



Short communication

Anti-spalling fibers for refractory castables: A potential application for recycling drinking straws

R. Salomão^{a,*}, V.C. Pandolfelli^b^a Materials Engineering Department, São Carlos School of Engineering, University of São Paulo, Avenida Trabalhador São-carlense 400, São Carlos, SP, Brazil^b Materials Engineering Department, Federal University of São Carlos, Rodovia Washington Luís, km 235, São Carlos, SP, Brazil

ARTICLE INFO

Keywords:

Drinking straws
Polymeric fibers
Recycling
Drying behavior
Refractory castables
Drying additives

ABSTRACT

Disposing plastic after using is an urgent worldwide issue, and recycling is usually pointed out as the best solution. Recycled thermoplastic polymers, however, in most cases, present inferior properties when compared to their as-produced equivalent grades. Re-exposure to processing temperatures and intense shear results in significant thermo-mechanical-oxidative degradation of the polymeric material. Typical consequences lead to a reduction in the average molecular mass and melting temperature, and modifications in the composition, microstructure and rheological behavior. Although such effects usually hinder using these materials in their original application, other technological uses can be found. In the present paper, a methodology used to produce thin continuous fibers from recycled polypropylene (PP) and high-density polyethylene (HDPE) drinking straws is presented. The processing parameters for melt-extrusion and chopping the fibers were set to produce staples of similar length and diameter as the conventional PP fibers currently employed as anti-spalling additives during the first heating of self-flow refractory castables. Because of their lower melting temperature and high melt flow index, recycled polymers cannot be used to produce new straws, however the fibers produced with them increased the castables' permeability at lower temperatures than those observed for the conventional ones. Therefore, besides helping the environment, the castables' overall risk of explosive spalling and total drying time can be successfully reduced.

1. Introduction

1.1. Polymeric fibers as drying-aid additives for refractory castables

Refractory castables are intrinsically low permeability materials due to their highly packed and low porosity structure [1–3]. Such features, which are useful for their thermo-mechanical behavior and corrosion resistance during service life [4], also make water vapor withdrawal difficult during the first heat-up [5–7]. Therefore, different types of polymeric fibers have been added to these materials compositions to reduce the explosive spalling likelihood [8–12]. The drying-aid behavior of polymeric fibers is based on the controlled increase of castables' permeability [12–15] and has two major requirements. Firstly, it must release large amounts of pressurized water vapor [16–18]. Secondly, it must be carried out at the lowest possible temperature, because water vapor pressure rises exponentially above its boiling point (100 °C) [19–23]. As a consequence, the higher the level of permeability increase generated and the lower the fiber melting temperature, the smaller the explosive spalling likelihood [15].

Polymeric fibers are usually dry mixed in castables' formulations and, during setting and hardening, the space occupied by them is preserved (Fig. 1a) [24,25], resulting in a large number of fibers per volumetric unity of castable. For instance, for cylindrical fibers measuring 20 µm diameter and 2 mm length and added to a high-alumina composition in volumetric amounts of 0.3 vol%, there are approximately 4750 fibers per cubic centimeter of castable and the average distance amongst two consecutive fibers is 60 µm [16,17]. Increasing fiber content or reducing its diameter enhances the total number of unities per volumetric unit of castable. During the first heat-up, fibers experience several thermal transitions that unblock the paths and connect low permeability matrix-aggregate interfaces, closed pores, as well as their canals with the surface of the solid structure [10–12]. The pressurized water vapor is then quickly released through these highly permeable paths, reducing the explosive spalling likelihood [21–23].

There are three main permeability-increase mechanisms associated with polymeric fibers. The first one uses hot-water soluble fibers, such as poly (vinyl alcohol) (PVAL) [23]. These fibers are inert during the castables' mixing step and dissolve during the initial stages of heating in

* Corresponding author.

E-mail addresses: rsalomao@sc.usp.br (R. Salomão), vicpando@ufscar.br (V.C. Pandolfelli).

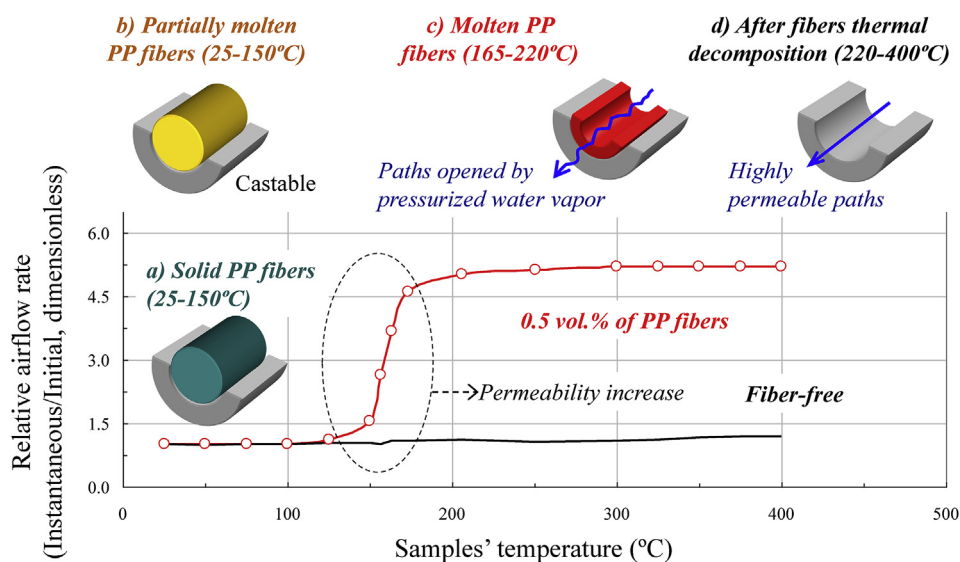


Fig. 1. Fluid dynamic behavior of polypropylene (PP) fibers containing refractory castables (hot-air permeametry) and schematic representation of fibers' permeability increase mechanism [12,13,15].

contact with the hot water present in the structure. The resulting polymeric solution is absorbed by the surrounding matrix leaving a partially empty canal. For the second one, natural cellulose-based fibers (such as jute and paper-pulp) swell in contact with water during mixing [19–21]. This expanded shape is preserved after setting. During drying, the fiber's diameters shrink, generating partially permeable paths along the fiber's length. An advantage of this mechanism is that it can be carried out even at room temperature and improves water withdrawal by evaporation. The third and most common mechanism is based on thermoplastic polymer-based fiber addition, such as polypropylene (PP) (Fig. 1a) [12,13,15]. During their melting ($T_{M,PP} = 165\text{--}170\text{ }^{\circ}\text{C}$), the polymer molecules become soft and deformable by the pressurized vapor flowing through the permeable paths (Fig. 1b) [12]. High-melting-point fibers (such as poly (terephthalate ethylene), PET, $T_{M,PET} = 235\text{ }^{\circ}\text{C}$; and polyamide, PA, $T_{M,PA} = 250\text{ }^{\circ}\text{C}$) require higher temperatures to result in permeability increase and, therefore, do not present a suitable behavior during the drying step [19,20]. For all three mechanisms, as heating proceeds, the remaining polymeric material decomposes generating fully accessible permeable paths (Fig. 1c) [12,24].

Recently, low molecular weight polyolefin-based copolymers were applied to produce fibers that melt between 70 and 90 °C and generate a permeability increase at approximately 95 °C [15,22,23]. Consequently, water vapor pressurization, the risks of explosive spalling and the total drying time were significantly reduced. These results suggest that other low-melting-point polymeric materials, which are easily degradable when heated could be used to produce drying-aid fibers. As it will be discussed in the next section, recycled plastics can be suitable candidates for such applications.

1.2. Challenges and opportunities in recycling plastic waste

Disposing of plastic after use is a worldwide concern [26–28]. Over the past decades, approximately 8 million volumetric tons of polymeric material-based waste was dumped every year at uncontrolled landfills and ended-up in rivers and oceans [26,27]. Recent studies showed that, in most cases, these materials originate from flexible film and packages, and deteriorate into microparticles and flakes after a few weeks of exposure to sunlight and water [27,29]. Due to their floatability, such particles can travel easily through continental distances causing irreversible environmental damage [29,30]. Therefore, there is an urgent need to reduce plastic consumption and search for processes and

applications that can reuse or recycle such materials and prevent them from being improperly disposed.

There are two concerns that make recycling most polymeric materials difficult. Firstly, preventing mixing different types of plastic waste [31–33]. Due to their chemical composition, some are incompatible and react at the molten state [32,33]. The second and most important one, is the physico-chemical changes observed after a second or third melting and shearing cycles [31,34,35]. The exposure of polymers to an oxidative atmosphere at high temperature and shear rate levels introduces chemically aggressive species (such as free-radicals and carboxylic side groups) that reduces their average molecular length and mass [31,34]. Therefore, recycled polymers present lower levels of rigidity, tensile and impact strength and transparency, as well as changes in color, melting flow index, and heat distortion and melting temperatures [35]. Due to this, in most cases, they must be used in different applications. For instance, PET-bottles, after recycling, are used to produce textile yarns and other disposable goods due to the loss of transparency and tensile strength [33].

The loss of performance is particularly deleterious for recycled PP and high-density polyethylene (HDPE) drinking straws [31,34,35]. These products are produced by blow-extrusion at a rate of approximately 180,000 ton per year⁻¹ (conservative studies report a daily consumption of 1×10^9 straws worldwide) [26,27]. The polymer grades used to produce them have a narrow distribution of molecular mass so they can present high elasticity in the molten state [36]. This feature is essential to ensure a stable elongational flow through the blow-die and to produce tubular films of suitable strength. When these straws are recycled, the high average molecular mass is reduced, and the polymers are no longer suitable for blow extrusion of thin films [35,37,38]. Besides this, because such grades are specifically synthesized for food and drink contact, they do not contain antioxidants and other protective additives that could facilitate recycling them [35]. Nevertheless, although used straws cannot be used to produce new ones, their low melting point and high melting flow index can be suitable features for producing drying-aid fibers for refractory castables.

In this study, thin fibers made of recycled PP and HDPE were manufactured from drinking straws by melt-extrusion and tested as drying additives for self-flow refractory castables. The extrusion parameters (die diameter and temperature, melt flow rate and stretching rate) were previously set to generate 40–80 μm diameter continuous fibers that were chopped as 3 mm staples and mixed with refractory castable raw materials. Permeability measurements (k_2 non-Darcy) were

Table 1
Physical properties of the straws and fibers tested.

Physical properties of the as-received straws	Straw type		
	Straws of PP		Straws of PE
Composition	Isotactic polypropylene (iPP)		High-density polyethylene (HDPE)
Melting temperature (°C)	170		148
Thermal decomposition (°C, under synthetic air atmosphere)	220–375		322–556
Solid density (g.cm ⁻³)	0.952		0.895
Length (cm)	22		22
Inner diameter (mm)	10		5
Average wall-thickness (μm)	300		200
Physical properties of the fibers produced and tested	Fibers' grades		
	PP	R-Straw-PP	R-Straw-PE
Composition	Unprocessed iPP	Recycled straws of iPP	Recycled straws of HDPE
Melting temperature (°C)	168	161	141
Thermal decomposition (°C, under synthetic air atmosphere)	218–370	194–357	320–521
Solid density (g.cm ⁻³)	0.951	0.945	0.895
Content added to the castable formulation (vol%/wt%)	0.36/0.067	0.36/0.065	0.36/0.061
Average diameter (μm)	23.5 ± 4.7	46.3 ± 11.4	83.4 ± 19.8
Initial length (mm)	4.3 ± 0.7	5.6 ± 0.8	4.7 ± 0.9
Length after mixing (mm)	3.4 ± 1.0	4.8 ± 1.8	4.5 ± 1.2

constant, before and after thermal treatment at 900 °C [6,39–41] and hot air permeametry (HAP) [12,41] results were related to the thermal characterization of the fibers (differential scanning calorimetry, DSC, and thermogravimetric analysis, TGA) [42,43]. Drying tests were conducted under continuous heating in freshly demolded humid samples [7,15,22,23]. A composition containing conventional PP fibers (that were produced using unprocessed material and have a similar diameter and length) and a fiber-free one were also tested as references.

2. Materials and methods

2.1. Preparation and characterization of fibers from discarded polymeric straws

Two types of polymeric drinking straws were collected from the authors' university cafeterias. Their average length was the same (22 cm), but with different inner diameters, wall thicknesses, composition and typical usage (Table 1). The thinner ones were made of high-density polyethylene (HDPE), were flexible and were used to drink water and soda. The larger ones, on the other hand, were more rigid, made of isotactic polypropylene (PP) and suitable for viscous beverages such as pulp juices and milkshakes. Ten disposed straws of each type were collected, washed five times in distilled water and dried at 80 °C overnight.

Four straws were flattened by hand (Fig. 2a–b), folded twice in half along their length (Fig. 2c) and placed inside a fifth one generating denser polyethylene or polypropylene rods (Fig. 2d). Each rod was inserted in a hot-melt glue-gun with temperature control (200 ± 10 °C, PKP18E, Bosch, Germany) (Fig. 2e). The molten polymers were forced through a 1 mm orifice die and collected by a rotating barrel (20 cm in diameter, operating at 10 rpm) aligned with the glue gun's nozzle. Due to the high speed at the surface of the collector, the semi-solid filament of molten polymer was stretched approximately ten times before total solidification. The polymeric rod feeding rate, the temperature of the glue-gun, and the rotating speed were previously set to produce thin continuous fibers with a diameter as close as possible to the commercial PP fibers used for the same purpose (typically 25 μm) [16–18]. The melt-spun fibers were manually chopped into 3–4 mm long staples using scissors (Fig. 2f).

The melting point and thermal degradation temperature range of the HDPE and PP straws and fibers produced and commercial fibers of PP (tested as reference) were determined by differential scanning calorimetry (DSC, Netzsch, Germany, 25–250 °C) and thermogravimetry

(TGA-Q50, TA Instruments, USA, 25–800 °C), respectively, at 5 °C.min⁻¹ heating rate, under synthetic air atmosphere [42,43]. Because castables' mixing is an aggressive step [25], fibers' diameters and lengths (before and after mixing) were measured using the images acquired by field emission scanning electron microscopy (FEG-SEM, Inspec-F50, FEI, Netherlands) and ImageJ software (at least 1600 measurements were carried out for each sample). Their solid density was measured by Helium pycnometry (Ultrapyc 1200e, Quantachrome Instruments, USA).

2.2. Preparation and characterization of polymeric fibers-containing refractory castables

A self-flowing high-alumina refractory castable was prepared dry mixing fine calcined alumina particles (24 wt%, D_{particle} < 100 μm, Almatis, Germany), coarse aggregates of electrofused white alumina (74 wt%, D_{Maximum} = 4750 μm, Elfusa Geral de Eletrofusão, Brazil), calcium aluminate cement (2 wt%, Secar 71, Imerys, France), the polymeric fibers (0.36 vol%) and a poly (ethylene glycol)-based dispersant (0.1 wt%, FS10, BASF, Germany). The particles' size distribution was previously designed using the Andreasen's particle packing model (q = 0.21) [44]. Following that, 4.5 wt% of distilled water was added to the composition and the homogenization step was carried out in a paddler mixer operating at 33 rpm for 180 s. The compositions were cast under vibration as cylinders of 70 mm diameter by 26 mm thickness for the permeability measurements, and 40 mm diameter by 40 mm thickness for the drying tests. A thin K-type thermocouple was placed 1 mm beneath the surface and at the half-height of the cylinders to measure the samples' temperature during the drying tests.

The curing step took 15 days at 10 °C under an environment of relative humidity close to 100%. These conditions were previously defined to produce structures of very low permeability and, therefore, of high likelihood to undergo explosive spalling during the initial heating [7,15,41,45]. Samples for permeability measurements and hot air permeametry tests were dried in silica gel for another three days at 10 °C to withdraw uncombined water. Those selected for the evaluation of the drying behavior were tested immediately after this curing period, just after demolding.

The effect of polymeric fibers on castable samples' permeability before and after thermal treatment (heating rate of 5 °C.min⁻¹ up to 900 °C, 5 h hold, cooling rate of 5 °C.min⁻¹) was evaluated using pressure *versus* flow rate data fitted over the Forchheimer's Equation (Equation (1)) [6,39–41]:

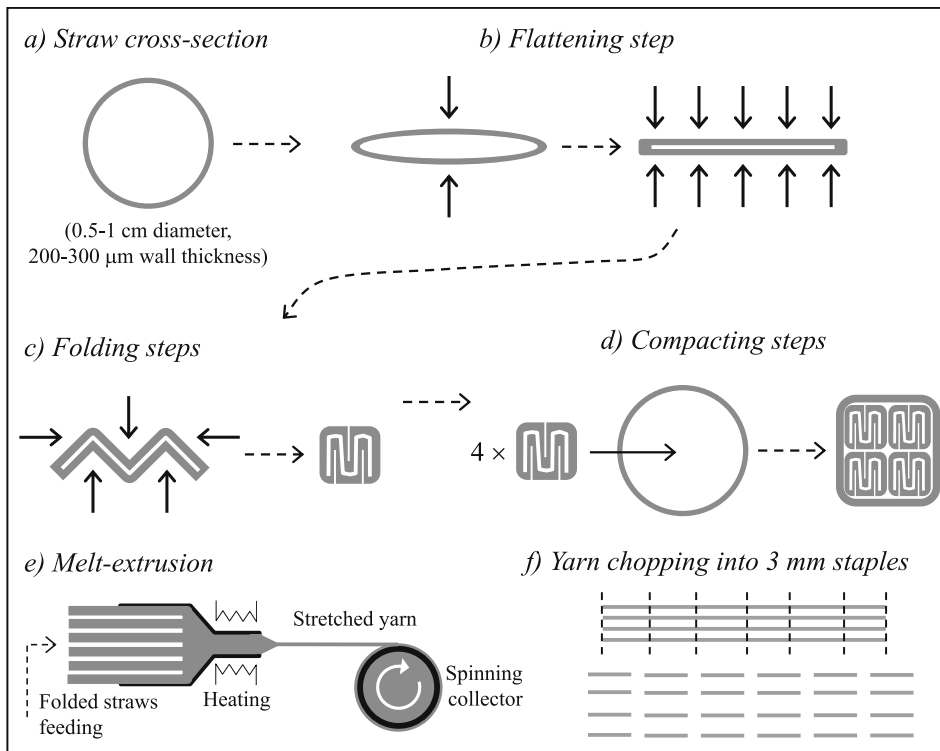


Fig. 2. Schematic view of the melt-spun process used to produce staples of recycled PP and HDPE straws.

$$\frac{P_i^2 - P_0^2}{2P_0L} = \frac{\mu}{k_1} V_s + \frac{\rho}{k_2} V_s^2 \quad (1)$$

where P_i and P_0 are, respectively, the inlet and outlet absolute air pressure at the sample; V_s is the fluid velocity, L is the sample's thickness; μ and ρ are the viscosity and density of air, and k_1 and k_2 are the Darcyan and non-Darcyan constants. Whereas the linear term ($\mu v_s/k_1$) of Forchheimer's equation represents energy losses due to viscous friction at lower flow rates, the quadratic one ($\rho v_s^2/k_2$) is associated to pressure drops caused by inertial effects and turbulence, when the fluid moves at a higher speed [40,41]. Both constants are useful tools to evaluate the impact of microstructural changes which occur in castable samples during thermal treatment. In this paper, the permeability variations were studied using the k_2 constant, which is more sensitive to changes in the tortuosity of the permeable paths such as those generated by polymeric fiber addition [12,16–18].

For the hot air permeametry (HAP) technique, permeability changes can be observed as a function of the temperature of thermal treatment and associated with effects such as polymeric fibers melting or dehydroxylation of hydrated phases of cement, for instance Refs. [7,12]. It is based on the airflow generated by a high-pressure gradient (0.3 MPa) throughout the sample's thickness under heating (20–400 °C, 5 °C.min⁻¹). Any permeability variation triggered by temperature induces an instantaneous change in the airflow rate.

The castables' drying behavior was assessed using thermogravimetric equipment that records mass variations simultaneously associated with water withdrawal and the temperature profile at the surface of the samples [7,15]. The mass loss (W , %) carried out during the initial heating (25–400 °C, 10 °C.min⁻¹ heating rate) and its time derivative (mass loss rate, %·min⁻¹) were calculated using Equations (2) and (3):

$$W(\%) = 100\% \times \frac{(M_0 - M_i)}{(M_0 - M_F)} \quad (2)$$

$$\left(\frac{dW}{dt}\right)_i = \frac{(W_{i+10} - W_{i-10})}{(t_{i+10} - t_{i-10})} \quad (3)$$

where M_0 , M_i , and M_F are, respectively, the initial mass of the sample just after demolding, the instantaneous mass recorded at a specific time t_i during the heating stage, and the final mass value collected at the end of the drying process. Detailed descriptions of the equipment and mathematical models used can be found elsewhere [7].

3. Results and discussion

3.1. Fiber characterization

The fibers produced from recycled PP and HDPE straws presented a small but significant reduction in melting and decomposition temperature ranges (Fig. 3 and Table 1) when compared to the correspondent original material. This behavior results from the exposure to high temperature and oxidative atmosphere during a second processing cycle [34,35,42,43]. The recycled PP and HDPE fibers presented a smooth cylindrical surface (Fig. 4) and were significantly thicker and

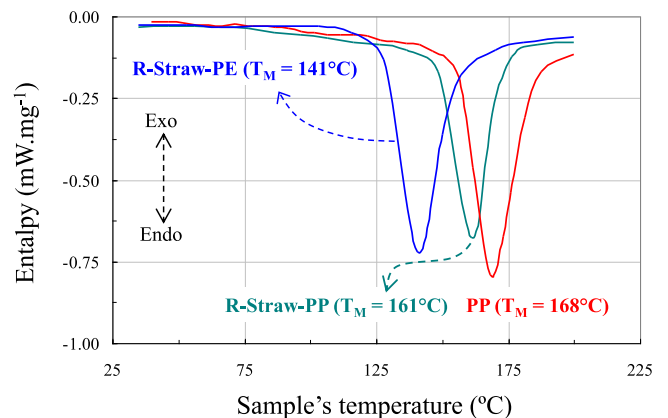


Fig. 3. Differential scanning calorimetry (DSC) analysis for the fibers produced (commercial fibers produced with unprocessed PP were also tested as a reference).

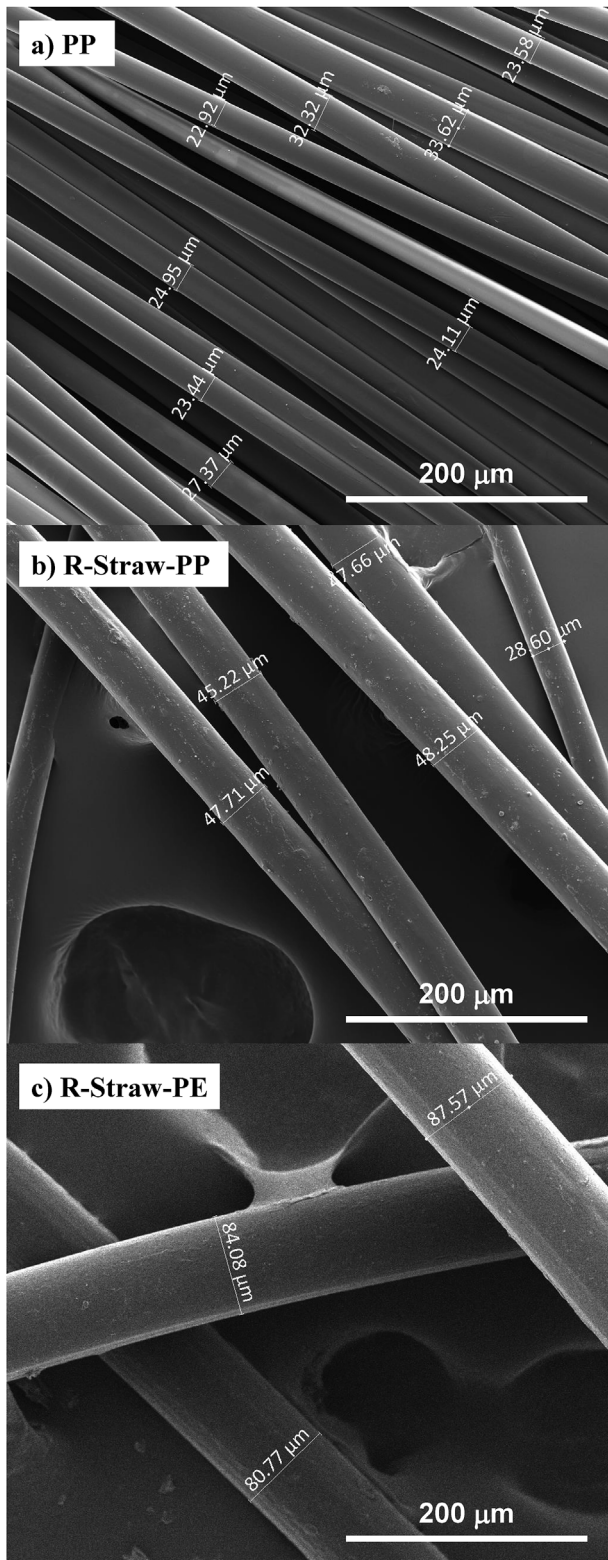


Fig. 4. Scanning electron microscopy images of the as-produce fibers (before mixing with castables raw materials).

longer (before the mixing) than those produced commercially from as-received PP and tested in this study as a reference (Fig. 5). Such a difference in geometry occurred because the equipment and processing parameter used in melt-spinning and chopping the continuous yarn were laboratory-scaled and, therefore, not as reproducible as the industrial ones [22,37,38]. The length and diameter measurements

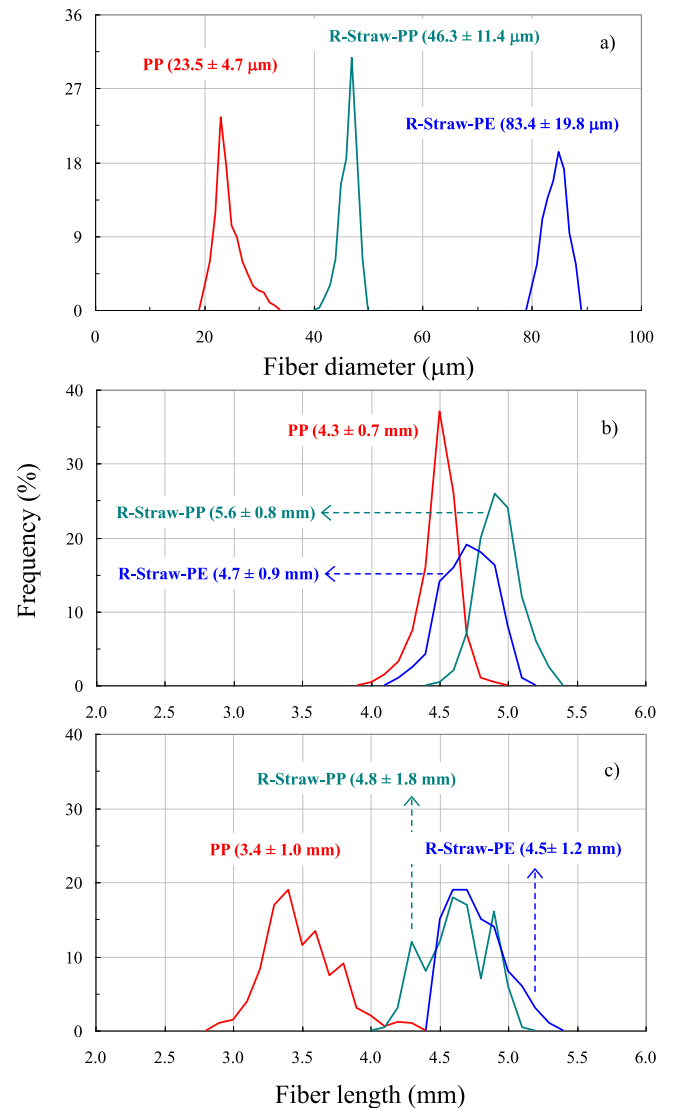


Fig. 5. Measurements of fibers a) diameter and length b) before and c) after mixing with castables raw materials.

carried out after mixing indicate that all fiber grades survived the intense shear and friction and presented a morphology able to result in a significant permeability increase during the first heating (diameter below $100\ \mu\text{m}$ and length above $2\ \text{mm}$) [16–18].

3.2. Permeability and drying behavior of polymeric fibers-containing castables

Fiber-free high-alumina refractory castables present a typical drying behavior where the water initially present in their composition is withdrawn in three-sequential steps (Fig. 6a) [5,7,45]. From room temperature up to $100\ ^\circ\text{C}$, water is removed from outer layers of the material as evaporated vapor, at lower rates and without significant pressurization of the structure [7,45]. Between $100\ ^\circ\text{C}$ and $250\text{--}300\ ^\circ\text{C}$, water is above its boiling point, and a large amount of pressurized vapor is formed [7,45]. Depending on the structure's permeability, the tensile strength generated can cause explosive spalling. Based on that, explosions and other mechanical damage usually occur at this temperature range [7,15]. Above $300\ ^\circ\text{C}$, the hydrates of the hydraulic binder begin to decompose, slowly releasing small amounts of vapor that are usually not critical for the drying process [7,46].

Adding fibers did not affect the samples' permeability before

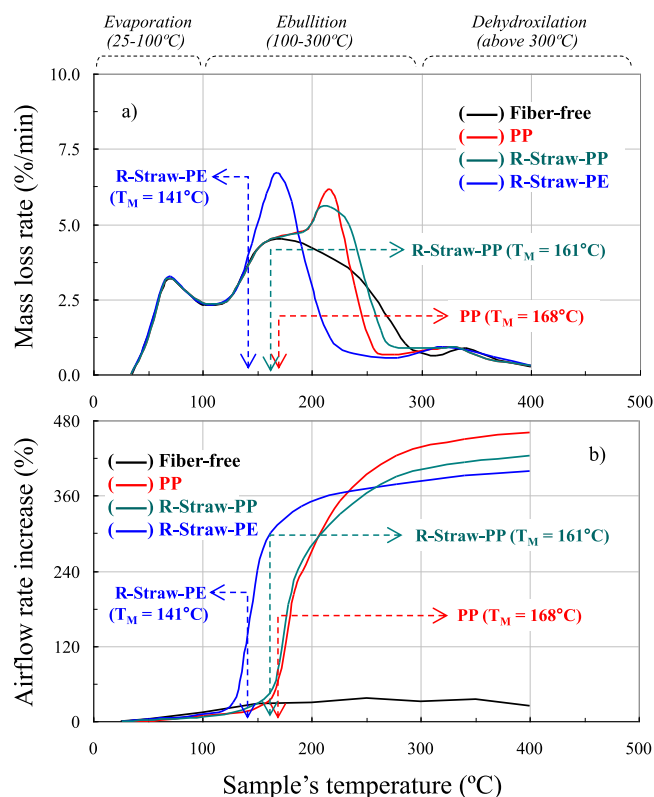


Fig. 6. a) Drying and b) fluid dynamic behavior of refractory castables containing the fibers produced from unprocessed polypropylene (PP), from recycled PP straws (R-Straw-PP) and recycled HDPE straw (R-Straw-HDPE) (the fiber-free composition is also shown as a reference).

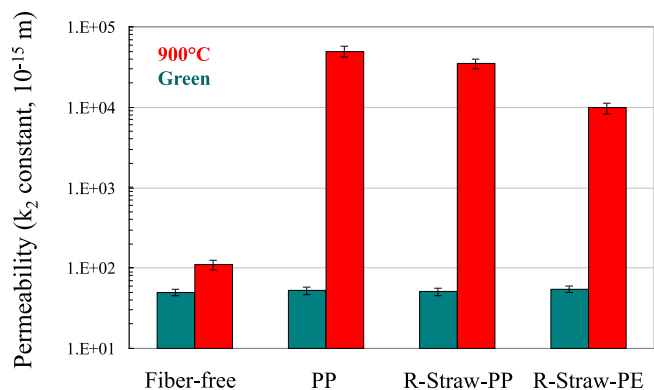


Fig. 7. Permeability measurements (non-Darcyan constant, k_2 , before and after thermal treatment at 900 °C, 3 h) for refractory castables containing the fibers produced from unprocessed polypropylene (PP), from recycled PP straws (R-Straw-PP) and recycled HDPE straw (R-Straw-HDPE) (the fiber-free composition is also shown as a reference).

thermal treatment (Fig. 7) and the drying behavior up to 144–170 °C (Fig. 6b), because before melting them, thermoplastic polymer fibers such as HDPE and PP perform as solid bodies clogging the permeable paths [19,20]. On the other hand, the similar levels of permeability of the green compositions is an indication that no processing defects, such as trapped air bubbles of fiber clusters, were generated during casting [13,25].

When compared to the fiber-free reference, the compositions containing PP and R-Straw-PP fibers presented significant and similar air flow rates, and their drying rate increased at approximately 170 °C. These effects can be associated with these fibers' melting temperature at 161 °C and 168 °C (Fig. 3), respectively, and the resultant permeability

increase generated as they decompose above 194 °C and 218 °C (Table 1) [12]. The small but noteworthy differences regarding the maximum airflow and drying rates attained occurred because these fiber grades present close melting points but different average diameters and lengths after mixing (Fig. 5) [16–18]. As reported elsewhere, for the same volumetric amount added to the composition, thinner and longer fibers result in a more significant permeability increase as a greater number of highly connected permeable paths are generated [18,24].

For the composition containing R-Straw-PE fibers, on the other hand, the airflow rate increased at a much lower temperature (at 144 °C) as these fibers melt at 141 °C. Consequently, because the permeability increase was carried out at the beginning of the water vapor generation, this sample presented the highest drying rates and the shortest total drying time, despite the lower permeability level after thermal treatment at 900 °C (Fig. 7).

These results present great technological relevance due to two aspects. Firstly, it was shown that recycled straws could be used as feedstock to produce drying additives for refractory castables. They showed similar or better performance when compared to the conventional fibers produced from unprocessed polymers. Secondly, due to the lower temperature permeability increase, the total drying time during castables' first heating can be significantly reduced, as well as the risks of explosive spalling.

3.3. How many straws can be recycled per year in this application?

According to recent studies that monitored North American coastlines over the past decade, plastic straws correspond to less than 0.25 wt% of the total mass of plastic waste dumped into the oceans [27,28]. Because they are a non-necessary disposal good, many cities in different countries developed programs to ban or significantly reduce their use [26]. Despite the lack of reliable data about their production, the straw consumption worldwide in 2018 was approximately 1×10^9 unities per day, which corresponds to an annual mass of 182,500 tons (for an average mass of 0.5 g per straw times 365 days) [26–28]. In the same year, the production of monolithic refractories in Japan, for instance, was approximately 700,000 tons [47]. The addition of 0.1 wt% of fibers of recycled straws to these materials would consume only 700 tons of them per year. Similarly, even if the estimated entire world annual production of pre-shaped and monolithic refractories were considered (approximately $8\text{--}10 \times 10^6$ tons [48]), it would require almost 20 years of refractory consumption to use the straws produced in a single year.

On the other hand, low melting point polymeric fibers can also be used to protect civil construction castables from spalling during fire accidents. As these materials contain approximately 20–30 vol% of water chemically bonded due to Portland cement hydration and they can be rapidly heated up to 400–1000 °C when exposed to open flames, explosions triggered by vapor pressurization constitute a significant concern [10,11]. As the 2008–2018 worldwide annual production of civil concrete was approximately $5\text{--}30 \times 10^9$ tons [49–51], it would require $5\text{--}30 \times 5.10^6$ tons of recycled fibers, which is roughly 30–160 times greater than the annual production of straws. Such a difference also suggests that other sorts of plastic waste could be used for this application as an alternative for their recycling.

4. Conclusions

Recycled polypropylene (PP) and high-density polyethylene (HDPE) from used drinking straws were used to produce continuous thin filaments. After chopping and mixing with a self-flowing refractory castable formulation, fibers' staples presented suitable geometry (thinner than 100 μm diameter and longer than 2 mm) to generate a net of interconnected permeable paths throughout their structure. Due to their lower melting point and decomposition temperature range

(compared to the correspondent as-received materials), these fibers resulted in earlier permeability increases in refractory castable samples during their first heating, reducing the total drying time and the overall risks of explosive spalling. The most significant technological impacts of such benefits are the reduction of energy and time consumption during the installation of refractory castables and the possibility of finding a better way of disposing of plastic waste using recycled material for such applications. The results attained also suggest that, besides using as drying additive for refractory castables, these fibers can also be used in civil construction concretes to prevent explosive spalling during fire situations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors acknowledge the Brazilian Research Foundations FAPESP (2002/00378-9; 2010-19274-5; 2017/06738-2; 2018/19773-3) and CNPq (305877/2017-8) for supporting this research, and Almatis (Brazil and Germany), Elfusa Geral de Eletrofusão (Brazil) and Imerys (France) for kindly supplying raw material samples. They are indebted to the Electron Microscopy Laboratory of Advanced Materials Research Support Center, EESC/IFSC for the SEM images. They also declare that, to the best of their knowledge, no competing interests (financial or personal) affected the results reported in this paper and that they cited all funding and supporting sources.

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