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Alumina developed by pre-gelling starch consolidation (PSC)

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Abstract

In this paper, pre-gelling starch was used to consolidate alumina-dense ceramic suspensions. The colloidal processing of the ceramic was prepared with alumina and commercial potato starch, and slips were prepared with 55 vol% of solids and 0.5 wt.% of starch. This small amount of starch was possible because of a previous pre-gelling starch treatment, resulting in more homogeneous suspensions and particles smaller than starch granules. Additionally, sucrose was also used as a dispersion aid. After sintering, the samples were analysed according to their mechanical properties. These processes produced ceramics with a 93% relative density, 325 MPa flexural strength, and a Weibull module whose value was $m = 10$, maintaining the capacity of this process to produce complex geometric shaped ceramics.

Keywords: Pre-gelling starch consolidation; Al_2O_3; Dense ceramics

1. Introduction

Several methods have been developed for obtaining ceramic suspensions, such as that of Lyckfeldt and Ferreira[1] in their studies of porous ceramics developed using the starch consolidation method. Since then, new works aimed at process optimisation and the use of new precursors have continued to be released.[2–7] The use of starch to control porosity has also been employed in other forming methods, such as slip and tape casting techniques,[8] foaming combined with gelling,[9] and extrusion.[10]

Starch consolidation is a “direct casting method” based on gel casting[11,12] in which the polymerisation reaction is substituted with a binder agent, the starch. Ceramic powder, water, and starch are mixed together to obtain a colloidal suspension. The slip is poured into an impermeable mould, which is put into an oven at 55–80 °C for gelling. Rigid body consolidation is reached without compaction or water removal. The starch absorbs water and swells until it becomes a gel, forcing the powder particles into contact with each other so that a rigid structure is formed. The mould is placed into the oven again for drying to allow for demoulding.

After drying, the sample is subjected to calcination (pre-sintering) for removal of the organic material (resulting in a porosity proportional to the starch amount used), and finally to a sintering process in which the ceramic acquires a substantial mechanical resistance. This forming technique via starch consolidation allows for the use of a vast series of simple and complex impermeable moulds.[1–7]

Chemically modified potato starches were the basic material used in the first works on this subject.[1–4,8,9] Since then, commercial starches, without modification, have been studied, mainly due to low cost and easy reproduction. The presence of possible toxic addictives was avoided.[5–7,13,14]

In general, research that uses the “direct casting method” has been utilized for obtaining porous ceramic or low density bodies (less than 90% of theoretical density). The largest limitation to obtaining dense ceramics by these techniques is the ability to include a small enough amount of binder agent to produce a ceramic body green with reduced porosity and enough mechanical resistance.[15]

The use of this process for production of dense ceramics was presented in a PhD thesis published in Brazil in 2001, in which the materials used included alumina (58 vol% of solids) and 5 vol% of commercial corn starch, which resulted in a relative densification of 87.5%.[16]

The characteristics of alumina ceramics obtained with a high theoretical density (98% or more) are shown in Table 1.[17] These

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Table 1
High density alumina ceramic characteristics

Parameters	Value	Unit
Flexural strength (σ_{50})	380 ± 50	MPa
Characteristic strength (σ_0)	395 ± 25	MPa
Weibull module (m)	11	–
Hardness (Vickers, 1 kg)	15 ± 2	GPa

values, however, depend on several factors, such as the material purity, the addictives used for densification, the forming technique, the temperature and sintering atmosphere, and the superficial finish, among others. In the uniaxial pressing method, for instance, a mechanical resistance around 300–400 MPa is obtained, as can be observed in the following examples. Moraes et al.[18] obtained ceramic bodies with a 99.13% relative density and 397 MPa of mechanical resistance using Alumina SG-1000 compacted with a 55 MPa uniaxial pressing operation and sintered to 1600 °C. In contrast, Maensiri and Roberts[19,20] obtained a 345 MPa mechanical resistance for sanded ceramic bodies and a 371 MPa for polished bodies, both prepared with a 42 MPa uniaxial pressing and subsequent hot isostatic pressing (CIP) with 200 MPa before sintering to 1600 °C.

The use of smaller granuled starch allows for a large ceramic powder involvement. This guarantees better resistance to green when there is a small amount of starch. However, to obtain dense ceramics, it is necessary to maximally reduce the water and starch amounts. This not only reduces the resistance to green but tends to produce samples with lesser homogeneity, diminishing the densification.[15]

An alternative found for obtaining dense ceramics is the substitution of powdered starch for the pre-gelling starch. This way, slip homogeneity is obtained and starch granule swelling for porosity formation is eliminated from the process. When "powdered" starch is used, size, form, and distribution of granules are parameters of great importance for controlling the porosity and final density of the ceramic.[13]

The characteristics of the starches in terms of size and form vary according to the type of starch. Rice starch has the smallest granule size, while potato starch has the largest grain size. In Table 2, the distribution of granule sizes for the starches is shown (according to International Starch Institute).[21]

However, with the use of the pre-gelling starch, size and form of the granules are irrelevant and other characteristics should be considered, such as viscosity curves or a viscoamylograph, which can be measured using rheometers like the Brabender viscoamylograph or Rapid ViscoAnalyser (RVA), which are used widely in the food industry.

Table 2
Granule size of various starches

Starches	Granule size range (μm)	Average size (μm)
Corn	5–25	14
Cassava	3–30	14
Arrowroot	9–40	23
Potato	10–100	36
Waxy rice	2–15	6

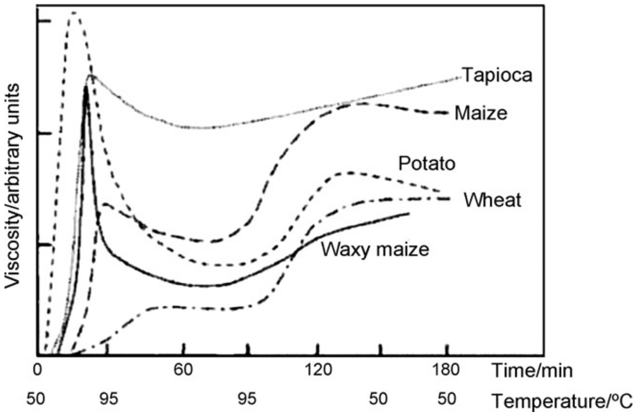

Fig. 1. Brabender amylograph curves of various starches.[22]

The viscoamylograph, displayed schematically in Fig. 1, shows the effect of temperature on the swelling and dissolution of native starch granules and the corresponding effect on the viscosity of the diluted system, which is about 10% solids.[22]

The graph in Fig. 1 depicts an area of significantly increasing viscosity during the heating phase, indicating swelling of the starch granules. Soon afterwards, the viscosity decreases due to the subsequent breakage of the swollen granules. This occurs because of the weakness of the granules and agitation of the solution produced by the equipment. After a period of constant temperature and during cooling, the viscosity increases once again, due to gelling of the starch paste.[22]

Potato starch possesses a larger capacity for swelling. Long ramified chains of amylopectin, with a polymerisation degree 3.75 times larger than corn starch, make this starch the most viscous of the commercial starches.[23]

To complement the existing body of research and to understand the behaviour of the available starches used in the trade for pre-gelling (and ultimately to select the starch most suitable for the processing of dense ceramic), pre-gelling and gelling trials were conducted.

In this work, after selecting a starch and adjusting the processing conditions, it was possible to reach a 93% densification and 325.6 MPa mechanical resistance using alumina and potato starch pre-gelling. These values were obtained without additives and without a superficial sample finish.

2. Materials

Materials used are listed in Table 3.

Alumina was chosen for its excellent properties, such as small particle size and high purity, which are ideal conditions for densification.

Sugar was used as a deflocculation aid, based on several studies which show the use of very small oligo and polysaccharide concentrations (1–3 wt.%),[24] as well as mono- and di-saccharides[25] used for ceramic-forming applications. These additives improve the processing of dense ceramics significantly, because of viscosity reduction raises the content of solids in the slurry.

Table 3
Materials used in this work

Material	Manufacturer	g/cm^3
Corn starch[a]	Refinações de Milho, Brasil Ltda	1.52
Potato starch[a]	YOKI Alimentos S.A.	1.54
Cassava starch[a]	Dafap's Ind. e Com. de Produtos Alimentícios Ltda	1.55
Rice starch[a]	YOKI Alimentos S.A.	–
Arrowroot starch[a]	Mariza Alimentos S.A.	–
Alumina (A-1000 SG)	ALCOA S.A.	3.98
Disperlan (PL1-A)	LAMBRA Produtos Químicos Auxiliares	–
Sugar (sucrose)[a]	Copersucar	–

[a] Materials purchased at stores without chemical modifications.

Moreover, in Pradhan and Bhargavaw's article,[26] sugar was used with a dispersant (polyacrylate), resulting in viscosity reduction and in green bodies with higher resistance.

3. Experimental procedure

The trials for pre-gelling/gelling of the starches was accomplished in a previous stage of this work, and consisted of evaluation of the properties of several commercial starches.

Samples of corn, potato, cassava, arrowroot, and rice starches (in powder form—25% in mass) were placed into suitable plastic containers labelled from A to E, respectively. They were added to the same amount of distilled water and were homogenized. Soon afterwards, the containers were covered and placed in the greenhouse.

Heating was slow and controlled so that, starting from 50 °C, there was an increase of 5 °C every 15 min (0.33 °C/min). During this procedure, the samples were observed and photographed at regular intervals of time and temperature.

After reaching 85 °C, the greenhouse heating was turned off, and after cooling, the samples were uncovered and partially dried at 45 °C for 30 min. The tablets of starch that resulted were demoulded for qualitative evaluation of drying time and ease of demoulding.

The objective of this first trial was verification of the main properties of the commercial starches, such as the gelling temperature, consistency, and resistance of the gelling, in order to best to evaluate the gelling process, as well as to select the most appropriate starch for formation of dense ceramic bodies.

In a second stage, ceramic suspensions were prepared with 55 vol% of solids and 0.5 wt.% of potato starch (selected in the first stage as the most appropriate starch). Additionally, 0.5 wt.% of sugar was used to aid deflocculation. Other solutions were made with 3 vol% of powdered potato starch.

The materials used for the 3 vol% powder starch ceramics were mixed directly in a ball mill. The solution prepared with 0.5 wt.% of starch was initially pre-gelled; that is, the starch was placed in a water flask, covered, and heated to 80–85 °C for 10 min under agitation so that the pre-gelling could occur, resulting in almost a paste of starch. The components were then mixed and homogenized directly in a ball mill. Breakage of the starch granules occurred during material mixture or grinding because the swelling weakens the granules. This produces a much smaller and more uniform granulation than that produced by the powder starch alone.

Fig. 2 shows the differences in starch gelling in the ceramic matrix. In (a), the starch granules swell and form pores, while in (b), when pre-gelled, the starch paste starch allows closer proximity among the alumina grains.

The slip obtained by pre-gelling was submitted to a de-airing process via low pressure for air removal. Next, the slip was poured into an impermeable mould, which was covered in order to avoid water evaporation, and placed in an oven for 30 min so that gelling could take place at a heated temperature range of 80–85 °C.

The material remained in the oven for cooling, and then the mould was uncovered for drying at 110 °C for 2 h. The samples were then demoulded.

The samples obtained showed good mechanical resistance and were placed in an oven for pre-sintering at 1000 °C and then sintering at 1600 °C for one hour. The use of various cycles

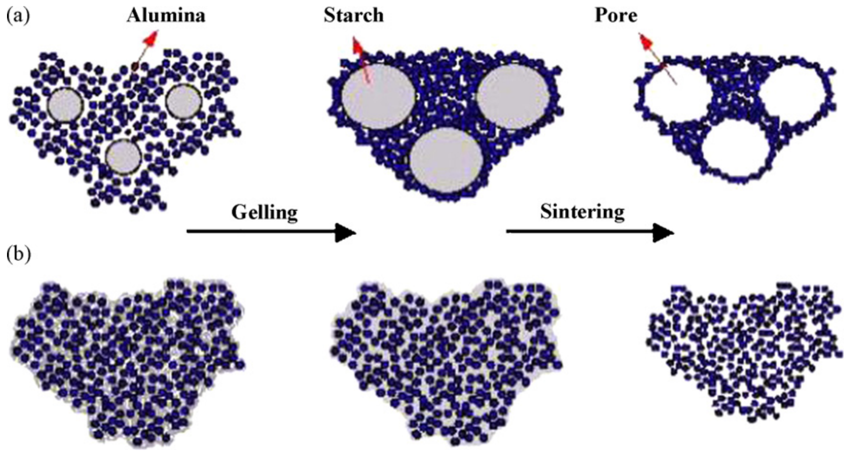

Fig. 2. Starch gelling in (a) granule form: water absorption and swelling with subsequent pore formation and (b) pre-gelled: reduced water absorption without pore formation, resulting in a more dense material.

for burning and sintering is a very low cost resource and allows for better control of the heating rates.

The ceramic was characterized in terms of linear shrinkage, mass loss, porosity, and apparent density (ASTM C20-00 norm), and flexural strength using the three-point flexural test (ASTM C1161-02c norm), with results analysed according to the Weibull statistical method and optical microscopy.

4. Results

Evaluation of commercial starches consisted of determination of the pre-gelling and gelling temperatures, the consistency, and the resistance of starch gelling, which was conducted through visualization of the swelling and colour alteration of the starches during heating, as well as observation of the characteristics of the formed tablets.

4.1. 1st stage:

4.1.1. Temperature of pre-gelling

Swelling was observed by the elevation of the level of starch in proportion to the water. The most significant images of this stage are shown in Fig. 3, accompanied by the respective temperature ranges.

The trials demonstrated that swelling of the starch does not occur until the temperature reaches 50 °C. Starting from a certain temperature, the starch that is initially decanted in the bottom of the container begins to dilate with the absorption of water, until it has taken in the entire volume of water, forming a whitish paste.

4.1.2. Temperature of gelling

After the swelling of the granules of starch, with continuing heating, an alteration is observed in the coloration of the pastes, which progresses from a whitish colour to a more or less transparent gel, according to the type of starch, as shown in the images in Fig. 4.

Based on this trial, illustrated in Figs. 3 and 4, it was observed that the gelling of starches can be divided into two stages: the first consists of pre-gelling of the starch (absorption of water and swelling of the granules, forming a whitish paste), and the second is marked by the gelling of the starch (conversion of the paste to a gel accompanied by alteration from whitish coloration to transparency). The corresponding temperatures of these two stages are described in Table 4.

The best results from this trial were obtained for a diameter/height ratio equal to 2.5 for the solution of starch in water. The initial tests were done in test tubes, which were then discarded in favour of plastic containers, as shown in the images.

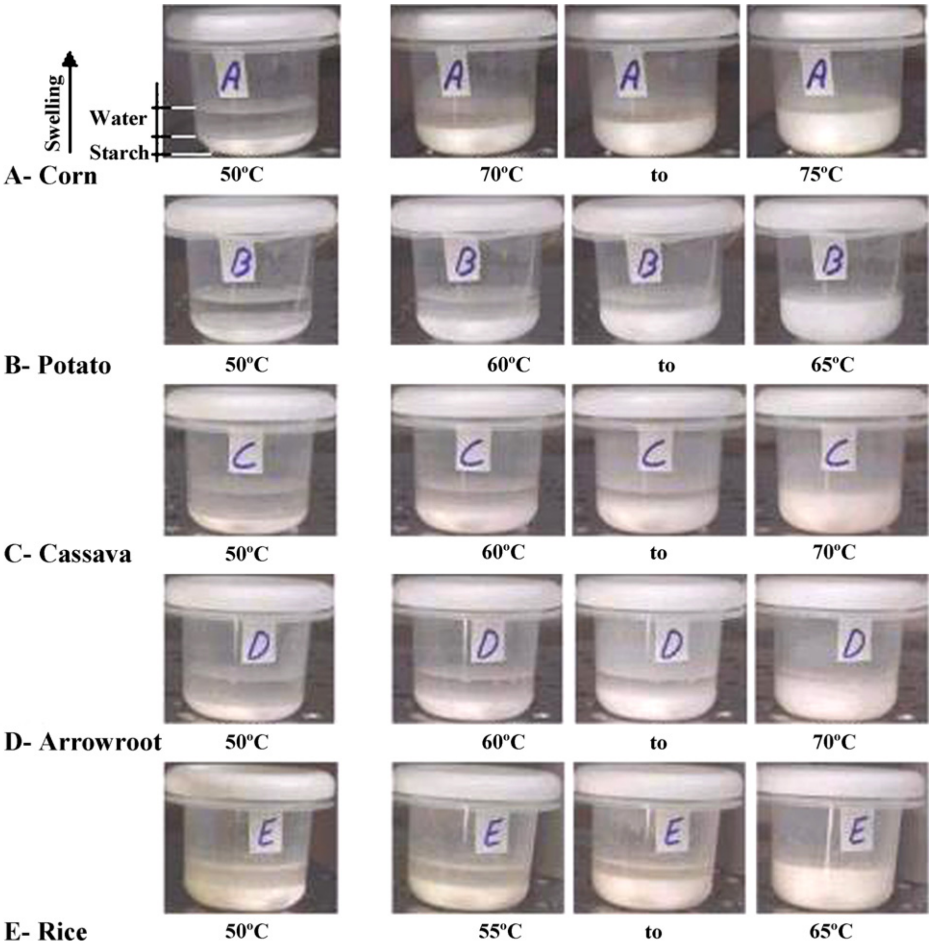

Fig. 3. Images of starch samples gelled by elevation of the temperature, exhibiting the swelling of the starch (proportion starch/water).

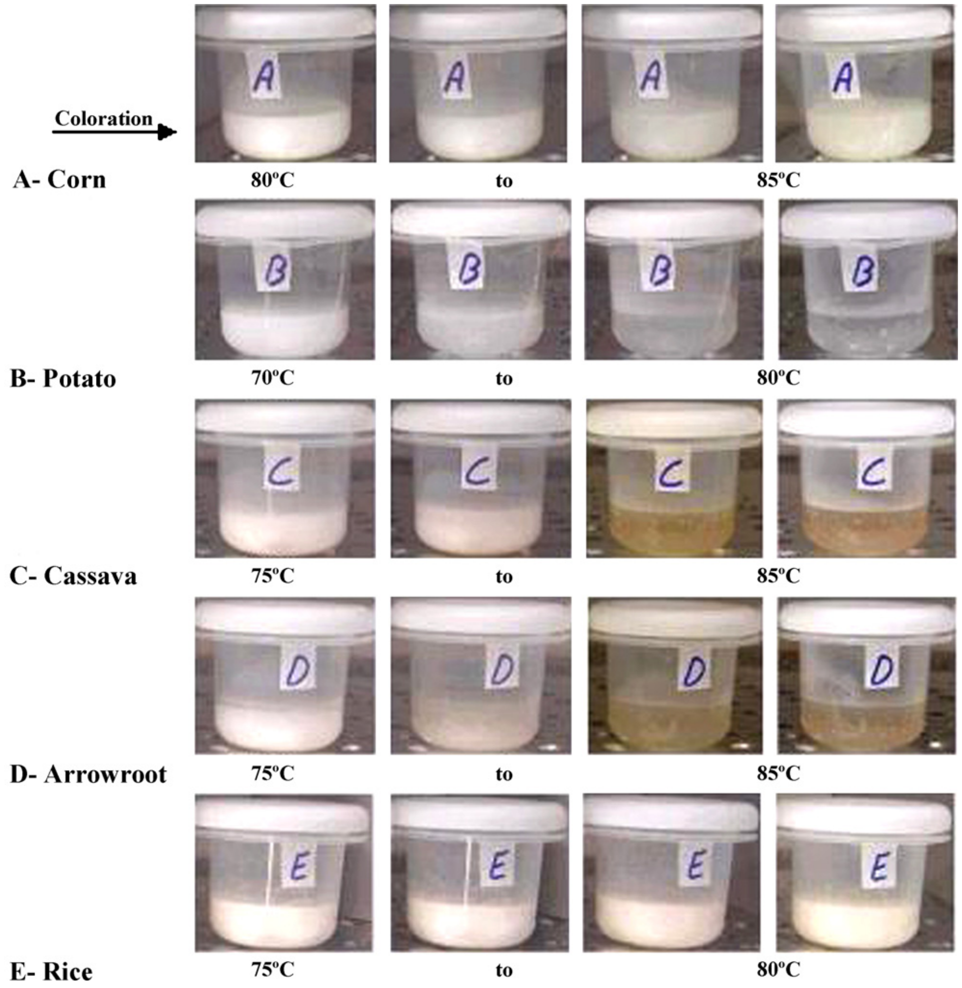

Fig. 4. Images of samples of starch gelled by elevation of the temperature, exhibiting the ranges of coloration alteration.

It was also observed that in low concentrations, the starch swells but does not occupy the whole volume of the water. This effect is more accentuated in the starches with smaller swelling ability such as the starches from cassava, arrowroot, and rice (according to the viscosity curves of Fig. 1). In these cases, the agitation during the heating favours the formation of a more homogeneous paste during pre-gelling.

4.1.3. *Characteristics of the starch tablets*

The plastic containers were uncovered and partially dried at 45 °C for 30 min. The resulting starch tablets were then demoulded (Fig. 5a), and their qualitative characteristics were observed (Table 5).

The difference in final coloration among the starches is due to the presence of other substances, indicating the degree of purity. For example, because the potato starch tablet was the most transparent, it was probably the purest.

During drying to room temperature, the corn and rice tablets split and deformed, as shown in Fig. 5b. The cassava and arrowroot tablets presented similar characteristics, initially showing a sticky consistency soon after demoulding, but forming a resistant and elastic external film with time. The potato starch tablet formed a resistant external film and was firm.

Based on these trials with commercial starches, we can observe that the potato starch produces firmer and more resistant gels with easy demoulding and good resistance after drying. The

Table 4
Pre-gelling and gelling interval for the commercial starches

Starch	Pre-gelling (°C)	Gelling (°C)
(A) Corn	70–75	80–85
(B) Potato	60–65	70–80
(C) Cassava	60–70	75–85
(D) Arrowroot	60–70	75–85
(E) Rice	55–65	70–80

Table 5
Qualitative characteristics of the starch tablets

	Starch	Initial aspect	Demould	Aspect after 24 h
A	Corn	Firm and smooth	Easy	Split
B	Potato	Firm and smooth	Easy	Resistant and firm
C	Cassava	Soft and sticky	Difficult	Resistant and elastic
D	Arrowroot	Soft and sticky	Difficult	Resistant and elastic
E	Rice	Brittle and smooth	Medium	Split

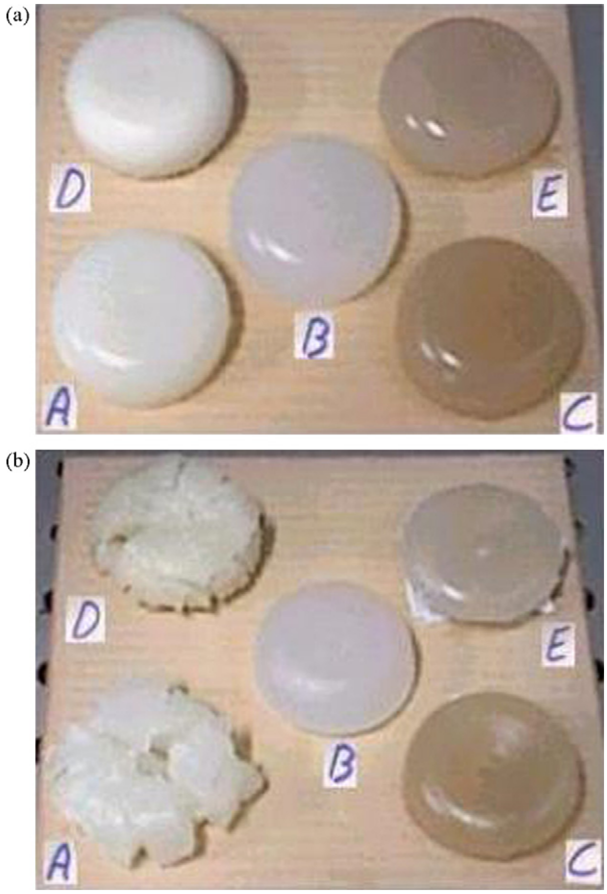

Fig. 5. Tablets from starch gelling: (a) recently demoulded and (b) after 24 h.

largest capacity for swelling is due to the dissolution of a larger amount of amylose and of amylopectin, which are responsible for the gelling and resistance of the gel, respectively.

Based on the best results, potato starch was chosen for the formation of the dense ceramic pieces.

4.2. 2nd stage:

4.2.1. Trials with the obtained ceramic

The values obtained in the trials with ceramic were confirmed with 55 vol% of solids containing 3 vol% of powdered potato starch (for comparison) and 0.5 wt.% of potato starch with and without 0.5 wt.% of sugar (to aid of deflocculation); results are shown in Table 6.

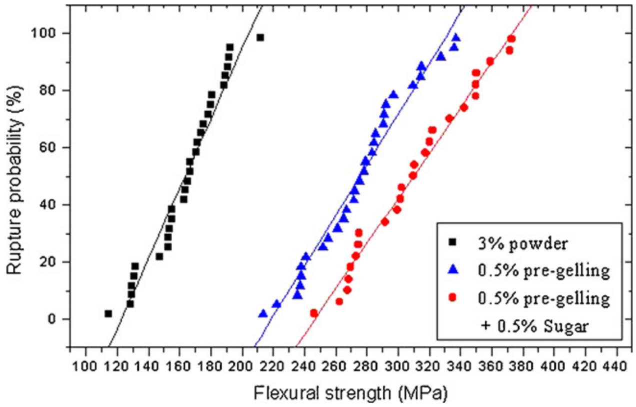

Fig. 6. Weibull graph for probability as a function of flexural strength for ceramic obtained by using 3 vol% of power starch and 0.5 wt.% of pre-gelling starch, with and without 0.5 wt.% of sugar addition.

With a small amount of starch, the contraction was practically unchanged (around 15%) while the mass loss (proportional to the amount of used starch) was smaller for the compositions with smaller amounts of starch.

In the work of Campos[16] using commercial potato starch, compositions with 55 vol.% of solids and 10–20% starch had mass loss around 33% and the contraction figure was around 17%.

In general, in the starch consolidation casting technique (and whenever it makes use of fugitive material), increases in the baking temperature of the pieces should be slow to allow the liberation of gases produced by the fugitive material (starch). With the pre-gelling technique, a smaller amount of starch is used. Additionally, the starch will be dissolved and better distributed in the ceramic matrix. As a consequence, this technique allows larger mean heating rates to be used during pre-sintering because there is an only a small amount of organic matter to be eliminated.

It was observed that the use of the smallest possible starch amount (3 vol%), results in a 90% apparent density versus 93% with the pre-gelling starch, giving an increase of 3.5%, as well as a 173 MPa characteristic strength (σ_0) compared to 326 MPa for the pre-gelling starch. The presence of sugar did not affect the porosity or the produced piece density significantly, but did produce an increase in the mechanical resistance. This result is due to the viscosity reduction of the ceramic suspension and consequent homogenisation improvement. The difference in the mechanical resistance can be better visualized in Fig. 6, which shows the rupture probability as a function of flexural strength.

Table 6
Results

Parameters	3 vol% powder	0.5 wt.% pre-gelling	0.5 wt.% pre-gelling + 0.5 wt.% sugar
Linear shrinkage (%)	15.72 ± 0.52	14.51 ± 0.43	15.00 ± 0.18
Mass loss (%)	2.39 ± 0.04	1.45 ± 0.05	1.60 ± 0.03
Apparent porosity (%)	1.36 ± 0.14	1.43 ± 0.19	1.50 ± 0.10
Bulk density (g/cm^3)	3.58 ± 0.04	3.72 ± 0.02	3.71 ± 0.01
Apparent density (%)	90.03 ± 0.98	93.45 ± 0.52	93.23 ± 0.36
Flexural strength (σ_{50}) (MPa)	163.40	275.4	309.80
Characteristic strength (σ_0) (MPa)	173.20	289.20	325.60
Weibull module (m)	8.30	10.20	10.10

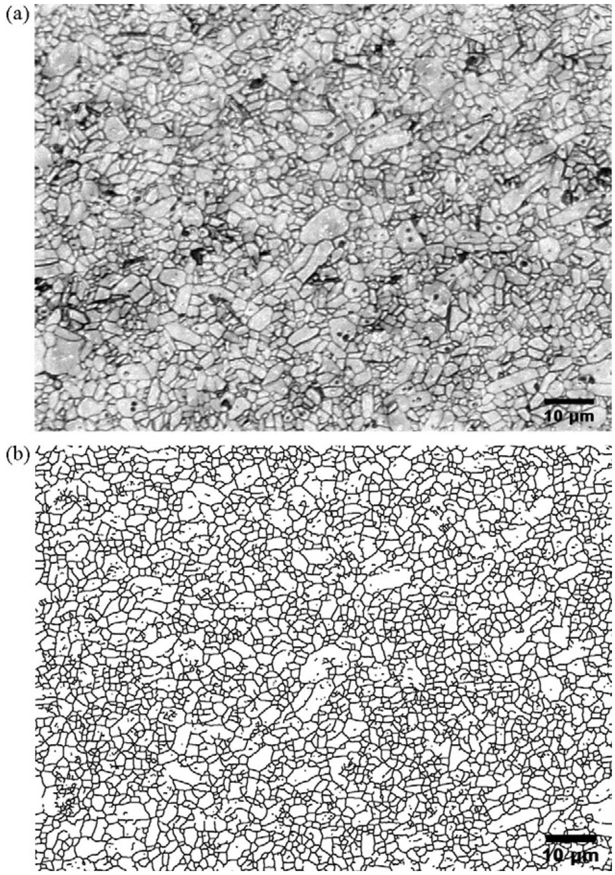

Fig. 7. (a) Alumina ceramic surface, obtained with pre-gelling starch and sugar, observed by optical microscopy and (b) grain boundaries after digital image processing.

In Fig. 7a, an optical image of a sample surface is shown. This sample was obtained with pre-gelling starch and sugar, making the reduced porosity clearly visible with an efficient alumina grain packing; in Fig. 7b, the alumina grain boundaries are shown after digital image processing.

Table 7 shows grain morphology parameters after digital image processing. The optical microscopy analysis shows a 2.1 µm average grain size, which in addition to the aspect ratio and circularity results, shows that the granules are not large and prolonged.

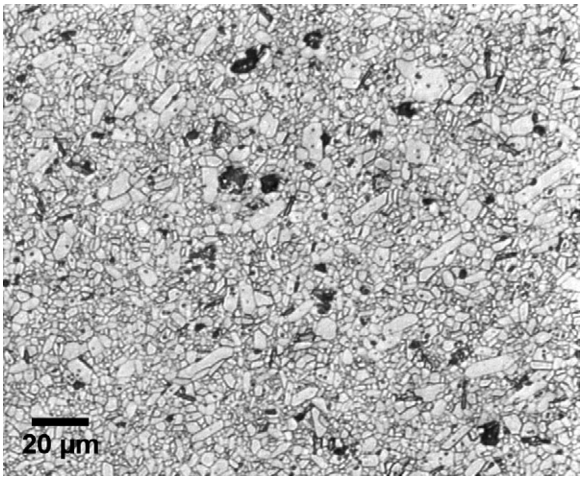

Fig. 8. Smaller pores due to residues of starch and larger pores due to air bubbles.

Fig. 9. Alumina dense ceramics obtained by pre-gelling starch consolidation.

Table 7
Alumina granule parameters

Parameters	Value
Area (μm^2)	2.61 ± 3.57
Length (μm)	2.08 ± 1.39
Feret's diameter (μm)	2.31 ± 1.53
Circularity	0.68 ± 0.16
Aspect rate	1.66 ± 0.47

However, the presence of pores was observed in the material (Fig. 8), most probably originating from particles of starch that did not pre-gel completely (residues) or did not dissolve during the grinding/homogenisation of the slip. Air bubbles present in the slip also can result in the formation of these pores. This indicates that for better results and elimination of pores, the best preparation practices of pre-gelling starch (avoidance of residues) and better preparation of the slip (avoidance of air bubbles) should be employed.

In spite of the difficulty in eliminating the porosity completely due to residues of the starch and air bubbles, the process using starch pre-gelling for production of dense ceramics has the advantages of low cost, the capacity for complex forms (Fig. 9), better homogenisation and material uniformity, and mechanical properties that are close to or superior to the ones obtained via uniaxial pressing processing.

5. Conclusions

The gelling of starches occurs during two stages, pre-gelling (absorption of water and swelling of the granules to form a whitish paste) and gelling (conversion of the paste to a gel accompanied of alteration of the whitish coloration to transparency).

Potato starch produces firmer and more resistant gels, and the large capacity for swelling allows for the dissolution of a larger amount of amylose and of amylopectin, which are responsible for the gelling and the resistance of the gel, respectively. For these reasons, it is the most suitable starch for the formation of dense ceramic pieces through pre-gelling.

The commercial starch granule size, which is larger than that of the ceramic powders, produces porosity and makes it difficult to obtain high density ceramics. Pre-gelling is a simple and viable procedure that eliminates this problem with the starch granule size.

The use of small starch amounts (3 vol%) produces samples with low apparent density and mechanical resistance, while the pre-gelling step allows the use of very low starch amounts (0.5 wt.%) to obtain ceramics with efficient mechanical resistance of the green compact.

The results obtained here, an apparent density of 93% and a characteristic strength (σ_0) of 325 MPa, confirm the viability of the method.

Better preparation of the starch for pre-gelling via grinding and screening can avoid the presence of starch residues which are the causes of pores. Notably, de-airing of the slip, as practiced in this work, was not enough to avoid the presence of air bubbles in the slip. Avoiding these problems will yield better results.

The technique presented in this work allows the production of dense ceramics with complex forms, with properties close to or superior to the ceramics obtained by uniaxial pressing. The use of commercial starch and a commercial aid additive (sugar) prevents harm to the environment.

The limits of the starch and commercial sugar amounts will be determined by rheological tests. This is the topic of future work already underway regarding diametrical compression.

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