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Microwave-assisted sintering of dental porcelains

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Abstract

An investigation was made of hybrid microwave-assisted sintering of dental porcelains, using five commercial ceramic frits employed in the production of dental porcelains. The powders were characterized, transformed into prismatic test specimens, and subjected to conventional and microwave sintering. Microwave sintering was performed at a frequency of 2.45 GHz, using a susceptor material and in the absence of vacuum. The apparent density and apparent porosity of the sintered samples were characterized based on the Archimedes principle. They were also analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM), and their flexural strength and microhardness were determined by the Vickers method. The powders, which showed a broad particle size distribution with a high fraction of particles of dimensions larger than 30 μm, were composed of amorphous phase and leucite particles. Microwave sintering yielded ceramic bodies whose apparent porosity (*t*-test, $p < 0.05$) was the same or very similar to that of the conventionally sintered samples, while the apparent density (*t*-test, $p < 0.05$) of most of the microwave sintered samples was the same or slightly lower. Although the microwave sintered samples showed larger average pore sizes (*t*-test, $p < 0.05$), four of the five samples used in this study showed the same flexural strength (*t*-test, $p < 0.05$) and all the ceramics under study showed the same surface microhardness (*t*-test, $p < 0.05$).

Keywords: A. Sintering; D. Porcelain; Dental ceramics; Microwaves

1. Introduction

Dental structure loss is caused by a variety of factors that range from diseases to trauma, as well as preventable hygiene-related problems. Allied to today's lifestyles, which require a good appearance, this problem has led to an increasing demand for dental restorations. This demand is translated into an increasing need for dental materials that allow for the creation of teeth esthetically and functionally similar to natural teeth [1].

In view of the above, increasing interest has focused on dental ceramic materials, due to their chemical and bio-inertness in the oral environment, suitable strength, and particularly the fact that these materials are the ones that best mimic the appearance of natural teeth. Dental ceramics include porcelains, which are ceramic materials that provide the best esthetic characteristics in dental restorations [2–4].

The vast majority of dental porcelains today consists of two phases: a feldspathic vitreous matrix and a dispersed phase of leucite particles ($K_2O\ Al_2O_3\ 4SiO_2$) (tetragonal leucite at room temperature) [5–7]. They are sometimes referred to as feldspar dental ceramics or feldspathic porcelains.

These materials are prepared by dental technicians, in the form of previously processed glass frit (glass powder), by means of fast firing cycles and they are extremely sensitive to processing cycles. This sensitivity may be caused by the considerable amount of liquid phase that develops very rapidly

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Table 1
Commercial products and their firing cycles recommended by the manufacturers.

Porcelain	Product/manufacturer/batch number	Drying time (min)	Standby temperature (°C)	Heating rate (°C/min)	Final temperature (°C)	Holding time (min)
A	EX-3/Noritake/019354	8	600	45	930	–
B	Starlight Ceram /Denstsply/1109000543	6	575	55	910	1
C	VMK 95/Vita/13380	6	600	55	930	1
D	VM 13/Vita/23160	4	500	55	880	1
E	VM 7/Vita/26470	9	500	55	910	1

in response to heating, or by possible changes in the quantity of leucite and the size of its crystals, which may also occur during heating and cooling. Inadequate firing cycles can affect the densification of the end material as well as its physical and mechanical characteristics [6,8,9].

In recent years, several studies [5,8,10,11] involving dental porcelains have used heating cycles produced by pressure or laser to sinter these materials. However, few studies have focused on the use of microwave-assisted heating cycles to consolidate dental porcelains, despite the known benefits of using microwaves in rapid heating cycles when compared with conventional heating in processing ceramic materials [12–15].

The literature on dental materials technology contains studies that used microwave energy to sinter zirconia [16–18]. These studies found that microwave processed materials showed similar mechanical properties, and in some cases smaller grain size and higher translucency, than conventionally processed materials. In the glazing step [19] of dental porcelain processing (not in the ceramic body consolidation step), microwave vitrified ceramics were found to present lower surface roughness.

As mentioned earlier, very few studies have focused on the use of microwave energy in the consolidation of dental porcelain bodies. Only two studies on this theme were found, one of them [20] involving the sintering of dental porcelain coating on the metal substructure of a Ni–Cr dental alloy, which reported an increase in porcelain–metal adherence with the use of microwaves. The second study [21], which used natural raw materials and not glass frit to produce dental porcelain bodies, reported higher values of strength when using microwave processing, but also found that the bodies were highly susceptible to the development of thermal runaway.

Microwave processing is characterized by the material's interaction with electromagnetic radiation and its volumetric heating [22,23], which allows for the use of high heating rates and the minimization of thermal gradients in the ceramic body, resulting in more homogeneous and refined microstructures. However, when there are marked changes in the material's dielectric loss in response to heating, part or all of the material may undergo uncontrolled heating, over-firing, deformation, melting, cracking, etc., making it very difficult to control the sintering process. This behavior is expected of materials that develop large amounts of glassy phase during heating, such as porcelains processed from glass frits.

An alternative to achieve better control of microwave heating of this type of material is the use of hybrid heating, which combines microwave heating and infrared sources [24,25] and allows for the minimization of uncontrolled heating (thermal runaway), better thermal homogeneity in the material during heating (because it minimizes thermal gradients) and rapid heating at low temperatures [24–28]. Thus, in this regard, in view of the paucity of studies on the theme and its technological potential, this work focuses on the hybrid microwave-assisted sintering of dental porcelains.

2. Materials and methods

Five low-fusing dental porcelains were used in the development of this research, as described in Table 1. The powders (glass frit) were characterized to determine their chemical composition by X-ray fluorescence (XRF) spectroscopy (Shimadzu EDX 750, equipped with a WDS detector); their particle size (the material was ultrasonically dispersed in distilled water) (CILAS 1064 particle size analyzer); by X-ray diffraction (step scan mode with CuKα radiation and 40 kV and 30 mA as working conditions) (Shimadzu XRD-6000); and by scanning electron microscopy (SEM, LEO 130).

After characterizing the powders, the test specimens were prepared one at a time by mixing the powders with distilled water on a glass plate to produce a ceramic paste. The quantity of powder and water was standardized for all the test specimens of each group of materials. The mixture was transferred to a metal mold (6 mm × 3 mm × 30 mm) and condensed under vibration (on a vibrating table). Excess water was removed from the surface of the test specimens using an absorbent cloth.

The test specimens were sintered conventionally in a furnace for dental ceramics (EDG, Alumni 50), applying the firing cycles recommended by the manufacturers (Table 1). All the test specimens were subjected to a heating process under vacuum, from standby to final temperature (including the holding time). The specimens were sintered in a multimode microwave oven (made in the laboratory), at 1.44 kW of radiation power and using a susceptor material (calcium hexaaluminate [29]) as heating aid (hybrid heating of the ceramic bodies). The microwave oven had two magnetrons of 900 W output power each, a microwave stirrer (mode stirrer), to achieve a more homogeneous distribution of microwaves in the chamber, and a chamber with dimensions of 400 × 300 × 400 mm^3. The microwave oven presented a power controlling system, which could operate with just one or with the two magnetrons. An optimal firing time was established for