator. The extruder screw rotation rate was 145 rpm with a throughput of 280 kg/h, and it filtered out about 1 wt% of mainly non-thermoplastic contamination in order to form the WEEEER material. The pre-made compatibilizers studied are shown in Table 1. To make the cost of the blended material comparable to virgin material, low amounts of the compatibilizers were added.

Table 1. The four different compatibilizers (wt%) blended with WEEEER for tensile testing.

Compatibilizers Used	Description	Amounts of Compatibilizer
Kraton [®] G1652 E	SEBS (30 wt% styrene)	0.83, 1.25, 2.5, 5, 10, 20 wt%
Kraton [®] FG1901 E	SEBS-g-MAH	2.5, 5, 10 wt%
Royaltuf [®] 372P20	SAN modified with EPDM	5, 10, 20 wt%
Fusabond [®] P353	PP-g-MAH	0.83, 2.5, 5, 10 wt%

Kraton Performance Polymers is the producer of Kraton[®] G1652 E and FG1901 E, both based on a linear triblock copolymer of styrene-b(ethylene-co-butylene)-b-styrene (SEBS), with a styrene content of 30 wt%. In addition, FG1901 E was grafted with 1.4 wt%–2 wt% maleic anhydride (MAH). Addivant provided Royaltuf[®] 372P20, which is styrene acrylonitrile (SAN) modified with ethylene-propylene-diene elastomers (EPDM). Fusabond[®] P353 is based on polypropylene (PP) with a high degree of grafted MAH and produced by DuPont[™].

2.2. Processing Equipment

The WEEEBR was blended with the different compatibilizers in a melt process with a co-rotating twin screw extruder, Werner and Pfleiderer ZSK 30 M9/2 (1984), with five heating zones along the barrel and one heating zone for the die. The screw length was 966 mm with a barrel bore diameter of 31 mm. The screw configuration was optimized for mixing and compounding. The temperature profile was set to 160-190-190-200-200-190 °C to avoid over-heating and resin degradation and the screw rotation rate was 60 rpm [17]. The extruded material was oriented and flattened with a 3 + 2 roll puller, Brabender 843316003, to a thickness of 0.4–0.9 mm. Test specimens, in the shape of dog bones, were produced by die punching to a shape according to ISO 527-5A. For the impact test, new batches with compatibilized materials were prepared in another co-rotating twin screw extruder, Coperion ZSK 26K, 10.6, manufactured by Coperion, Germany. The residence time in the extruder was 50 s, the screw speed 180 rpm, and the throughput 6 kg·h⁻¹. The blends are presented in Table 2. It is noteworthy that none of the properties of the material produced using the second machine were inconsistent with the properties of the product made using the first machine.

Table 2. WEEEBR blends studied for impact testing with the compatibilizer Kraton® G1652 E (0–10 wt%).

Material	Amount of Kraton® G1652 E (wt %)
WEEEBR reference	0
WEEEBR	2.5
WEEEBR	5
WEEEBR	10
WEEEBR (gamma-irradiated granulate)	0
WEEEBR (gamma-irradiated granulate)	2.5
WEEEBR (gamma-irradiated granulate)	5
WEEEBR (gamma-irradiated dog bones)	0

The gamma irradiation used in the impact test was performed in a Gammacell 220 (Atomic Energy of Canada, now trading as Norion) with a dose rate of 8 kGy·h⁻¹ on average, and the dose rate was measured with the ferrous-cupric sulphate dosimeter test (4 February 2014). The temperature within the chamber was approximately 50 °C. The WEEEBR granulate and dog bones were irradiated with a dose of 50 kGy.

2.3. Material Characterization

The mechanical properties of modulus of elasticity, yield strength and elongation at break were measured with a Zwick/Z 2.5 tensile tester equipped with pneumatic grips and a 500 N load cell. The measurements were performed at 22 ± 2 °C and 40 % ± 10 % relative humidity. Seven specimens were evaluated for each type of sample and the average values were calculated with ± one standard deviation.

The Charpy impact properties were evaluated according to ISO 179/1eA by an Edgewise single notch test and compared with un-notched samples. The samples were notched with an Instron CEAST AN50, 0.1 mm each time to a final notch depth of 2 mm. The impact test equipment was an Instron CEAST 9050, with an impact energy of 0.5, 1.0, and 4.0 J. The impact specimens were prepared and evaluated at Swerea IVF. The results reported were based on ten test specimens evaluated for each material and given as average values and ± one standard deviation.

The thermal storage modulus and loss tangent were determined by Dynamic Mechanical Thermal Analysis (DMTA) using Rheometrics Solids Analyzer RSA II together with TA Orchestrator RSA2 software. Two curves were produced for each material. Unless they matched, a third measurement was made. The first of the matching curves was used. The melt flow rate was determined according to

ISO 1133 and measured on a Modular Melt Flow 7024, produced by Ceast, Italy. One measurement was performed per material.

3. Results and Discussion

Previous tests performed on WEEEBR have shown low elongations at break (ϵ_b) and low impact strengths [13], indicating poor compatibility between the phases, suggesting it is a very brittle material. To increase the ductility, compatibilization with compatibilizers was considered.

3.1. Tensile Properties

As expected, it can be seen in Figure 1 that the most effective compatibilizer was a styrene-ethylene/butadiene-styrene triblock copolymer (SEBS G1652 E). This substance caused a significant increase in the ductility of WEEEBR. Even at as low as 2.5 wt% compatibilizer, the ϵ_b -values increased over five times. This is considerably higher than has previously been reported for PS/PP blends compatibilized by SEBS (up to 25 wt%) [18]. At least two explanations exist for this improving effect of the SEBS. Firstly, the SEBS is a block copolymer with sections that are similar and, thus, compatible, to the two types of polymers (polystyrene and polypropylene) in the blend. It could act as a surface-active material, reducing the surface tension between the two liquid phases during processing [19]. This reduction in interfacial tension should result in a reduction of the domain size can be expected to improve the properties of the mixture [20]. An alternative explanation offered by La Mantia is that the SEBS is not a true compatibilizer, instead it reduces the brittleness of at least one of the phases instead of accumulating at the interfaces [21].

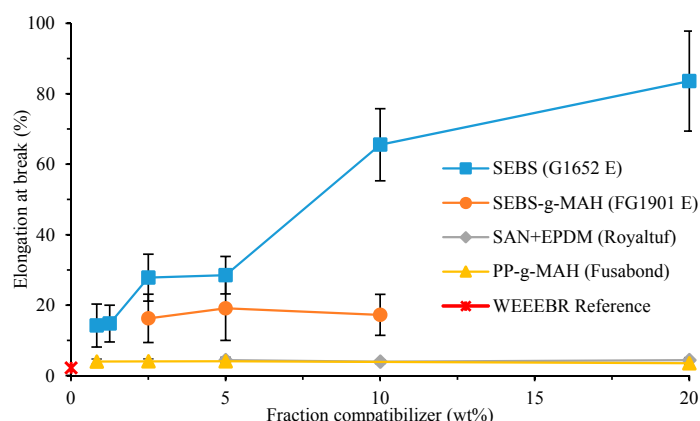


Figure 1. Elongation at break measured for WEEEBR with the compatibilizers used and non-compatibilized WEEEBR (as reference). The error bars represent the standard deviation.

However, while SEBS grafted with maleic anhydride (MAH) (FG1901 E) increased the ϵ_b -value, this increase was smaller than for the unmodified SEBS. Unlike the unmodified SEBS, increasing the quantities of the MAH-grafted SEBS did not cause further improvement in the plastic.

While it could be thought that a MAH-grafted PP should be a good compatibilizer for the WEEEBR blend, it turned out to be ineffective. It has previously been reported by Parameswaranpillai *et al.* that a PP-g-MAH compatibilizer for a PP/PS was effective for stabilizing the morphology and improving the impact strength. However, the other mechanical properties such as tensile strength, flexural strength, and elongation at break gave irregular results compared with the reference blend [22]. A study by Yilu *et al.* reported a novel approach to graft PP with dual monomers (PP-g-(MAH-co-St)), overcoming the inability of MAH to homopolymerize, which causes little MAH to be grafted onto PP and severe degradation of the PP backbone [23,24]. The sluggish reaction of MAH with the electron-poor radicals, formed by the reaction of MAH with a radical, can be overcome by adding styrene (St) as a second monomer onto PP. This not only increases the grafting proportion of MAH onto

PP (gMAH) but also reduces the PP chain scission degradation [25]. The styrene works as a medium to bridge the gap between the PP macroradicals and the MAH. The co-polymer St can first graft onto the PP backbone and then react with the MAH [26].

Focusing on the best SEBS-containing compatibilizer, different amounts (1.25 wt%–20 wt%) were blended with the WEEEBR, and the transition from a brittle (non-compatibilized) to a ductile material is shown in Figure 2. The strain at break increased significantly for all added amounts, but from an engineering point of view an addition of about 5 wt% compatibilizer, giving an increase in strain at break of about 25 %, can be considered enough to replace virgin plastics used in electronic equipment with WEEEBR.

As seen in Figures 3 and 4 both the stiffness (σ_b) and the yield stress (σ_y) decreased with increasing amounts of compatibilizer. The stiffness was lower compared with the reference sample for all the compatibilized samples and appeared to have a linear dependency on the compatibilizer content. On the other hand, low compatibilizer levels (<2.5 wt%) resulted in a slight decrease in yield stress but had only a very small influence on the σ_y -values between 2.5 wt% and 20 wt%.

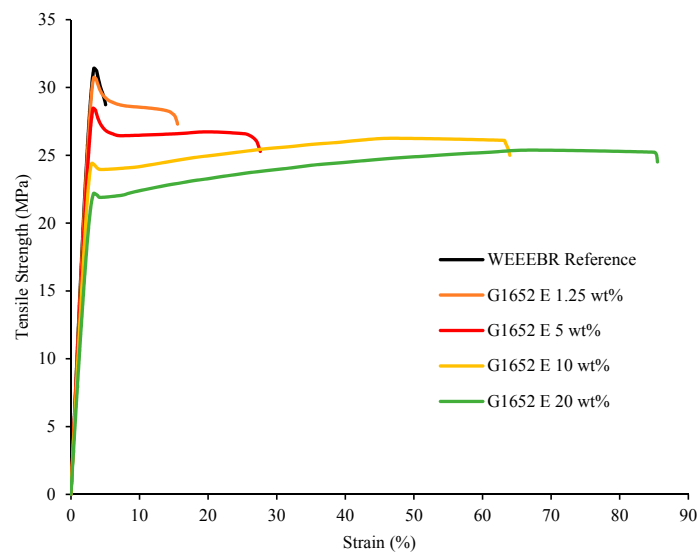


Figure 2. Tensile strength *versus* strain for non-compatibilized WEEEBR (reference) and compatibilized WEEEBR samples studied.

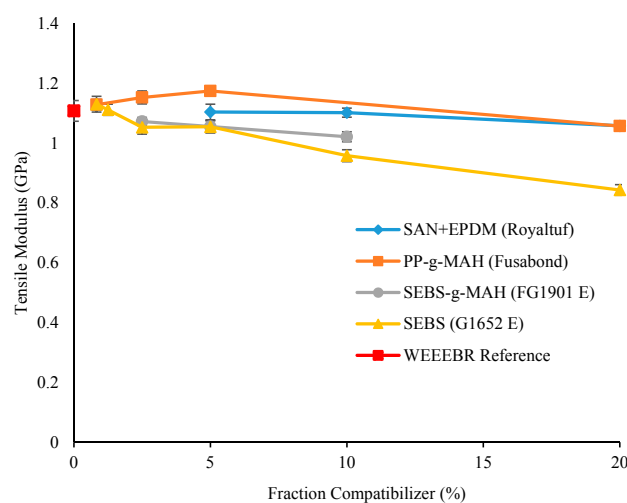


Figure 3. Tensile modulus of WEEEBR with the compatibilizers used and non-compatibilized WEEEBR. The error bars represent the standard deviation.

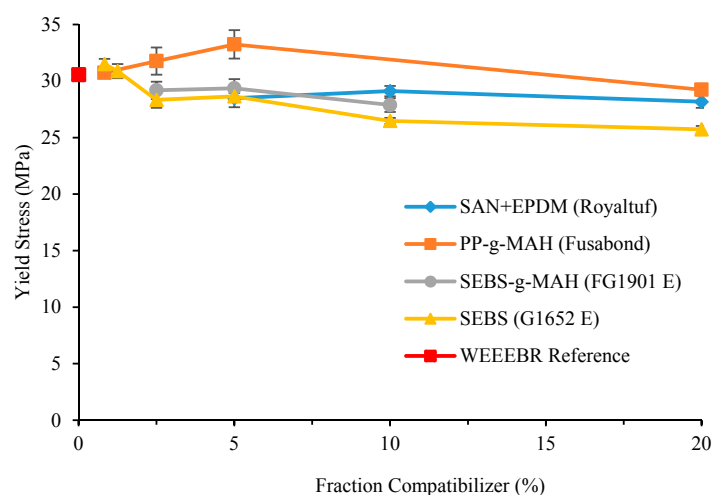


Figure 4. Yield stress of WEEEER with the compatibilizers used and non-compatibilized WEEEER. The error bars represent the standard deviation.

It is interesting to note that while an increase in toughness is normally associated with a decrease in stiffness and strength [18,27], in our case the addition of the SEBS only caused a moderate decrease in these important mechanical properties.

3.2. DMTA

The DMTA results of the storage modulus (G') and loss tangent ($\tan \delta$) studied in compression mode for WEEEER mixed with different amounts of the compatibilizer Kraton[®] G1652 E can be seen in Figures 5 and 6. The storage moduli were around 1 GPa (room temperature, 22 °C), in compliance with previous results by the authors [13], and fairly stable up to 85 °C. As expected from Figure 3, the storage modulus at temperatures below 85 °C was somewhat lower with increasing amounts of compatibilizer. It is interesting to note, however, that the compatibilizer does not seem to have any negative influence on the thermal stability, since no real difference in the softening (>100 °C) of the materials could be seen between the reference and the compatibilized samples.

The T_g can be found as the $\tan \delta$ peak of WEEEER and the compatibilizer in Figure 5 at about 110 °C.

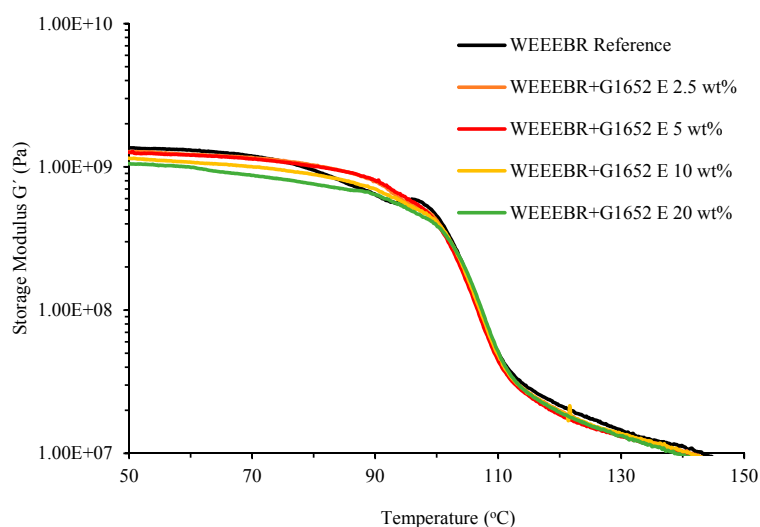


Figure 5. Storage modulus as a function of temperature for WEEEER with different amounts of compatibilizer.

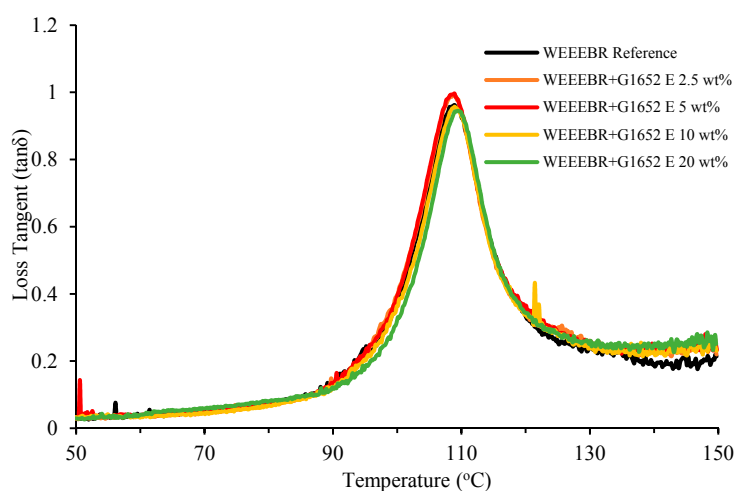


Figure 6. The loss tangent as a function of temperature for WEEEER reference and WEEEER with different amounts of compatibilizer.

3.3. Impact Strength

The impact strength increased with increasing amounts of compatibilizer for both notched and un-notched samples; see Table 3. For the notched samples, the impact strength increased by 28.6% for the 2.5 wt% compatibilized sample, 81.0% for the 5 wt% compatibilized sample, and an astonishing 138.1% for the 10 wt% compatibilized sample. The standard deviation is based on ten samples. Un-notched samples gave a higher standard deviation than notched samples, as could be expected.

Table 3. Impact testing of WEEEER blended with the compatibilizer Kraton® G1652 E (0–10 wt%) and gamma-irradiated samples (50 kGy).

Material	Amount of Kraton® G1652 E (wt%)	Notched Samples		Un-Notched Samples	
		kJ/m ²	Standard Deviation	kJ/m ²	Standard Deviation
WEEEER reference	0	2.1	0.3	7.9	1.0
WEEEER	2.5	2.7	0.1	13.2	1.5
WEEEER	5	3.8	0.3	16.8	2.0
WEEEER	10	5.0	0.1	24.5	4.1
WEEEER (50 kGy)	0	1.3	0.1	6.7	0.9
WEEEER (50 kGy)	2.5	2.3	0.1	10.7	1.1
WEEEER (50 kGy)	5	2.6	0.0	12.5	1.9
WEEEER (50 kGy) *	0	2.1	0.5	8.0	1.1

* Gamma-irradiated dog bones.

The impact strength of gamma-irradiated samples decreased while the compatibilizer showed similar behaviour to that within the non-irradiated samples.

No change was seen in the mechanical properties of gamma-irradiated samples (same value as WEEEER reference). This effect could be due to the fact that hydroperoxides require thermal activation to form hydroxyl and alkoxy radicals [28]. The gamma irradiation of polyvinyl chloride (PVC) results in the formation of hydroperoxides, which later form these radicals [28]. The hydroxyl radicals will abstract hydrogens at random sites, and the alkoxy radicals can undergo a beta-scission reaction forming a carbonyl group and a new but smaller macromolecular radical. This thermal activation of the hydroperoxides leading to chain scission has been specifically reported for polypropylene [29]. Thus, it is the granules that need to be gamma-irradiated, and then mechanically and thermally processed for a reaction resulting in a change of mechanical properties to occur.

When molding samples of WEEEER were prepared for the impact test, it was noted that the injection molding pressure increased with increasing amounts of compatibilizer and also that the

increase was smaller for the gamma-irradiated samples; see Table 4. These observations indicated a viscosity decrease in the gamma-irradiated blends. A decrease in the mechanical strength of one component in a composite material can cause the mechanical properties of the material taken as a whole to decrease, as polypropylene is known to be more sensitive to radiation than polystyrene [30] and to undergo mainly chain scission, rather than crosslinking [31]. We reason that the loss of strength in the polypropylene domains can explain the effects of irradiation followed by thermal processing of the WEEEBR.

Table 4. Injection molding pressures used for molding the WEEEBR blends with the compatibilizer Kraton® G1652 E (0–10 wt%).

Material	Amount of Kraton® G1652 E (%)	Injection Molding Pressure (bar)	Increase in Injection Molding Pressure (%)
WEEEBR reference	0	435	-
WEEEBR	2.5	508	17
WEEEBR	5	544	25
WEEEBR	10	544	25
WEEEBR (50 kGy)	0	435	0
WEEEBR (50 kGy)	2.5	471	8
WEEEBR (50 kGy)	5	508	17

3.4. Melt Flow Rate

The MFR decreased with increasing amounts of compatibilizer for the WEEEBR; see Table 5. Thus, the material became more viscous, indicating better compatibility between the phases, which can be explained by better phase adhesion [32] and possibly co-continuous structures [33]. It is notable in Table 5 that the major decrease in MFR is seen between 0 and 2.5 wt% SEBS and further increases of the compatibilizer amount only resulted in a slight decrease of MFR. This indicated that 2.5 wt% SEBS, or less, was enough to significantly change the flow characteristics of the polymer blend and should have a positive effect on the phase adhesion in the WEEEBR material. Gamma irradiation was instead found to increase the MFR, which can be explained by a degradation of the polymer chains subjected to gamma irradiation expectedly resulting in chain scission and decreased molecular weight. The increase in MFR with gamma irradiation was seen for all the different compatibilization amounts compared with the non-irradiated samples; hence, the viscosity decreased and the material flowed more easily.

Table 5. Melt Flow Rate of WEEEBR blended with the compatibilizer Kraton® G1652 E (0–10 wt%) and gamma-irradiated samples (50 kGy).

Material	Amount of Kraton® G1652 E (wt%)	Melt Flow Rate (g/10min) @220 °C, 5 kg	
		Non-Irradiated	50 kGy Gamma-Irradiated
WEEEBR reference	0	29	45
WEEEBR	2.5	21.7	38.2
WEEEBR	5	21.1	34
WEEEBR	10	19.6	-

- Means not measured.

4. Conclusions

The addition of SEBS greatly increased the ductility of WEEEBR, even at low levels (2.5 wt%). On the other hand, maleic anhydride-grafted polypropylene failed to improve the properties of the mixture of plastics. The addition of SEBS to the mixture caused only a small decrease in the storage modulus, while a considerable increase in the impact strength was seen, from 2.1 kJ/m² to 3.8 kJ/m², with 5 wt% compatibilizer. At temperatures below 85 °C, the storage modulus was reduced as the

amount of compatibilizer increased. It was interesting to note, however, that the compatibilizer did not seem to influence the glass temperature of the plastics blend, since no significant difference in the softening of the materials was seen between the reference and the compatibilized samples. The MFR decreased with increasing amounts of compatibilizer for the WEEEBR and, thus, the material became more viscous, indicating better compatibility between the phases, which can be explained by better phase adhesion and possibly co-continuous structures. As the addition of SEBS only caused a moderate increase in the viscosity of the molten plastic it will not prevent the use of either extrusion or injection molding as means of forming objects from the recycled plastic. It was found that gamma irradiation (50 kGy) before thermal processing expectedly caused a moderate reduction in the mechanical strength of the plastic due to chemical degradation of the plastic.

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Conflict of Interest: The authors declare no conflict of interest.

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