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Micro-structured alumina multi-channel capillary tubes and monoliths

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ABSTRACT

In this study, a continuous and single-step fingering inducing phase-inversion process for fabricating micro-structured alumina multi-channel capillary tubes and monoliths, which consist of a plurality of radial micro-channels, has been developed. In addition to the geometrical similarity to conventional ceramic monoliths, the unique radial micro-channels created in the walls of the multi-channel capillary tubes (or monoliths) can reduce mass transfer resistance and increase surface area, which are critically important factors for filtration and catalytic reactions. Furthermore, the enlarged cross section area of the multi-channel configuration enhances the resistance to external impacts. The technique described in this study not only can be scaled up for fabricating monoliths of commercial sizes, but can also be scaled down to make multi-channel capillary tubes, solving the issue of insufficient mechanical property in current ceramic hollow fibres.

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1. Introduction

In the last decade, a single-step fingering inducing phase-inversion process [1] has been well investigated for developing micro-structured ceramic hollow fibres/capillary tubes that can be used in various applications, such as oxygen separation [2–5], membrane reactors [6–10] and solid oxide fuel cells (SOFC) [11–14]. In contrast to conventional ram-extrusion-based routes, this technique creates a plurality of self-organised micro-channels in the cross section of the tube walls [15,16], and allows single-step formation of multi-layer hollow fibre/capillary tubes (up to 3 layers) [12]. Apart from flexible control over morphologies and microstructures, a wide range of ceramic materials can also be adopted. Moreover, the presence of radial micro-channels significantly increases the accessible surface area, which is critically important for catalytic reactions when employed as a catalyst substrate, or greatly reduces the permeation resistance to fluids as a membrane. However, these radial micro-channels compromise the membrane's mechanical property, and thus it is preferred to bundle the hollow fibres/capillary tubes of this type together in order to meet industrial expectations.

To increase the tube strength to resist external impacts, the most efficient way is to increase the cross section area of the ceramic tubes. Two strategies can be adopted to achieve this: one is to use a single-channel configuration and increase the area of the cross section by increasing the diameters and/or the wall thickness; and the other option is to use a multi-channel configuration. The former strategy seems straightforward but in practice faces problems during the phase-inversion fabrication process. When the thickness or the diameter exceeds a certain value, the precipitation rate of the nascent fibre reduces quickly and the suspension cannot be solidified in time, hence the fibre will not be maintained in the desired dimensions because of severe elongation induced by gravity. Due to this reason, the diameter and the thickness of ceramic hollow fibres are limited. In fact, even if larger ceramic hollow fibre/capillary tubes can be made, it will compromise the surface area to volume ratio as well as their transport convenience.

Multi-channel ceramic monoliths, or sometimes known as ceramic honeycombs, have been widely studied and used in industries [17], especially in the fields of catalyst substrates and membrane filtration. Its current fabrication process is largely based on extruding a ceramic paste through a die or mould, similar to the way that commercial ceramic tubes are fabricated. The multi-channel monoliths fabricated through this method have symmetric-structured walls. Even though the monoliths obtained in such a way can have high surface area to volume ratios, it is still low when compared with the requirements of a catalyst carrier, and thus an extra coating of γ-alumina is often washed onto the inner surface of the channels to further enhance the surface area. Thus, the resistance of mass/heat transfer is also significantly increased due to its microstructure.

Applying the fingering inducing phase-inversion technique to the multi-channel configuration is theoretically advantageous from aspects of both the fabrication process and the performance of the multi-channel monoliths. In this research, the idea of multi-channel monolith membranes via the fingering inducing phase-inversion method is implemented by delicately designed spinnerets as shown in Fig. 1. The designed spinnerets use certain numbers of well-arranged parallel nozzles to deliver the internal coagulant, and the

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rest of the encompassed circular area is filled with the ceramic suspension. With such multi-channel spinneret configurations, the problem of slow precipitation in larger-dimension single-channel hollow fibres will no longer be an issue, because the thickness of the internal walls between the channels and the outer wall has been largely reduced. Furthermore, the internal walls come into contact with the internal coagulant on both sides, thus the precipitation rate of the suspension is further improved. With the multi-channel spinnerets, it is now possible to produce ceramic monolith/capillary tubes of large dimensions. It is also expected that multi-channel ceramic monoliths produced through this method will minimise the drawbacks of traditional monoliths by introducing self-organised radial micro-channels in the cross section of the walls, which will not only reduce the resistance of mass/heat transfer, but also greatly increase the geometric surface area which will be beneficial for catalytic reactors.

Aiming to combine the advantages of phase-inversion induced asymmetric structures and multi-channel monolith tubes, in this prove-of-principle study, 1-channel and 3-channel hollow fibres as well as 7-channel and 19-channel capillary/monolith tubes were successfully fabricated through the fingering inducing phase-inversion method. The macro- and micro-structures of the multi-channel tubes were characterised, the mechanical property was investigated, and the mass transport property of the tubes was evaluated from water permeation results. This paper also theoretically explored the influence of the macro-structure to the mechanical property, as well as presenting a feasible route of improving water permeation through optimised sealing patterns of the multi-channel tubes.

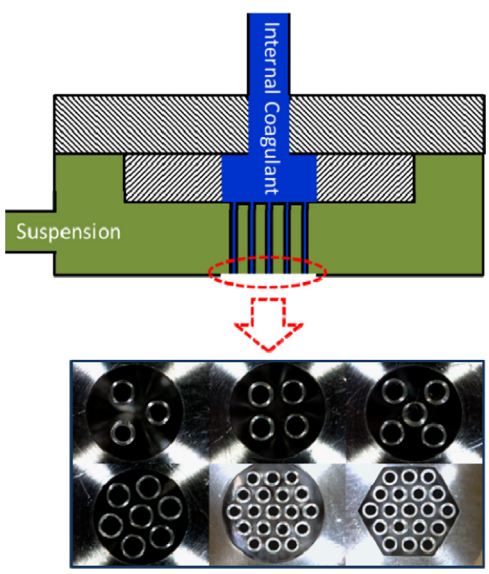


Fig. 1. The multi-nozzle spinneret designs.

2. Theoretical analysis of the geometrical influence to the fractural loading

Long, slim shaped materials are most likely to be damaged by a shear force perpendicular to the longitude direction, which is particularly important for brittle materials like ceramics. Hence for the case of ceramic hollow fibres and capillary tubes, the resistance to a bending force is one of the most important mechanical properties. This resistance can be properly evaluated by three-point bending tests, which give the maximum bending force needed to fracture a ceramic sample. In general, a three-point-bending fractural stress of a circular bar with a symmetric cross section is determined by

$$\sigma_{max} = \frac{M \times R}{I}$$

where σ_{max} is the fractural stress which is determined by the material and the microstructure and irrelevant to the geometry of the sample. R is the outer diameter of the sample. M is the bending moment which is determined by the fractural loading force F and the span L used during the test

$$M = \frac{F \times L}{4}$$

And I is the second moment of area defined by

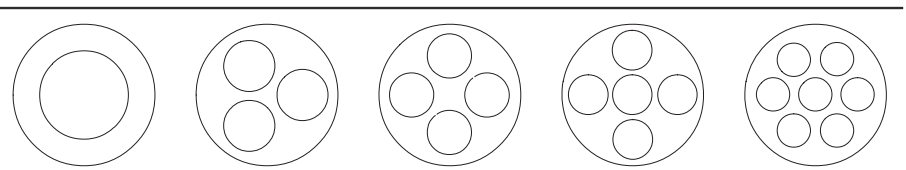
$$I = \int y^2 dA$$

y is the coordinate of the force direction relative to the centre of the cross section, and the integral over the solid area of the cross section. Apparently I is geometry dependent. Hence the fractural loading F is expressed by

$$F = \frac{4I \times \sigma_{max}}{L \times R}$$

It would be useful to analyse how the geometry of the cross section affect the fractural loading of the ceramic tubes. For comparison, the multi-channel and single-channel tubes of same material with a fixed outer diameter R were considered, so that the fractural loading only varies with I. To be fair, the area of the cross section was also fixed so that the fractural loadings obtained were from the same amount of material. A typical dimension of a single-channel ceramic capillary tube was adopted, say, the outer diameter of 4 mm and the inner diameter of 2.5 mm for this case study. The corresponding radius of the channel of a multi-channel tube was set so that the area of the cross section was the same as the single-channel tube. The configuration of each tube and the relative fractural loading compared to the single-channel tube are shown in Table 1. It can be seen from the calculation that the fractural loading of multi-channel tubes is actually lower than the single-channel tube with same cross section area. The 19-channel monolith is not included in Table 1, because the second moment of area I is difficult to calculate due to the complexity of the cross section geometry. Shi et al. also experimentally studied the

Table 1
Relative fractural loading of multi-channel tubes.

Configuration[a]					
Internal radius (mm)	1.25	0.7217	0.6250	0.5590	0.4725
Distance between the tube and channel centres (mm)	N/A	1.0	1.0	1.3	1.2
Relative fractural loading (%)[b]	100	88.96	90.46	83.25	86.98

[a] The outer diameter of the tube is 4 mm.
[b] The fractural loading is normalised by the value of the single-channel tube.

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