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Study of the physico-chemical properties of recycled polymers from waste electrical and electronic equipment (WEEE) sorted by high resolution near infrared devices

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A B S T R A C T

Recycling of plastics from waste electrical and electronic equipment (WEEE) requires large scale high-performance sorting and identification technologies. The aim of this project is to evaluate the sorting performances of near infrared (NIR) devices, developed by the company PELLENC ST, for three styrenic polymers from WEEE: acrylonitrile-butadiene-styrene (ABS), high-impact polystyrene (HIPS) and ABS/polycarbonate blend (ABS/PC) and to study the physico-chemical properties of recycled materials. The use of an improved near infrared technique allows high levels of efficiency and performance to be achieved. This study can be used to evaluate the substitution ratio of virgin styrenic polymers by recycled ones for life cycle assessments (LCA) of reclaimed materials. Mechanical properties and fire properties of the sorted polymers were investigated. Mechanical properties of the polymers, particularly these of HIPS, were slightly modified after sorting. However, a degradation of the polybutadiene phase was observed. Fire tests revealed the presence of flame retardants in ABS and HIPS. Hence, NIR process could be used after a first sorting of brominated plastic wastes, in order to respect the RoHS directive. This project offers industrially viable alternatives for the recovery of plastics waste and the use of secondary raw materials.

Keywords:
Polymer
Recycling
WEEE
Near infrared sorting
Mechanical properties

1. Introduction

Electronic industry has lived several technological revolutions these last decades. The constant evolution of technologies coupled with a planned obsolescence of products leads to a huge amount of waste electrical and electronic equipment (WEEE). WEEE generated in the European Union was appraised at around 9 million tonnes per year in 2005 (Huisman et al., 2007). A recent report of the UNEP has estimated a tremendous increase of electronic waste for developing countries: by 2020, WEEE in South Africa and China will have soared to 200–400 per cent from 2007 levels and to 500 per cent in India (UNEP, 2009). Illegal exportation of WEEE to developing countries, especially Africa and China, has been pointed out by several non-governmental organizations (Puckett et al., 2003; Hosoda, 2007; SBC, 2011).

To limit this exponential growth of EEE wastes, the European Commission adopted in 2002 the Directive 2002/96/EC on WEEE which defines strict recycling quotas on 10 WEEE categories (European Commission, 2003a). Quotas imposed are ambitious: 50–75% for recycling rates and 70–80% for recovery rates.

With an average polymer content of about 30 wt% in WEEE (Menad et al., 1998; Taurino et al., 2010), recycling and waste recovery of plastics from WEEE is a key issue to achieve directive quotas. There are several strategies to recycle plastics and thus to avoid landfilling (Vilaplana and Karlsson, 2008). Using mechanical recycling, a secondary raw material is produced from plastic waste through processes based on physical phenomena. Feedstock recycling consists in cracking and depolymerizing plastic waste to obtain a series of petrochemical products or monomers (Aguado et al., 2006; Garforth et al., 2004). Energy recovery enables to produce energy through incineration processes of plastic wastes, thanks to their high calorific content (Mirza, 1999). Others recycling processes, such as chemical recycling, can solubilize both thermoplastics and a few fraction of thermosets polymers (i.e. sheet moulding compounds) by solvolysis (Perrin et al., 2006).

Life cycle assessment (LCA) is a standardized method used to evaluate the environmental impacts of a product or a service (ISO 14040). Many studies deal with LCA of plastic wastes recycling (Jenseit et al., 2003; Perugini et al., 2005; Shonfield, 2008;

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Finnveden et al., 2005). Most of time, they compare the different routes used: energy recovery, mechanical, chemical, feedstock recycling or landfilling for thermoplastics polymers, which represent 80% of total plastic consumption (Al-Salem et al., 2009). Results vary strongly according to the selected route. For Salem et al., feedstock recycling is the most sustainable solution as it recovers valuable petrochemicals while producing energy (heat, steam, etc.). They emphasize the disadvantages of mechanical recycling as the necessity to obtain a high quality, clear, clean and homogenous recycled material to compete with virgin polymer or the degradation of polymers chains occurring during re-processing. Lazarevic et al. has reviewed LCA of plastic waste recycling and they conclude that mechanical recycling is generally the environmentally preferred treatment option (Lazarevic et al., 2010). However, they specified that some critical parameters can reverse the trend, especially the organic contamination content and the virgin material substitution ratio. Indeed, this organic contamination engenders an additional step of washing, inducing a waste water treatment, whereas the organic content increases the heating value of the waste and thus favours the energy recovery option (Frees, 2002). The virgin material substitution ratio, defined as the ratio at which recycled plastic substitutes virgin plastic at equivalent properties, is essential to evaluate the ecological impacts. Indeed, when recycled plastic has a lower quality than virgin one, a mix with the virgin polymer is considered in order to lead to a recycled material with the same properties than virgin one, and thus environmental impacts linked to the virgin polymer production and transport are included in the LCA. When it is lower than 0.5, the benefit of mechanical recycling compared to incineration is less evident (Lazarevic et al., 2010). It can be noticed that in most of studies the substitution ratio is assumed to be 1, by lack of data about recycled polymers. Quality of recycled polymer is directly linked to purity. Thus, to ensure environmental and economic benefits, purity of recycled polymers must be the highest possible, a level of 5 wt% is acceptable whether the polymer considered. A complete study of the Waste and Resources Action Programme (WRAP) (Shonfield, 2008) about the LCA of management options for mixed waste plastics compared different scenarios of recycling with different technologies. They concluded that landfilling and incineration were the worst options. They also noted that the substitution ratio was a critical point to ensure the benefit of the mechanical recycling. LCA for waste plastic management is difficult to perform due to the lack of data about recycled polymers properties. Thus, it is essential to evaluate substitution ratios, which are directly linked to the sorting process.

Moreover, the interest of the mechanical recycling of thermoplastics is limited due to the quality of the recycled material. Thermoplastics polymers show only a limited compatibility among each other. Hence, contaminated fractions and mix of different polymers lead to a drastic decrease in the plastic properties and thereby entail limited applications for the recycled material. Then, to recover mixed thermoplastics waste, mechanical properties have to be improved by adding additives such as impact modifiers (Elmaghor et al., 2004) or by mixing recycled polymers with virgin ones (Mehat and Kamaruddin, 2011). Mechanical recycling can only be viable if the recycled polymers can compete, technically and economically, with virgin ones.

To preserve environment, the use of some hazardous substances is restricted by the RoHS European Directive (2002/95/CE European Commission, 2003b). Since 2006, EU members' states have to ensure that all new electric and electronic devices do not contain lead (Pb), mercury (Hg), cadmium (Cd), hexavalent chromium (Cr (VI)), and four brominated flame retardants. Most of plastics from WEEE contain contaminants as heavy metals or brominated flame retardants (BFRs), mainly polybrominated diphenylether (PBDE). BFRs are widely used to improve the fire reaction of polymers. By thermal degradation, those contaminants are suspected of promote the production of highly toxic compounds, such as halogenated dioxins and furans (Ebert and Bahadir, 2003). Consequently, an essential step to WEEE plastics recycling is the separation of flame-retarded plastics fraction containing halogens from the other plastics, to exclude them from the recycling loop.

Engineering polymers such as acrylonitrile-butadiene-styrene (ABS), high impact polystyrene (HIPS) and polycarbonate (PC)/ABS blends, constitute around 50 wt% of the recovered plastics of end-of-life electronics. ABS consists of styrene-acrylonitrile copolymer (SAN) mixed with a polybutadiene (PB) rubber phase. PB particles are generally grafted with acrylonitrile and/or styrene to improve the adhesion with SAN matrix (Xu et al., 2005). Chemical and heat resistance, toughness, rigidity and easy processing are provided by SAN while PB provides impact strength. HIPS is a mix of polystyrene (PS) and PB, which improves the impact strength properties of PS. PC main features are high impact resistance and good fire behaviour, despite it is relatively expensive and very notch sensitive. To improve PC properties, it is often mixed with ABS to obtain PC/ABS blends which combines the good mechanical and thermal properties of PC and the ease of processability, notched impact resistance and the sometimes lower price of ABS. ABS content is generally around 25–30 wt%. Mechanical properties of ABS and HIPS, especially impact properties, are highly dependent on the PB particle size and volume fraction (Bucknall et al., 1986).

To separate mixed plastics, technologies based on different polymer's properties are used, like density (Ferrara and Meloy, 1999; Pongstabodee et al., 2007), electromagnetic (Dodbiba et al., 2005) or surface properties (Shibata et al., 1996; Buchan and Yarar, 1995). However, those techniques do not permit to separate polymers of the same family, since densities are too similar, or polymers charged with an important level of additives, since densities are too modified. For instance, Shen et al., were not able to distinguish ABS from PS by flotation with several surfactants (Shen et al., 2002). Thus, a direct identification of the polymers nature is more reliable, in order to sort them automatically from a waste flow. Several techniques can be used to identify polymers and additives: mass spectroscopy (Luederwald, 1981), X-ray fluorescence (Boeykens and Vázquez, 2004), plasma spectroscopy (Anzano et al., 2010), vibrational spectroscopy as infrared spectroscopy (Weyer, 1985; Bledzki and Nowaczek, 1994; Taurino et al., 2010) or Raman spectroscopy (Florestan et al., 1994; Mailhot and Gardette, 1996). Near infrared spectroscopy of polymers is influenced by additives presence, whether their chemical structures, concentrations and thus infrared absorbance. Indeed, this technology has been used to quantitatively measure some additives concentrations (Solera et al., 1999; Karstang and Henriksen, 1992). However, a characteristic spectrum is measured for each polymer and then identification is possible even with additives, except black carbon.

Infrared spectroscopy is a relevant method for automatic sorting of polymers as it is a non-destructive and fast technique. Both the near-infrared (NIR) (wavelengths between 0.8 μm and 2.5 μm) and mid-infrared (MIR) ranges (2.5–30 μm) can be used for spectroscopic analysis of physical and chemical polymeric properties. NIR spectroscopy is more adapted than MIR to automatic sorting as spectrum acquisition is faster (Huth-Fehre et al., 1995; Wahab et al., 2006). The accessibility of NIR sensors led to the development of this technique. NIR spectroscopy presents a lot of advantages: remote high speed measurements, high penetration depth of the NIR radiations, which prevents a sample pre-treatment, and an important signal-to-noise ratio. The main disadvantage of NIR spectroscopy is the incapacity to detect black polymers as radiation is totally absorbed by this kind of material. In addition, NIR photons excite two or more different molecular vibrations simultaneously, and then absorption is weaker and featureless than with MIR. Thus, to exploit NIR spectroscopy as a

Table 1
Size of plastic flakes of the studied samples.

PW	Polymers	$s < 10$ mm	10 mm $< s <$ 20 mm	$s > 20$ mm	m (kg)
1	ABS, ABS/PC	8%	92%	0%	0.78
2	ABS, HIPS, PP, non plastic parts	0%	0%	100%	4.00
3	ABS, HIPS, ABS/PC, PVC, non plastic parts	1%	8%	91%	155.70

fast, accurate, non-destructive and remote sensing identification technique, an indirect calibration of the spectra is necessary. This calibration consists in the measurement of a representative numbers of samples with the desired polymer composition. This step, called the machine learning, requires standard samples with known composition. NIR technique has already been studied to identify plastics, as polyethylene terephthalate (PET), polyvinyl chloride (PVC), polypropylene (PP), or polyethylene (PE), in household waste or building and construction waste (Serranti et al., 2012) and also for the more recent biobased polymer polylactid acid (PLA) (Ulrici et al., 2013). Classification of measured spectra can be carried out using an artificial neural network (Huth-Fehre et al., 1995; Scott and Waterland, 1995) or using classical techniques, such as linear discriminant analysis (DLA) and partial least square (PLS) (Van den Broek et al., 1998).

The aim of this paper is to present the sorting performances of automatic NIR sorting devices, developed by PELLENC ST Company, devoted to WEEE plastics and the properties of the sorted materials. Mechanical and fire properties of three of the sorted polymers: ABS, HIPS and ABS/PC were evaluated. The originality of this work consists in the study of polymer materials separated on line, as in industrial conditions, using near infrared spectroscopy as sorting technology. Results of mechanical properties can then be used to evaluate the substitution ratio for an LCA of plastics from WEEE sorted by NIR automating technology.

2. Experimental

2.1. Sorting system

2.1.1. Materials

Plastic waste (PW) samples of WEEE from different sources were used to test the sorting performances of NIR devices. For the device 1, a mix of several plastic from differents WEEE sources was used. For the device 2, three distinct lots were studied: firstly, a reference lot: PW1, with only ABS and ABS/PC, was made by selecting already identified samples of ABS and ABS/PC from computer and information technology products. The nature of polymers of PW1 was identified with a MIR spectrophotometer (Bruker IFS 66 FTIR). The second one, PW2, corresponded to an actual waste deposit from household appliances such as fridges and stoves. It was mainly composed of ABS, HIPS and PP, but also of non-plastic materials such as metal and wood. A third lot, PW3, also from a waste deposit of computers was composed of "grey plastics": ABS, HIPS, ABS/PC, PVC and of a small part of non-plastic materials. Size of plastic flakes (s) after grinding and weight of each lot are presented in Table 1. The size of flakes was determined using sieves of 10 mm and 20 mm. The weight content of each polymer differs in lots, with a predominance of styrenic polymers but also black polymers (Table 2).

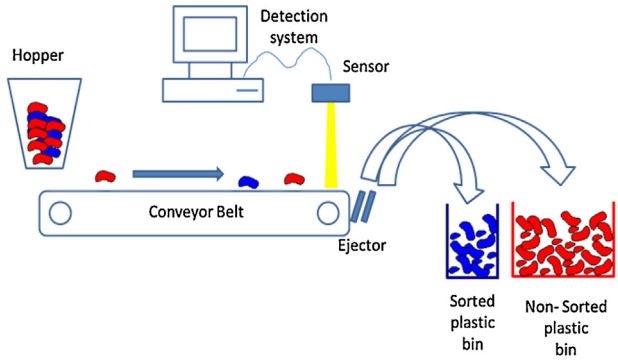

Fig. 1. Illustration of the automated sorting system.

2.1.2. Sorting principle and characterization methods

The high resolution near infrared (NIR) technology, called Mistral series, developed by PELLENC ST (Pertuis, France) aims to sort various kinds of materials on the range of 1000–2100 nm. The spectrum of the objects located on a conveyor belt is obtained and the composition of each material is recognized. The detection system was developed by PELLENC ST, first, the machine learns the models spectra from standard samples, which define the reference database. Then each analyzed sample is compared to the models spectra. A specific mathematical treatment leads to the distance between the analyzed spectra and a model one, a minimal distance is needed to separate two polymers. An upgradeable software application allows the identification of new materials to be performed. Simultaneous recognition allows different plastics to be sorted from each other:

- Commodity plastics: PET, high density PE, low density PE, PP, PS, PVC.
- Engineering plastics: ABS, ABS/PVC, ABS/PC, HIPS, PC.
- Fibrous materials: elasthane, paper and cardboard.

The distance between the detection zone and the ejection is deliberately very close to avoid errors resulting from the trajectory of objects on the conveyor belt.

The main components of the sorting system are illustrated on Fig. 1. Samples are deposed on a conveyor by a hopper; they are transported under the NIR light and sensor and are ejected in the collecting bins if desired. The system permits to sort one plastic type only. The conveyor has a length of 4 m and a speed of 2.5 m/s. The absorption spectrum of each sample is acquired by a spectrometer

Table 2
Mass composition of each studied lot.

PW	ABS	ABS/PC	HiPS	Other polymers	Black polymers	Other material
1	63%	37%				
2	39%		22%	16%	21%	2%
3	40.3%	14.4%	2.6%	2.4%	31.2%	9.2%

Table 3
Sorting system characteristics of device 1.

Work width	500 mm
Conveyor speed	1.5 m/s
Capacity	800–1000 kg/h
Sorted polymers	ABS, ABS/PC, PVC, PC, HIPS
Dark or black samples	Non sorted
Size of analyzed samples	10–20 mm

Table 4
Characteristics of the new device 2.

Work width	800 mm
Conveyor speed	2.5 m/s
Capacity	2 t/h
Sorted polymers	11
Dark or black samples	Non sorted
Size of analyzed samples	5–200 mm

and on line analysis is performed using a computer. Analysis time is typically around 40 μs.

The system learns the spectroscopic fingerprint of materials which have to be sorted: several samples having the same composition are analyzed; the spectrum obtained for each plastic type defines a model. This stage is essential to ensure a good sorting. The first material database (A) learned by the system is made of 5 types of plastic: ABS, ABS/PC, PVC, PC and HIPS. The characteristics of this basic system, noted 1, are presented in Table 3.

Performances of the sorting system are determined by means of two indicators: the removal efficiency and the purity. Removal efficiency is defined as the yield of the sorting; it is calculated as the ratio of the mass of a given sorted material to the initial total mass of this material. The purity of the sorting reveals the error of sorting; it is defined as the ratio of the mass of desired sorted material to all materials sorted in the same bin. The nature of the sorted plastic samples was identified with a Bruker IFS 66 FTIR spectrophotometer. With PW3, a quartering process was first carried out to limit the number of analysis. A Perkin Elmer Diamond differential scanning calorimeter (DSC) was also used to determine the nature of impurities of the sorted plastic samples. ABS and ABS/PC samples were heated from 20 °C to 260 °C at 10 °C/min under nitrogen atmosphere and kept at 260 °C for 1 min. The cooling ramp was similar. X-ray diffraction was used to determine the presence of brominated flame retardants with a Bruker D8 diffractometer using CuKa radiation. Data were collected between 1 and 13° by step of 0.02° using an X-ray generator with $\lambda = 0.15406$ nm.

2.1.3. Improvement of the sorting system

The sorting system was optimized to handle with large WEEE waste flows. Several modifications of the basic device 1 were made to lead to a new one (noted 2). Algorithms used to treat infrared spectra were the same for the two devices. The new characteristics are presented on Table 4. The detection system was highly improved in order to detect samples with a minimal size of 25 mm^2 instead of 100 mm^2 for the basic device. The acquisition frequency was increased: 2.5 more spectrums are analyzed for each run. The range of analysis of the NIR spectrometer was enlarged to better distinguish ABS and HIPS. A new database (B) containing 11 polymers: ABS, ABS/PC, PVC, PC, HIPS, polyamide (PA), poly(phenylene oxide)(PPO)/PS, polyoxymethylene (POM), SAN, PC/PET, and acrylonitrile styrene acrylate (ASA)/PC, was set up in the system. These plastics were selected according to the main plastics found in WEEE. The use of a material database (A or B) is independent of the device.

Table 5
Notations of sorted samples.

Plastic 1A	Plastic sorted once with device 1 and database A
Plastic 2B	Plastic sorted once with device 2 and database B
Plastic $(2B)^2$	Plastic sorted twice with device 2 and database B

2.2. Polymer recycling and testing

2.2.1. Materials

The materials from PW3 sorted using both devices (1, 2) and both databases (A, B) were studied. Only PW3 was tested, because mass of lots PW1 and PW2 were not sufficient to realize test specimens. Three polymers were sorted: ABS, ABS/PC, and HIPS with a total mass of 4 kg for each sorting process. Those polymers were chosen because of their predominance in WEEE plastic wastes. After sorting, each fraction of plastics was ground using a Rotoplex grinder with a rotation speed of 1500 rpm to obtain fragments with a final size of 6 mm. Each piece was inspected before and after grinding stage. Two successive sorting were only tested for ABS as it is the main polymer found in WEEE. Samples notation is presented in Table 5.

Virgin materials studied are ABS (Toyolac 100MPF, Toray), ABS/PC (Establend 4500, Cossa Polimeri) and HIPS (Escrimo SB, GmBH). They were used as reference to compare recycled polymers properties. Indeed, styrene resins ABS, HIPS and ABS/PC copolymers are representative of most of the waste of electrical and electronic equipment for example as the casings of TVs, phones and computer screens. Furthermore, in order to achieve a reliable comparison taking into account the type of WEEE used from styrene resins, raw polymers having a similar degree of injection moulding (MFI between 20 and 23) have been used. On the other hand, the mass composition of terpolymer block ABS and PC/ABS (30–70) is 25% of acrylonitrile, 25% of butadiene and 50% of styrene.

2.2.2. Sample preparation

Sorted ABS, HIPS and ABS/PC were processed using a twin screw extruder with a screw length of 900 mm (L/D = 36, co-rotating, Clextral BC21). Temperature profiles were adapted to recycled plastics: 240 °C for ABS/PC, 220 °C for HIPS and 230 °C for ABS. Granulates were injection moulded with a Sandretto 95 t injection moulding machine to obtain standardized specimens. ABS/PC was dried at 120 °C for 4 h before extrusion and injection.

2.2.3. Mechanical and fire behaviour characterizations

Tensile tests were performed on dogbones specimens, according to ISO 527-1 standard, using a Zwick Z010 tensile tester. Young's modulus measurements were made at a crosshead speed of 1 mm/min, and elongation measurements were made at 50 mm/min. The reported values for all tensile properties are the average of 10 determinations. Impact tests were carried out on according to ISO 179 standard, with a Zwick 5102 Charpy pendulum impact tester. Only ABS/PC specimens were notched. Indeed, tests performed on unnotched ABS/PC specimens led to values outside the energy range of the pendulum (7.5 J). Impact results are the average of tests run with 15 specimens.

Fire reaction properties of the samples were studied using UL 94 V fire test and Glow Wire Ignition Test (GWIT) according to IEC 60695-2-12. The tests were respectively carried out on 12.5 mm × 12.5 mm × 3.2 mm injection moulded barrels and 50 mm × 50 mm × 4 mm injection moulded square sheets. We report the GWIT maximum temperature of each sample after three consecutive tests for the same composition.

3. Results and discussion

3.1. Sorting performances

The purity and removal efficiency obtained for ABS, HIPS and ABS/PC sorted with NIR sorting devices are presented on Fig. 2 and

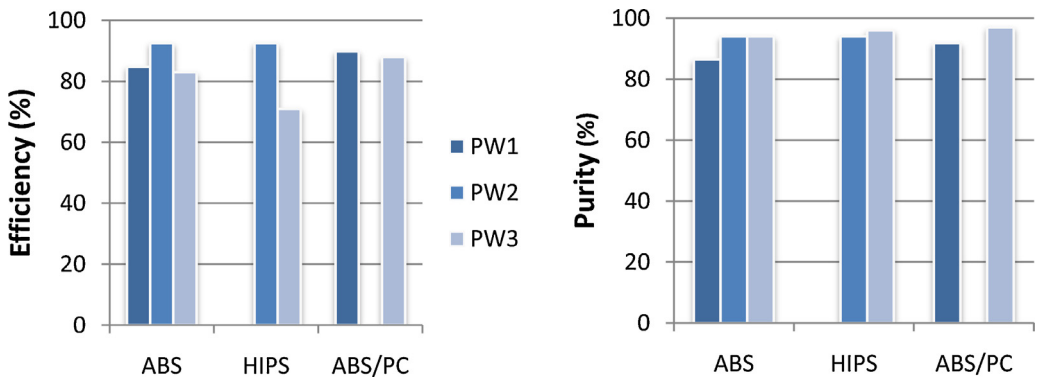

Fig. 2. Sorting performances of the device 2 with database B.

Table 6. With device 1, efficiency is at 80% for all the studied polymers. Purities of HIPS and ABS are at 70%, while ABS/PC is at 85%. The presence of PC helps in detecting the ABS/PC which leads to a higher purity for ABS/PC, while the distinction between ABS and HIPS was not successful. With device 2, PW2, which only contains pieces with sizes higher than 20 mm, presents the best results of removal efficiency (>90%). Except for HIPS from PW3, all efficiencies obtained were higher than 80%. This result can be explained by the low concentration of HIPS in PW3 (2.6 wt%). PW1, only constituted of ABS and ABS/PC, displays rather poor purity results.

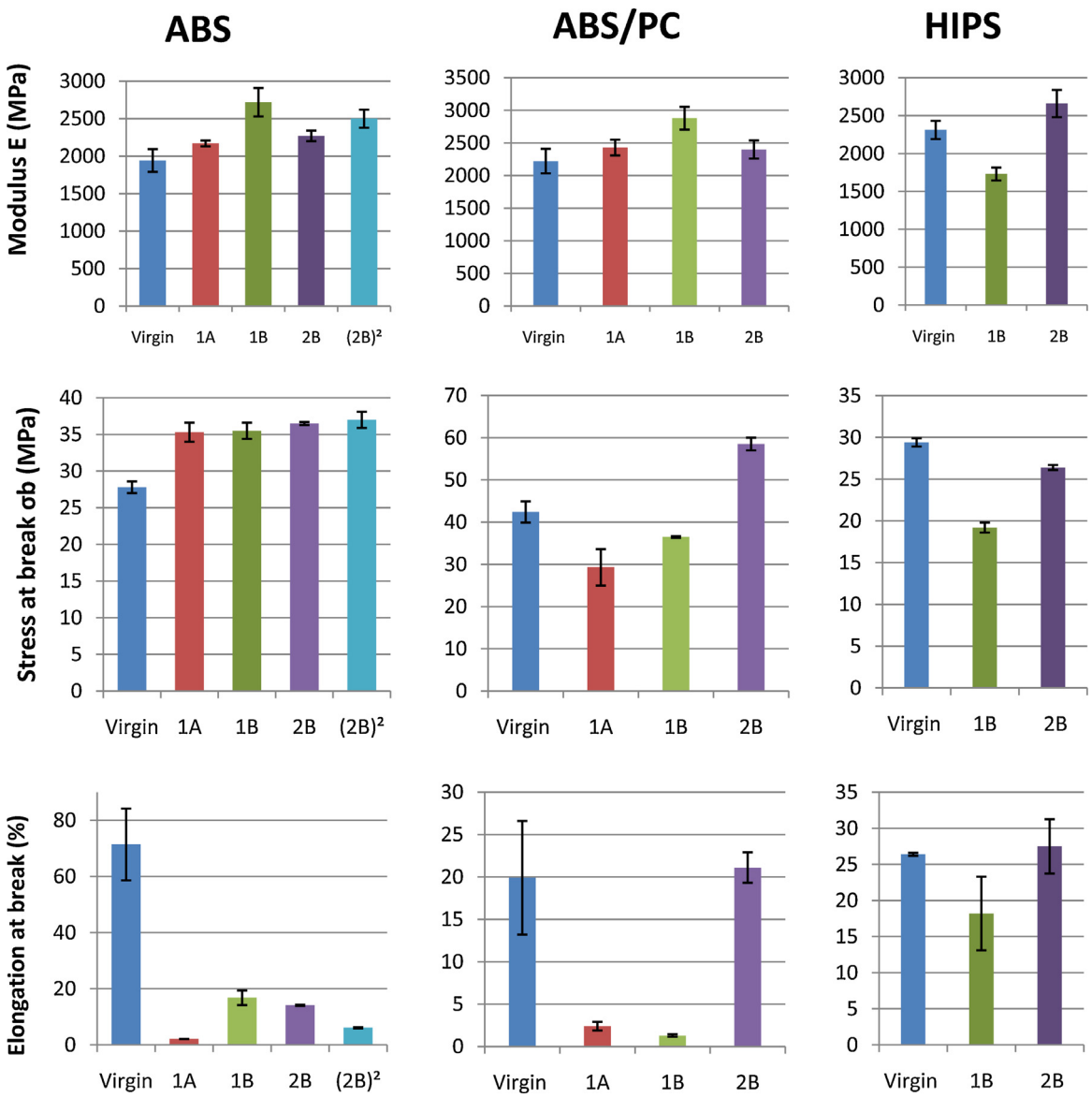

Fig. 3. Mechanical properties of sorted ABS, ABS/PC and HIPS (error bars represent one standard deviation).

Table 6
Sorting performances of device 1.

	ABS	HIPS	ABS/PC
Purity	70%	70%	85%
Efficiency	80%	80%	80%

This can be ascribed to the difficulty in distinguishing ABS/PC from ABS. Purity obtained with actual WEEE deposits, PW2 and PW3, were higher than 90%. Other sorting technologies in development lead to the same performances. Indeed, results of purity for ABS and HIPS are similar to the performances obtained recently by Hui et al., with a dissolved air flotation sorting method in two stages, by using specific wetting agents (Hui et al., 2012). A purity of around 50% for ABS in a binary mixture of ABS/PS was obtained using a triboelectrostatic separation process (Dodbiba et al., 2005). Separation of ABS and HIPS was carried out using froth flotation with the use of selected wetting agents (Pascoe, 2005); however no tests were performed on actual deposit of WEEE mixed plastics.

Improved NIR-based sorting process leads to purities higher than 90% for all sorted polymers and enables to separate polymers of the same family of styrenic.

3.2. Mechanical properties

3.2.1. Tensile properties

As expected, tensile properties of the three recycled polymers are notably different from the reference polymers (Fig. 3). For ABS and ABS/PC, elongation at break decreases drastically while modulus is quite similar or higher than virgin one. Stress at break increases for ABS, but not for ABS/PC, for which the results depends on sorting process. 2B leads to the best tensile properties for ABS/PC. Conversely, no specific trend is observed regarding the sorting process used for ABS: elongation at break decreases, while other properties increase in comparison with virgin ABS. With a second sorting ($2B^2$), mechanical properties are close as with only one sorting (2B).

Recycled HIPS 1B has lower tensile properties than virgin HIPS. Sorting process 2B allows tensile modulus and elongation at break to be improved.

3.2.1.1. Discussion about tensile properties of ABS. Few studies about recycling of styrenic polymers have been made using post-consumer materials (Arnold et al., 2010; Brennan et al., 2002; Barthes et al., 2011). Most of times, virgin polymers are reprocessed to simulate recycling loops (Pérez et al., 2010; Vilaplana et al., 2006). This leads to only take into account melt processing degradation on mechanical properties, without considering the influence of impurities and ageing phenomena, occurring during the useful life of end-of-life products. In the case of post-consumer materials, impurities resulting from automatic or manual sorting and ageing of polymers can lead to a slight diminution of molecular weight (Scaffaro et al., 2012).

Generally for ABS, similar trends are observed: tensile strength and modulus are slightly modified, whereas elongation at break considerably decreases (Bai et al., 2007; Karahaliou and Tarantili, 2009; Brennan et al., 2002; Salari and Ranjbar, 2008). One can note that elongation at break of virgin ABS can vary between 20% and 70% depending on the grade used and thus the PB loading. The important decrease of elongation at break is often ascribed to the PB degradation as well as to the mix of various grades of ABS.

Loss of mechanical properties during recycling can be explained by several phenomena. Bai et al. suggest that the loss of small volatiles molecules during processing, as lubricants for instance, induces a loss of mechanical properties (Bai et al., 2007). Increase of tensile strength and Young's modulus can be explained by the presence of impurities and especially ABS/PC. In the case of the model lot PW1, it can be noticed that after sorting ABS contains around 15 wt% of ABS/PC. The presence of impurities was confirmed on the SEM micrograph of the fracture surface of ABS after tensile test (Fig. 4), the nature of impurities was determined by X-ray analysis: calcite and clay on this sample.

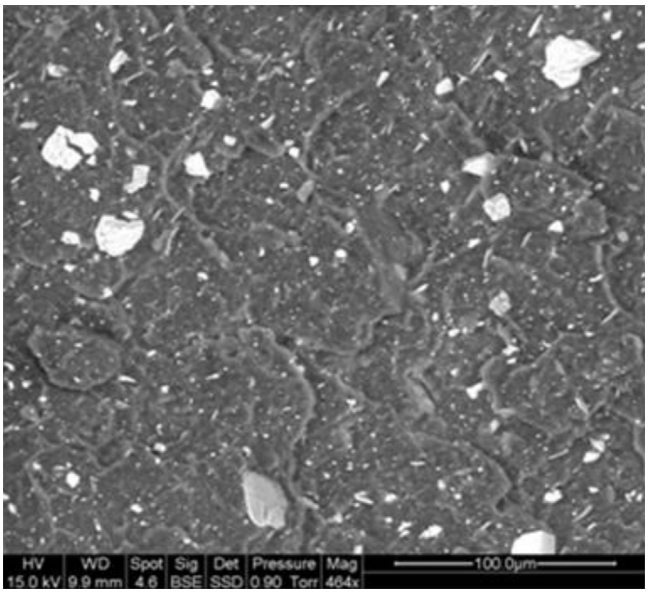

Fig. 4. SEM micrograph of fracture surface of ABS after tensile test.

Tarantili et al., observed that the incorporation of PC into ABS leads to an increase of tensile strength and elasticity modulus, as predicted by the law of mixtures. The degradation of polymeric phases, as polybutadiene and/or SAN, causes chain-scission or crosslinking, leading to the formation of oxidized moieties (Scaffaro et al., 2012; Salari and Ranjbar, 2008). Karahaliou studied ABS thermomechanical degradation by Fourier transform infrared spectroscopy (FTIR) on virgin ABS reprocessed five times: hydroxyl and ester peaks intensities increased with the number of extrusion cycles (Karahaliou and Tarantili, 2009). It was suggested that cross-linking reactions could be predominant since tensile strength and Young's modulus increased notably, thanks to the network formation (Karahaliou and Tarantili, 2009).

Whatever the conditions of sorting, elongation at break of recycled materials is always significantly lower than the virgin ones. As for other tensile properties, the degradation of the butadiene rubber phase and the loss of small molecules (monomers, thermal stabilizers, functional molecules, etc.) are the explanations generally proposed. The results obtained, showing an increase in the modulus of elasticity compared with the reference ABS seems to confirm these explanations. Polybutadiene phase degradation was confirmed by IR spectroscopy made on the Bruker IFS 66 FTIR spectrophotometer, sorted ABS from WEEE was compared to virgin ABS and virgin styrene-acrylonitrile polymer (SAN) (Fig. 5). The characteristic bands of carbon double bonds around 900 cm^{-1} drastically decrease for sorted ABS from WEEE, this zone is more similar than the spectrum obtained with SAN polymer, which does not contain polybutadiene phase. Other interpretations are related to both the presence of impurities, which facilitates the initiations and propagations of cracks, and microstructure modifications of ABS. The improvement of the impact strength by further sorting could confirm the negative role of the impurities about the ultimate properties.

3.2.1.2. Discussion about tensile properties of ABS/PC. Mechanical properties of the different sorted ABS/PC are greatly influenced by

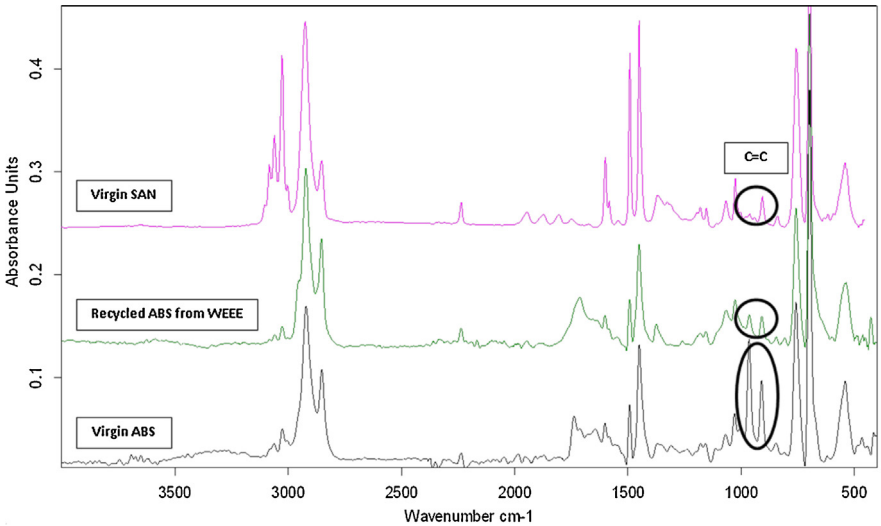

Fig. 5. NIR spectra of virgin ABS, recycled ABS from WEEE and virgin SAN.

the presence of impurities. Using process 1A or 1B, a significant decrease in elongation at break is observed. ABS/PC elongation at break for 2B process is at the same level as virgin polymer blend. ABS/PC properties depend on the amount of each monomer in the ABS terpolymer (Inberg, 2001) as well as the ABS/PC ratio. Moreover, ABS is generally the minor phase in the ABS/PC blend, thus the polybutadiene degradation phase is less notable than for pure ABS. Studies on PC reprocessing have shown that tensile properties are, in most of cases, few affected by the first cycles of processing (Pérez et al., 2010; Aguirre Flores and Sanchez-Valdes, 1999). However, Eguiazabal and Nazabal observed that tensile strength and elongation at break of ABS/PC began to decrease after the third cycle of reprocessing and ascribed it to a large degradation of butadiene rubber phase (Eguiazabal and Nazabal, 1990).

Since high levels of mechanical properties are reached for ABS/PC 2B thanks to high purity, it could be expected that a second sorting step ($2B^2$) could entail better mechanical properties, due to a higher purity of ABS/PC. It will be confirmed by further works.

3.2.1.3. Discussion about tensile properties of HIPS. HIPS was only sorted with the database B (Fig. 3). Device 1 has lower sorting performances, which is confirmed by the mechanical properties of HIPS 1B. The presence of a minor phase of others polymers, such as PE and PP polyolefins, induces a decrease of tensile properties. Without the use of compatibilizers, adhesion between polyolefins and styrenic polymers is weak (Santana and Manrich, 2002; Horák et al., 1996). Moreover, polystyrene matrix displays a good resistance to degradation for the first reprocessing cycles (Remili et al., 2011). Because of the lower content of PB in HIPS than in ABS (10 wt% often used in HIPS vs whereas 10–25 wt% often used in ABS), tensile properties of HIPS are less affected by recycling. Studies about influence of reprocessing on tensile properties of HIPS present various results and conclusions. For Brennan et al., reprocessing cycles do not have noticeable effects on the tensile strength and elastic modulus of HIPS, whereas Santana et al. observed a notable decrease in the tensile properties (Brennan et al., 2002; Santana and Manrich, 2002). Vilaplana et al. also observed a weak influence of first reprocessing cycle on tensile properties of recycled HIPS (Vilaplana et al., 2006). Nevertheless, influence of reprocessing on tensile properties is rarely noticeable after the first re-extrusion cycle, but thermochemical ageing must also be considered (Vilaplana et al., 2011). Influence of processing parameters is also not negligible, but cannot be compared for the above publications. The results of Fig. 3 highlight the improvement of the mechanical properties thanks to the improvement of the NIR sensitivity, and thus of its resolution. The reduction of the stress at break of HIPS 2B compared with virgin HIPS can be explained by the presence of impurities having a higher modulus of elasticity (probably ABS), but also leading to microstructure modifications. Nevertheless, tensile properties of HIPS using 2B process are very close to virgin HIPS ones. Consequently, a good sorting of HIPS in a real WEEE deposit can be expected.

3.2.2. Impact properties

Most of studies about recycling of styrenic blends have concluded that impact properties are the most affected by recycling and should be decisive for the use of recycled polymers in many applications requiring minimum values of impact strength. Impact properties of sorted polymers are presented on Fig. 6.

Device 1 always leads to a reduction of the impact strength, except for ABS 1B. This can be mainly ascribed to a high content of impurities, such as polyolefins, as they present poor interfacial interactions with the matrix. As device 1 has weak sorting performances, distinction of ABS and HIPS polymers should not be done. Thus, the increase in impact strength noticed for ABS 1B in comparison with all sorted ABS could be explained by the presence of a small fraction of HIPS which exhibits higher values of this property as in the case of our reference materials. This minor fraction of HIPS could also present a less degraded polybutadiene fraction.

With 2B sorting, ABS and ABS/PC are more affected than HIPS which exhibits impact strength close to virgin HIPS one. Impact resistances of ABS and ABS/PC are ruled by the polybutadiene phase, while SAN polymer presents low impact properties. Degradation of the PB phase, occurring during reprocessing and ageing, leads naturally to a reduction of the ABS and ABS/PC fracture energy. Grafting presents on PB phase should be degraded, leading to an agglomeration of the PB particles and then to a reduction of the toughness of ABS (Xu et al., 2005). Moreover, the presence of impurities in ABS and ABS/PC could modify the blend microstructure, entailing also an agglomeration of PB particles.

As well as for the tensile properties of ABS, the presence of ABS/PC could decrease the impact properties of ABS. Barthes et al., studied the incorporation of PC into ABS and observed that even at low PC content, impact properties of ABS were significantly affected (Barthes et al., 2011).

Luzuriaga et al. studied the influence of thermochemical ageing and reprocessing on HIPS properties; they showed that impact

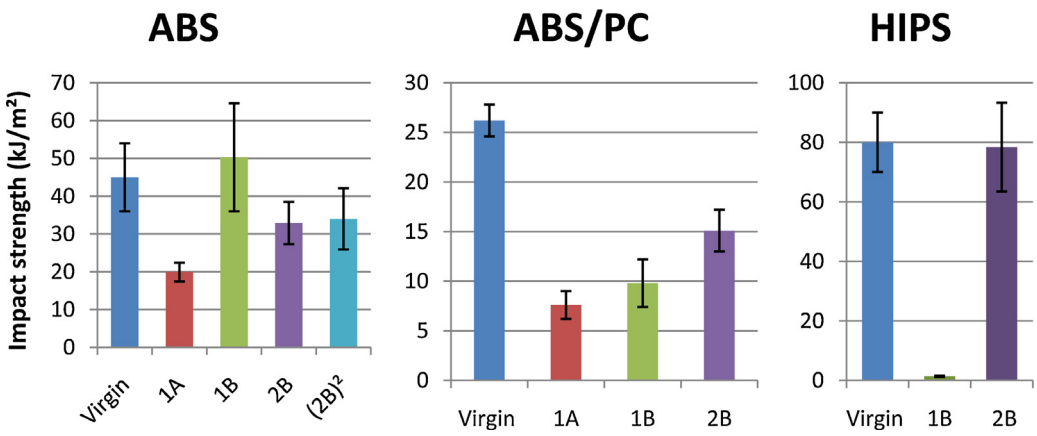

Fig. 6. Impact properties of sorted ABS, ABS/PC and HIPS (error bars represent one standard deviation).

strength of HIPS submitted to photochemical ageing decreased because of a surface degradation, whereas reprocessing led to a recovery of the initial impact strength thanks to the mixing at molten state of the material (Luzuriaga et al., 2006). Repeated extrusion had no clear effect on HIPS impact properties (Santana and Manrich, 2002), certainly because of the high thermal stability of PS and the low PB loading.

Finally, it appears that the increase of purity achieved with the 2B process leads to restored impact properties for HIPS. For ABS and ABS/PC, impact strength is reduced; however, those recycled materials can still be convenient for many applications

3.3. *Fire reaction tests*

Flame retardants are present in 30% of plastics of EEE (Vehlow et al., 2003). Brominated flame retardants (BFR) are generally used with styrenic polymers, and particularly for HIPS and ABS which present lower limiting oxygen index values than ABS/PC and PC due to the auto-extinguishing ability of PC. Fire reaction tests were performed for HIPS and ABS sorted with process 2B, in order to confirm the presence of FR additives since brominated flame retardants have been identified using X-ray diffraction on plastic parts, prior to grinding stages. Results showed that the recycled polymers are classified favourably (Table 7). $(2B)^2$ process for ABS even exhibits a high level of fire performance, since V0 is the highest class.

Therefore, the likely presence of several kinds of flame retardants for a same type of polymer does not appear to play a detrimental effect on flame retardancy. Hence, flame retardant compounds could offer complementary effects or even synergistic effects on fire behaviour.

Some of the brominated flame retardants (BFR) present in the samples are possibly restricted of use by the RoHS Directive (European Commission, 2003b). However, as no technology enables to identify automatically the chemical nature of BFR, all brominated plastic waste must be separated from the mechanically recycled plastic stream. NIR technology should be used with a complementary analytic tool, able to identify the presence of brominated FR, as laser induced breakdown spectroscopy (Kaski et al., 2004) or hand held X-ray fluorescence (XRF) device as it was recently done by Menad et al. (2012).

Table 7
Fire behaviour of virgin[a] and recycled polymers sorted with process 2B.

	Virgin HIPS	HIPS 2B	Virgin ABS	ABS 2B	ABS $(2B)^2$
UL 94 classification	HB	V1	HB	V1	V0
Max GWIT temperature (°C)	650	850	750	850	960

[a] Values of virgin polymers come from technical data sheets.

4. Conclusion

Improvement of a NIR sorting device enabled to reach high levels of purity for three of the most used plastics in WEEE: ABS, ABS/PC and HIPS, with impurities content varying between 5 and 8% in weight. Mechanical properties of materials obtained by reprocessing of the sorted polymers were determined. Recycled HIPS sorted with the improved device and database (2B) presents mechanical properties very close to virgin one. A degradation of the polybutadiene phase of ABS and ABS/PC was observed and confirms various previous works. Hence, elongation at break and impact strength decreased compared to virgin polymers. Despite further improvement of the sorting device is required, substitution ratio of a recycled HIPS sorted by NIR automating technology can be assumed to be 1 in view of the mechanical properties of HIPS 2B. Indeed, mechanical properties of HIPS 2B are closed of those of virgin HIPS, and thus it could be used instead of virgin HIPS, without the addition of virgin HIPS. This result can only be applied to the HIPS from the studied deposit, with this sorting device. For ABS and ABS/PC, complementary studies should be carried out to evaluate it accurately. However, substitution ratios should be quite near of 1 for these polymers, because of the low degradation of mechanical properties. The presence of flame retardants in recycled plastics from WEEE was confirmed by fire reaction tests and X-ray diffraction. NIR technology did not permit to identify these additives; however it can be coupled with X-ray fluorescence device in order to separate brominated polymers from recycled polymers. Obtaining sorted materials with similar performances to reference materials can allow iso-function applications to be envisaged, but also various beneficial applications, leading to ecological and economical gains. Degradation of the visual aspect of recycled polymers is still a concern for reusing them in visible pieces. Future works should be performed on the acceptability of such materials by consumers through the improvement of sensorial properties of these recycled polymers.

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