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Processing multi-channel alumina membranes by tape casting latex-based suspensions

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Abstract

Tape casting has been used to produce thin, multi-channel alumina ceramic membranes using aqueous-based suspensions with latex as the binder. Three different kinds of latex binders, vinyl/acrylic, vinyl acetate/acrylic and acrylic/styrene latexes, each plasticized with dibutyl-*o*-phthalate or glycerol, were evaluated in terms of their slurry rheology and green tape mechanical properties. The results showed that the different binder systems had a substantial effect on the rheology of the system as a function of the dispersant concentration; the acrylic/styrene system proving to be the most useful. As a plasticizer, glycerol was not found to be effective; however, dibutyl-*o*-phthalate was much more successful, particularly in the acrylic/styrene system where it enabled the strain-to-failure of the green tape to be doubled with only a modest decrease in tensile strength. As expected, both binder content and sintering temperature significantly affected the final porosity and fracture strength of the ceramic membranes; 15 wt.% binder and a sintering temperature of 1500 °C were found to yield a membrane with a porosity of ~40% and strength of ~95 MPa. Simple multi-channel substrates could be easily assembled by bonding strips of the green tapes together with the ceramic slurry. Adhesion between the tapes was excellent with no interface being observed and on sintering no warping or deformation occurred.

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Keywords: A. Tape casting; D. Al_2O_3; E. Membrane; Latex binders

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1. Introduction

Ceramic membranes can offer a number of advantages over their polymeric and metallic counterparts for processes such as separation, filtration and catalytic reactions in the food, pharmaceutical and electronic industries [1]. For example, they can possess high thermal and chemical stability, which lead to a long life expectancy, as well as offering a catalytic function and good defouling properties when required [2]. Their principal drawbacks, however, are related to their high working pressure, which arises because of their minimum thickness, large size and comparatively high cost. In an attempt to address some of these issues, the present work sought to fabricate membrane substrates only about 1 mm thick via tape casting using a range of different binder systems.

Tape casting is the primary technique for producing thin, flat, ceramic sheets such as substrates [3] and has been used for the fabrication of ceramic membranes by a number of investigators including the present authors [4–6]. The slurries required for the process are typically prepared by dispersing a ceramic powder in a solvent with the addition of appropriate dispersants, binders and plasticizers such that after casting the evaporation of the solvent will yield a flexible tape. Whilst the process has traditionally involved the use of organic solvents [7,8], there is now a trend to move towards aqueous-based systems because of the volatility, toxicity and cost of the organics involved and the fact that water-based systems potentially offer lower costs, health and

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environmental risks [9]. Nevertheless, current aqueous-based systems currently suffer from four main drawbacks [10,11], viz. (1) a low evaporation rate during drying; (2) the need to use a high binder concentration if a good tensile strength is to be achieved; (3) the requirement for a de-airing step and the use of a defoaming agent to eliminate air bubbles; and (4) the fact that the green tape is more susceptible to defects such as orange peel, fish-eye and cracking due to the inherent lack of compatibility between the surfactant and the defoaming agent.

A wide range of water-soluble binders exists and several have been evaluated for tape casting [12–15] including the derivates of cellulose ethers such as hydroxyethycellulose and hydroxylpropylmethylcellulose [16], gelatine [17] and acrylic polymers [18]. However, the addition of only small quantities of the cellulose and polyvinyl alcohol-based binders increase the viscosity significantly, limiting the amount that can be incorporated without the slurry becoming difficult to tape cast, compromising the mechanical strength of the green tape. The main advantage of latex binders, such as acrylic polymer emulsions [19–23], compared with water-soluble binders lies in the fact that the slurry can be prepared with a high solids loading and polymer content whilst retaining a relatively low suspension viscosity, a short drying time and a high mechanical strength for the green tape.

The present work reports an investigation into the preparation of thin alumina membranes with a multi-channel structure via tape casting using three different latex binders. In order to understand the role of each of the organic components used, viz. dispersant, binder and plasticizer, and to allow optimization of the system, the influences of the organic components on the rheological behaviour of the slurries and the tensile strength and strain-to-failure of the green tapes have been examined in detail. Finally, the membranes were sintered at a range of different temperatures and the potential for creating thin, multi-channel systems evaluated.

2. Experimental

2.1. Materials

The tape casting slurries were prepared using 75 wt.% of α-Al_2O_3 with an average particle size of 0.7 μm (99.5% purity, Alcoa, USA), deionised water and ammonium polyacrylate dispersant, the latter being prepared by adding analytical grade ammonium into polyacrylic acid with pH adjustment to 9. The anti-foaming agent used was analytical grade tributyl phosphate. Three different latex binders were investigated, see Table 1, all three had a solid content of 50% and were supplied by National Starch and Chemical Co. Ltd., USA. Finally, two plasticizers were also investigated, viz. dibutyl-*o*-phthalate and glycerol; both were analytical grade.

Table 1
Latex binders used during the present work and their properties

Binder	Average particle size (μm)	Inherent pH	T_g (°C)	Ionic nature
Vinyl/acrylic latex	0.3–0.5	4.3	28	Anionic
Vinyl acetate/acrylic latex	0.3–0.5	2.5	21	Anionic
Acrylic/styrene latex	<0.1	8.3	18	Non-ionic

2.2. Experimental

The tape casting slurries were prepared by adding the alumina powder into deionised water containing a pre-determined concentration of dispersant; mixing was accomplished by ball milling for 4 h. Following addition of the binder and plasticizer, the slurry was ball milled for a further 2 h and then the anti-foaming agent was added followed by a final 2 h of ball milling. Subsequently the slurries were de-aired in a chamber connected to a vacuum pump and then stirred at low speed for 4 h to homogenize them. The final slurries had an Al_2O_3 content of 75 wt.%, a dispersant concentration in the range 0–0.7 wt.%, an anti-foaming agent content of 0.05 wt.% and a binder content of 10–20 wt.% (all by mass of the Al_2O_3) and a plasticizer content of 1–4 wt.% (by mass of the latex binder). Rheological measurements of the slurries as a function of the different levels of additives used were performed using a rotating cylinder viscometer (DV-II+, Brookfield Engineering Laboratories Inc., USA) at a shear rate that continuously increased from 0.02 to 41.8 s^{-1} over a 1 min internal.

The slurries were cast on a polyethylene terephthalate film using a tape casting machine (Cerlim Equipment, Limoges, France) at a casting speed of ~1 cm s^{-1}. The tapes were initially dried at room temperature, ~20 °C, for 8 h before being removed from the backing film and placed in an oven at 50 °C for further 8 h. The dried tapes all had a thickness in the range 1.2–1.5 mm. To obtain their tensile strength and strain-to-failure values, the tapes were punched into 'dog-bone' shaped samples and then measured under uniaxial tension on a universal mechanical testing machine (M30K, J.J. Lloyd Instrument Ltd., UK) at a cross-head speed of 2 mm min^{-1}. Each reported value represents the average of four samples.

Based on the slurry rheology and mechanical property data obtained for the different binder systems, the acrylic/styrene latex was selected for further study. In order to determine the decomposition characteristics of the organic species within the green tapes and hence the optimum heating rate for sintering, TG-DTA analysis (Netzsch TG 209, Germany) was performed in an air atmosphere using a heating rate of 10 °C min^{-1}. The tapes were then punched into bars measuring 80 mm × 10 mm × 1.2 mm and sintered at 1500, 1525 and 1550 °C for 3 h. A heating rate of 1 °C min^{-1} 1 was used up to 300 °C and then 0.5 °C min^{-1} to 354 °C where the temperature was held for a period of 2 h whilst the organics burnt out. Heating was then resumed at a rate of 2 °C min^{-1} up to the sintering temperature. After sintering the

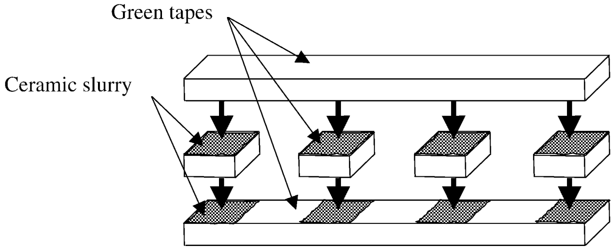

Fig. 1. Schematic showing construction of the prototype multi-channel membrane substrates.

samples were allowed to cool at 5 °C min^{-1}. The densities of the sintered tapes were measured using the Archimedes principle with water as the liquid medium. Prior to measurement, the samples were boiled in water for 2 h to ensure that the air in the interconnected porosity was completely eliminated. The fracture strengths of the tapes were obtained using the 3-point bend test with a loading rate of 1 mm min^{-1}. Scanning electron microscopy (JSM-6700F, Jeol Ltd., Japan) was used to examine the microstructures of both the green and sintered tapes.

Prototype multi-channel membrane substrates were produced by assembling 15% acrylic/styrene latex-based green tapes into the structure illustrated in Fig. 1. The tapes were cut into the appropriate sized pieces and bonded together using the same alumina slurry. A compressive force of 2 N cm^{-2} was used to hold the tapes together during drying, after which the structure was sintered at 1500 °C for 3 h. Finally, the porosity and pore size distribution were measured using mercury intrusion porosimetry (Poremaster, Quantachrome Instruments, USA).

3. Results and discussions

3.1. Rheological behaviour of the slurries

The effect of varying the dispersant concentration on the Al_2O_3 slurry viscosity is shown in Figs. 2 and 3. It can be seen that the slurry displayed shear thinning behaviour with the viscosity passing through a minimum at a dispersant concentration of ~0.1 wt.%. Similar behaviour has been observed before for ammonium polyacrylate-dispersed alumina suspensions and has been explained in terms of the stabilisation mechanism of the ceramic particles by the polyelectrolyte [24–27].

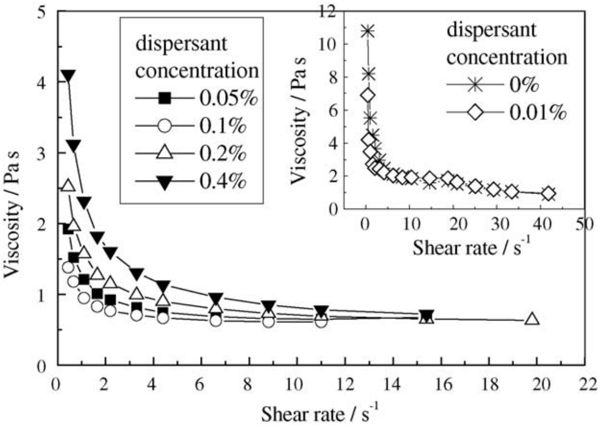

Fig. 2. Variation in viscosity as a function of shear rate for 75 wt.% solid content alumina suspensions prepared with different dispersant concentrations.

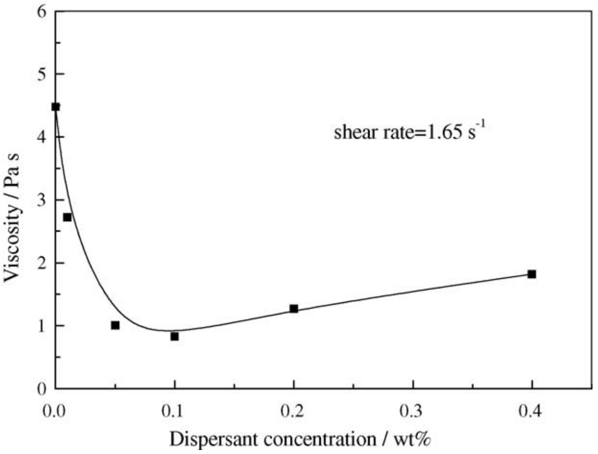

Fig. 3. Variation in viscosity as a function of dispersant concentration for 75 wt.% alumina suspensions at a fixed shear rate of 1.65 s^{-1}.

The effect of the addition of 15 wt.% of each of the three different binders on the slurry viscosity as a function of dispersant concentration at a fixed shear rate of 1.65 s^{-1} is shown in Fig. 4. It can be seen that each binder affected the slurry differently, changing both the viscosity and the optimum dispersant concentration, a result also observed by Kristoffersson et al. [22]. This behaviour may be explained by the pH of the different suspensions. Whilst it was 9.9 for the alumina suspension without any latex binder present, when 15 wt.% of the binders were added it decreased to 9.3 (acrylic/styrene latex), 9.0 (vinyl/acrylic latex) and 4.7 (vinyl acetate/acrylic latex).

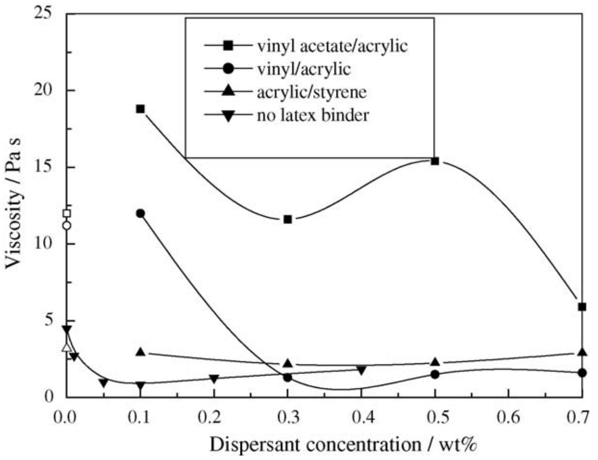

Fig. 4. Variation in viscosity as a function of dispersant concentration for 75 wt.% alumina suspensions containing 15 wt.% binder additions at a fixed shear rate of 1.65 s^{-1}. The viscosities of the latex binders without dispersants or alumina are also plotted in the figure for comparison.

It can be seen that the greater the deviation of the suspension pH from that of the original slurry, the more the rheology also varied. Hence, the suspension containing the acrylic/styrene latex binder showed the most similar behaviour, although the (very shallow) minimum appeared to shift to ∼0.4 wt.%. The latter could be due to increased adsorption of the dispersant as a result of the slight pH change increasing the quantity of positive charges on the alumina particle surfaces and decreasing the dissociation of the polyelectrolyte [6,26]. A broadly similar result was obtained when vinyl/acrylic latex was used. Again, the minimum viscosity was shifted to a higher dispersant concentration although the viscosity at dispersant concentrations <0.3 wt.% was significantly higher, presumably due to inadequate dispersion since the pH was closer to the isoelectric point for the alumina, ∼pH 8.6 [6]. In contrast, although the general shape of the curve when 15 wt.% of vinyl acetate/acrylic latex (pH 2.5) was added to the suspension was similar to that for the vinyl/acrylic latex system, the viscosity was much higher at all dispersant concentrations, presumably because the resulting pH of ∼4.7 was well below the isoelectric point of the alumina. Whilst the precise explanation for this rheological behaviour is not fully understood, it is possible that it is associated with the adsorption of both the dispersant and latex particles onto the alumina particle surfaces. At such a low pH, the alumina particle surfaces will have been highly positively charged and the degree of dissociation of the –COONH$_4$ moiety of the polyelectrolyte will have been very low [26]. Therefore, it is possible that the anionic latex particles could have been adsorbed onto the particle surfaces via a degree of electrostatic interaction, causing them to aggregate and the viscosity to increase. Irrespective of the actual mechanism involved, the viscosity was still practical for tape casting.

In order to examine the effect of binder content on the porosity of the membrane, three binder contents were used to prepare the slurries for tape casting, viz. 10, 15 and 20 wt.%. Whilst the viscosities of all the slurries increased with increasing binder content, all could still be tape cast satisfactorily. In addition, for all the binder systems the addition of small amounts of plasticizer and anti-foaming agent had no significant effect on the suspension viscosities.

3.2. Characterization of the green tapes

Drying is one of the most important steps during the tape casting process since it has a great influence on the final properties of the tape. The benefit of incorporating latex as a binder is that it is a stable colloidal dispersion of a polymer in an aqueous solvent and forms a film around the ceramic particles as the water phase evaporates, providing a degree of flexibility to the tape. To be successful, two important conditions must be met during the film formation [28]: first, compressive forces must develop on the latex particles as the solvent evaporates, and second, the glass transition temperature, T_g, of the latex must be lower than the working temperature. The former is important because it forces the latex particles to distort and form a continuous, cross-linked film within the tape, whilst the latter ensures that, once formed, the film confers sufficient flexibility at the working temperature to allow easy handling and storage.

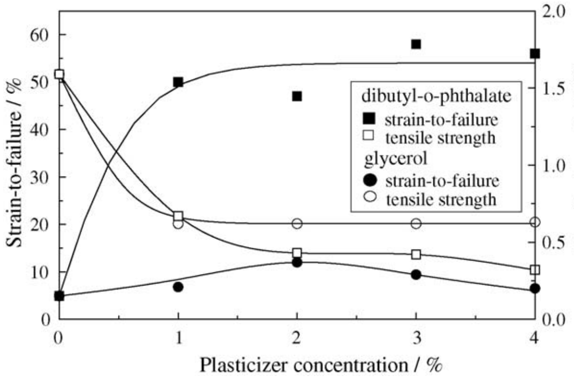

Fig. 5. Strains-to-failure and tensile strengths of green tapes prepared with 15 wt.% vinyl/acrylic latex binder as a function of plasticizer concentration.

The primary role of the plasticizers that are generally added to tape casting suspensions is to interact with the binder and reduce its T_g to below room temperature, thus allowing (a) a wider range of binder materials to be considered; and (b) lower working temperatures to be used. The most common plasticizers are the glycols and phthalates, such as glycerol and dibutyl-*o*-phthalate. However, whilst the use of a low molecular weight plasticizer increases the flexibility of the green tape, it is at the expense of the tensile strength. Hence, to obtain the optimal flexibility and strength, the correct binder/plasticizer system must be selected and the relative concentrations adjusted appropriately.

Figs. 5–7 show the tensile strengths and strain-to-failures of the green tapes prepared with various binder/plasticizer combinations. For both the vinyl/acrylic and vinyl acetate/acrylic latex systems, as expected the green tapes exhibited the highest strengths when unplasticized, however they also had failure strains of only ∼5% as a result of the relatively

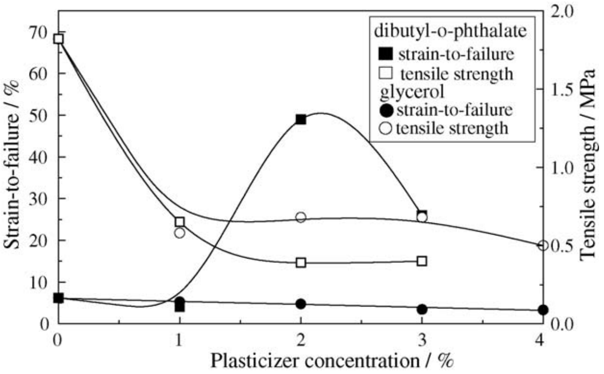

Fig. 6. Strains-to-failure and tensile strengths of green tapes prepared with 15 wt.% vinyl acetate/acrylic latex binder as a function of plasticizer concentration.

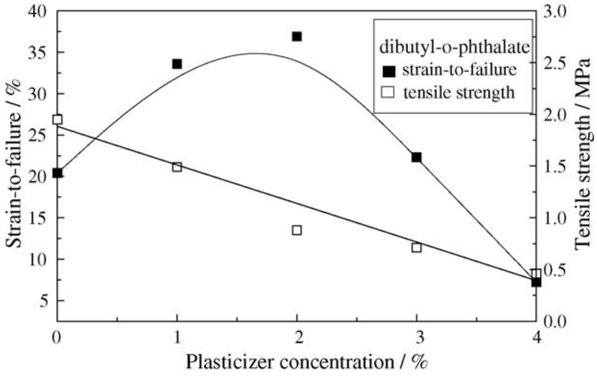

Fig. 7. Strains-to-failure and tensile strengths of green tapes prepared with 15 wt.% acrylic/styrene latex binder as a function of plasticizer concentration.

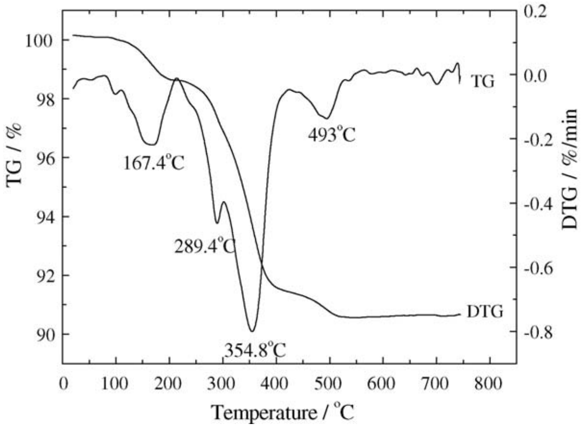

Fig. 9. The TG/DTA curve of a green tape containing 10% acrylic/styrene latex binder.

high T_g values of both binders, 28 and 21 °C, respectively. For the vinyl/acrylic latex binder system, whilst both plasticizers decreased the tensile strength by more than 50% with only 1 wt.% additions, the glycerol had relatively little effect in terms of improving the strain-to-failure at any additive level. In contrast, the dibutyl-*o*-phalate increased it by an order of magnitude, although additions beyond 1 wt.% were not beneficial. The results were broadly similar for the vinyl acetate/acrylic latex binder system in that the addition

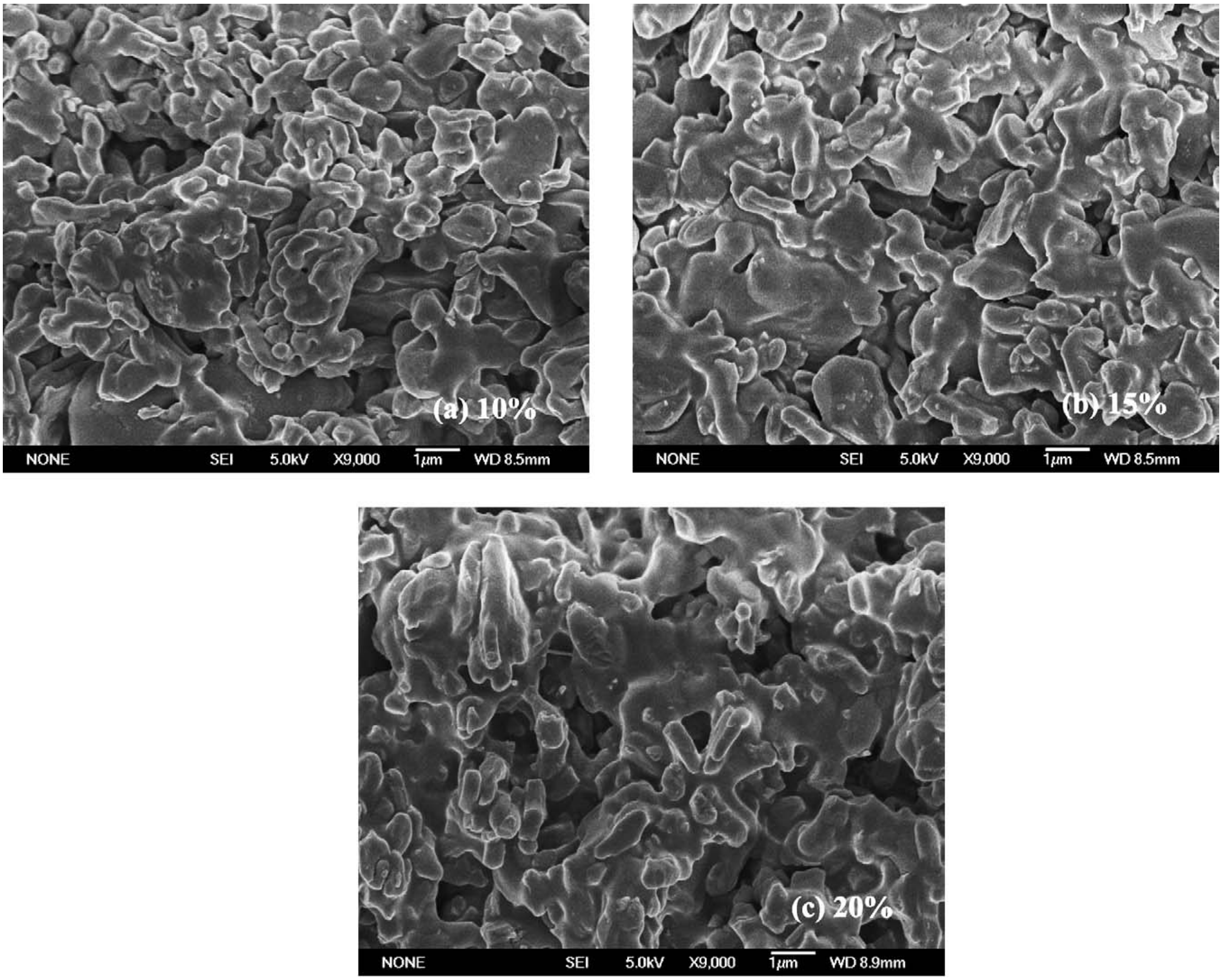

Fig. 8. Microstructures of the green tapes prepared with acrylic/styrene latex binder contents of (a) 10%; (b) 15%; and (c) 20%.

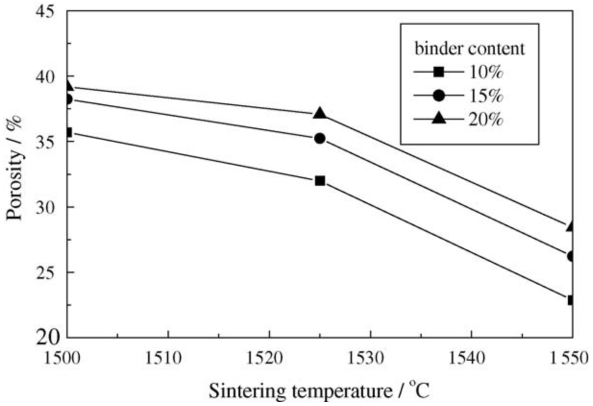

Fig. 10. Porosity as a function of sintering temperature for the acrylic/styrene latex-based tapes with different binder contents.

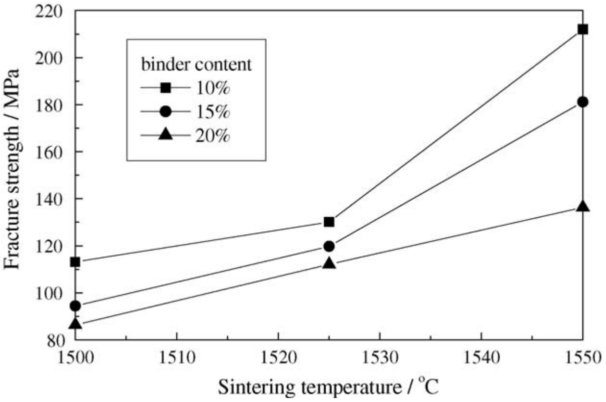

Fig. 11. Fracture strength as a function of sintering temperature for the acrylic/styrene latex-based tapes with different binder contents.

of only 1 wt.% of either plasticizer decreased the tensile strength substantially. However, whilst this time the glycerol actually decreased the strain-to-failure, the dibutyl-*o*-phalate only resulted in a sharp maximum occurring at approximately the 2 wt.% addition level. These results suggest that whilst the glycerol was unsuitable as a plasticizer in both binder systems, probably because it worked as a lubricant without interacting with the polymer network, the dibutyl-*o*-phthalate acted as a true plasticizer. According to Descamps et al. [7], the mechanism is believed

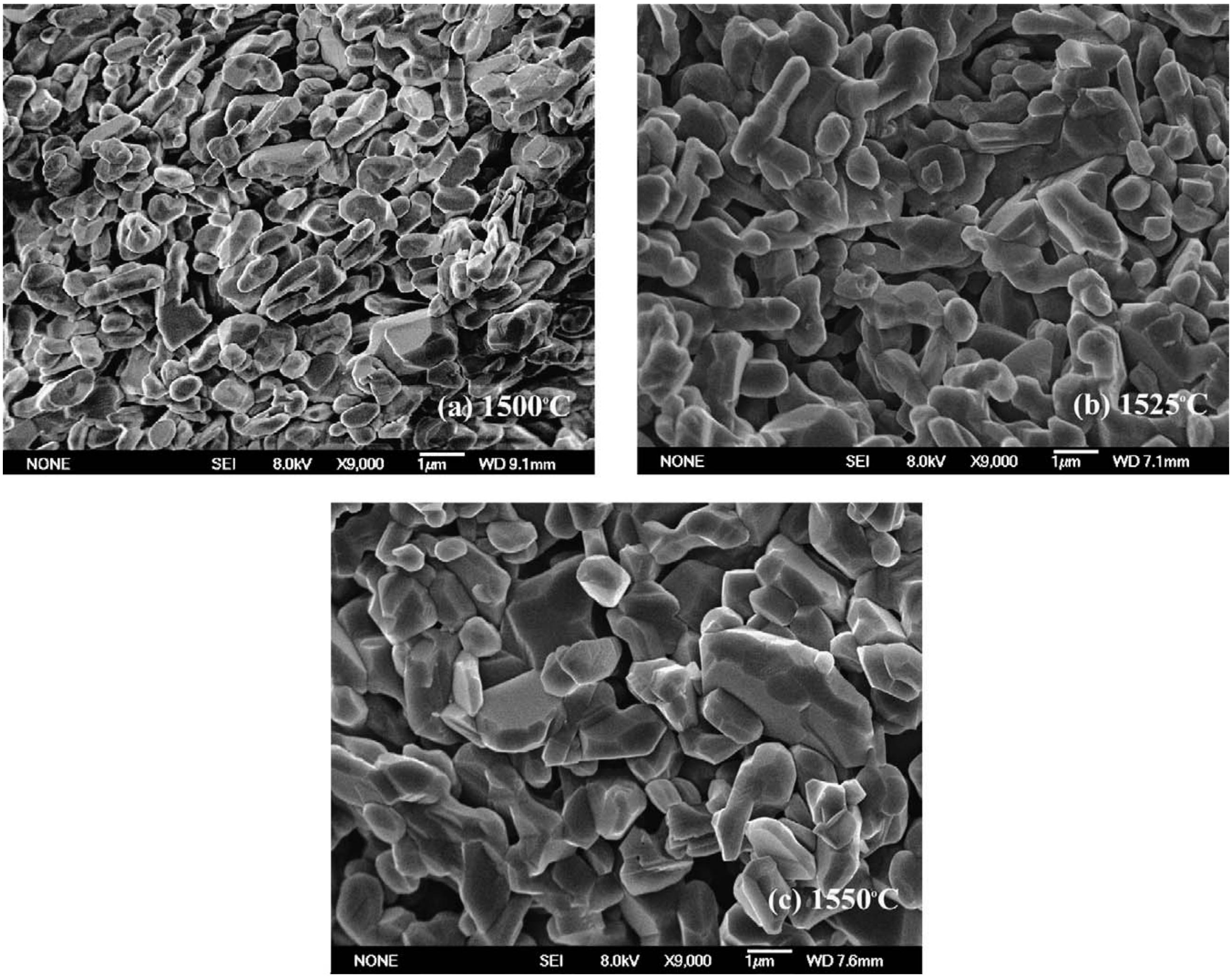

Fig. 12. Membrane microstructures after sintering at (a) 1500 °C; (b) 1525 °C; and (c) 1550 °C for 3 h.

to be via insertion into the latex film formed and the destruction of the local intermolecular strength responsible for the mechanical cohesion of the network.

As a result of the low T_g of 18 °C, the tapes based on the acrylic/styrene latex system showed reasonable flexibility at room temperature even without additions of a plasticizer, as evidenced by a strain-to-failure of ~20%. However, this was not sufficient to permit the production of tapes with a thickness greater than ~1.2 mm; the flexibility was not high enough to allow handling, especially rolling. Given the lack of success with glycerol, only the dibutyl-*o*-phthalate was investigated (Fig. 7). Whilst the strain-to-failure could be approximately doubled with ~2 wt.% additions, this was not as large an increase as that observed with this plasticizer in either of the other binder systems. Nevertheless, an added benefit was that the tensile strength was much higher and only decreased slowly, and roughly linearly, with increasing plasticizer concentration. At a strain-to-failure of 30%, the tensile strength was about 1.5 MPa compared with about 1 and 0.5 MPa for the tapes prepared with vinyl/acrylic and vinyl acetate/acrylic latex, respectively, at the same failure strain. This result indicates that the flexibility of the tapes could be significantly improved with less sacrifice in strength.

Based on these results, further investigation focused on the acrylic/styrene latex binder system. Fig. 8 shows the microstructures of the green tapes formed with binder contents of 10, 15 and 20 wt.% and a dibutyl-*o*-phthalate concentration of 1 wt.%. As expected, the micrographs show homogeneously packed particles with less porosity and more polymer present as the binder content increased. The SEM observation also showed that no sedimentation of the large particles occurred during the drying process.

The TG/DTA analysis of the green tapes with a 10% acrylic/styrene latex binder content is shown in Fig. 9; decomposition appears to occur at four different temperatures with the bulk of it centred at 354 °C. Hence, a particularly slow heating rate was used from 300 to 354 °C and a 2 h hold inserted at the latter temperature.

3.3. Characterization of the sintered tapes

Figs. 10 and 11 show the porosity and fracture strength, respectively, of the sintered tapes as a function of sintering temperature and binder content. As expected, higher sintering temperatures and lower binder contents lead to lower porosity and higher strengths. The sintered microstructures in Fig. 12 for membranes made with a binder content of 15 wt.% and sintered at the three different temperatures confirm these results. Much greater densification, and grain growth, has occurred at the higher temperatures yielding a less useful pore structure. When taking into account the ability to de-air the slurry and the porosity requirement of the membrane, the optimal binder content was identified as 15% and the sintering temperature as 1500 °C. This yielded a substrate with a porosity of 38% as measured by the Archimedes method and 36% by mercury intrusion porsimetry. The pore size distribution is shown in Fig. 13; it is monomodal with an average diameter of ~0.5 μm. The tapes had a fracture strength of ~95 MPa, which is believed to satisfy the requirements of the ceramic membrane.

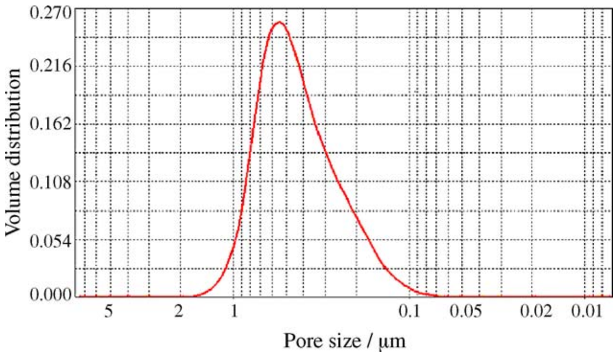

Fig. 13. Pore size distribution of a membrane as determined by mercury intrusion porosimetry.

A fracture surface of the sintered assembled tapes at the corner where two parts of the tape met is shown in Fig. 14a. The large void on the left-hand side is where the upper part of the tape had chipped slightly, as shown by the inset figure;

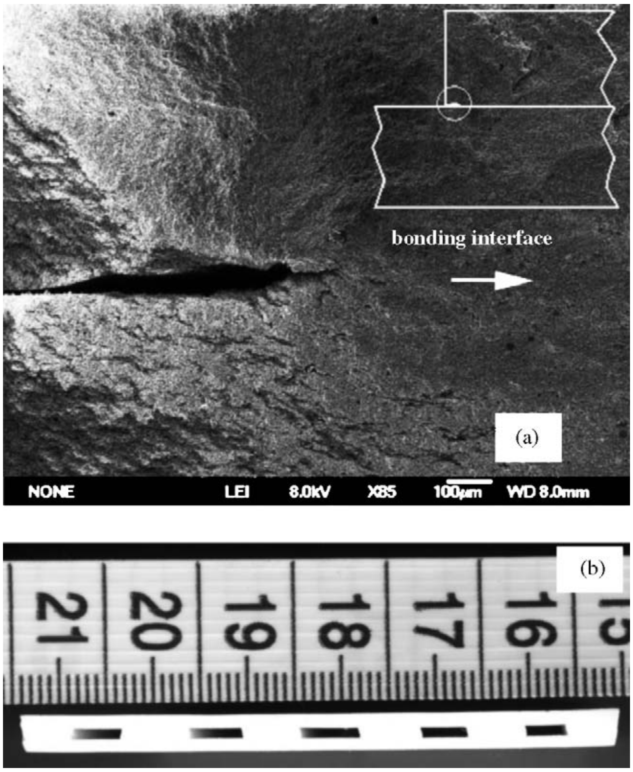

Fig. 14. (a) Fracture surface of the joint structure for 15 wt.% acrylic/styrene latex-based tapes; inset shows the region where the image was taken; and (b) a photograph of the cross section of the prototype multi-channel membrane substrate constructed as shown in Fig. 1 from the same tape.

however, the absence of an interface is clearly visible on the right-hand side of the image. A cross-section view of the sintered prototype multi-channel ceramic membrane is shown in Fig. 14b. The lack of any deformation or warping is clearly seen suggesting that each stage of the process has been optimized successfully.

4. Conclusions

Three latex binders and two plasticizers have been studied for the tape casting of porous alumina membranes. The acrylic/styrene latex system appears to be the most compatible with the alumina suspension in terms of the slurry rheology, possibly because of the non-ionic nature of the latex, and also yielded higher strength green tapes for the same strain-to-failure. The plasticizer dibutyl-*o*-phthalate proved to be the more successful of the two investigated, with glycerol failing to significantly increase the strain-to-failure for any of the binder systems. As expected, both binder content and sintering temperature affected the porosity and fracture strength of the final membrane. The optimal system in terms of processability, porosity and strength as determined in the present work involved using 15 wt.% acrylic/styrene latex as the binder, 1 wt.% dibutyl-o-phthalate as the plasticizer and a sintering temperature of 1500 °C. This lead to a membrane porosity of $\sim$37% and fracture strength of 95 MPa as well as less grain growth within the microstructure. The work also demonstrated that relatively simple assembly methods could be successful in terms of fabricating thin, multi-channel ceramic membrane substrates.

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