EFFECT OF THE POLYMERIC FIBRES ADDITION ON THE PERMEABILITY OF CERAMIC CANDLE FILTER

A.L. Souza[1], *L. Simão*[2], *F. Raupp-Pereira*[1], *S. Arcaro*[1], *M.D.M. Innocentini*[2], *O.R.K. Montedo*[1]

[1] University of the Extreme South of Santa Catarina, Av. Universitária 1105, Criciúma (SC), Brazil, okm@unesc.net

[2]University of Ribeirão Preto, Av. Costábile Romano 2201, Ribeirão Preto (SP), Brazil

Abstract: Adding polymeric fibres to ceramic filters is an efficient way to increase their permeability substantially. In this study, the effect of polymeric fibre content added to a ceramic composition was evaluated, and the permeability was analysed. The powder was prepared with 30 wt.% plastic clay, 11 wt.% kaolin, 34 wt.% feldspar, and 25 wt.% limestone and polymeric fibres (23, 24.5, and 26 vol.%). This formulation was extruded, fired at 1050 °C and characterised with respect to apparent porosity, water absorption, compressive strength, and microstructure. The formulation containing 26 vol.% of polymeric fibres showed apparent porosity and water absorption of approximately 50%, compressive strength of 15 MPa, k_1 of $3.78 \times 10^{-14} \pm 0.24 \times 10^{-14}$ m^2, and k_2 of $2.69 \times 10^{-10} \pm 0.42 \times 10^{-10}$ m. Thus, the obtained tubular filters are applicable to aerosol filtration.

Key-words: Fibres, ceramics, porosity, permeability, filters.

1. INTRODUCTION.

The requirement to improve the living conditions of the population and the performance of the industries, aligned with the sustainable development goals (SDGs) of the United Nations Organization (SDG 3 Good health and well-being, SDG 9 industry, innovation, and infrastructure, SDG 11 sustainable cities and communities, and SDG 12 responsible consumption and production), in addition to meeting the regulatory and legislative requirements, has encouraged research and development in different areas, such as the reduction of pollutant particles emitted into the atmosphere, soil, and water [1,2].

Filtration is a process for separating particles from a fluid, where the filter medium can consist of a polymeric, metallic, ceramic, and composite material, each possessing various physicochemical and mechanical characteristics [1,3].

In some filtration ranges (for example, microfiltration), the permeate fluid flow rate is primarily dependent on the microstructural characteristics of the porous medium, such as its thickness, pore size, and porosity, thus affecting its permeability [4,5].

In the production stage of the filter membrane, one of the attributes to be analysed is permeability, which refers to the dynamic interaction between the fluid and the porous medium when the fluid flows through it. This causes a variation of energy between the inlet and outlet of the filter, often measured in pressure drop, which can be described by the Darcy's law and considers the characteristics of the fluid and filter medium [1,4].

Studies [6-8] reveal that adding polymeric fibres to ceramic compositions can create a network of intercommunicating pores in the matrix after the fibre decomposition stage and provide advantages over other materials, which can also be used for this purpose. One advantage is that the fibres present an elongated geometry, which increases the probability of interconnecting pores and interfacial zones with each other and improves the porosity of the material. Another advantage is that the decomposition of the fibres results in less tortuous permeable paths, decreasing the flow resistance. Both characteristics favour the increase of permeability [-6-8].

In recent years, ceramic filters have increasingly been used because of their resistance to temperatures above 1000 °C and low production and maintenance costs, despite the difficulty in obtaining their commercial data. Among the various technological applications of ceramic filters, we have highlighted the use of domestic, hospital, and industrial waste in incineration and catalytic cracking, metal refining, diesel combustion in automotive vehicles, and hot-gas filtration [1,9-11].

In an ideal model, filters should offer minimum resistance to fluid drag; commercial filters with a porosity between 70 and 90% can present mechanical resistance between 0.5 and 2.0 MPa [1,12].

Thus, the main objective of this study is to analyse the influence of polypropylene fibres on the permeability of low-cost porous ceramic samples (total porosity greater than 50%) that shows adequate mechanical resistance for microfiltration at high temperatures (up to 1000 °C).

2. MATERIALS AND METHODS.

A ceramic powder composed of plastic clay (40 wt.%), kaolin (15 wt%), and feldspar (45 wt.%) (Colorminas Colorifício e Mineração, Brazil) was prepared. This composition was wet milled (40 wt.% water) in a ball mill (Servitech, Brazil) for 24 h at 300 rpm. The suspension obtained was sieved on a 35-mesh sieve (<500 µm) and dried in an oven at 100 °C for 24 h. The powder obtained was then crushed using a mortar to add limestone.

Separately, limestone (Colorminas Colorifício e Mineração, Brazil) was wet milled (40 wt.% water) in a ball mill (Servitech, Brazil) for 24 h at 300 rpm and subsequently in a high-energy planetary mill (Retsch PM 100, Germany; 450 rpm for 35 min. The suspension was passed through a 500-mesh sieve (<25 µm) and separated, presenting an average particle size of 1.8 µm, evaluated using a laser particle size analyser (Cilas 1064, France). Then, the suspension was dried in an oven at 100 °C for 24 h, and the obtained powder was crushed using a mortar. The final ceramic composition was obtained by mixing 25 wt.% of limestone powder and 75 wt.% of previously obtained ceramic powder.

To this ceramic composition containing the limestone, polypropylene microfibers were added (FLINCO; Brazil; 20 µm x 500 µm) at different volume percentages: F1 (0.0 vol.%, reference), F2 (23.0 vol.%), F3 (24.5 vol.%), and F4 (26.0 vol.%). Isopropyl alcohol was added to each formulation containing polypropylene fibres at a 3:2 alcohol-to-powder weight ratio to promote fibre dispersion in a Y-type agitator at 30 rpm for 1 h. The mixtures were subsequently dried in an oven at 45 °C for 24 h, crushed in a mortar and passed through a 35-mesh sieve (<500 µm) for deagglomeration. The adequate dispersion of the fibres in the ceramic powder was confirmed under an optical microscope (Olympus BX41M-LED, Japan).

Polyvinyl alcohol (PVA) solution with a concentration of 9.1 wt% was added as a binder to formulations F2, F3, and F4 at contents of 5.0, 5.5, and 6.0 wt.%, respectively. Each powder was dried at 80 °C for 3h, humidified using 8 wt.% water, passed through a 35-mesh sieve (<500 µm) for granulation and kept for at least 24 h in an adequately closed container for homogenisation.

The powders were pressed into a disc design (4 mm × 60 mm) using a hydraulic press (Gabbrielli Press 110 T, Italy) at a specific pressure of 60 MPa.

The pressed specimens were heat treated in a rapid-firing furnace (FORTELAB QR 1300/3, Brazil) at a heating rate of 3 °C/min up to 300 °C and 5 °C/min up to 950 °C and a plateau of 5 min. The maximum firing temperature was determined by evaluating the linear shrinkage curve as a function of the temperature obtained in an optical dilatometer (MISURA HSM ODHT, Italy).

The selected formulation (F4) was subsequently extruded in a vacuum extruder (CT-083/1, Brazil) to obtain tubular-shaped specimens. The firing occurred at a heating rate of 3 °C/min from room temperature to 300 °C and 5 °C/min from 300 °C to 1050 °C with 5 min landing time.

The fired tubular specimens were characterised as a function of water absorption (HA), and apparent porosity (P) through the boiling test performed following ASTM C20 - 00/2015. The compressive strength was measured in a universal mechanical testing machine (EMIC DL 10000, Brazil), while the microstructure of the fractured samples was evaluated in a scanning electron microscope (ZEISS EVO MA10, Germany).

The permeability was evaluated using a permeameter shown in Figure 1 [9,10]. The air permeability test was performed using a permeameter with cylindrical samples submitted to direct airflow filtration at a steady state. The sample was fixed and sealed in a cylindrical sample holder, as shown in Figure 1.

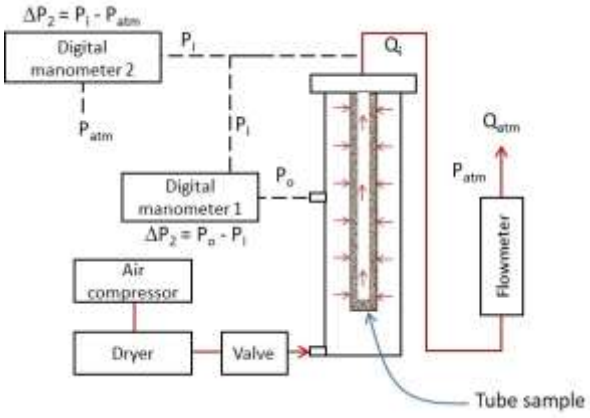

Figure 1. Schematic diagram of the permeameter used to measure the permeability of the tubular ceramic samples.

Air dried in silica gel was permeated into the porous medium with the help of a compressor. The air surface velocity (v_s), as well as the inlet (P_e) and outlet air pressures (P_s) were measured. the Forchheimer equation for compressible flow (Eq. 1) was used for the correction of the Darcian (k_1) and non-Darcian (k_2) permeability constants:

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} v_s^2 \qquad \text{Eq. 1}$$

where L is the sample thickness, and µ and ρ are the absolute viscosity and air density, respectively. The permeability constants k_1 and k_2 represent the properties of the porous medium regardless of the type of fluid or the flow velocity.

The ΔP is obtained using Eq. 2:

$$\Delta P = \frac{P_e^2 - P_s^2}{2P_s} \qquad \text{Eq. 2}$$

After passing through the sample, the resulting volumetric flow rate was adjusted using a valve and measured using a rotameter. A digital manometer measured the air pressure drop with outlets before and after the sample. The pressure drop in each set flow rate was measured. Ten pairs of flow and pressure data were collected, and the flow rate (Q) was converted to air surface velocity (v_s) using the flow area (A) ($v_s = Q/A$).

The flow and pressure drop data were treated according to the Forchheimer equation (Eq. 1) for compressible flow, allowing the permeability constants k_1 and k_2, which are properties of the porous medium and do not depend on the type of fluid or the flow velocity.

3. RESULTS AND DISCUSSION.

To obtain an appropriate firing temperature for the samples, with the best relationship between permeability and mechanical resistance of the ceramic filter, the thermal analysis of optical dilatometry was performed, as shown in Figure 2.

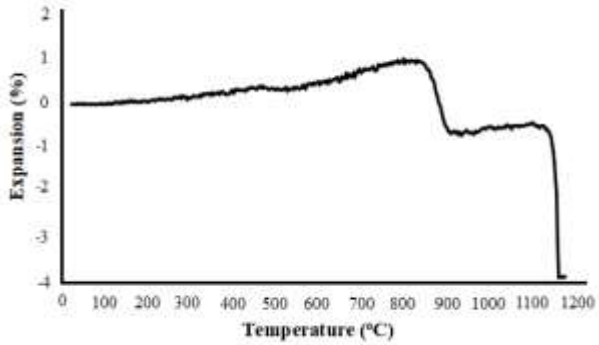

Figure 2. Thermal expansion in relation to the temperature of pressed F4.

Figure 3 shows that the thermal expansion of the sample occurs at a temperature range of 25 to 825 °C due to the expansion of the material and decomposition of calcium carbonate [13]. Between 825 and approximately 910 °C, a strong shrinkage was observed in the material due to the sintering process. Although sintering continues to occur at this temperature range, between approximately 910 and 1120 °C, an expansion of the material is observed, contributing to an increase in permeability. Thus, based on Figure 2, the temperature of 1050 °C was selected for the firing of the samples, since above 1100 °C the ceramic composition presents significant shrinkage, leading to a reduction in the porosity and, consequently, in the permeability of the material.
Figure 3 shows the result of P in relation to the volumetric content of polymeric fibres. The P increases with an increase in the volumetric content of polymeric fibres due to the formation of a network of interconnected voids (pores) [12]. The same behaviour was observed in relation to HA (Figure 4).

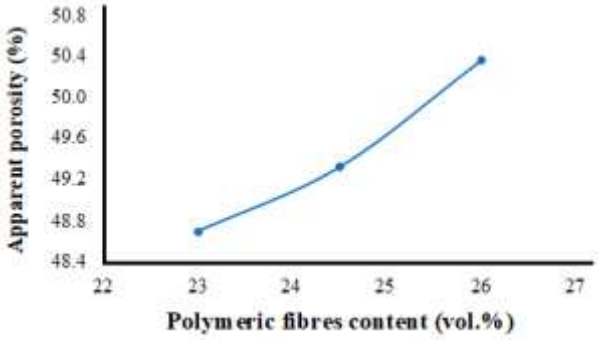

Figure 3. Apparent porosity in relation to the polymeric fibre content of F4.

Figure 5 shows the effect of the polymeric fibre content on the compressive strength as a function of the fibre content of the ceramic composition studied. The polymeric fibre content for the studied levels did not influence the compressive strength.

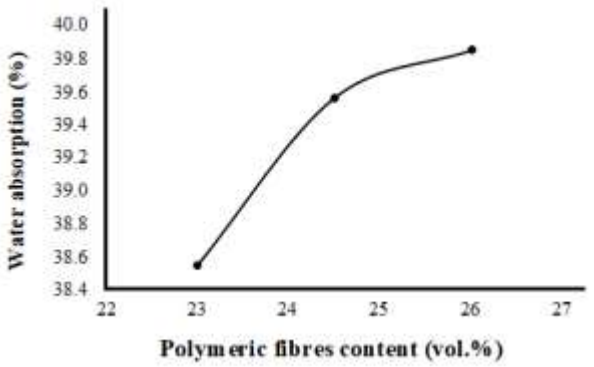

Figure 4. Water absorption as a function of the polymeric fibre content of F4.

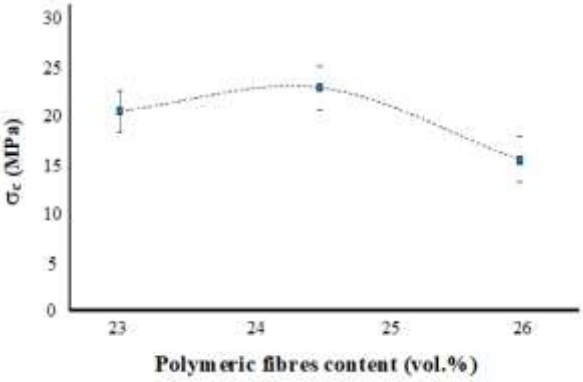

Figure 5. Compressive strength (σ_c) as a function of the polymeric fibre content of F4.

Figure 6 shows the microstructure of F4, while Figure 6(a) shows the porosity of the limestone-rich regions; this porosity is generated from the decarbonation of $CaCO_3$. Figure 6(b) illustrates the limestone-rich region (1) in relation to the remaining material (2). In contrast, Figure 6(c) shows the porosity generated by the elimination of polymeric fibres during the firing of the material. Figure 6(d) illustrates the pore of this elimination, indicating that the fibres had a diameter of approximately 100 µm.

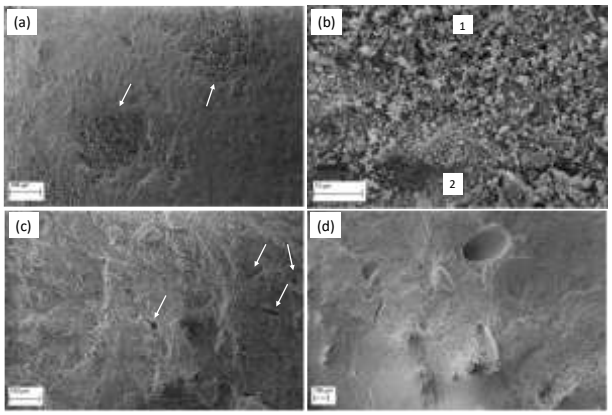

Figure 6. Scanning electron microscopy images of the fractured samples of F4.

Thus, considering the results presented, the tubular F4 formulation samples containing 30 wt.% plastic clay, 11 wt.% kaolin, 34 wt.% feldspar, 25 wt.% limestone and

26 vol.% polymeric fibres was chosen to determine permeability.

As expected, the pressure drop increases with air velocity (Figure 7), and the permeability increases significantly with the use of polymeric fibres. Using Eq. 1, k_1 is estimated as $3.78 \times 10^{-14} \pm 0.24 \times 10^{-14}$ m^2 and k_2 as $2.69 \times 10^{-10} \pm 0.42 \times 10^{-10}$ m. Simão *et al.* [13] studied the permeability of a similar ceramic composition without polymeric fibre content and obtained a k_1 of $0.22–0.59 \times 10^{-14}$ m^2 and a k_2 of $0.19–0.48 \times 10^{-10}$ m. Thus, the obtained tubular filters are applicable to aerosol filtration [14].

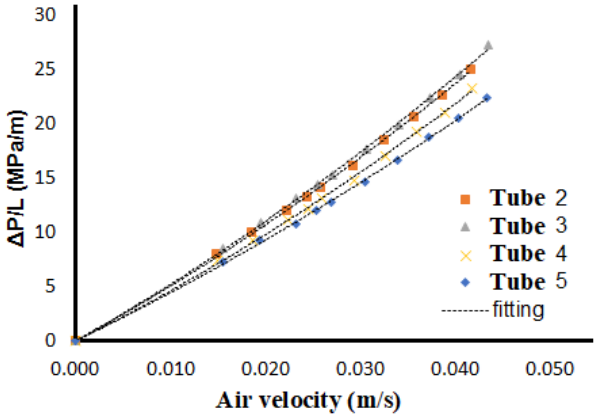

Figure 7. Pressure drop (ΔP/L) as a function of air velocity of tubular F4 samples.

4. CONCLUSIONS.

A ceramic formulation containing 30 wt.% plastic clay, 11 wt.% kaolin, 34 wt.% feldspar, and 25 wt.% limestone with additions of 23, 24.5, and 26 vol.% of polymeric fibres were used to create ceramic tube samples for gas filtration at high temperatures (up to 1000 °C). The P and HA increased with the polymeric fibre content, but the mechanical compressive strength remained constant. The composition containing 26 vol.% of polymeric fibres presented the best results, with P and HA of approximately 50%, compressive strength of 15 MPa, k_1 of $0.22–0.59 \times 10^{-14}$ m^2, and k_2 of $0.19–0.48 \times 10^{-10}$ m. Thus, the obtained tubular filters are applicable to aerosol filtration.

5. ACKNOWLEDGEMENTS.

The authors would like to thank the National Council for Scientific and Technological Development (CNPq/Brazil; process n. 307259/2018-8, 306992/2019-1, 307761/2019-3, and 310328/2020-9).

6. REFERENCES.

[1] de Freitas, N.L., Maniero, M.G., Coury, J.R., "Filtration of aerosols at high temperatures using double-layer ceramic filters: influence of particle diameter on collection efficiency", Ceramics, 50 (2005). 355-361.

[2] de Silva, G.M.C., "Desempenho de filtros cerâmicos na filtração de gases a altas temperaturas", UFSCar, 2008.

[3] Tanabe, E.H., "Performance of filter media in gas filtration at high pressures", UFSCar, 2011.

[4] Huisman, I.H., Dutré, B., Persson, K.M., Trägårdh, G., "Water permeability in ultrafiltration and microfiltration: Viscous and electroviscous effects", Desalination, 113 (1997), 95-103.

[5] Persson, K.M., Gekas, V., Trägårdh, G., Study of membrane compaction and its influence on ultrafiltration water permeability, Journal of Membrane Science, 100 (1995), 155-162.

[6] Salomão, R., Cardoso, F.A., Bittencourt, L.R.M., "Effect of polymeric fibers on refractory castable permeability", American Ceramic Society Bulletin, 82 (2003), 51-56.

[7] Solomon, R., Zambon, A.M., Pandolfelli, V.C., "Polymeric fiber geometry affects refractory castable permeability", American Ceramic Society Bulletin, 85 (2006).

[8] Innocentini, M.D.M., Salomão, R., Ribeiro, C., Cardoso, F.A., Pandolfelli, V.C., Rettore, R.P., Bittencourt, L.R.M., "Permeability of fiber-containing refractory castables", American Ceramic Society Bulletin 81 (2002), 34-38.

[9] Hotza, D., Müller, D., Rambo, C.R., Moreira, E.A., Coury, J.R., da Silva, G.M.C., Innocentini, M.D.M., "Fibrous ceramic filters for high temperature gases", Exacta, 6 (2011) 49-50.

[10] Salvini, V.R., Pupim, A.M., Innocentini, M.D.M., Pandolfelli, V.C., "Optimisation of the processing for the fabrication of filters in the Al O_{23} -SiC system", Ceramics, 47 (2001) 13-18.

[11] Nettleship, I., "Applications of Porous Ceramics", Key Engineering Materials, 122-124 (2009), 305-324.

[12] D. Muller, W. Acchar, G.M.C. Silva, E.A. Moreira, J.R. Coury, M.D.M. Innocentini, D. Hotza, C.R. Rambo, S. Carlos, Processing and characterization of fibrous ceramic filters, Ceram. 55 (2009) 318-325.

[13] Simão, L., Caldato, R.F., Innocentini, M.D.M., Montedo, O.R.K., "Permeability of porous ceramic based on calcium carbonate as pore generating agent", Ceramics International, 41 (2015), 4782-4788.

[14] Simão, L., Montedo, O.R.K., Caldato, R.F., Innocentini, M.D.M., Paula, M.M.S., Angioletto, E., Dal-Bó, A.G., Silva, L., "Porous ceramic structures obtained from calcium carbonate as pore generating agent", Materials Science Forum, 775-776 (2014), 1956-1963.