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Fabrication of multilayer glassy ceramic filters by fine particle migration during slip casting

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Abstract

Cylindrical, glassy ceramic filters with multilayer compaction, consisting of filtration layers of varied pore sizes on granular assemblies with specific interlayers, were shaped by a fine particle migration phenomenon during slip casting. The interlayer provided structural integrity of the filter material and thus the occurrence of the fine particle migration is vital for the success of the casting process. The production of non-crack, dry compaction was investigated from the experiments, which were designed to systematically combine varied sizes of mixture powder particles (quartz–zeolite–frit glass) and its granules. This technique can be applied to the production of ceramic filters with the median pore size of about 0.35 μm. The problem in dealing with finer pore sizes is in the fabrication process: a low casting rate is obtained with finer particles, which results in a low viscous drag on the particles, preventing particle migration.

Keywords: A. Slip casting; B. Microstructure-final; B. Porosity; E. Membranes

1. Introduction

Research on the ceramic filters has focussed on two main topics, which are: filters prepared from low-cost ceramic powders and the fabric produced by a low-cost and simple process. However, the filter media must also have a high porosity, narrow pore size distribution, and high bend strength as well as high chemical resistance to filtered liquids. The hydrophilic nature of the filter is very important in relation to the material used for water filtration. It provided capillarity during filtration and reduced the particle compaction within the filter pores and thus the recovery of the filter increases [1–6].

Recently, the author has produced ceramic filters from a low-cost ceramic powder mixture (quartz, zeolite and frit glass) and the material that was obtained had superior properties. It also had other operational advantages: the filter indicated the formation of a grain-coating of glass due to the sintering that was applied, which was above the fusion temperature of the additives (frit glass and zeolite). The microstructure of this type of material has glassy pore walls indicating high hydrophilic action; the author had been the first person to introduce this microstructure [5]. Later, the water-contact angle of the filters was measured by goniometry [6] using a thin layer wicking approach [7], which obtained significantly low values (10–11°). The high hydrophilic nature makes the material better to use in the dewatering of mineral slurries [8] and the filtration of spring water [6] and of some waste waters [4,5].

The filters under discussion have been fabricated by conventional techniques such as slip casting, pressing and filtration coating. Such fabrications are costly: multilayer compaction required more than one stage including the preparation of a substrate and coating its surface with finer sizes. It is obvious that coating a micro porous filtering layer on the substrate that has large pores, is very difficult. Usually, more than one layer is required to obtain lower pore sizes in the external filtering layer and each additional stage of the process increased the cost of the filters. Instead of the conventional application, the author recently proposed a new approach, which indicated the possibility of fabricating a multilayer ceramic membrane filter with an apparent density gradient using a simple and single stage to process the cast [9]. The process has a high migration phenomenon, obtained by using a mixture of cast slurry designed as powder slurry with granules.

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The granules were deposited, and the fine particles were carried by the liquid that was flowing through the pores and those deposits were close to the surface of the mould surface and thus formed the gradient compaction. This is the first time the technique has been applied to the fabrication of glassy pore wall filters and then tested with relatively coarse particles [9]; the average size of the particles (δ_{50}) was 1.82 μm and the size of the granules was between 500 μm and 1000 μm. The investigation had focused on determining the influence of slurry concentration on particle migrating phenomena; the results led to the conclusion that the best concentration was 10% solids in weight. The same experiment was also used for fine sizes of powder particles but the outcome was not successful because the dry product indicated surface cracking. This development of cracks may be controlled by decreasing the granule size, which should, in turn produce a finer pore size so that a glassy membrane filter can be obtained. It is obvious that the fabrication of fine pore size of a glassy pore wall filter is advantageous during filtration. The fine pore size increased the ability to selectively separate filter material and, more significantly, results in the increase of filtration capacities and recovery of the filtered material. The hydrophilic nature of the fine pore sizes increased the recovery of the filter [5,6] and also led to high capillarity during water filtration, according to the Laplace equation:

$$\Delta P_{cap} = \frac{2\sigma \cos\theta}{r} \tag{1}$$

where ΔP_{cap} represents the capillary pressure in the porous ceramic body, σ represents the surface tension, r represents the capillary radius and θ represents the contact angle between the liquid and the solid. According to this equation, the high filtration capacity could be obtained with a small pore size of filter material. In light of this, it is the aim of this investigation to show how it is possible to obtain a multilayer, glassy pore wall filter with varied pore sizes, especially finer sizes, using the new low-cost and simple cast process, so that the capillary ceramic filters can be better used for water filtration applications and can enhance a wider usage of the filters in liquid/solid separation.

1.1. Fine particle migration during shaping

The process of fine particle migration was discussed in detail in the filtration studies [10–16] and some approaches were presented in relation to ceramic compaction [17–22]. During casting, the fine particles migrated through the previously formed cast bodies and filled the voids between the granules, which led to the shaping of a ceramic body that had an apparent density gradient. However, the non-uniform ceramic compaction process led to development of cracks and deformations during the drying, and later the sintering, process. In addition to these problems, the author presented a successful study on the fabrication of a multilayer ceramic membrane filter with the migration conditions set out above [9]. The study was conducted with relatively coarse particles, designed as an assembly of powder and granules. Analysing the reliability of the new cast approach for the compaction of finer sized particles, is the motivation behind this investigation.

During the cast approach outlined above, it is obvious that serious problems will arise during the compaction of finer sized particles: (i) a low migration phenomenon, which is determined by the viscous drag on the particles, (ii) a low casting rate, which is the result of a high resistance of the cast body, and (iii) local densification, which may occur due to the non-uniform migration phenomenon caused by the clogging of either the cast bodies or the pores of the mould. As well as the compaction troubles detailed above, the sintering of fine particles, with high fusible additives, is dangerous: the efficacy of the pores was reduced by the particles coalescing, which is due to the process of compaction taking place in a non-uniform manner because of the clogging of mould pores.

2. Experimental

The starting material for fabrication of the glassy membrane filter was the powder mixture of high-purity quartz (99.09% SiO_2), natural zeolite (clinoptilolite) and frit glass (lead borosilicate). The filter matrix was of a quartz type and the additives (frit glass and zeolite) caused the filters to have glassy pore walls (high hydrophilic nature) as the sintering temperature that was applied was above their fusion temperature.

The powder mixture was ground a number of different times using alumina balls in an aqueous system and later oven dried at 105 °C for 24 h. The material ground for the least amount of time (δ_{50} = 6 μm) was used for production of the granules. Water was added to the powder (5 wt.%) in order that it agglomerated and sintering was applied at 900 °C for 20 min, with a heating rate of 5 °C/min and the materials were cooled in the furnace. The granules were sieved through different sieve sizes and four groupings were obtained: (i) 125 + 75 μm; (ii) 125 + 250 μm; (iii) 500 + 250 μm and (iv) 1000 + 500 μm.

The above powder mixture was also ground for more periods of times leading to the production of powder samples with varied sizes: (i) δ_{50} = 0.22 μm; (ii) δ_{50} = 0.65 μm; (iii) δ_{50} = 0.98 μm and (iv) δ_{50} = 1.82 μm. The particle size of the ground powders was measured by using a laser particle size analyser (Malvern Mastersizer 2000). Then each group of the powder particles was used to prepare cast slurries, with the solid concentration being 10% solids in weight. The slurries were mechanically agitated for 30 min and then casting was undertaken.

The casting unit used was unique and was presented for the first time in a recently published paper [9]; it consisted of a hollow, cylindrical mould, a cylindrical glass tube and a cover plate (see Fig. 1). The mould was made of plaster-of-paris in which gypsum was added to water as 70 wt.% of the water content, and the gypsum slurry was stirred for 1 min, and then degassed under vacuum. The gypsum slurry was poured into a resin mould and obtained a hollow cylinder (ϕ20 × 100 mm). The mould was dried at 40 °C before slip casting. The glass tube had a 20 mm internal diameter and a height of 100 mm; it was placed tightly into the mould cavity. The 200 ml of powder

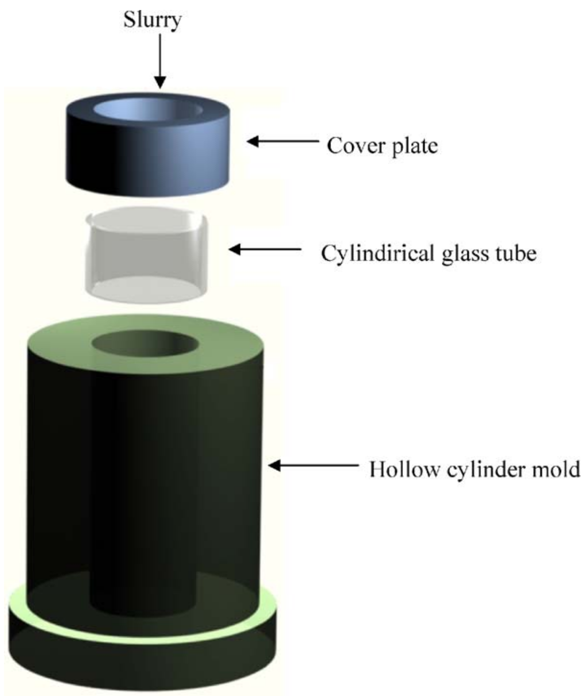

Fig. 1. The casting unit for the multilayer compaction of membrane filters.

slurry that had been prepared previously, was poured into the mould and some of the slurry remained in the glass tube and was used as a reservoir for casting. The water of the cast slurry was absorbed into the walls of the mould whereupon a thin filtering layer formed on the inner surface. After that the granular particles were placed into this same mould over laying the powdered slurry. The non-stop casting was achieved by feeding the excess slurry, which remained in the glass tube, into the granular medium producing a secondary compaction due to the fine particle migration. Thus an interlayer had been obtained.

The migration phenomena will be discussed in terms of particle sizes through the same casting conditions: (i) a fresh mould was used for the each casting experiments, by this way the applied capillary pressures being the same. The mould preparation procedure of our work was the same as the previously study conducted by Hotta et al. [23], this study indicated the capillary pressure of the gypsum mould was of 1.5×10^5 Pa, (ii) the slurries have a pH = 7 and the same temperatures (~20 °C), (iii) the same solid concentration (10% solids in weight) and the agitation time (30 min) before the cast operation.

The shaped filters were dried overnight in a room at 60% humidity, and then kept in ambient conditions for a day, and finally oven dried at 105 °C for 12 h. The bodies were sintered at 1000 °C for 20 min with a heating rate of 5 °C/min. The microstructure of the filters was investigated by scanning electron microscopy (Zeiss EVO-50 EP). The ranges of pore sizes of the filtering layers were determined by the mercury porosimetry technique (Quantachrome PoreMaster). The apparent porosity was measured by a water immersion technique according to the Archimedes' principle.

Rheological measurements were carried out a rotational controlled shear rate rheometer (Bohlin Instruments-CVO). The measuring configuration adopted was a cup and bob (C25 DIN 53019) measurement system was performed in the shear rates range from about 0.2 to 1500 s^{-1}. Before starting a measurement, pre-shearing was performed at a shear rate of 200 s^{-1} for 10 s followed by a rest of 10 s in order to transmit the same rheological history to whole suspension being tested.

3. Results and discussion

The starting point of the new cast approach is similar to other conventional studies; the significant difference is the addition of granular particles into the mould while the casting process is in progress. The moment when the granules were added into the system was very important because it is at this point that the desired thickness of the cast layer was achieved. When it was introduced later on in the casting process it did not influence the thickness of the cast layer. In this system, the cast that is formed is the filtering layer and its thickness directly determines the performance of the filter product. A thick layer increased the water resistance, so the cast layer should be thinner. It is obvious that the cast layer is a part of our gradient compaction and its mechanical strength is of great importance during the processing of the material, which includes its removal from the mould and the process of drying and sintering. Also, the mechanical strength of the sintered material is important due to the pressure applied during processing both in the filtration operation and the back flushing, which is used for filter recovery. Therefore the thickness of the filtering layer must not be made too thin.

Thus the casting rates of the powder samples were determined by the powders being four groups arranged by size and the prepared cast slurries had the same solid contents (10% solids in weight). The thickness of the filtering layer could be easily determined from the conventional plot: the cake thickness (L) versus square root of casting time ($t^{1/2}$) [24]. Fig. 2 shows a representative plot, where L versus t curves are displayed for the casting tests. The good linear dependence of L on $t^{1/2}$ is in agreement with the conventional casting rate equation and indicating the casting was implemented well. The experimental results indicated that the casting rate is significantly less for the finer sized particles. This result does not significantly influence the thickness of the cast layer; only a

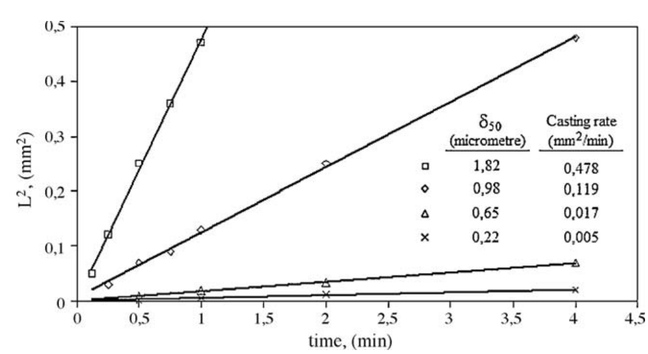

Fig. 2. The cake thickness (L) as a function of the square root of time ($t^{1/2}$) for

little more time is required in our process in order to obtain the same thickness. The influence of the casting rate on the following casting process will be discussed later. A cast thickness of about 500 μm was selected for the fabrication of the present filters. The thickness was previously tested for our system and obtained good enough product samples [9].

After the required thickness was achieved, the granular particles were poured into the interior of the cylindrical mould. At that point, dead-end casting was performed. The slurry reservoir helped the cast progress through the granular medium. The particles were carried by the liquid flowing through the interstices of the granules. At this stage, two different cast phenomena were observed: (i) the particles flowed freely, so they migrated and most of them were deposited into the granular medium, nearly to the filtering layer (this is how the interlayer was formed) and (ii) the granular medium clogged. At this stage, the particle migration was not sufficient for the production of an apparent density gradient. This was an undesirable phenomenon for the processing of our cast.

It is obvious that the particles were flowing freely because of the relatively large size granules. As the size of granules is reduced, then the size difference compared with the powder particles is less and the consequence of this is the clogging phenomenon within the granular medium. This produced particles that were dispersed through the granular medium and thus the multilayer compaction only contained two layers, which is not the aim of this study. The clogging phenomenon may be partially useful as it increased the viscous drag on the particles by applied high pressure difference during casting.

The high number of migration phenomena, obtained using a large size difference between the powder particles and granules, is shown to be advantageous for multilayer compaction but it should also be recognised that achieving success for only one type of casting is not good enough, the following processes such as removal of the mould, drying and sintering, need to be carried out without any crack developing. Therefore it may be necessary to produce more migration phenomena rather than the higher ones. Table 1 shows the influence of powder and granule size on the production of the multilayer filter by the new casting route. The experiment's results indicate that there are limitations due to the possibility of no surface cracking of the dry product and using finer sized particles cause without problems through the mould remove of the sample. The following are the benefits to using certain combinations of powder particles and granules: (i) fine size particles ($\delta_{50} = 0.65$ μm) with fine size granules (125–250 μm); (ii) moderate size particles ($\delta_{50} = 0.98$ μm) with moderate size granules (250–500 μm) and (iii) coarse size particles ($\delta_{50} = 1.82$ μm) with coarse size granules (500–1000 μm). The finer size particles ($\delta_{50} = 0.22$ μm) did not lead to the production of filter material; both used the granules as coarser to finer sizes. An analysis of these systematic combinations indicates that there is a relationship between the sizes of the powder and granules, but given the critical size ratio, more data from more experiments was required, testing particles with narrower size intervals. In light of this, it was expected that non-crack production would be possible using the combination of finer sizes of particles ($\delta_{50} = 0.22$ μm) and finer sizes of granules (75–125 μm). However, this experiment was not successful and the cast body had some cracks in it and it could not be removed from the mould without causing damage.

Fig. 3a and b shows the fractured surface of the dry products from the casting, operated through the combination of coarse powder with coarse granules and fine powder with fine granules, respectively. The sample produced coarse particles, which indicates multilayer compaction, in which the occurrence of an interlayer is clearly shown. However, the finer particles show compaction without an interlayer; the granular medium with fine particles formed a homogenous substrate. As a result of this process, two layers of compaction were obtained and the structural integrity of the layers failed (see Fig. 3b). It is clear that the response of inferior compaction is the result of insufficient particle migration. Particle migration through the granular medium required a high viscous drag on the particles but the casting rate obtained after the filtering layer was compacted with finer particles, is relatively low (see Fig. 2). Therefore the low viscous drag did not lead to particle migration and so the interlayer could not be obtained. The filter without interlayer produced cracking and later separation of the filtering layer from the granular substrate and thus, an inferior casting condition with the new casting approach was obtained. The low casting rate that occurred with the finer particles through the plaster mould is normal; the pressure obtained by the plaster mould is limiting and the resulting filtering layer with finer particles has more resistance to flowing liquid. This system may require pressure casting, and through this process a high viscous drag may result from the production of particle migration.

The migration phenomena should be also discussed with respect to rheology of slurries. It is the fact that the rheology of suspensions greatly determined by the solid concentration of suspensions where the particle size and particle size distribution are the other relevant factors. In this study, the rheological behaviour of suspensions is investigated for two different

Table 1
The influence of powder and granule sizes on the production of the multilayer membrane filter.

Powder sizes (δ_{50}) (μm)	Size interval of granules, μm			
	$500 < \delta < 1000$ (coarse)	$250 < \delta < 500$ (moderate)	$125 < \delta < 250$ (fine)	$75 < \delta < 125$ (finer)
0.22 (finer)	Surface cracking after drying	Surface cracking after drying	Surface cracking	Crack observed after casting
0.65 (fine)	Surface cracking after drying	Surface cracking after drying	Well production	–
0.98 (moderate)	Surface cracking after drying	Well production	–	–
1.82 (coarse)	Well production	–	–	–

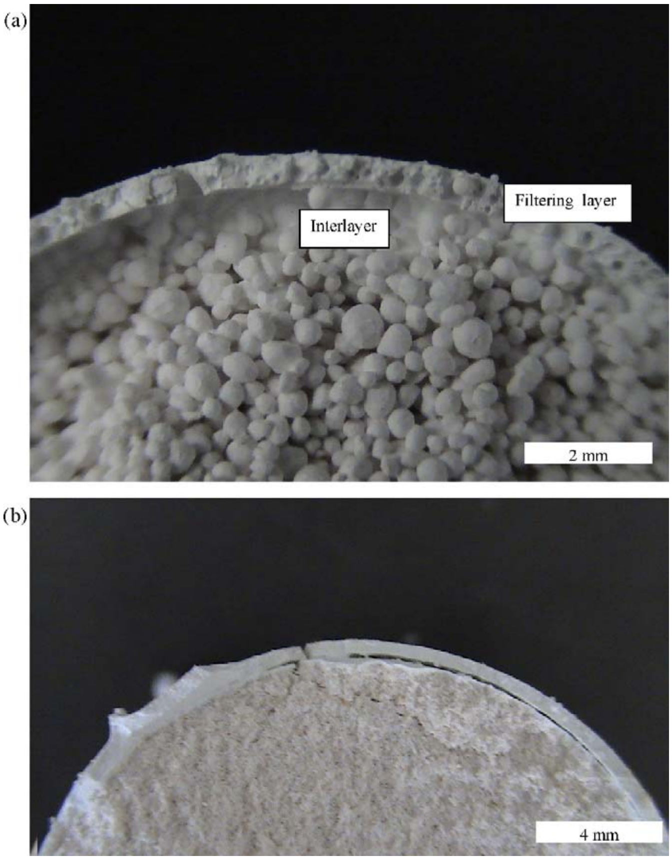

Fig. 3. (a) Photos of the fracture surface of the cast sample prepared from the coarse sizes of powder (δ_{50} = 1.82 μm) and granules (500 < δ < 1000 μm) [9]. (b) Photos of the fracture surface of the cast sample prepared from the finer sizes of powder (δ_{50} = 0.22 μm) and granules (75 < δ < 125 μm).

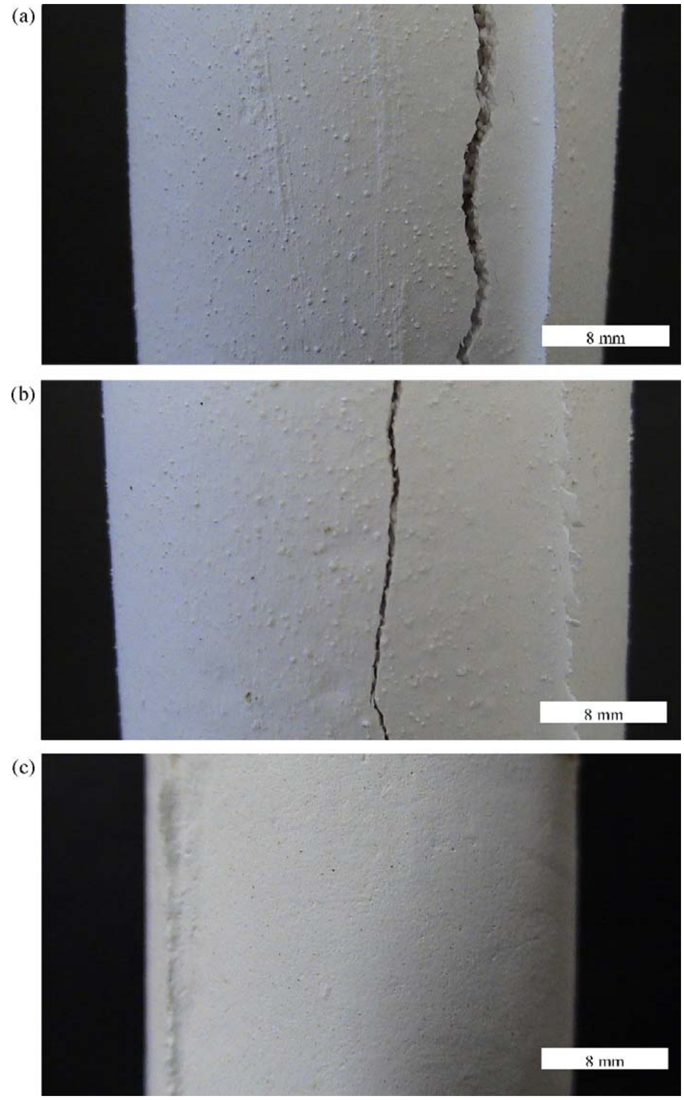

Fig. 4. (a) Photos of the surface of filter after drying for the casting with the powder size and granules being δ_{50} = 0.650 μm and 500 < δ < 1000 μm, respectively. (b) Photos of the surface of filter after drying for the casting with the powder size and granules being δ_{50} = 0.650 μm and 250 < δ < 500 μm, respectively. (c) Photos of the surface of filter after drying for the casting with the powder size and granules being δ_{50} = 0.650 μm and 125 < δ < 250 μm, respectively.

particle sizes (δ_{50} = 0.65 μm and δ_{50} = 0.22 μm) in which coarse sample is sufficiently migrated and the fine sample being failed. The rheological behaviour of the suspensions is given in Fig. 7. The results show that the rheological behaviours of the suspensions have no more difference. It may be the results of close particle sizes and the other factor may be the low solid loading (10% solids in weight). Generally, the influence of particle size on the rheological behaviour has been studied with high solid loadings [25–27].

The cracks developing due to the size of granule particles with the same powder particles (δ_{50} = 0.65 μm) is shown in Fig. 4a–c for the coarse, moderate and fine granules, respectively. The sample obtained with the coarse granules has a larger amount of cracking at the surface than when the moderate-sized-granules were used. The size of granules decreased to the fine size, which obtained a surface with no cracks. With this combination, the interlayer that was produced was shown to be good enough to compensate for the different rates of shrinkage of the surface layer and the granular medium. The interlayer acts as a wetting material for the filtering layer and the granular substrate medium.

The non-cracked, dried samples were sintered at 1000 °C and their surfaces were examined. The microscopic studies indicated that there was no cracking. Furthermore, examination is necessary to predict whether the material can be successfully used for water filtration. The properties of the surface layers, such as the porosity and pore size distribution as well as their hydrophilic nature, were investigated. The study using coarse powder with coarse granules was previously determined [9] and results indicated that the product sample shows promising engineering properties for a capillary system. These properties include a glassy pore wall could be obtained and the average pore size and porosity could be 1.15 μm (pore size interval: 0.3–4.5 μm) and 53.94%, respectively. The fine powder, used for making the filtering layer, the porosity was 44.26% and this is acceptable for filtering purposes. Also, a fine size, with mono modal pore size distribution and with a narrow size interval (0.15–1.4 μm), could be obtained (see Fig. 5), meaning that a high selectivity can be obtained in filtration applications. Fig. 6 shows the scanning electronic microscope (SEM) micrograph

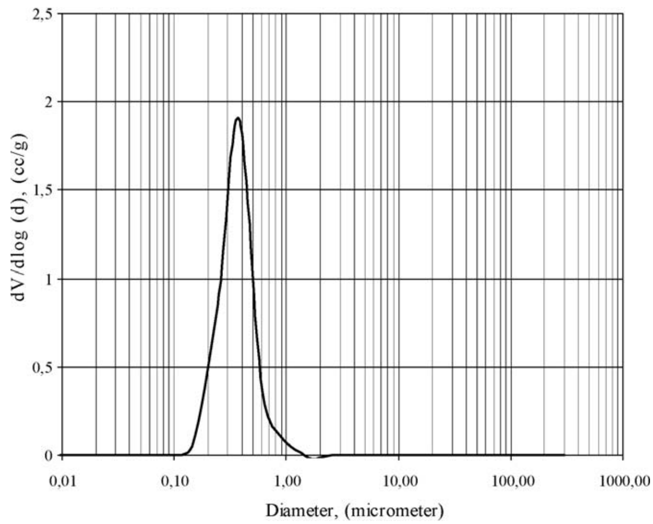

Fig. 5. Pore size distribution of the filtering layer obtained using fine size powder sample ($\delta_{50} = 0.650$).

(back scattering mode) of the filter, which shows a glassy nature. The grain coating by glass phenomenon was previously discussed [7,28] in which the white grains, with high a atomic number, consist of a lead containing glass homogeneously distributed throughout the microstructure. This type of microstructure with multilayer compaction is believed to have many potential applications for high performance capillary ceramic filters.

Industrial filter usually has a tubular configuration with one or several channels; however the present filters show cylindrical shapes, the interior space of the mould completely filled by granules during the casting. The production as tubular configuration may possible using freely a nonporous bar inserted at the centre of the mould. The product is also required a large size of fabrication. A scale-up study with tubular configuration will be further studies to enhance wider use of the filters in liquid/solid separation.

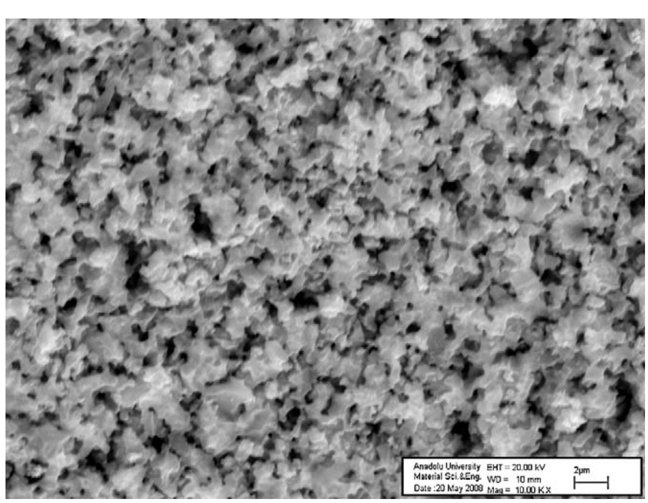

Fig. 6. Scanning electronic microscope (SEM) micrograph of the filtering layer (back scattered mode) obtained using fine size powder sample ($\delta_{50} = 0.650$).

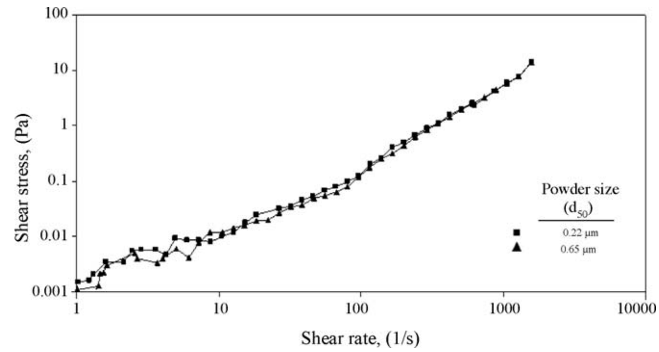

Fig. 7. The log–log plots of shear stress as a function of shear rate for the suspensions with different particle sizes ($\delta_{50} = 0.65$ and $\delta_{50} = 0.22$ μm).

4. Conclusions

Cylindrical shaped ceramic filters, with an apparent density gradient, have been fabricated by a simple and single stage cast process using a mixture of particles designed as powder and granules. The particles composed quartz-natural zeolite-lead borosilicate glass. Besides the advantages of the cast, the filter has high hydrophilic surfaces in which the pore walls of filter were glassy in nature. The additives (frit glass and zeolite) fused during sintering and obtained the microstructure of grains coated by glass. The filter fabrication is limiting with respect to the slurry concentration of the used powder particles [9] and the size ratio of powder and granule particles. It is obvious that the large size difference increased the fine particle migration but the product indicates surface cracking after drying. On the other hand, the use of finer particles with finer sized granules adversely affected the migration phenomenon and thus the structural integrity of the filter was destroyed. For the mould casting system, the new approach is useful for the production of multilayer glassy ceramic membrane filters, using powder particles of an average size (δ_{50}) of about 0.65 μm. The system also required the granules to have a size interval of between 125 and 250 μm. The insufficient migration phenomena observed with finer particles may be accomplished using high pressure casting.

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