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Toward recycling unsortable post-consumer WEEE stream: Characterization and impact of electron beam irradiation on mechanical properties

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22 **Abstract**

23 The work reported here is the first study aimed at providing a full screening of a real
24 unsortable non-recycled post-consumer WEEE stream free of brominated flame retarded
25 plastics, separated using on-line X-ray detection, toward its recycling. In the existing sorting
26 lines, up to 40% of plastics from waste electrical and electronic equipment (WEEE) stream
27 can be rejected, herein named unsortable plastics, To have the most representative
28 homogeneous sample for physico-chemical characterizations, a sampling method was
29 developed to overcome the heterogeneity of the investigated 500 kg batch. The batch
30 screening on both representative samples (~500 μm size) and 100 plastic fractions (~20 mm
31 size), by means of routine techniques used in the plastic industry, has allowed to quantify
32 reliably the main polymers included in the studied batch; ~50 % styrene-based polymers, ~15
33 % polypropylene (PP), ~15 % polycarbonate (PC), ~1-4 % polyamide (PA), polyethylene
34 (PE), polyvinyl chloride (PVC), poly(ethylene terephthalate) (PET), poly(methyl
35 methacrylate) (PMMA) and ~8 % of multi-layer plastics, paints and thermosets. The
36 identification of the ~8.0% inorganic phase by X-ray fluorescence spectrometry revealed the
37 presence of several additives/charges commonly incorporated in plastic materials, such as
38 calcium carbonate and talc. The studied batch was then subjected to electron beam irradiation
39 at 50 and 200 kGy doses, as a means of compatibilization between the batch components. The
40 mechanical properties and thermal behavior of irradiated samples pointed out the crucial role
41 of the residual free radical scavenger agents present in post-consumer WEEE streams, leading
42 to significantly different properties compared to those of irradiated virgin polymer blends
43 highlighted in the literature.

44 **Keywords:**

45 Unsortable plastics, Post-consumer WEEE, Electron beam irradiation, Sampling method,
46 Plastics recycling, Mechanical properties

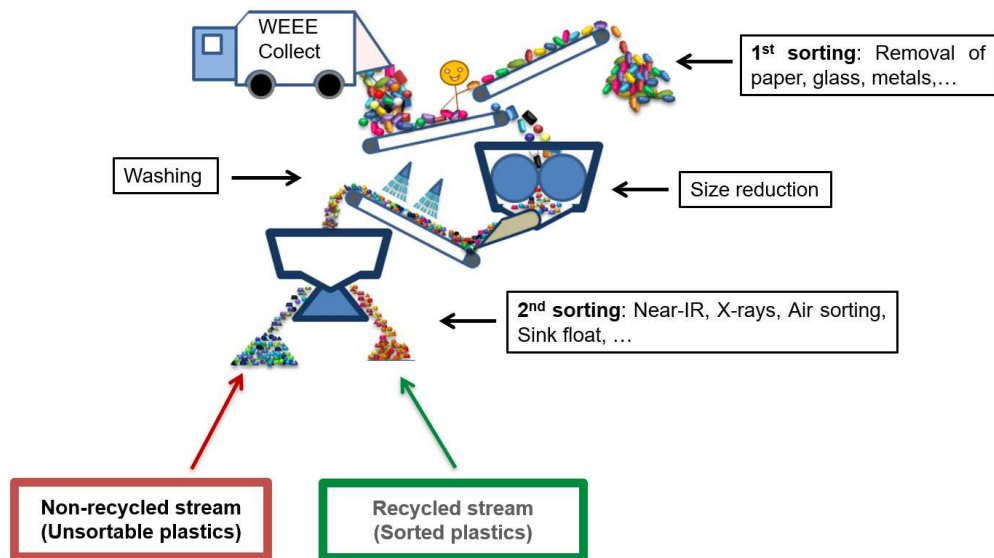
47

48 **1. Introduction**

49 Waste electrical and electronic equipment (WEEE) is the fastest growing source of waste,
50 potentially rising 4% per year (Sahajwalla and Gaikwad, 2018). This increasing waste stream
51 has led to the publication of directive 2012/19/EU by the European Commission, targeting a
52 WEEE recycling rate between 55% and 80% (2012/19/EU, 2012). WEEE is a complex stream
53 consisting of a mixture of metals (Kyere et al., 2018), ceramics, glass and plastics. The
54 proportion of the latter, depending on the six WEEE categories defined in directive
55 2012/19/EC (2012/19/EU, 2012), is ranging between 10 wt.% and 33 wt.% (Gramatyka et al.,
56 2007; Kang and Schoenung, 2005; Parajuly and Wenzel, 2017; Vazquez and Barbosa, 2016;
57 Wang and Xu, 2014; Widmer et al., 2005). Therefore, to achieve the objectives set out in
58 directive 2012/19/EC, WEEE plastic recycling is imperative. Furthermore, plastic waste
59 management has become an ecological issue given the need to limit the impact of plastics on
60 our environment (Dolores et al., 2020; Hamaide et al., 2014; Ismail and Hanafiah, 2019;
61 Milad et al., 2020); From an economical point of view, studies on the plastic composition in
62 WEEE reported in the literature highlighted the presence of different types of polymers, the
63 amount of which varies with the source (Alwaeli, 2011; Bovea et al., 2016; Chancerel and
64 Rotter, 2009; Dimitrakakis et al., 2009; Maris et al., 2015; Martinho et al., 2012; Stenvall et
65 al., 2013; Taurino et al., 2010). Considering the economic effectiveness of the waste utilized
66 as substitutes for primary materials, as discussed by Alwaeli (2011), only the most
67 representative polymers of the stream, that can be technically sorted and valorized according
68 to the regulatory, are separated individually for further mechanical recycling operations. The
69 plastic composition of WEEE issued from small electrical and electronic equipment (EEE)
70 recycling units in Europe consists mainly of acrylonitrile-butadiene-styrene copolymer
71 (ABS), polypropylene (PP), polystyrene (PS), and high-impact polystyrene (HIPS)

72 (Dimitrakakis *et al.*, 2009; Maris *et al.*, 2015; Martinho *et al.*, 2012). Up to now, the
73 complexity and financial cost of plastic sorting makes difficult to consider the separation of
74 the plastics from each other to facilitate recycling of a single type of polymer.

75 In the past decade, near infrared spectroscopy (NIR) has been integrated in the recycling
76 process of WEEE (Menad, 2016), and allows to separate automatically on line the ABS, PP
77 and PS from waste white plastics with a high accuracy up to 99% (Li *et al.*, 2019). This
78 technique can potentially detect other plastics. However, it is not always relevant to the
79 amount of each plastic and the economic issues related to sorting schemes. As a result, about
80 40% of WEEE plastics are rejected from the sorting line, herein named unsortable (Fig. 1),
81 because i) they do not comply with sorted resins, ii) they contain dark pigments, named black
82 plastics (Maris *et al.*, 2015), which are not recognized by the classical NIR equipment or iii)
83 because they contain brominated flame retardants (BFR), estimated to be ~25% of WEEE
84 plastics (Hennebert, 2017). Using on-line X-ray detection, BFR-containing plastics are
85 separated as specified in the directive 2019/1021/EU (2019/1021/EU, 2019). Indeed, the
86 recovery of brominated flame retarded plastics has been restricted to treatments that destroy
87 or irreversibly transform the substances, as stated in 2012/19/EC directive and Stockholm
88 convention (2012/19/EU, 2012; Stockholm Convention, 2017).



89
90 **Fig. 1.** General sorting steps for post-consumer WEEE plastic waste
91

92 Based on this observation, Tostar *et al.* (2016) have studied recycling of a polymer blend
93 based on 80 wt.% styrene-based polymers and 10 wt.% PP from WEEE, by adding a
94 compatibilizer or by gamma irradiation. The irradiation of polymeric materials with ionizing
95 radiation appears to be a promising tool to chemically modify polymers as it leads to the
96 formation of free radicals along the backbone even on low reactive polymers, such as
97 polyolefins (Burillo *et al.*, 2002; Chmielewski *et al.*, 2005). The compatibilization is ensured
98 by (i) direct crosslinking between macroradicals of two polymers to form new covalent bonds
99 (Gu *et al.*, 2014; Li *et al.*, 2009) or (ii) indirect crosslinking via a coupling agent binding two
100 polymers (Lambla and Seadan, 1993, 1992; Xanthos and Dagli, 1991). The resulting
101 copolymers act as a compatibilizing agent which concurrently lower the interfacial tension at
102 phase boundaries and enhance their adhesion, leading to an improvement of the mechanical
103 properties (Maris *et al.*, 2018; Utracki, 2002). For example, the mechanical properties of a
104 blend of seven virgin polymers frequently found in plastic solid waste: low-density
105 polyethylene (LDPE), high-density polyethylene (HDPE), poly(vinyl chloride) (PVC), PS,
106 HIPS, PP, and poly(ethylene terephthalate) (PET) were improved through the addition of

107 peroxides (Vivier and Xanthos, 1994). Similarly, Said *et al.* (2013) have reported an
108 enhancement of the tensile properties of different PET/LDPE mixture compositions subjected
109 to gamma irradiation at 25 and 50 kGy doses. Blends of waste polyethylene/LDPE 70/30,
110 exposed to electron beam (EB) irradiation at doses up to 300 kGy, showed higher ductility,
111 toughness, and resistance to oxidative degradation. These irradiated blend materials were
112 successfully blow molded to make bottles (Satapathy *et al.*, 2006).

113 Apart from Taurino *et al.* (2010) investigations on characterization of two sorted black/grey
114 plastic waste categories (i.e. personal computers and televisions), the work reported in this
115 article is the first study aimed to analyze and provide a full screening of a real unsortable non-
116 recycled post-consumer WEEE stream.

117 In this contribution, for the first time the composition of unsortable post-consumer WEEE
118 plastics stream free of BFR, sorted using on-line X-ray technique as specified in the directive
119 2019/1021/EU, was investigated. The studied 500 Kg batch was collected in France from the
120 following streams: cooling appliances, household electrical equipment, and information
121 technology (IT), such as computers, printers and phones. The polymer and additives/charges
122 composition of unsortable plastics has been determined using Fourier-transform infrared
123 (FTIR) spectroscopy, X-ray fluorescence spectrometry, differential scanning calorimetry
124 (DSC), and thermogravimetric analysis (TGA).

125 Burillo *et al.*, 2002; Fel *et al.*, 2016; Lambla and Seadan, 1993; Numata and Fujii, 1995;
126 Said *et al.*, 2013; Satapathy *et al.*, 2006; Tostar *et al.*, 2016; Vivier and Xanthos, 1994 have
127 reported the impact of irradiation process on polymer blend. In contrast to these cited studies
128 where polymer blend recycling was simulated on the basis of virgin polymers or unsorted
129 plastics, the present work provides a comprehensive investigation on EB-irradiated unsortable
130 post-consumer WEEE batch prior to melt-process. EB irradiation has the advantage of being a

131 clean and continuous process that is already commercially well-established (Drobny, 2010).
132 Thus, the impact of EB irradiation doses (50 and 200 kGy) on thermal behavior and
133 mechanical properties of the studied batch was investigated.

134 **2. Experimental Section**

135 **2.1. Materials**

136 A 500 kg big bag of unsortable post-consumer WEEE, collected in April 2015, was
137 supplied by Veolia (France). Liquid nitrogen used to grind the sample was provided by Air
138 Liquide (France).

139 **2.2. Sampling procedure**

140 The sampling procedure was carried out on a 500 kg batch with heterogenous fraction sizes
141 (<70 mm) from unsortable post-consumer WEEE stream. In order to have the most
142 representative homogeneous sample for physico-chemical characterizations, a sampling
143 method is necessary to overcome the heterogeneous constitution and size distribution of the
144 tested batch. For this study, we developed a sampling method (see Fig. 2) based on Gy (1998)
145 work and the XP CEN/TS 17188 standard (Afnor, 2018), and adapted from our research team
146 previous work (Epsztein *et al.*, 2014).

147 **2.3. Melt Processing and Characterization Methods**

148 **2.3.1. Twin-screw extrusion**

149 Extrusion experiments were carried out using a co-rotating twin-screw extruder Coperion
150 ZSK18 (L/D = 40). The barrel temperature along the interpenetrate screws (feeding to die)
151 was set at 190, 190, 200, 200, 200, 200, 200, 210, 210 and 210 °C with a screw speed of 250
152 rpm. The obtained extrudates were pelletized after cooling.

153 **2.3.2. Injection molding**

154 Dog-bone specimens 1A ISO 527 type and impact bars (80 x 10 x 4 mm³) were injection
155 molded using a Krauss Maffei EX80-380 injection molding extruder. The detailed processing
156 parameters are summarized in Table S1.

157 **2.3.3. Fourier-transform infrared spectroscopy (FTIR)**

158 Infrared spectra were collected in the wavenumber range 400–4000 cm⁻¹ using a Fourier
159 transform spectrometer Nicolet 380 DTGS in transmission mode for micro-ground samples,
160 and ATR (attenuated total reflection) mode for bulk samples. Spectra were recorded at a
161 resolution of 2 cm⁻¹ and 128 scans. The pellet samples for the transmission mode were
162 prepared by grinding micro-ground sample (~500 μm) and KBr powder with a ratio of
163 10:100, and then pressing the mixture into pellets.

164 **2.3.4. Differential scanning calorimetry (DSC)**

165 Differential scanning calorimeter analysis was carried out on ~7 mg representative sample
166 using a Q100 TA Instrument under standardized conditions ISO 11357. Samples were
167 equilibrated at -50 °C, ramped at a heating rate of 10 °C/min to 280 °C, cooled down to -50
168 °C and re-heated to 280 °C at the same heating rate, under nitrogen atmosphere. The reported
169 data represent the cooling and the second heating cycles for three replicates.

170 **2.3.5. Thermogravimetric analysis (TGA)**

171 Thermal behavior of the studied samples was investigated on ~13 mg representative
172 sample, following the ISO 11358 standard, using a TGA1 analyzer from Mettler Toledo.
173 Tests were performed from 50 to 900 °C under nitrogen atmosphere (flow of 45 mL/min) at a
174 heating rate of 10 °C/min. After 1 min isothermal at 900 °C under nitrogen, the atmosphere
175 was switched to dry air and kept for 10 min at 900 °C. The repeatability of the measurement
176 was evaluated on the basis of three replicates.

177 **2.3.6. Energy dispersive X-ray fluorescence (XRF)**

178 The XRF data were collected with a wavelength dispersive X-ray spectrometer PW2404-
179 DY 750 Philips equipped with a rhodium X-ray tube anode and operated at 2.4 kW power for
180 all measurements using vacuum conditions. To investigate possible interferences, different
181 scans were performed using a 27 mm collimator and five crystals: LiF 200, LiF 220, Ge, InSb
182 and PX1. The XRF test specimens were obtained by compression molding the representative
183 sample powder at 160 °C, under manual pressure of ~15 bars. The reported values represent
184 the average of three replicates.

185 **2.3.7. Electron beam irradiation**

186 EB irradiation was performed on extruded pellets and micro-ground samples by Ionisos SA
187 (France) using a commercial EB accelerator source of ⁶⁰Co, under room temperature and air
188 atmosphere. The tested irradiation doses (50 and 200 kGy) were controlled by varying the
189 automatic conveyor speeds. Acceleration energy was set at 0.7 ± 0.2 MeV.

190 **2.3.8. Tensile strength**

191 Tensile strength tests were carried out on injection molded dog-bone specimens 1A ISO
192 527 type, according to ISO 527 standard, using MTS Criterion® 43 test machine. Samples
193 were equilibrated at ~23 °C and ~50% relative humidity (RH) for at least 48h before testing
194 them under the same environmental conditions. The reported values represent the average of
195 ten replicates for tensile stress and tensile stains, and of five replicates for Young moduli.

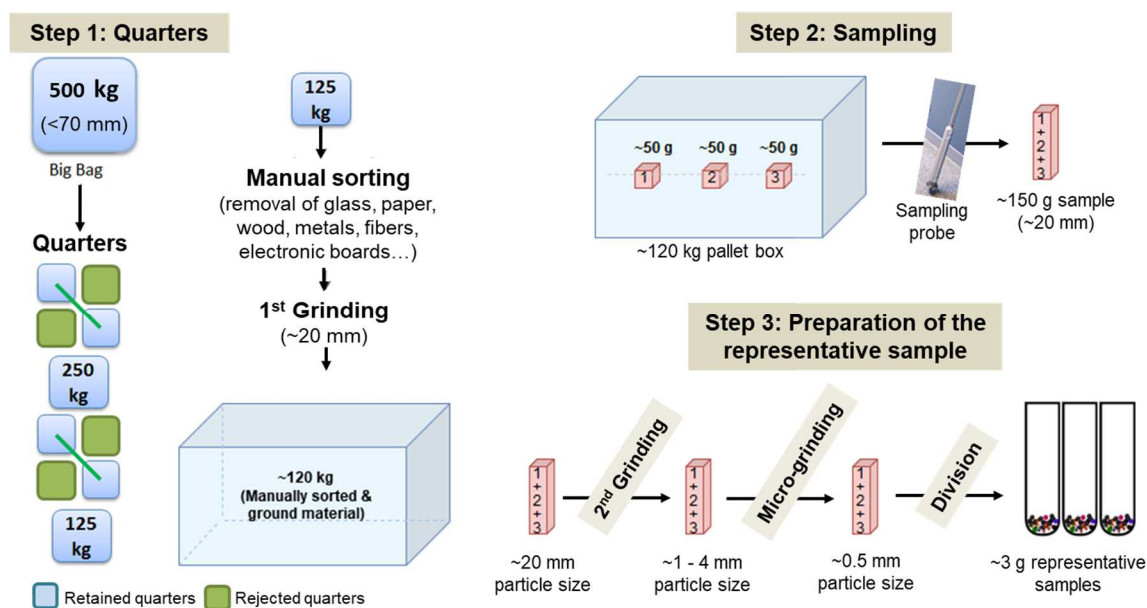
196 **2.3.9. Impact toughness**

197 Horizontal Charpy impact tests were carried out as per ISO 179 standard on unnotched
198 injection molded sample bars, using ZWICK 5102.202 impact pendulum device. Samples
199 were equilibrated at ~23 °C and ~50% RH for at least 48h before testing them under the same
200 environmental conditions. The reported impact toughness values were calculated for ten
201 replicates.

202 **3. Results and discussion**

203 **3.1. Composition of the studied unsortable WEEE batch**

204 Being aware of the composition and fraction size distribution (<70 mm) heterogeneity of
205 the studied batch, a sampling method turned out to be necessary to obtain the most
206 representative sample from the supplied big bag. Thus, we adapted a sampling method based
207 on our research team previous work (Epsztein *et al.*, 2014), as detailed in Fig. 2.



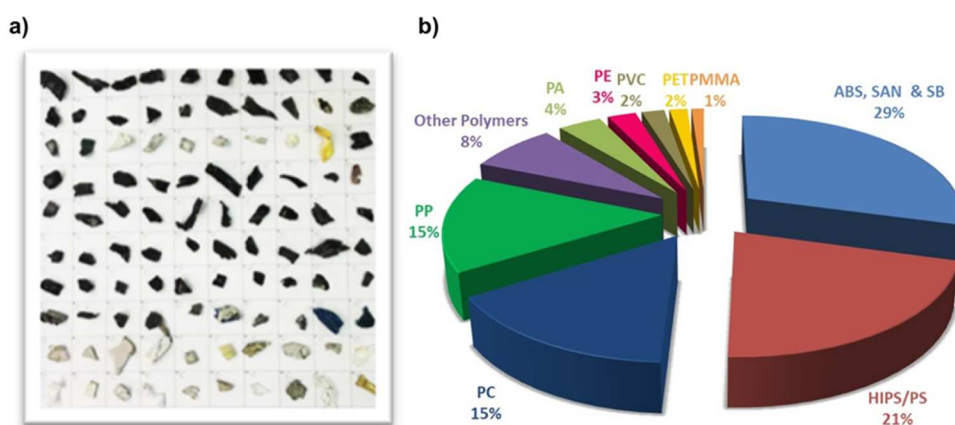
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209 **Fig.2.** Sampling method used in the present study

210 The 500 kg batch was first spread out and divided into four quarters twice, and at each
211 time, only two quarters were retained to finally obtain a 125 kg batch sample. After a manual
212 sorting, aimed at removing residual metals, glass, paper...etc, the sample batch was ground to
213 a particle size of ~20 mm, and three ~50 g samples were collected from the pallet box central
214 horizontal axis using a sampling probe. The three collected samples were mixed, ground to a
215 particle size of 2-4 mm, then to 500 μm. Finally, the ~150 g sample was divided using a
216 rotating divider to obtain homogeneous representative sample fractions, of approximately 3 g,
217 used for physico-chemical analysis.

218 **3.1.1. Analysis of bulk samples**

219 In order to quickly estimate the composition of the studied unsortable batch, 100 ground
220 samples of ~20 mm size (Fig. 3a) were collected arbitrary from the ~120 kg pallet box (Fig.
221 2) and characterized by attenuated total reflection Fourier-transform infrared spectroscopy
222 (ATR-FTIR). The polymer nature corresponding to each plastic fraction was identified by
223 comparing the recorded ATR-FTIR spectra to that of the Omnic software database. Fig. 3b
224 depicts the sum of assigned spectral polymers gathered from the 100 analyzed samples.



225
226 **Fig. 3.** a) 100 ground samples of ~20 mm size used to estimate b) the polymer composition, in
227 %, of the studied unsortable batch using ATR-FTIR analysis.

228
229 The composition heterogeneity of the batch under investigation is clearly seen in Fig. 3.
230 The collected sample is mainly constituted of styrene-based polymers (~50 %) such as
231 acrylonitrile-butadiene-styrene (ABS), polystyrene (PS), high impact polystyrene (HIPS),
232 poly(styrene-*co*-acrylonitrile) (SAN) and poly(styrene-*co*-butadiene) (SB). Polypropylene
233 (PP) and polycarbonate (PC) represent about 15 % each. Polyamide (PA), polyethylene (PE),
234 polyvinyl chloride (PVC), poly(ethylene terephthalate) (PET) and poly(methyl methacrylate)
235 (PMMA) are present in a very low quantity (i.e. 1-4 %). It is worth noting that the ~8 % of
236 “other polymers” include unidentified polymers such as multi-layer plastics, paints and
237 thermosets. This preliminary polymer identification (Fig. 3) allows to determine qualitatively
238 the polymer composition in the batch. Although this identification cannot be considered as

239 representative of the batch, the obtained results agree well with the data reported by Maris *et*
240 *al.* (2015). Similarly, other studies carried out in Germany (Dimitrakakis *et al.*, 2009), and
241 Portugal (Martinho *et al.*, 2012) on post-consumer plastic waste batches derived from small
242 EEE showed that ABS, PS/HIPS (~50-56 wt.%) and PP (~25-30 wt.%) account for at least 70
243 wt.% of the total plastic weight, and that the weight percent of each polymer depends on the
244 equipment type and WEEE categories (e.g. small household appliances, IT devices...).

245 **3.1.2. Analysis of the batch representative sample**

246 Likewise, the representative sample was characterized using FTIR, in the transmission
247 mode, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and energy
248 dispersive X-ray fluorescence (XRF) to identify more reliably and quantitatively the
249 composition of the studied batch before its melt processing and for further mechanical
250 characterization. Through the different cited physico-chemical analysis, the following
251 polymers, additives, charges and fillers were identified.

252

253 **3.1.2.1. Styrene-based polymers**

254 Table 1 presents the absorption bands observed on the FTIR transmission spectral
255 signature (Fig. S1) and their assignment for the studied batch representative sample. It is
256 clearly seen that the main screened constituents are styrene-based polymers. Indeed, the
257 strong out-of-plane C-H bending bands at 697 and 757 cm^{-1} , and the aromatic C-C stretching
258 bands at 1452, 1493 and 1601 cm^{-1} are characteristic of the aromatic substitution pattern.
259 Additionally, the two (out of four) aromatic combination bands in mono-substituted benzene
260 (1879 and 1941 cm^{-1}), usually observed for PS (Liang and Krimm, 1958; Munteanu and
261 Vasile, 2005), confirm the presence of styrene pattern. The other two aromatic combination

262 bands (1745 et 1800 cm^{-1}) might be hidden due to the band overlapping as a consequence of
 263 the complex composition of the studied batch.

264 **Table 1.** Observed FTIR absorption bands and their assignment for the representative sample
 265 of the studied unsortable WEEE batch.

Wavenumber (cm^{-1})	Intensity	Assignments	Polymer type
697	vs		
757	m	δ C-H aromatic	Styrene-based polymers
1452	m		
1493	w	ν C-C aromatic	Styrene-based polymers
1601	w		
1879	vw	Aromatic combination bands in mono-substituted rings	PS
1941	vw		
2236	vw	ν $\text{C}\equiv\text{N}$	ABS, SAN
3025	w	ν C-H aromatic	Styrene-based polymers
3059	vw		
1640	w	ν C=C of 1,2-vinyl butadiene	ABS, HIPS, SB
909	w	δ CH_2 1,2-butadiene	
964	w	δ CH_2 <i>trans</i> -1,4-butadiene isomer	
1163	m	ν C-O & ν C-C aromatic	PC
1193	m	ν C-O & ν C-C aromatic	
1229	m	ν C-O	
1772	w	ν C=O	
1731	w	ν C=O	PET or PMMA
1376	w	δ CH_3	PP
2849	w	ν CH_2 polymer chains	PE
2917	m		
3298	vw	ν NH or ν OH	PA or degraded ABS respectively

266 vs: very strong; m: medium; w: weak; vw: very weak; ν : stretching vibrations; δ : bending vibrations

267
 268 On the other hand, the absorption peaks of butadiene pattern recorded at 967 and 909 cm^{-1}
 269 are representative of ABS, SB and/or HIPS; these polymers are mainly characterized by C–H

270 deformation in *trans*-1,4-butadiene isomer and in 1,2-butadiene units at 965 and 910 cm⁻¹
271 respectively. The other butadiene characteristic band (i.e. 729 cm⁻¹ for δ CH₂ in *cis*-1,4-
272 butadiene) (Lacoste *et al.*, 1996; Silas *et al.*, 1959) could not be detected. Nevertheless, the
273 peak at 1640 cm⁻¹ reflects the stretching vibrations of the C=C interaction in the 1,2-butadiene
274 group (Munteanu and Vasile, 2005). Adding to that, the stretching vibrations of the nitrile
275 group (C \equiv N), typical for ABS and SAN, was observed at 2236 cm⁻¹.

276 The presence of styrene-based polymers in the studied batch is also supported by DSC
277 (Fig. 4a). As it is clearly seen in Fig. 4a, a glass transition temperature (T_g) around 99°C,
278 distinctive of ABS, PS, HIPS and SAN, was identified.

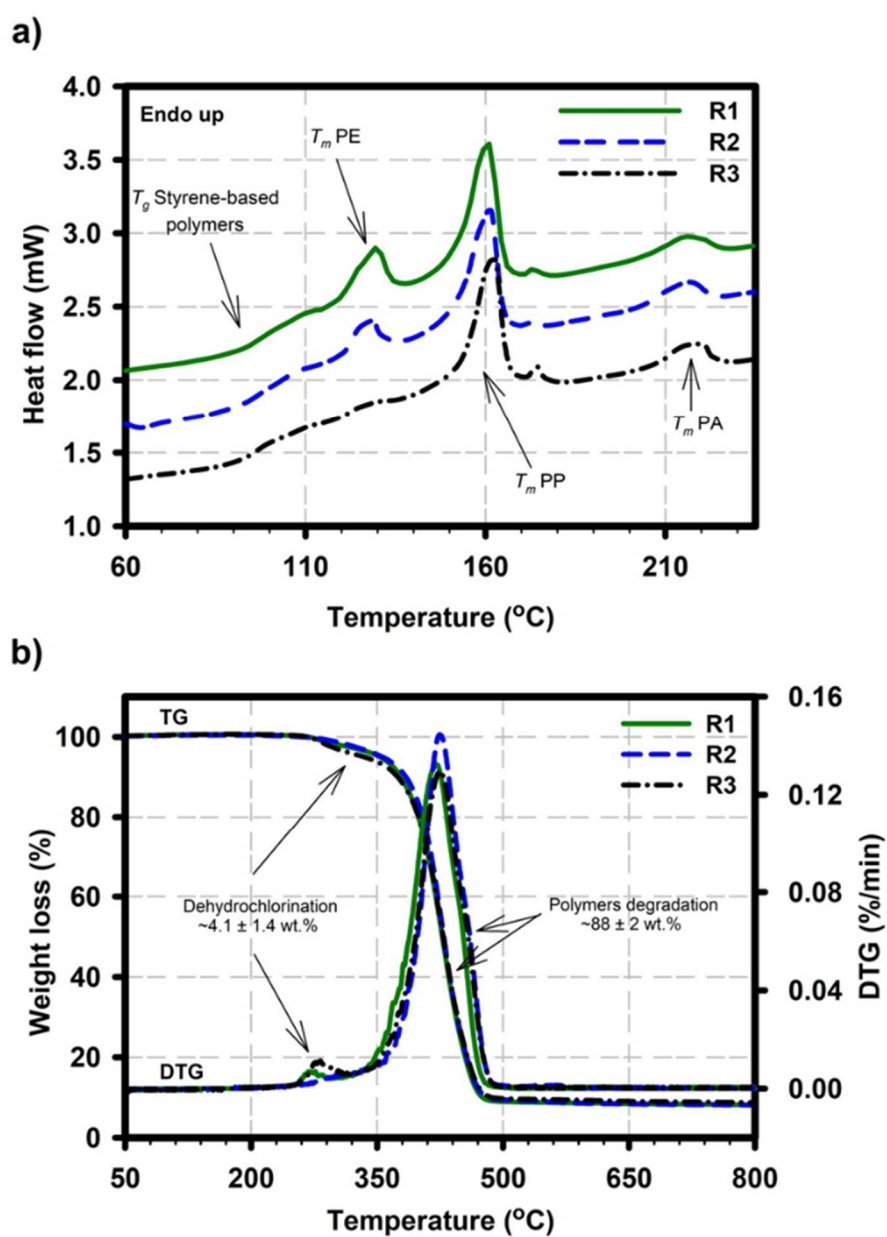
279 **3.1.2.2. Polyolefins**

280 The DSC thermograms (Fig. 4a) depict two distinctive endothermic peaks at ~129-133°C
281 and ~162-165 °C, typical of PE and PP melting temperatures (T_m PE and T_m PP),
282 respectively. Besides, the cooling exotherms in Fig. S2 display two single crystallization
283 peaks at temperatures corresponding to PP (T_c = ~120-121 °C) and PE (T_c = ~144-146 °C).
284 Moreover, compared to the PP crystals melting enthalpy (~5.9-8.3 J/g), that of PE (~0.3-2.7
285 J/g) indicates a very low weight concentration of PE in the studied batch, which perfectly
286 matches the FTIR data for bulk samples (Fig. 3b).

287 Interestingly, a higher temperature melting peak next to the main PP endothermic peak was
288 observed at ~175 °C for two replicates (Fig. 4a). It is not clear whether this extra melting peak
289 is a consequence of different PP lamellar arrangements that may result from polymer
290 oxidation phenomenon, or simply the presence of α -phase isotactic PP crystals (Huy *et al.*,
291 2004) subsequent to different crystallization conditions that may have occurred during the
292 manufacture processes.

293 **3.1.2.3. Other polymers**

294 **Polyamides.** The maximum absorption peak at $\sim 3298\text{ cm}^{-1}$ (Table 1 and Fig. S1) could be
295 assigned to stretching vibrations of NH bond, characteristic of polyamides. However, it has
296 been shown that the degradation of ABS can lead to the OH moiety formation with a vibration
297 band at $\sim 3400\text{ cm}^{-1}$ (Li *et al.*, 2017). Furthermore, the large melting peak at $\sim 220^\circ\text{C}$ (Fig. 4a)
298 and the crystallization one at $\sim 190^\circ\text{C}$ (Fig. S2) can be attributed to PA6, PA610 and/or
299 PA612.



300

301 **Fig. 4.** a) 2nd heating DSC thermograms, and b) TG and DTG curves for three replicates (R1,
302 R2, R3) of unsortable WEEE representative sample.

303

304 **PC, PMMA, PET.** The very low concentration of PMMA and PET in the studied batch
305 (Fig. 3b) as well as the hiddenness of the PC T_g , covering the 145-150°C range, by other
306 polymers thermal events (Fig. 4a and Fig. S2) make their detection by DSC impossible.
307 Nonetheless, Fig. S1 and Table 1 exhibit several absorption peaks that may be associated with
308 PC, PMMA or PET. Bands at 1772 cm⁻¹, assigned to stretching vibrations of C=O bond, and
309 the absorption peaks between 1128 and 1235 cm⁻¹ (Fig. S1), corresponding to stretching
310 vibrations of C-O bond, involve the presence of PC (Ghorbel *et al.*, 2014). The band at 1731
311 cm⁻¹ can also be attributed to stretching vibrations of C=O interactions of the ester function in
312 PET or PMMA (Ghorbel *et al.*, 2014; Zhu and Kelley, 2005).

313 Although the mentioned absorption peaks emphasize the presence of PC, PMMA and PET,
314 the same functional groups can also be ascribed to the degradation of styrenic polymers,
315 either via thermo- (Karahaliou and Tarantili, 2009; Vilaplana *et al.*, 2006) or photo-oxidation
316 (Gardette *et al.*, 1995) mechanisms.

317 **PVC.** The identification of PVC in the studied batch was achieved on the basis of TGA
318 analysis. The first degradation observed in thermogravimetry (TG) and derivative
319 thermogravimetry (DTG) curves at about 285°C (Fig. 4b) could be attributed to the presence
320 of PVC and allows the estimation of $\sim 4.1 \pm 1.4$ wt. % of PVC in the sample. Indeed, it has
321 been demonstrated that the degradation of PVC occurs in two stages (McNeill *et al.*, 1995): i)
322 the first one (250-360°C) corresponds to the dehydrochlorination and accounts for ~ 50 wt.%
323 of the weight loss, and ii) the second one (360-500°C) is related to the polymer chains
324 degradation. The latter accounts for ~ 25 wt.% weight loss. PVC content in the representative
325 sample determined from TGA is twice as high as estimated by the ATR-FTIR analysis on the

326 100 plastic fractions (Fig. 3b). On the other hand, $\sim 1.9 \pm 0.4$ wt.% of chlorine (Cl), coming
327 mainly from PVC, was obtained from XRF analysis (Table 2). The over-estimated PVC
328 concentration by TGA may be due to potential interactions between degradation products of
329 the batch components that could enhance the dehydrochlorination rate of PVC, particularly in
330 the presence of ABS and/or PET (Czégény et al., 2012).

331

332 **3.1.2.4. Additives, charges and fillers**

333 The complexity of the studied batch comes not only from the different types of polymers,
334 as described above, but also from the numerous inorganic/organic additives commonly
335 incorporated in plastic materials to improve their properties either mechanical, physico-
336 chemical, thermal, rheological or even esthetic. TGA analysis (Fig. 4b) showed
337 approximately $8.0 \pm 0.3\%$ under nitrogen residue, confirming the presence of an inorganic
338 phase. XRF analysis of the studied batch has allowed a reliable screening of its elemental
339 composition (Table 2). The validity of the sampling method is demonstrated by the low
340 standard deviation of the replicates. Several additive systems were identified as a result of the
341 elements examination.

342 Magnesium (Mg), aluminum (Al) and silicon (Si) are commonly used in the composition
343 of mineral fillers, usually in the form of silicates such as talc (hydrated magnesium silicate,
344 $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$) or kaolin (hydrated aluminum silicate, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$). Mg and Al
345 elements can also be issued from the presence of hydrotalcite compounds ($\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16},$
346 $4(\text{H}_2\text{O})$), used as a heat co-stabilizer for PVC (Bao *et al.*, 2008). Calcium carbonate (CaCO_3)
347 is the main Ca-based molecule frequently used in plastic industry with the purpose to decrease
348 the final cost of the material and increase its mechanical properties. Nonetheless, calcium

349 oxide (CaO) in combination with silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃),
 350 originated from fiberglass (Maris *et al.*, 2015), should not be ignored.

351 **Table 2.** Weight % of the different elements detected in the unsortable WEEE representative
 352 sample as determined by XRF.
 353

Elements	Concentration (wt. %)		Elements	Concentration (wt. %)	
	Average	Error*		Average	Error*
H	7.917	0.050	Ba	0.143	0.042
C	82.366	0.665	S	0.112	0.015
N	1.865	0.021	Fe	0.145	0.012
O	2.175	0.192	Ni	0.011	0.001
Ca	0.859	0.082	Cu	0.011	0.001
Mg	0.342	0.056	Cr	0.014	0.002
Si	0.646	0.046	Zn	0.072	0.004
Al	0.152	0.135	Mn	0.002	0.000
Cl	1.867	0.400	Sn	0.002	0.000
Br	0.070	0.002	Pb	0.006	0.000
Sb	0.114	0.021	Na	0.107	0.021
Ti	0.571	0.065	K	0.035	0.003
P	0.390	0.041	Sr	0.009	0.002

354 *The calculated error is based on three replicates

355 The recorded concentration of total bromine (Br; 0.070 ± 0.002 wt.%) along with that of
 356 the other regulated elements such as lead (Pb; 0.006 wt.%) and chromium (Cr; 0.014 ± 0.002
 357 wt.%), complies with RoHS (Restriction of Hazardous Substances) regulation, hence allowing
 358 the recovery of the studied batch by means of mechanical recycling. Once again, the very
 359 small standard deviation reflects the reliability of the sampling method. It is worth mentioning
 360 that BFR-containing plastics were separated from the studied batch using an on-line X-ray
 361 detection as defined in the directive 2019/1021/EU (2019/1021/EU, 2019).

362 Br, antimony (Sb) and phosphorus (P) indicate the presence of flame retardants (FR) in the
 363 studied batch. Sb, in the form of antimony trioxide (Sb₂O₃), is used as a synergic agent of
 364 BFR thereby enhancing the bromine release from BFR by forming Sb₂Br₃ (Grause *et al.*,
 365 2010). Additionally, P-based FR such as phosphates, phosphites and melamine phosphates
 366 have been widely used in the last decades as value-added FR systems.

367 The small concentration of Zn (zinc) and Cr elements in the batch can be attributed to
368 Ziegler-Natta and Philips catalysts used for polymerization process of PP and PE (Bichinho et
369 al., 2005). Similarly, residual sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$), a water-soluble initiator, used in
370 the emulsion polymerization processes (Kumar and Gupta, 2003), can explain the presence of
371 sodium (Na) and sulfur (S). Sulphites, are another S-based molecule commonly found in
372 plastic parts because of their hydroperoxide inhibition properties.

373 The low Ti content ($\sim 0.57 \pm 0.07$ wt.%), originated from titanium dioxide (TiO_2), is related
374 to the fact that unsortable WEEE streams are mainly constituted of dark colored plastic
375 fractions, whereas TiO_2 is usually used as white pigment in PC/ABS blends (Taurino *et al.*,
376 2010).

377 Iron (Fe), nickel (Ni), and copper (Cu) XRF signals, can be explained by the presence of
378 residual metallic parts that have not been removed during the sorting steps. Furthermore, the
379 Fe-based barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) is frequently found in electrical equipment operating at
380 microwave/GHz frequencies due to its electrical properties (Pullar, 2012).

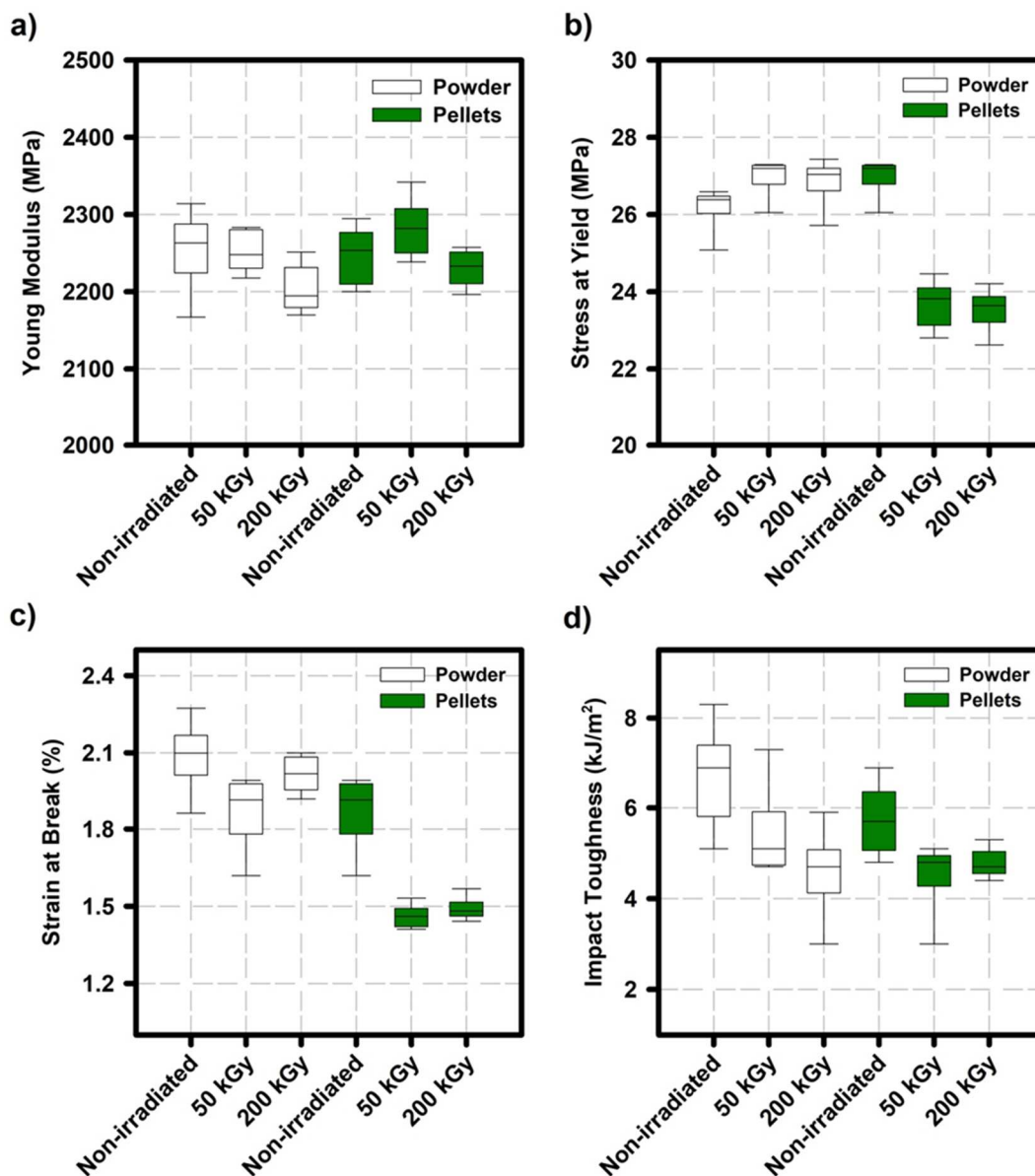
381 The screening of the 500 Kg batch gives an interesting insight into the composition of a
382 real unsortable post-consumer WEEE streams. The characterization of either bulk samples
383 (~ 20 mm size) or batch representative samples has allowed a reliable quantification of the
384 main polymers used in the manufacture of small EEE devices; styrene-based polymers (e.g.
385 ABS, PS and HIPS), PP and PC account for more than 70% of the total organic components.
386 The heterogeneity and complexity of the stream arise also from the detected inorganic/organic
387 additives frequently used in the plastics industry. This phase accounts for 8 wt. % and
388 includes calcium carbonate, mineral fillers, and fiberglass. More importantly, the recorded
389 concentration of the RoHS restricted substances enables for mechanical recycling.

390 ***3.2. Relevance of EB irradiation to mechanical properties of unsortable WEEE plastics***

391 Given the complex composition of the studied batch, electron beam (EB) irradiation was
392 considered as a means of compatibilization between the batch components. Indeed,
393 processing a complex polymer blend may give rise to materials with poor mechanical
394 properties as a consequence of polymers incompatibility and heterogeneity (Maris *et al.*,
395 2018). Irradiation of a complex polymer blend results in *in-situ* cross-linking reactions at the
396 interface leading to polymer compatibilization (Sonnier *et al.*, 2012). From a potential
397 industrial application perspective, EB-based ionizing radiation was adopted. Indeed, EB
398 technology is already used on an industrial scale for commercial purposes, does not generate
399 nuclear waste, is characterized by its short processing time and complies with restrictions on
400 volatile organic compounds emission (International Irradiation Association, 2011).

401 In the present work, two experimental pathways were investigated; the first one has
402 involved EB irradiation of micro-ground sample (< 500 μm particle size) prior to twin-screw
403 extrusion step, hereafter denoted as irradiated powder, while the second one has dealt with
404 irradiated extruded pellets, hereafter denoted as irradiated pellets. Non-irradiated pellets,
405 extruded irradiated powder and irradiated pellets were then injection molded and
406 mechanically characterized. Fig. 5 illustrates tensile strength and impact toughness results on
407 irradiated unsortable WEEE samples as a function of irradiation dose and experimental
408 procedure. The applied irradiation on powder has not a significant effect on the mentioned
409 properties. Except for elastic moduli (Fig. 5a), the mechanical behavior of the investigated
410 samples highlights irradiation dose and process dependency; stress at yield (Fig. 5b) of
411 irradiated pellets decreases after irradiation, while the EB irradiation undertaken on powder
412 does not show a significant evolution of the mentioned property. Additionally, the irradiation
413 dose is with no noteworthy impact on the irradiated powder elongation at break (Fig. 5c)
414 compared to that on the irradiated pellets, where a drop of the elongation at break was
415 observed. Regarding the impact toughness (Fig. 5d), the EB irradiation led to a slight decrease

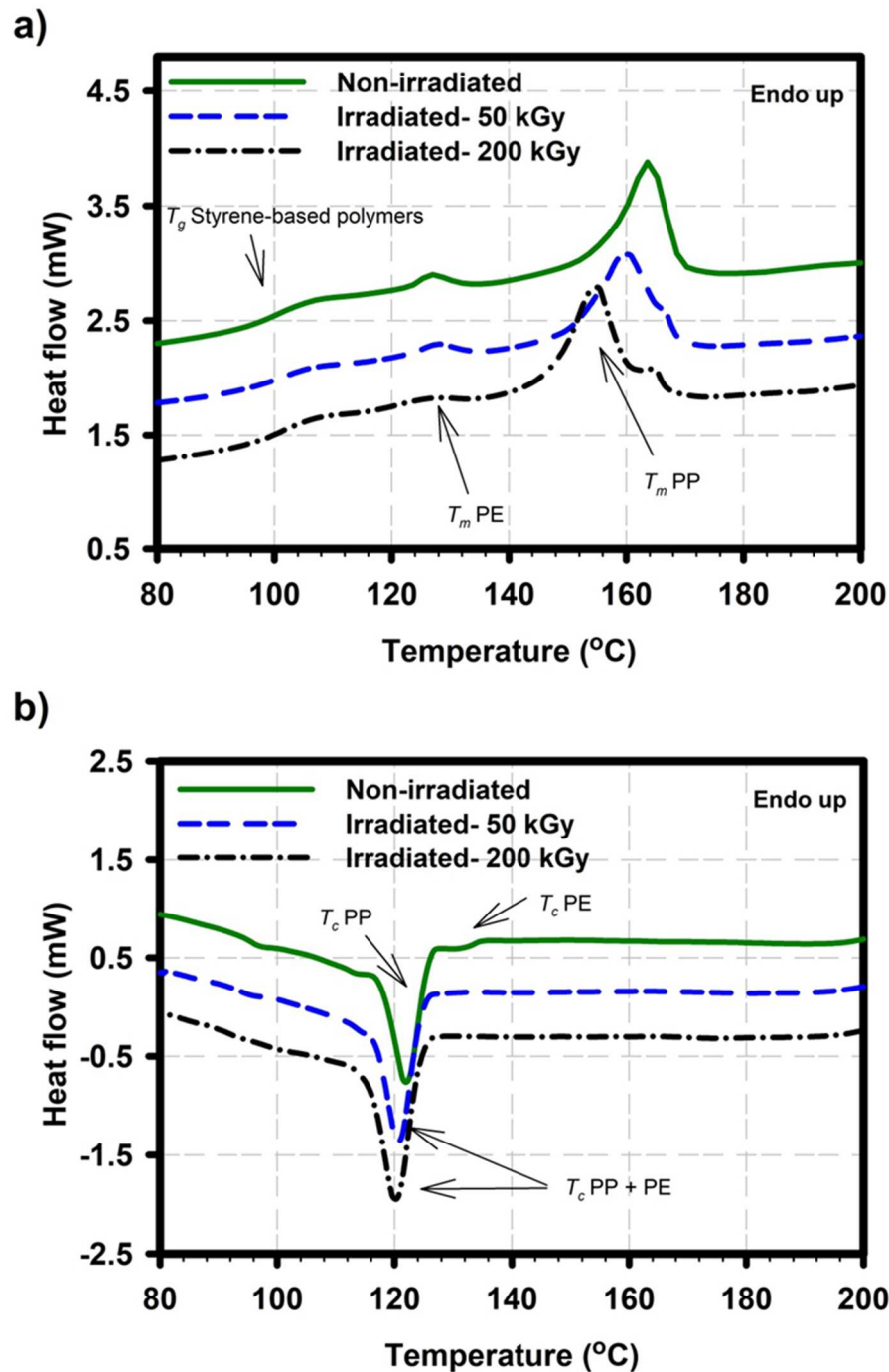
416 of the sample toughness median value regardless of the applied irradiation dose and the
 417 experimental procedure. Nevertheless the wide range of the boxplot limits (i.e. Min-Max) of
 418 non-irradiated material highlights the material heterogeneity that probably hides the real effect
 419 of EB irradiation.



420
 421 **Fig. 5.** a) Young modulus, b) stress at yield, c) strain at break and d) impact toughness for
 422 irradiated unsortable WEEE samples as a function of irradiation dose and experimental
 423 procedure (dog-bone specimens from irradiated powder or from irradiated pellets).

424

425 Based on these findings, it can be assumed that EB irradiation process does not really
426 increase the adhesion through *in-situ* crosslinking and compatibilization between the different
427 component phases of the investigated batch. Moreover, the decrease of elongation at break
428 and impact toughness of the irradiated samples may result from a potential polymer chain
429 degradation. In order to give more insight into this hypothesis, DSC analysis was carried out
430 on injection molded bar specimens used for mechanical testing. The recorded DSC
431 thermograms are displayed in Fig. 6. First, the heating DSC thermograms (Fig. 6a) of non-
432 irradiated bars exhibit phase transitions similar to those for representative samples (Fig. 4a);
433 two distinctive endothermic melting peaks around ~ 127 °C (T_m PE) and ~ 164 °C (T_m PP), as
434 well as a styrene-based polymers glass transition at ~ 99 °C. On the contrary, the
435 crystallization of PE component occurs at a delayed temperature (~ 131 °C), closer to that of
436 PP, for non-irradiated processed sample (Fig. 6b), than for non-processed representative
437 sample (Fig. S2). These observations might be indicative of PE dissolution in PP matrix in the
438 molten state (Blom et al., 1998). Besides, EB irradiation, particularly at 200 kGy, leads PP
439 melting point to shift meaningfully to a lower temperature (T_m PP = ~ 154 °C) and PE melt
440 enthalpy to decrease significantly (0.48 J/g for 200 kGy irradiated sample vs. 0.83 J/g for non-
441 irradiated sample). In addition, the EB irradiation process gives rise to a single slightly shifted
442 exothermic peak (Fig. 6b), representative of simultaneous crystallization of PP and PE,
443 whereas it shows no impact on T_g of the styrenic polymers. It should be noted that DSC
444 analysis on bar samples injection molded from irradiated pellets (Fig. S3) exhibits a similar
445 trend to that presented in Fig. 6 with a PE crystallization occurring later than that of PP
446 resulting in a broader PP melt peak.



447

448 **Fig. 6.** DSC thermograms for bar specimens injection molded from irradiated then extruded
 449 powder at different irradiation dose (0, 50 and 200 kGy). a) 2nd heating cycle, and b) cooling
 450 cycle.

451

452 It has been demonstrated for PE/PP blends that the depression of both melting temperature
 453 and crystallinity rate indicates the miscibility phenomena (Li *et al.*, 2001), and that potential
 454 chemical interactions between macroradicals of irradiated PP and PE are favorable during

455 melt processing (Fel *et al.*, 2016). However, it is worth pointing out that melt temperature
456 decrease is also indicative of degraded polymers, and that crystallinity rate drop may be due
457 to their crosslinking. Given the complex composition of the studied batch, DSC was not able
458 to estimate the impact of irradiation on the other batch polymer components, that would have
459 provided supporting explanations for the observed mechanical behavior of irradiated samples.

460 Other methods such as electron paramagnetic resonance (EPR) and 1,1-diphenyl-2-
461 picrylhydrazyl (DPPH) (unpublished results) failed to detect the persistence of macroradicals
462 in irradiated samples. Several key assumptions can be made: (1) EPR spectra are difficult to
463 interpret because of the complex batch composition; (2) The presence of Fe-based molecules
464 gives rise to anisotropic EPR spectra as the sample position changes; (3) The remaining
465 pigments in the batch hinder UV-absorbance of DPPH• radicals; (4) Residual antioxidants and
466 free radical scavengers react with DPPH• radicals.

467 Based on all the observations and discussion in this section, it can be hypothesized that upon
468 irradiation, PP undergoes β -chain scissions followed by crosslinking reactions with irradiated-
469 PE during melt processing stages, which may explain the decrease in the elongation at break
470 and impact toughness. On the other hand, although the irradiation may have led to free radical
471 production, the subsequent *in-situ* crosslinking reactions were not efficient enough to allow
472 compatibilization between the different polymer phases, and consequent mechanical
473 performance improvement of the final material. This can be explained by the complex
474 composition of the unsortable post-consumer WEEE stream and the presence of residual free
475 radical scavengers.

476

477 **4. Conclusions**

478 Mechanical recycling of plastics derived from post-consumer WEEE is an important waste
479 management strategy. However, ~40% of this waste stream, named unsortable, are rejected
480 from the classical NIR sorting lines as they don't comply with the sorted plastic streams and
481 also because of their dark color. For the first time, a comprehensive examination of a real
482 unsortable WEEE fraction has been investigated in the present work. The screening of the
483 studied batch composition, based on FTIR analysis of 100 plastic parts (~ 20 mm size) and
484 physico-chemical characterization of representative samples, allowed a reliable quantification
485 and identification of different polymer types; including mainly ~50 % styrenic polymers, ~15
486 % PP, and ~15 % PC, and showed the existence of ~8 wt. % inorganic phase (calcium
487 carbonate, talc and fiberglass). Most importantly, the concentration of total bromine, lead and
488 chromium, in agreement with RoHS (Restriction of Hazardous Substances) regulation, gives
489 evidence for the batch mechanical recycling.

490 Given the heterogenous composition of the 500 kg batch under investigation, EB
491 irradiation, as a means of compatibilization between the batch components, was investigated.
492 The potential recycling of the studied batch was evaluated through mechanical properties as
493 function of irradiation dose (50 and 200 kGy) and experimental procedure (irradiated powder
494 and irradiated pellets). Unlike irradiated powder, a significant drop of the elongation at break
495 and stress at yield was detected for irradiated pellets regardless of the applied dose.
496 Nevertheless, the impact toughness property was found to decrease independently of the
497 applied irradiation dose and the experimental procedure. DSC analysis have shown potential
498 PP β -chain scissions followed by crosslinking reactions with irradiated-PE during melt
499 processing stages. This study has provided evidence that the behavior difference between a
500 EB-irradiated unsortable post-consumer WEEE stream and a EB-irradiated virgin polymers
501 blend, reported in the literature, is due to the complex composition of the stream and the
502 presence of residual free radical scavengers which hinder *in-situ* crosslinking reactions

503 leading to the compatibilization between the different polymer phases. The present study
504 shows that mechanical recycling simulation based on virgin polymer blend or sorted polymer
505 waste is not representative of real post-consumer unsortable streams. This work may spur
506 further studies on other ways of compatibilization in order to improve mechanical properties
507 and cost-effectiveness.

508 **Author contributions section**

509 **Imane Belyamani:** Conceptualization, Methodology, Validation, Formal analysis,
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512 analysis, Investigation, Data Curation. **Sylvie Bourdon:** Conceptualization, Supervision,
513 Writing- Review and Editing, resources, funding acquisition. **Jean-Michel Brossard:**
514 Conceptualization, Validation, Supervision, Writing- Review and Editing, resources, funding
515 acquisition. **Laurent Cauret:** Conceptualization, Methodology, Validation, Supervision.
516 **Laurent Fontaine:** Conceptualization, Methodology, Validation, supervision. **Véronique**
517 **Montembault:** Conceptualization, Methodology, Validation, Project administration,
518 Supervision, Writing- Review and Editing.

519

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523

524

525 Additional Supporting Information may be found in the online version of this article.

526

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