Review Article

Use of manufacture residue of fluidized-bed catalyst-cracking catalysts as flame retardant in recycled high density polyethylene

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\begin{abstract}
The Municipal Company of Urban Cleaning uses plastic lumber to manufacture urban furniture items, whose process generates a post industrial waste scrap that is recycled once again. The Fábrica Carroca de Catalisadores produces catalysts for oil cracking in fluidized bed. This production process creates an inorganic solid residue with prospective flame retardant property in polymeric materials. This inorganic residue is currently sent to licensed landfill. The goal of the present work was to test the use of this inorganic solid waste from the manufacture of fluidized-bed catalytic cracking catalysts as flame retardants agent in polymer–matrix composites of rHDPE. The materials were processed in a Haake internal mixer, and the rHDPE/inorganic residue composites were compounded in the 100/0, 80/20, 60/40 and 40/60 proportions, by weight percentage. The composite materials were characterized by: flammability tests, tensile tests; TG/DTG, DSC and SEM. The results of the horizontal firing tests performed clearly showed that the incorporation of inorganic residue into the polymer material fostered the increase on fire resistance. The TG curves showed that the thermal decomposition of the material occurred between 480 and 500 °C. The DSC curves of the residue showed a characteristic peak of alumina dehydration, below 300 °C. The tensile test results demonstrated that when 20% residue was added, the Young’s modulus of the rHDPE increased by about 17% but the tensile strength was similar to rHDPE. The incorporation of inorganic particles into the polymeric matrix resulted in a change in the behavior of the material – from ductile to brittle – as observed by SEM micrographs.

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\end{abstract}

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1. Introduction

The earliest applications of some polymeric materials were intended to replace materials from animal origin, such as celluloid used to replace ivory in the production of billiard balls, and Bakelite replacing turtle shell, horns, corals and nacre in the production of combs, cables, buttons, toys and bijoux. The so-called “plastic” earned the ecological appeal, despite their eminent fossil and nonrenewable origin [1]. Over time and after the discovery of new materials, other applications were developed. The production increased and the mass production greatly reduced the pricing of polymer materials, which became central to life in contemporary world. According to Dasari et al. [2], more than 50% of the world’s plastics production is used in short-life applications (packaging, electrical and electronic industry, disposable utensils, among others). On the one hand, these applications mean practicality and comfort for consumers, but on the other hand they contribute to the main current problem related to the use of polymeric materials: their inappropriate disposal. Because of their great chemical inertia, when discarded in nature polymeric materials take a long time to degrade, and tend to accumulate and/or be ingested by animals – which end up dying because they cannot digest such materials. Although most of the polymers can be somehow reused, in most countries the current infrastructure is unable to manage the reuse of such a large quantity of produced, consumed and discarded plastics. Thus, over time, the ecological image of early stages plastics applications became a non-sustainability image [1–3].

The development of plastic materials with different properties, able to replace traditional materials like steel and wood with the advantages of having lower cost, lightness, easy processability, has greatly contributed to the consumption growth of these materials. However, some of the more widely used polymers – such as polyolefins – are intrinsically more easily combustible than traditional materials. This is a disadvantage of polymeric materials in applications that may result in fire situations. This is a growing concern, as there are records of several claims involving high-impact fires involving polymeric materials, such as the recent event at the Grenfell Tower in London. There are a number of available products designed to reduce the flammability of plastic materials, called flame retardants [4–6].

The recycling of plastics, as well as any other type of waste material, is an important solution to reduce the consumption of non-renewable raw materials, and their disposal in nature. The American Society for Testing and Materials (ASTM) defines plastic lumber as a manufactured product having a plastic content greater than 50% by weight, with a rectangular cross-section with dimensions typical of industrialized wood products. Plastic lumber is a material produced from recycled discarded polymer waste, mainly high density polyethylene (HDPE) with the addition of fillers and additives, aiming to improve the properties of the recycled polymer material [7, 8].

Comlurb uses plastic lumber to make urban furniture items, whose processing generates scraps – a post industrial waste that is recycled once again [9].

The Carioca Catalysts Factory (Fábrica Carioca de Catalisadores – FCCSA) produces catalysts for the petroleum cracking in fluidized-bed. The production process uses several compounds, many of them with potential to act as flame retardants. It generates an inorganic solid residue, which therefore contains these same compounds, and has the potential to act as flame retardant in polymeric materials. This inorganic residue is currently sent to licensed landfill.

The goal of the present work was to test the use of this inorganic solid waste from the manufacture of fluidized-bed catalytic cracking catalysts (FCC catalysts) as flame retardants agent in polymer–matrix composites of recycled high density polyethylene (rHDPE) – plastic lumber. The materials were processed in a Haake internal mixer, and the rHDPE/inorganic residue composites were compounded in the 100/0, 80/20, 60/40 and 40/60 proportions, by weight percentage. The inorganic residue was characterized by chemical composition, granulometric analysis, surface area, apparent density, loss on ignition test (LOI), moisture content, scanning electron microscopy (SEM), thermogravimetry/derivative thermogravimetry analysis (TG/DTG) and differential scanning calorimetry (DSC). The composite materials were characterized by flammability tests (ASTM D635-10) [10], tensile tests (ASTM D638–14) [11]; TG/DTG, DSC and SEM.

2. Experimental procedures

2.1. Collection and processing of inorganic waste

The inorganic residue used in this work was kindly donated by the Fábrica Carioca de Catalisadores S.A. Company, and was derived from the effluent treatment system. More specifically, it is the filtering wet cake resulting from decanted material in the sedimentation basins of the treatment system. Since it is a waste that gathered effluents from various areas, the composition can vary over time, depending on the plant units that were operating and the types of products being produced in the factory. In order to eliminate effects of seasonality, the material was manually collected in plastic bottles (approximately 500 g each) and sealed with lids. The collection was performed on random days, over a year, one sample per month, between...
September 2015 and August 2016. All samples were collected in the form of a filtering cake at the exit of the filter-press, a doughy material with high moisture content. We used equivalent aliquots of wet cake, taken from each of the 12 collected flasks. The processing of the inorganic residue consisted of drying the wet cake in an Ethiktechology oven, at 120 °C for 24 h, in order to eliminate free (non-structural) water. Then the dried material was manually crushed using porcelain gral and pistil; the crushed material was sieved in a BERTEL ind. Metalúrgica Ltda. stainless steel sieve with nominal granulometry of 100 mesh/150 μm. We obtained around 500 g of dry sifted material.

2.2. Characterization of the inorganic residue

The free moisture of the original filter cake was determined using nickel capsules and oven drying (105 °C) to constant weight. We analyzed an aliquot of each of the 12 samples, and calculated their average. The weighings were performed in a METLER analytical balance model AE200. The determination of the moisture in the filter cake is important, because it is related to future calculations about the economic viability of the proposed process – of using the inorganic residue material in composites. The remaining total moisture in the dry and sieved material – LOI analysis – was determined by drying in a Carbolite CFW1200 oven-muffling, at 815 °C, for 1 h. The average bulk density (ABD) of the dry-sifted inorganic residue was measured by weighing the material in a flask of known volume, without compaction. The particle density of the dry-sifted inorganic residue was measured by pycnometry, in triplicate, using calibrated pycnometers. The materials were compounded in an internal mixer and, in order to comply with the established limits of the equipment, this property of the dry particulate material was needed, to allow the calculation of inorganic and polymeric material quantities used in each batch. The chemical composition of the dry-sifted inorganic residue was assessed, in order to confirm the presence of potential compounds and chemical elements that may reduce the flammability of the composites, when compared to the neat polymer. We performed the following characterizations: Sulphate and Carbon content, in a LECO CS200; Al₂O₃, SiO₂, Iron, Sodium, Titanium, P₂O₅ and RE₂O₃ (rare earth oxides) by X-ray fluorescence (XRF), in a Pananalytical AXIOS equipment, using the Omnian semiquantitative method. Granulometry, surface area and morphology of the material were characterized by the following tests: particle size distribution and mean particle size (MPS) by laser scattering, using a Malvern Mastersizer 2000 equipment; BET surface area using Micrometrics TriStar equipment; the morphology was observed by scanning electron microscopy (SEM) using a JEOL-JSM-6490 LV microscope, with 20 kV of beam acceleration voltage, using cryogenic fractured samples, metallized with gold before the analysis.

2.3. Processing of plastic waste

The plastic waste used in this work was kindly collected and provided by the Companhia Municipal de Limpeza Urbana – Comlurb, as part of a partnership with the Universidade Estadual da Zona Oeste (UEZO). The plastic residue contained plastic processing scraps of different sizes – some of them too rough for the job – as well as a small amount of sawdust and some metallic residue. The processing of the received material initially consisted of manual separation, in order to eliminate coarse materials and sawdust residues. Thereafter, the material was manually cut into smaller sizes, to allow insertion into the feeder of the internal mixer. Finally, in order to attract small metal residues, a magnet was introduced amongst portions of the cut material, but none was found.

2.4. Composites preparation

Composites with high levels of inorganic residue were prepared in the following rHDPE/inorganic residue proportions: 100/0, 80/20, 60/40 and 40/60 percentage by weight, as seen in literature [2,5,14–20].

The compounding of the described formulations was carried out in a Haake internal mixer Polylab OS Internal RheoDrive 4 model, using roller type and 170 °C temperature. We considered the assessed particle density of the inorganic residue (determined by pycnometry) and the typical value for polyethylene density of 0.96 g/cm³, in order to calculate the amount of plastic residue and dry sifted inorganic residue of each batch. In total, there should be around 50 g of each material after the compounding. Based on these calculations, the quantities of inorganic residue and plastic residue required to prepare the batches of each formulation were weighed separately on a METLER PE360 scale, and segregated into suitably identified plastic bags. Aiming to obtain four batches of each composite, we weighed approximately 200 g of each formulation.

2.5. Materials characterization

2.5.1. Flammability test

For the horizontal firing test, we prepared specimens according to the ASTM D635–10, by compression molding in a Marconi MA908/A400 heated hydraulic press (adjusted to 170 °C) and a MA908/R15 cooled hydraulic press (adjusted to 25 °C). We used 9 tons pressure in both presses. The heated pressing time was 7 min. The specimens were produced in the dimensions of 125 mm × 13 mm × 3 mm (length × width × thickness). We used a carbon steel mold, manually filled, capable of producing two pieces at a time. Ten specimens of each formulation were prepared. ASTM D635–10 standardizes the conditions and procedures used for the horizontal firing test. Ten specimens of each of the 100/0, 80/20, 60/40 and 40/60 formulations were tested. The marked and numbered specimens were conditioned for 48 h at temperature conditions of the test environment, prior to their completion. One of the marks was made 25 mm away from one end (to be burned), and the other 100 mm away from the other end (to be clamped). The distance between the two marks was 75 mm.

2.5.2. Tensile test

The specimens for the tensile test, standardized by ASTM D638–14, were made by injection molding in a Battenfeld PLUS3S injection molding equipment. The injection temperature was adjusted to 170 °C, with an injection pressure.
of 10 MPa, settling pressure of 7–9 MPa and mold pressure of 14 MPa. The injection rate was 40–55% and the cooling temperature of the mold was 25 °C. We tested seven specimens of each formulation in an EMIC DL3000 universal testing machine. The tests were performed at a speed of 50 mm/min. The 40/60 formulation test specimens could not be injected, because this composite tripped the injection molding machine.

2.5.3. Thermogravimetry/derivative thermogravimetry (TG/DTG)
The thermal stability of the samples of dry sieved inorganic residue and the composites (100/0, 80/20 and 60/40 and 40/60) was evaluated by thermogravimetry (TG/DTG) analysis. We used a NETZSCH equipment STA 409 PC Luxx model, with platinum pan, temperature ramp from 30 to 550 °C, heating rate of 10 °C/min and nitrogen atmosphere.

2.5.4. Differential scanning calorimetry (DSC)
The DSC analysis was performed using a NETZSCH equipment STA 409 PC Luxx model, with platinum pan. The samples were analyzed under nitrogen atmosphere, according to the following cycles: in the first cycle, the sample was heated from 30 to 550 °C, at a heating rate of 10 °C min⁻¹, keeping the material at 550 °C for 2 min; the second cycle was done using a cooling rate of 10 °C min⁻¹, until 30 °C; at the third cycle, the same temperature range and heating rate of the first cycle were applied (except for the 2 min isothermal); and at the fourth cycle, the same temperature range and cooling rate of the second cycle were applied. We considered the data of the second heating curves.

2.5.5. Scanning electron microscopy (SEM)
The SEM analysis was performed using a FEI Company microscope Inspect 550 model, over specimens coated with platinum applied by an Emitech equipment K550X model. Cryogenically fractured transversal sections of the samples were assessed, and the images were obtained in 500× and 1500× magnification.

3. Results and discussion

The average moisture content found in the filtering cake inorganic residue was 55.14%, which means that about 45% of the collected material is inorganic solid material, usable for the composites preparation. The high moisture content is a negative factor for eventual industrial use of this residue, because greatly increases the cost of drying.

Table 1 shows the results of the chemical composition, texture properties and grain size of the inorganic residue. As expected, the chemical composition indicated high content of silicas and aluminas, which may be present in the form of aluminosilicates (kaolin and zeolites). The presence of Titanium points out the possible existence of kaolin. The result of the rare earth oxides (RE₂O₃) content is an indicator of the presence of zeolites in the residue. Another indicator of the zeolites presence is the micropores volume value (MIPV) and total surface area (SA) [21,22]. The high contents of Sodium and Sulphates indicated of the salinity of the material, which classifies it as non-hazardous and non-inert waste for environmental disposal purposes. The LOI value at 815 °C showed that the dry material still contains considerable moisture content (free + structural). If this moisture is associated with alumina (in the form of alumina trihydrate), then we could have a potential component acting as a flame retardant. The presence of silica, alumina, phosphates, rare earths, zeolites and clays (kaolin), besides the very inorganic nature of the material, indicated that the material may act as a flame retardant when incorporated into a polymeric matrix [14,15,23].

The low bulk density (ABD) is in agreement with the low average particle size of 31 µm. This value is lower than the 45 µm cited by Lima [24] as being the maximum limit for incorporation into polymer–matrix composites. In fact, the smaller the particle size, the better the dispersion obtained in the processing. The mean particle density of the material was determined by pycnometry and the mean value found was 2.0 g/cm³.

Khumalo et al. [12] stated that synthetic boehmite can be suitably incorporated into polyolefins without any previous treatment. Zhang et al. [13] studied the properties of high

<table>
<thead>
<tr>
<th>Elements</th>
<th>Amount (%)</th>
<th>Texture properties</th>
<th>Particle size (µm)</th>
<th>%</th>
<th>LOI at 815 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>35.7</td>
<td>Total surface area (m²/g)</td>
<td>187</td>
<td>&lt;149</td>
<td>94</td>
</tr>
<tr>
<td>Na</td>
<td>7.10</td>
<td>MIPV* (µL/g)</td>
<td>0.034</td>
<td>&lt;105</td>
<td>86</td>
</tr>
<tr>
<td>Fe</td>
<td>0.35</td>
<td>Mean surface area (m²/g)</td>
<td>114</td>
<td>&lt;80</td>
<td>78</td>
</tr>
<tr>
<td>RE₂O₃*</td>
<td>4.06</td>
<td>Low bulk density (g/ml)</td>
<td>0.34</td>
<td>&lt;60</td>
<td>69</td>
</tr>
<tr>
<td>SiO₂</td>
<td>48.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>0.03</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>1.26</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SO₄</td>
<td>4.63</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>C</td>
<td>0.30</td>
<td></td>
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</tr>
</tbody>
</table>

* Rare earth oxides.
* Micro pores volume.
* Mean particle size.
* Loss on ignition test.
density composites and particulate alumina in concentrations up to 50% by weight without any compatibilizing agent. The preparations made in the present work successfully followed such indications, meaning no previous treatment and no compatibilizing agent added.

Fig. 1(a) and (b) shows images of the dry sifted inorganic residue obtained by SEM, with magnifications of 30× and 500×. Fig. 2(a)–(f) presents the SEM images of the rHDPE (100/0) and the 80/20 and 60/40 composites, with 500× and 1500× magnifications. We observed in Fig. 1 the presence of agglomerates in the dry sieved residue, which was manually triturated (low efficiency). However, the 31 μm mean particle size is suitable for use. This tendency to form aggregates was also observed by Kiyohara et al. [25]. Although the formulation 100/0 is 100% plastic waste, we noticed in Fig. 2(a) and (b) the presence of some dispersed solid particles. The fact that the material is recycled explains this presence, since the post-consumption disposal and recycling processes exposed the collected polymer to foreign materials. In addition, some additives might have been used to improve the reprocessing of the plastic lumber. In Fig. 2(a) and (b) we also observed the formation of “alveoli”, typical of ductile fracture. In Fig. 2(c)–(f) the fracture aspect is fragile, although discrete formation of alveoli can still be seen in the 1500× magnification. In both cases, 80/20 and 60/40 composites, the amount of inorganic material incorporated is relatively high, which makes the images very similar.

The TG curves of the dry sieved inorganic residue and the 100/0, 80/20, 60/40 and 40/60 formulations are shown in Fig. 3(a). Fig. 3(b) contains the DTG curves of these samples. In the 100/0 sample, the residue content at the end of the thermogravimetric analysis indicates that the recycled material already contained some impurities that do not decompose thermally, since the pure polyethylene decomposition would have left no residue. This conclusion is in agreement with the micrography of this formulation, and corroborates the statements of Spinacé and De Paoli [3] and Guimarães [26] about the presence of contaminants in post-consumer recycled materials – which is the case of the plastic lumber. The residue content at the end of the TG analysis of the inorganic residue sample (73.59%) means that this sample lost 26.41% mass by heating to 550 °C. The value found for the LOI (at 815 °C) of this residue was 23.4%. Therefore, the moisture present in the aliquot analyzed by thermogravimetry was higher than the one originally analyzed. This fact can be explained by homogenization problems of the residue sample, or by an eventual re-absorption of moisture in-between analysis. The values of the residue contents at the end of the TG analysis are compatible with the dosed residue levels in the samples of each formulation. The TG and DTG curves of the 100/0, 80/20, 60/40 and 40/60 formulations had the typical HDPE TG and DTG curves aspect, with T_{onset} in the range of 450–480 °C and T_{endset} between 500 and 520 °C; and with peaks in the DTG curves between 484 and 499 °C, the typical ranges for this material. The DTG peaks were as intense as the content of polymer material in the formulation. The DTG curve of the inorganic residue had a mass loss speed peak at 58.2 °C. This mass loss corresponds to free moisture. The TG curves of the formulations 100/0 and 80/20 were practically overlapping, indicating that lower inorganic material contents did not affect the thermal behavior of the polymer material. The curves of the 60/40 and 40/60 formulations differ from the 100/0 at temperatures below the T_{onset}, due to the effect of mass loss of the higher amount of incorporated residue.

Fig. 4 shows the DSC curves of the inorganic residue, rHDPE (100/0) and composites (80/20, 60/40 and 40/60). The DSC curves of the formulations 100/0, 80/20, 60/40 and 40/60 have endothermic peaks between 136 and 143 °C, corresponding to the absorbed heat in the melting of the polymeric material, compatible with the HDPE melting range. These curves also showed heat absorption at higher temperatures, with endothermic peaks between 480 and 500 °C, referring to the decomposition of the polymeric material in an inert atmosphere, i.e. pyrolysis of the organic material without the occurrence of ignition. The DSC curve of the inorganic residue showed an endothermic peak at 64.3 °C, referring to the absorbed heat in the free humidity evaporation, and endothermic peaks between 260 and 280 °C related to the absorbed heat in the alumina dewatering (structural water elimination) [17]. From the analysis of the DSC curves, we observed that the residual mass indicated continuous mass loss throughout the analysis and total loss of 26.4%, compatible with the result of LOI at 815 °C (23.4%). The main endothermic peak occurred
Fig. 2 – SEM micrographs of fractured surfaces of rHDPE/residue: (a) and (b) rHDPE (100/0); (c) and (d) 80/20 composite; (e) and (f) 60/40 composite.

at 64.3°C, ascribed to probable free (non-structural) moisture evaporation.

Regarding the results of the flammability tests, the specimens of the 100/0, 80/20 and 60/40 formulations burned to completion. In the case of the 40/60 formulation, only two specimens burned to the end, which indicated that this formulation is more resistant to fire spreading than the others. We could also assume that the processing of this composite did not achieve good homogeneity, which can be explained by the presence of high amounts of particulate material. The burning residue of rHDPE (100/0) had the appearance of molten material, while the burning residues of the formulations with embedded particulate material have the aspect of carbonaceous residue mixed with off-white material (the inorganic residue). According to the definition in Appendix X.1 of ASTM D635-10, if the firing front reaches the 100 mm mark, but its propagation speed falls below 40 mm/min, the material is classified as “HB category”. The results obtained in the tests of the formulations 100/0, 80/20 and 60/40 and 40/60 fit this criterion. Nevertheless, it can be seen in Table 2 that this speed decreased as we increased the content of inorganic material in the formulation. The 40/60 formulation had the lowest flame propagation speed, almost seven times less than the flame propagation velocity in the recycled polymer (100/0). These observations support the hypothesis that the inorganic residue has a flame retarding effect when
used in the preparation of recycled HDPE polymer–matrix composites.

The effects of the residue on the mechanical properties of the rHDPE and the composites are listed in Table 3. It was noticed that when 20% residue we added, the modulus of the rHDPE was increased by about 17% but the tensile strength was similar to rHDPE. These results are similar to those achieved for Lei et al. [27] from
rHDPE/pine using maleated polyethylene (MAPE) as a coupling agent. The mineral powder fillers tend to increase the Young’s modulus value if compared to the original polymeric material [4,12,15].

4. Conclusions

The results of the horizontal firing tests performed in the rHDPE and their composites (80/20, 60/40 and 40/60), clearly demonstrated that the incorporation of inorganic residue from the manufacture of FCC catalysts into the polymer material fostered an increased fire resistance of the composite, translated by the reduced rate of flame propagation as we increased the content of inorganic residue. These results can be explained by the very inorganic and incombusible nature of the composite residue (which reduced the total amount of combustible material present in the material), and by the presence of substances such as alumina, zeolites, rare earths and others in the inorganic residue, which gave greater resistance to fire propagation.

The thermogravimetric curves of the residue and formulations showed that the residue still contained free moisture, and that the thermal decomposition of the polymer material occurred between 480 and 500 °C, compatible with literature of HDPE.

The DSC curves of the residue showed a characteristic peak of alumina dehydration, below 300 °C, confirming its presence in the material. The DSC curves of the formulations containing polymer material showed endothermic peaks of melting and decomposition at typical HDPE temperatures.

The tensile test results demonstrated that when 20% residue was added, the modulus of the rHDPE increased by about 17% but the tensile strength was similar to rHDPE. The incorporation of inorganic particles into the polymeric matrix resulted in a change in the behavior of the material – from ductile to brittle – as observed by SEM micrographs.

For applications where the fire resistance – translated by the flame propagation rate – is the preponderant factor, the 40/60 formulation is the most appropriate. However, the physical properties may limit its application. Both effects – flame retardance and resulting mechanical properties – need to be considered when selecting applications for the composites produced by the joint processing of chosen residues.

Conflicts of interest

The authors declare no conflicts of interest.

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