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Development of flame retarded self-reinforced composite from automotive shredder plastic waste

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ABSTRACT

Multilayered self-reinforced composites were developed from density-separated light fraction of automotive shredder waste of high polyolefin content, which can fulfil the current technical, safety and environmental requirements of structural materials. The significantly enhanced mechanical properties of the recycled composites were ensured by polypropylene fabric reinforcement; meanwhile, reduced flammability was obtained by modifying the matrix layers, made of secondary raw materials, with phosphorous-containing flame retardant additive. The results of the new flame retarded composite systems allowed the discussion of a novel mechanistic observation. The mechanical and flammability properties of the prepared self-reinforced composites were compared to conventional glass fabric reinforced composites and to compounds without reinforcement.

Keywords: recycling; self-reinforcement; multilayered composites; flame retardancy;

1. Introduction

The worldwide increasing plastic usage, owing to the achievable outstanding strength/density balance, processing flexibility and cost-effectiveness, produces an enormous amount of end-of-life (EOL) plastic products as a consequence. Their current treatment, due to the low volume/weight ratio

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and shortage of recycling, greatly burdens the landfills. The European legislation, in accordance with the waste treatment hierarchy (2008/98/EC Directive), aims to reduce the waste disposal and supports more environmentally friendly waste treatment technologies such as recycling.

In the case of mixed EOL plastics the conventional thermo-mechanical recycling is accompanied by property damage (downcycling) [1]. The main causes of downgrading (poorer quality and reduced functionality) of the reprocessed plastic mixture are the impurity (metals and dirt), inhomogeneity (large number of polymers with different molecular structure, melt behaviour, rheology and thermal stability) and incompatibility (different components form discrete phases with weak adherence) [2]. Additionally, there are economic constraints hindering the recycling of mixed plastic waste; the products obtained by conventional mechanical reprocessing do not meet the requirements of the market (neither in terms of mechanical properties nor in aesthetic aspects). Those products are almost unmarketable, which negatively affects the motivation for plastic recycling [3]. The solution for this negative tendency could be the transformation of the low-cost plastic waste into valuable composites or products that have special functions or properties, such as outstanding strength, impact resistance, and heat and/or fire resistance.

Among the mixed plastic waste the polyolefin fraction is the most suitable for thermo-mechanical recycling. Recyclable polyolefin fractions can be mostly obtained from automotive, electric and electronic equipments and packaging waste. To achieve economically effective recycling rates and high quality products, the mixed polyolefin fractions must be sorted by polymer types (polyethylene (PE) and polypropylene (PP)), and according to *Bakker et al.* [4] the fraction purity should be better than 97%. These requirements can only be ensured by new, effective separation techniques. Nevertheless, the compensation of the negative effects of impurity and inhomogeneity is indispensable to achieve mechanically suitable materials.

In order to enhance the mechanical properties of polyolefins, glass fibre reinforcement is generally used due to their stiffness, high strength, heat resistance, or natural fibres [5] due to their renewability, low-cost and light weight. Concerning further improvement of strength, longer (continuous) reinforcing fibres or fabrics can be applied. However, the glass or natural fibre reinforced composites are multi-component systems, where the use of dissimilar materials does not provide good adhesion. Furthermore, it encumbers the reprocessing and application of other waste treatment technologies, whereas their novel alternatives, the self-reinforced (SR) polymer composites [6], are easy to recycle

[7]. In the literature there are several contributions reporting a comparison of commercial glass fibre reinforced (GF) and SR composites [7,8,9,10,11] revealing that the most important advantages of SR composites are the potential of weight reduction, high tensile strength and impact resistance, excellent viscoelasticity and recyclability, thus the SR composites show continually increasing application potential.

Although the polymeric materials increasingly squeeze out the higher density and more expensive metallic structural units, their flammability still limits their applicability in many cases. Therefore it was assumed that development of flame retarded composites could acquire market benefit of recycled materials. Ammonium polyphosphate (APP) based intumescent flame retardants (IFR) are very commonly used in PP. Advantageously, in case of fire they produce low smoke density and do not emit corrosive gases. For homopolymers about 20-22 m/m% IFR is required to achieve acceptable levels of flame retardancy [12]. However, the simultaneous reinforcement and reduction of flammability are apparently antagonistic requirements, as the imperfect dispersion and the pure interfacial interaction between the IFR and the polymer considerably deteriorate the mechanical properties of the materials [13,14]. This fact is even more apparent when fibre reinforcement is applied [15,16]. The authors supposed to eliminate this contradiction by preparing multilayer composites including reinforcing and flame retarded sheets separately but well consolidated.

Our work aimed at developing environmentally advantageous composites from secondary raw materials accompanied with improved mechanical properties and reduced flammability at the same time. To reach this goal multilayered composites were prepared from the polyolefin fraction of automotive shredder waste. Polypropylene fabrics were applied as reinforcement, supporting the possibility of multiple recycling, while the flammability was reduced by modifying the recycled matrix layers with APP based flame retardant additive.

2. Materials and methods

2.1. Materials

Density-separated polyolefin fraction ($\rho < 0.92 \text{ g/cm}^3$) of automotive shredder waste, obtained from Alcufer Ltd. (Hungary), was used as matrix material of the prepared multilayered composites. On the basis of comprehensive analysis [17, 18] (TG, DSC, IR- and Raman spectroscopic measurements)

the main polymer component of the chosen fraction is polypropylene (but far from the advantageous purity of 97%, the PP content was 78 m/m%), its inorganic filler (mainly silicates and calcium-carbonate) content, determined by heating at 900°C, is lower than 0.75 m/m%.

In order to reduce the flammability of the composites intumescent flame retardant additive (APP: Exolit AP766, Clariant GmbH) was added to the recycled matrix material.

As reinforcement plain woven fabrics (Stradom S.A., Czestochowa, Poland; identification code: T-PPT-181) composed of highly stretched split PP tapes were used. The reinforcing fabrics have a melting temperature of $T_m = 172.4^\circ\text{C}$ (determined by DSC), a tensile strength of 128.8 ± 3 MPa measured on a 50 mm wide strip and an areal weight of 171.5 g/m^2 .

The mechanical and flammability properties of the recycled self-reinforced composites were compared to glass fabric reinforced recycled composites. The glass fabrics were obtained from Hexcel Co. (code of the fabric: HexForce 01717 1050 Z6224, type of yarns: EC9 68, strip tensile strength: 142.6 ± 6 MPa, areal weight: 160 g/m^2).

2.2. Preparation of composites

Three types of flame retarded recycled samples: compounds without reinforcement (REC-FR18), glass fabric reinforced (GF-REC-FR18) and self-reinforced (SR-REC-FR18) composites have been prepared with the same IFR content of 18 m/m%, the properties of which were compared to additive-free references: REC, GF-REC and SR-REC, respectively.

In the case of compounds without reinforcement the specimens (REC-FR18) were prepared by simple mixing 18 m/m% APP with the recycled matrix (REC) in a Brabender Plasti Corder PL 2000 type internal-mixer at 190°C , with a rotor speed of 30 rpm, for 10 min and then hot pressed to 4 mm thick specimens in a Collin P200E type laboratory hot press.

In the case of the multilayered composites (GF-REC-FR18 and SR-REC-FR18) first a higher amount (34 m/m%) of IFR was introduced into the recycled matrix, then from the obtained compounds $180 \mu\text{m}$ thick matrix layers were compression moulded at 190°C in a Schwabenthan Polystat 300 S laboratory hot press. The multilayered fabric reinforced composites were manufactured by lamination of 11 recycled matrix layers and 10 woven glass or PP fabrics according to the film stacking method [19]. The film-stacked packages were hot-pressed to composites in the same hot press at 162°C with a pressure of 6 MPa, for 2 min. The thickness of the prepared self-reinforced (SR) and glass fabric

reinforced (GF) composites was 3.78 mm and 2.78 mm, respectively. For the sake of better comparison between the GF and SR composites, the composition (number of layers) and the nominal reinforcement content (i.e. glass or PP fabric) of ca. 48 m/m% were also kept constant for each reinforced composite.

2.3. Methods

2.3.1. Mechanical tests

Comparative static tensile tests were performed on rectangular specimens of 20 mm × 150 mm (width × length) using a Zwick Z020 universal testing machine with a crosshead speed of 5 mm/min.

Static 3 point bending tests were performed on rectangular specimens of 80 mm × 20 mm using a Zwick Z020 universal testing machine. The crosshead speed was 5 mm/min and the span length was 64 mm.

Interlaminar (shear) strength was determined on rectangular specimens of 25 mm × 160 mm using Zwick Z020 universal testing machine according to the ASTM D 3167-97 standard with a crosshead speed of 5 mm/min.

Instrumented falling weight impact (IFWI) tests were performed on Fractovis 6785 (Ceast, Pianezza, Italy) using the following settings: maximal energy: 228.64 J; diameter of the dart: 20 mm; diameter of the support rig: 40 mm; weight of the dart: 23.62 kg and drop height: 1 m. 70 mm × 70 mm square specimens were subjected to IFWI tests. From the IFWI tests the specific perforation energy (E_p , the thickness-related total energy [J/mm]) was determined.

All of the above mentioned tests were performed at room temperature and at least five specimens were tested in all cases.

2.3.2. Flammability tests

The flame retardant performance of the prepared composites was characterized by limiting oxygen index (LOI) measurements according to ASTM D 2863 standard. The LOI value expresses the lowest ratio of oxygen of the atmosphere, where the specimen burning is still self-supporting.

Standard UL-94 flammability tests, according to ASTM D 635-77 were also performed. The UL-94 classification is used for determination of dripping and flame spreading rate.

Mass Loss type Cone Calorimeter tests were carried out by an instrument made by FTT Inc, following the procedures of ASTM E 906 standard method. Specimens (100 mm x 100 mm) were exposed to a constant heat flux of 50 kW/m², ignited, and heat release values and mass reduction were continuously recorded during their burning.

3. Results and Discussion

Experiments with real polymer wastes (i.e. not only mixtures of virgin polymers modelling polymer waste) and results for reprocessing of mixed plastic waste are quite rarely discussed in literature, therefore our results regarding the recycling of automotive shredder light fraction could not rely on antecedents.

Flame retarded self-reinforced composites were prepared by lamination and hot-pressing of polypropylene reinforcing fabrics and matrix layers made of secondary polyolefins. In order to reduce the flammability of the fabric reinforced composites, the recycled matrix layers have been modified by adding 34 m/m% IFR additive. The properties of easily recyclable self-reinforced composites (SR-REC) were compared to common glass fabric reinforced composites (GF-REC) (prepared by the same technological parameters) and to reference samples without reinforcement (REC).

3.1. Mechanical properties

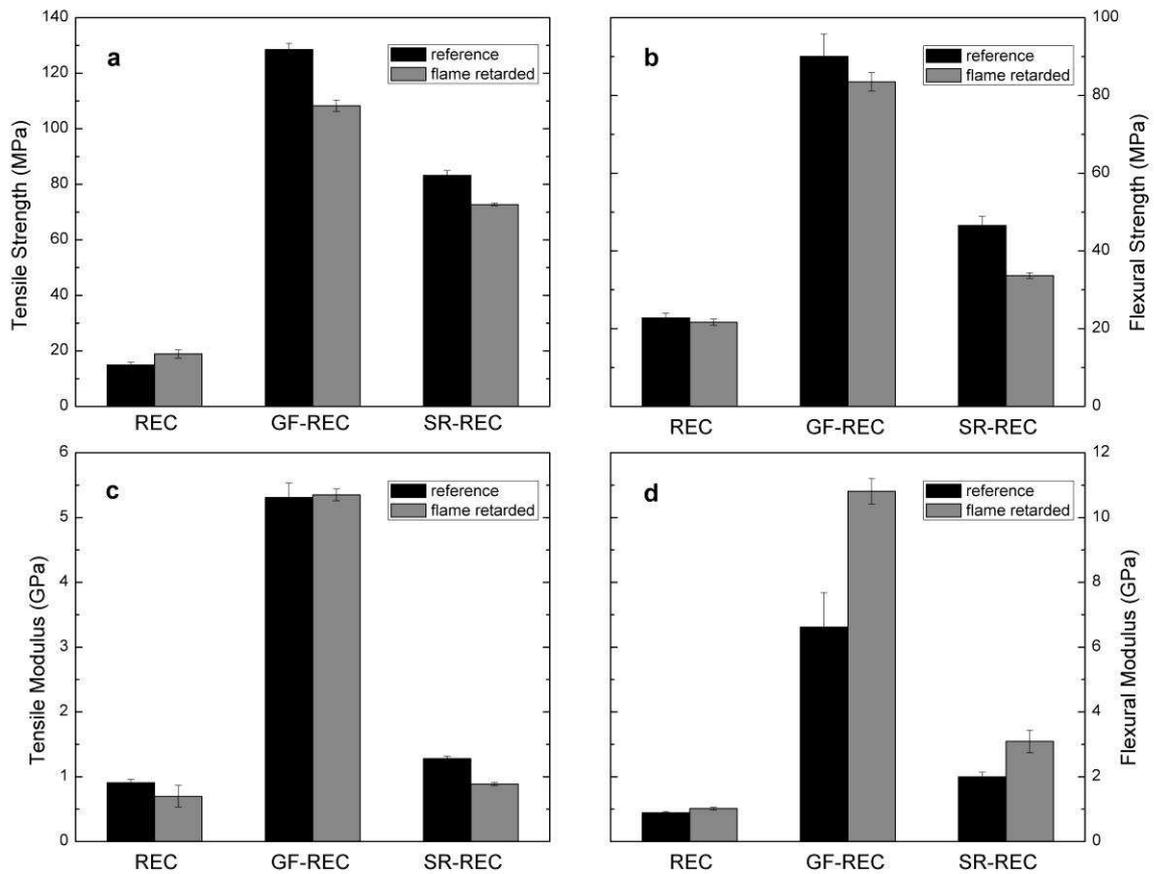


Fig. 1. Tensile and bending test results of the prepared recycled composites: (a) tensile strength, (b) flexural strength, (c) tensile modulus, (d) flexural modulus

The tensile and bending tests results are demonstrated by column charts in **Fig. 1 a-d**. It is quite clear that despite of the relatively low purity of the applied polyolefin waste for both glass- and self-reinforced composites (GF-REC and SR-REC) the tensile- and flexural strength of the polyolefin waste (REC-REF) could remarkably be increased. Although the original strip tensile strengths of the reinforcing fabrics are almost the same, the load-bearing capacity of the highly oriented PP tapes decreased during the heat treatment of the composite preparation (162°C for 2 min) leading to approx. 50% inferior tensile- and flexural strength values in the case of SR composites compared to GF composites (**Fig. 1 a and b**); but still, 5 times higher tensile strength and 2 times higher flexural strength could be achieved using reinforcing PP fabrics, than those of the unreinforced reference (allowing multi-fold recycling as well in this case). It can also be observed that the FR content of the matrix layers has only slightly decreased the prominent tensile- and flexural strength of the reinforced

composites. Consequently the prepared flame retarded recycled composites can also be suitable for several technical applications.

Considering the tensile and flexural moduli of the fabric reinforced composites (**Fig. 1 c and d**), it can be seen that the self-reinforced composites' moduli are almost as low as that of the reference matrix (REC-REF); meanwhile, the stiffness was significantly increased by glass fabric reinforcement. This is the expected consequence of the difference between the specific properties (stiffness, flexibility, etc.) of the applied reinforcing materials. Their selection and utilization depends on the application requirements. The different effect of the applied FR additive on the tensile- and flexural moduli (**Fig. 1 c and d**) of the composites can be explained by the dissimilarity of the two stress profiles i.e. bending involves some compression as well (**Fig. 1 d**).

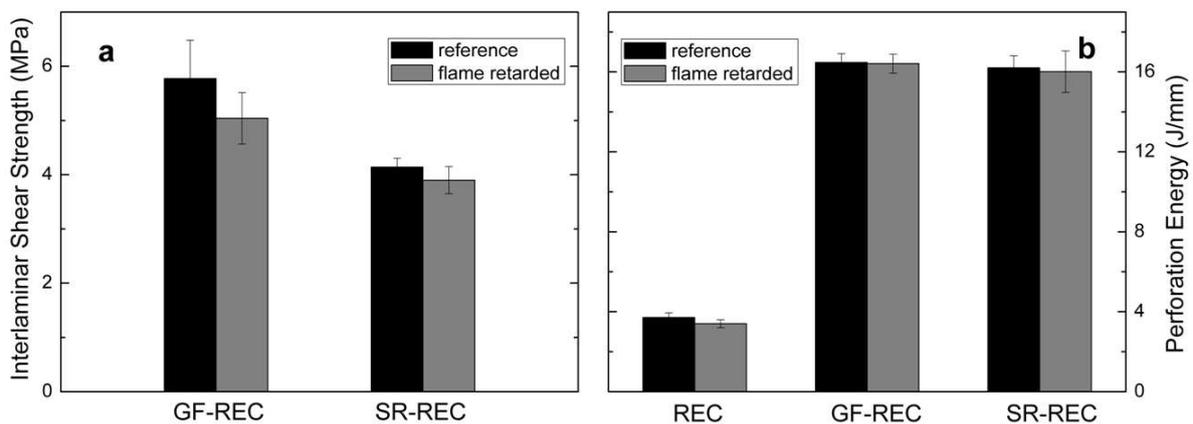


Fig. 2. (a) Interlaminar shear strength and (b) perforation energy of the prepared recycled composites

In order to investigate the adhesion between the layers of the prepared reinforced composites, their interlaminar shear strength has been determined (**Fig. 2 a**). The harmful effect of the FR additive on the adhesion between the recycled matrix and the glass fabrics has been manifested by the decrease of their interlaminar shear strength, as in the case of the self-reinforced composites it was concluded that the transcrySTALLINE layer, which is the key factor of the improved adhesion and consequently of the good mechanical performance [20,21], was not noticeably influenced by the added flame retardant. The lower initial interlaminar shear strength in the case of the self-reinforced

composites can be ascribed to the lower specific surface and thus lower contact area of the PP fabric than that of the glass fabric.

Photographs taken of the tensile and IFWI test specimens are shown in **Fig. 3** and **Fig. 4**, respectively. In the case of the self-reinforced composites (**Fig. 3 b,d** and **Fig. 4 b,d**) imperfect consolidation quality is reflected by slight delamination and tape pullout compared to the figures showing the GF systems (**Fig. 3 a,b** and **Fig. 4 a,b**), which is in close connection with their different interlaminar shear strengths [21] and also strongly influences their creep resistance [22]. The delamination process absorbs high energy [21,23], resulting in noticeable impact resistance (perforation energy), as shown in **Fig. 2 b**. In the case of the glass fabric reinforced composites just the opposite tendencies (less delamination, higher tensile-, flexural- and interlaminar shear strength) can be observed. However, these well-consolidated GF composites are not superior considering the IFWI test (**Fig. 2 b**), their perforation energy is about the same as that of the SR composites. The obtained high perforation energy (more than 4-times higher than that of the recycled reference matrix (REC-REF)) of the prepared multilayered recycled composites can be advantageous in many fields of application where shock-resistant protection (covers, equipment housing etc.) is required. The applied film-stacking method can be easily implemented in industrial scales by double belt press or roll forming technologies [24].

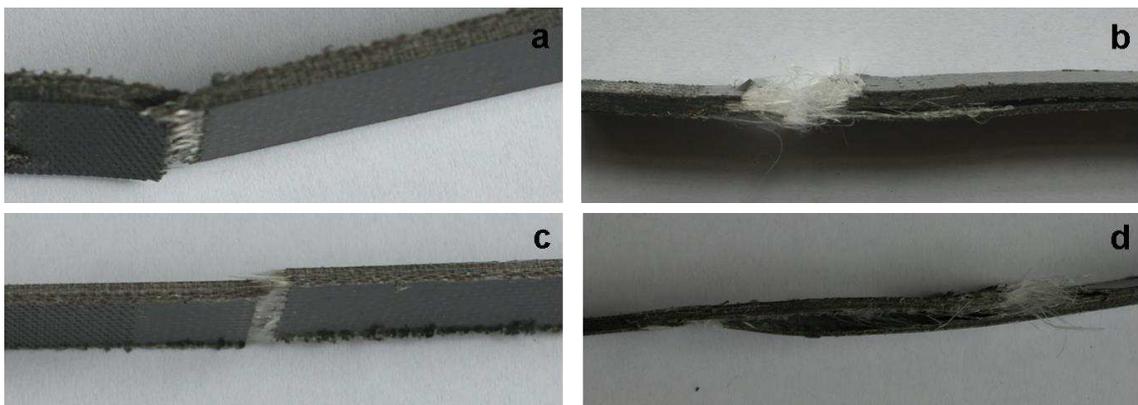


Fig. 3. Photographs taken of the tensile test specimens: (a) GF-REC-REF, (b) SR-REC-REF, (c) GF-REC-FR18 and (d) SR-REC-FR18

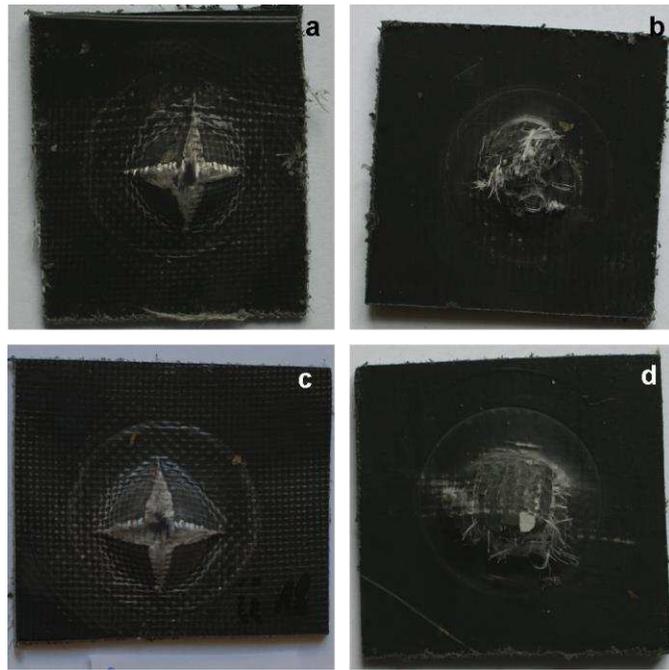


Fig. 4. Photographs taken of the IFWI test specimens: (a) GF-REC-REF, (b) SR-REC-REF, (c) GF-REC-FR18 and (d) SR-REC-FR18

3.2. Flammability

All the prepared flame retarded samples (multilayered composites and compounds without reinforcement) contain 18 m/m% FR additive (considering their total mass), thus relevant conclusions could be drawn on the bases of comparative flammability tests.

Sample	LOI	UL-94	Burning rate (mm/min)
REC	20	HB	31.5
GF-REC	21	HB	19.9
SR-REC	19	HB	34.6
REC-FR18	26	V-1	-
GF-REC-FR18	33	V-0	-
SR-REC-FR18	30	V-0	-

Table 1. LOI values and UL-94 ratings of the recycled composites with a flame retardant content of 18 m/m%

The results of the performed LOI and UL-94 flammability tests are summarized in **Table 1**. With 18 m/m% IFR content V-1 rating could be achieved in the case of REC-FR18 compound, which is in accordance with the fact that at least 20-22 m/m% IFR is required to achieve V-0 classification in PP [12]. Unexpectedly, the same IFR content proved to be sufficient for the V-0 rating when incorporated into multilayered composites (GF-REC-FR18 and SR-REC-FR18). It is also shown, that in spite of the same IFR content 7 and 4 v/v% higher LOI could be reached by preparing fabric reinforced composites. The lower flammability of GF-REC-FR18 can be explained by its high (ca. 48 m/m%) incombustible material (glass) content, which is also manifested by the low burning rate (19.9 mm/min) and relatively high LOI (21 v/v%) of the additive-free GF reference composite (GF-REC). Nevertheless, the favourable behaviour of the self-reinforced composite (SR-REC-FR18), which is entirely composed of combustible materials, is much more surprising and remarkable. This beneficial effect is assumed to be attributed to the physical interaction between the intumescent i.e. expanding structure and the highly oriented, consequently shrinking tapes. It is presumed that this system exposed to heat due to the shrinkage of the PP fabrics forms a special compact charred layer on the surface of the sample, where the vapourization of phosphorus compounds and the gas release of the nitrogen containing foaming agent occurs in a much smaller bulk, resulting in more effective fire extinction, as a consequence. In order to understand this phenomenon better, further investigations are in progress.

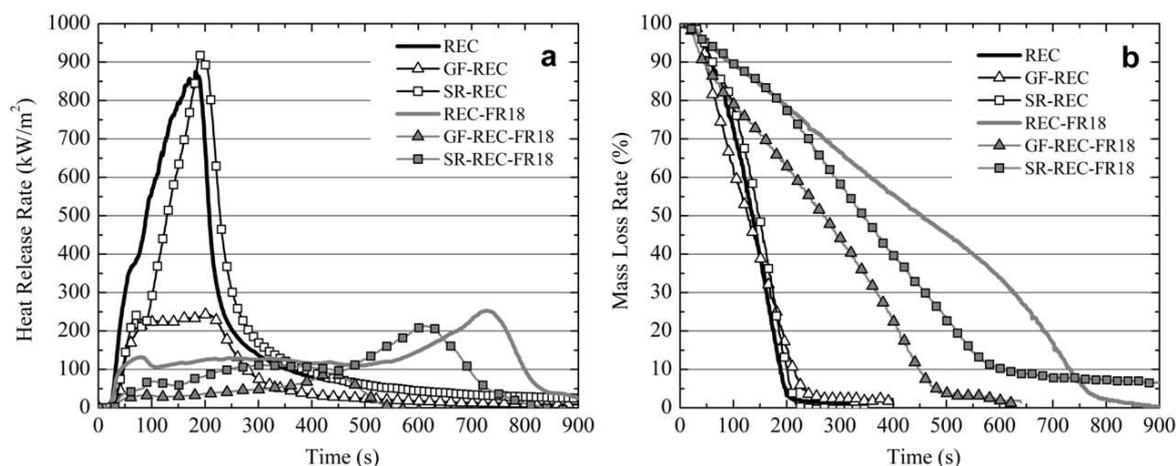


Fig. 5. (a) Heat release rate and (b) mass loss rate (of the combustible content) measured during the burning of flame retarded recycled samples

	Sample (g)	TTI (s)	pkHRR (kW/m ²)	pkHRR _{time} (s)	Time of burn (s)	Residue (mass%)	tHR (MJ/m ²)
REC	34.1	23	875.0	182	194	0.3	109.5
GF-REC	36.5	33	243.6	213	266	53.9	48.7
SR-REC	34.1	27	922.4	190	225	0.1	105.2
REC-FR18	39.8	24	253.6	728	841	3.7	107.6
GF-REC-FR18	35.8	26	90.9	435	515	59.3	23.0
SR-REC-FR18	36.9	25	213.1	613	740	8.1	78.0

Table 2. Results of Mass Loss type Cone Calorimeter measurements

(TTI: time to ignition, pk HRR: peak of heat release rate, pkHRR_{time}: time of peak heat release rate, tHR: total heat released)

The combustibility of the prepared recycled samples has been characterized under the conditions of a Mass Loss type Cone Calorimeter. The heat release rate (HRR) and the mass loss rate (MLR) curves of the examined specimens are given in **Fig. 5 a** and **b**, respectively. Significant reduction of the heat release rates of the recycled matrices can be observed in the cases of flame retardant and glass fabric containing samples. In connection with the protective effect of the formed heat insulating foams (as shown on photographs in **Fig. 6**) the measured pkHRR values were lowered with 71% in the case of the flame retarded matrix material (REC-FR18), with 63% in the case of the glass fabric reinforced composite (GF-REC-FR18), and with 77% in the case of the self-reinforced composite (SR-REC-FR18) when compared to the relevant reference samples. The quantitative results are summarized in **Table 2**. The remarkably lower heat release rate values (pkHRR and tHR) of the GF composites (GF-REC and GF-REC-FR18) compared to reference materials can not be explained just by their significantly lower (52%) combustible content. The significantly larger horizontal heat-conductivity of the glass fabrics containing composites has to be taken into account as well. As for the SR composite containing 18% IFR additive (SR-REC-FR18), the 16% reduction of the pkHRR can be ascribed to the compact surface structure of enhanced protective efficiency achieved by the shrinkage of the surface layer.

The mass loss rate data recorded in the case of glass fabric reinforced composites (GF-REC and GF-REC-FR18) have been corrected for providing better comparison. Thus only the mass loss rates of the combustible content are presented in **Fig 5 b**. (The real masses of the residues are shown in **Table 2**.) It can be seen that the combustible part of the flame retarded GF composite (GF-REC-FR18) burns out, due to the candlewick effect, faster than that of the SR-REC-FR18 sample i.e. glass fibres accelerate the transport of flammable mass to the burning area [15,23]. On the other hand, the charred residue that was detected in the case of flame retarded self-reinforced composite confirms the assumption that the shrinkage of the surface layer improves the protection capability of the of the IFR system. Comparing the released total heat (THR) of two materials with exactly the same composition even more remarkable difference can be found: the self-reinforced recycled sample (SR-REC-FR18) released 28% less total heat (THR) than the compound without reinforcement (REC-FR18). The difference is quite obvious when the photographs taken of the charred residues after combustion are compared (**Fig. 6**).



Fig. 6. Photographs taken of the charred residues of flame retarded samples obtained after combustion in Mass Loss type Cone Calorimeter

Based on the flammability test results of the flame retarded SR composite a special synergistic effect could be discovered between the APP based intumescent flame retardant additive and the highly oriented PP fabrics in the recycled matrix. This interaction resulted in improved flammability properties such as 4 v/v% higher LOI value, significantly lower intensity of burning and better UL-94 classification (V-0) than expected. Consequently, smaller amount of flame retardants are needed in order to achieve acceptable levels of flame retardancy, resulting in cost-effectiveness and higher market value of such flame retarded secondary products. In addition, it has to be highlighted that excellent mechanical properties can be guaranteed in the case of the recycled multilayered

composites with reduced flammability as well, thus the preparation of such composites can be a solution for the upgrading recycling of plastic waste.

4. Conclusion

In accordance with the increasingly strict European legislation, environmental and safety aspects of material development became relevant besides the economic efficiency. As a consequence the potential of those plastic industrial applications, where reduced flammability combined with reinforcement and recyclability are essential requirements, is continually increasing.

The reported work aimed at upgrading automotive shredder plastic waste by the development of flame retarded self-reinforced composites from density separated secondary polyolefin fraction. The mechanical and flammability properties of the prepared self-reinforced composites were compared to conventional glass fabric reinforced composites and to the secondary polymers.

The mechanical properties of the polyolefin waste have been successfully improved by applying reinforcing polypropylene fabrics: 5 times higher tensile strength, 2 times higher flexural strength and 4 times higher perforation energy were achieved. Although the tensile and flexural strength of the recycled matrix could be more effectively increased using glass fabric reinforcement, the prepared self-reinforced composites are given preferences from environmental aspects considering that their density is much lower, and in opposition to glass fibre reinforced composites, they are easy to recycle by simple reprocessing methods at the end of their life-cycle.

Significantly reduced flammability could be obtained by applying intumescent flame retardant additives and the prominent mechanical properties of the recycled multilayer composites have not been remarkably influenced by the FR content of their matrix layers. Special beneficial effect on the effectiveness of the applied phosphorus-based intumescent flame retardant additive was found in the self-reinforced system resulting in flame-extinguishing behaviour during horizontal and vertical burning (UL-94) test, LOI of 30 v/v%, furthermore in time shifted and by 75% reduced pkHRR during combustion. This novel phenomenon promoting the possibility of cost-effective fire retardancy is under further comprehensive investigation in our laboratory.

Based on the described results it can be concluded that products of high technical value can be obtained from secondary raw materials with the preparation of self-reinforced composites upgraded with flame retardancy. Considering that recently the scaling-up of fabric reinforced composite

preparation can be easily implemented, it is assumed that the safe, recyclable, self-reinforced polyolefin composites made of low-cost recycled materials could become especially attractive for the industry.

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