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The effect of wollastonite incorporation on the linear firing shrinkage and flexural strength of dental aluminous core ceramics: A preliminary study

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ABSTRACT

The aim of this study is to evaluate the effect of wollastonite (CaSiO3) material on the linear firing shrinkage and flexural strength of dental aluminous core porcelain. Twenty five specimens were prepared in a manner that each five of them contained 0%, 2%, 3%, 4%, and 5% wollastonite by weight. The linear firing shrinkage values were determined according to the formula given in the standard ANSI/ADA (American National Standards Institute/American Dental Association) 69 and the flexural strength of the specimens was evaluated using 3-point bending test. The results of this study showed that addition of wollastonite reduced the linear firing shrinkage and increased the flexural strength of dental aluminous core porcelain. The highest reduction (approx. 20%) in the linear firing shrinkage was obtained by the addition of 5% wollastonite. Kruskal–Wallis test found a statistically significant difference among the mean linear firing shrinkage values of the porcelain groups at the 95% confidence level ($p < 0.05$). Mann Whitney U statistical test was performed to demonstrate which porcelain groups show statistically significant difference. In spite of achieving 30% increase in the flexural strength by the addition of 2% wollastonite, no statistically significant difference was found among the mean flexural strength values of the porcelain groups ($p > 0.05$).

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1. Introduction

Dental ceramics are usually processed by sintering, but in the last few years, processing techniques used for high-technology ceramic materials have been applied to dental ceramics, leading to the development of castable ceramics, machinable ceramics, pressable ceramics and infiltrated ceramics. The sintering process can be described as a complex sequence of high-temperature reactions occurring above the softening point of the porcelain and leading to partial melting of the glassy matrix, with coalescence of the powder particles [1]. The conventional powder/liquid particle processing of dental porcelains results in a significant amount of shrinkage during sintering densification of the powder. This is due to individual porcelain particles conglomerating as a result of surface melting or sintering [2]. The percentage of shrinkage depends on the degree of firing temperature, the degree of condensation or compaction of the porcelain powder in the shaping of the restoration before firing operation, the brands of porcelain and porcelain modelling liquids and the manipulative techniques [2,3]. The linear shrinkage of glazed porcelain has been reported to be approximately 14% for low-fusing porcelain and 11.5% for high-fusing porcelain. On the other hand, the low-fusing porcelain has a volumetric shrinkage of from 32% to 37% and the high-fusing porcelain can shrink as much as 28–34% [4]. Not only the conventional powder/liquid particle processing of dental porcelains but also high-technology ceramics such as pre-sintered ceramic of Al_2O_3 based on machining with CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) systems were subjected to high sintering shrinkage (15–20%) when the pre-sintered blocks at the green stage were sintered at 1550 °C [5].

Clinically the amount of shrinkage constitutes a problem for both metal-ceramic and all-ceramic restorations. Unrestrained shrinkage of ceramic would lead to marginal inaccuracy and decreased strength of ceramic restorations and multiple firing schedules to produce the required tooth contour and accuracy of marginal fit [1,3,6]. Marginal and internal accuracy of fit is valued as one of the most important criteria for the clinical quality and success of all-ceramic crowns [7]. Increased marginal discrepancy of a crown causes cement dissolution and microleakage which can lead to inflammation of the vital pulp [8,9]. During multiple firing schedules, due to heating and cooling cycles of the porcelain, transient and/or residual stresses can occur. The transient stress, if high enough, can cause immediate cracking of the porcelain, while residual stresses might affect the restoration's longevity [10].

The relative strength is of utmost importance for the long-term clinical performance of ceramic restorations. Mechanical failure of ceramic materials is almost completely controlled by brittle frac-

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ture. This brittle behavior, combined with the presence of surface flaws, has resulted in relatively low ceramic strength and has limited the use of ceramic materials, especially those used in all-ceramic systems [11]. In order to solve this problem, most of the all-ceramic porcelain systems require the combination of two layers of ceramic material, such as strong ceramic core and a weak veneering porcelain with better optical properties. The application of the veneering porcelain onto the ceramic core is identical to the production of porcelain-fused-to-metal restorations, in which the veneering porcelains are directly applied in layers onto the metal substrate to reproduce final shape and esthetics of the natural tooth. For the production of high-strength ceramic core material, aluminium oxide, magnesium oxide, zirconium oxide crystals, high content leucite or lithium disilicate are used by a number of techniques, such as lost-wax, slip-cast, or CAD-CAM [1,10].

There are several fundamental strengthening mechanisms of dental ceramics, such as crystalline reinforcement, chemical strengthening, and thermal tempering. Strengthening by crystalline reinforcement involves the introduction of a high proportion of the crystalline phase into the ceramic to improve its resistance to crack propagation. The principle of chemical strengthening relies on the exchange of small alkali ions for larger ions below the strain point of the ceramic. Since stress relaxation is not possible in this temperature range, the exchange leads to the creation of a compressive layer at the surface of the ceramic. Finally any applied load must first overcome this built-in compression layer before the surface can be placed into tension, resulting in an increase in fracture resistance. Thermal tempering of ceramics involves rapid cooling of a ceramic by forced convective cooling in the air or in a liquid medium. This process results in a prestress on the glass surface and residual compressive stresses are induced, inhibiting the initiation and the growth of cracks [1].

Wollastonite material is a naturally occurring calcium metasilicate ($CaSiO_3$) with a theoretical composition of 48.3% CaO and 51.7% SiO_2. Natural wollastonite is an acicular (needle-like) mineral that occurs in triclinic and monoclinic varieties. It is typically white or cream in color, but also may be light yellow, pink, gray, or very pale shades of green and brown. The acicularity (aspect ratio) reportedly varies from 3:1 to 5:1 for low aspect ratio or powder grades, to 10:1–20:1 for high aspect ratio grades [12]. Because of its favourable properties such as low shrinkage, low moisture, high brightness and whiteness, good strength, lack of volatile constituents, body permeability, fluxing characteristics, it is widely used in ceramics, plastics, metallurgical applications, friction products [13,14]. Powdered (milled or low aspect ratio) wollastonite is used especially for the applications in ceramics and metallurgy. Due to its good bioactivity and biocompatibility wollastonite has been regarded as the candidate for the artificial bone and dental root in the clinics [15,16]. Very little knowledge has been found associated with the dental applications of wollastonite. In an in vitro study, Yılmaz and Korkmaz [17] used this material as a filler in two different provisional resin materials and concluded that wollastonite increased the fracture toughness of these materials significantly.

Although wollastonite has been used in industry and medicine for many years, no data have been reported previously related to its effect on the physical properties of dental ceramics. Therefore the purpose of this preliminary study is to evaluate the effect of wollastonite on the linear firing shrinkage and flexural strength of dental aluminous core porcelain.

2. Method

The linear firing shrinkage and flexural strength of dental aluminous core porcelain (Vitadur-Alpha, Vita Zahnfabric, Bad Sackingen, West Germany) with and without wollastonite were evaluated in this study. In order to determine the effect of wollastonite in aluminous core porcelain, a weight of 2%, 3%, 4%, and 5% of wollastonite, respectively, in the form of powder were added into the porcelain powder by excluding an equal amount of the main material at the same time. Thus the total volume of the mixture were kept the same. The physical properties and chemical composition of the wollastonite material used in this study were shown in Tables 1 and 2.

Table 1
Physical properties of wollastonite used in this study.

Aspect ratio	5
Loss	<5
Mohs hardness	4.5–5
Bulking value (g/cm^3)	2.8–3.1
Appearance	White
Morphology	Acicular
Molecular weight	116
Specific gravity	2.9
Melting point (°C)	1540

Table 2
Chemical composition of wollastonite used in this study.

SiO_2	>46%
CaO	>43%
MgO	=0.84%
Fe_2O_3	<2%

By using a mould having a cavity of nominal size 25 mm × 6 mm × 3 mm, twenty aluminous core porcelain specimens were formed in a manner that each five of them contained a weight of 2%, 3%, 4%, and 5% wollastonite, respectively. And additional five porcelain specimens were produced by aluminous porcelain powder without any wollastonite as a control group (Table 3). The identical specimens were formed by condensing Vitadur-Alpha aluminous core porcelain powder with Vita Modelling Fluid (Vita, Bad Sackingen, Germany) to produce a 'creamy' slurry consistency. The entire mixture was transferred to the mould using a brush and placed on a vibrating table for 90 s where excess liquid was brought to the surface and removed with absorbent tissue. By using a razor blade excess material was removed from the overfilled mould. The specimens were transferred to a silicon nitride firing slab and fired in a vacuum furnace (Vitamat 30, Vita Zahnfabrik, Bad Sackingen, West Germany), preheated to 600 °C and fired according to the manufacturer's recommendation. The initial drying time (air fired) was 360 s at 600 °C and then the temperature was increased at a rate of 87 °C/min to 1120 °C and held for 120 s. The fired bar specimens were allowed to cool to room temperature and the surfaces of the specimens were checked and if any of them had surface defects or cracks, they were discarded. Twenty-five specimens were stored in a desiccator maintained at 23 ± 1 °C to prevent hydrolysis of the silicate bonds until they were required for grinding.

After the firing process, randomly selected specimens were gold-coated by sputtering (Polaron SC502, Fisons Instruments, Uckfield, UK) and examined under SEM. (Scanning electron microscope; model JSM-6060LV, JEOL Ltd. Tokyo, Japan) at 10 kV. Then the linear firing shrinkage, L_{fs}, of each specimen was calculated by using the following formula [18]:

$$L_{fs} = [(L_m - L_s)/Lm] \times 100$$

where L_s is the length of each fired specimen (mm) and L_m is the length of the cavity (mm). The L_s and L_m values were determined to an accuracy of ±0.01 mm.

Table 3
Statistical summary of the linear shrinkage values for dental aluminous core porcelain groups.

Wollastonite weight ratio (%)	*n*	Mean value	SD	Minimum	Maximum
0	5	9.86	0.19	9.65	10.00
2	5	8.89	0.37	8.27	9.31
3	5	8.82	0.75	7.93	10.00
4	5	8.34	0.44	7.93	8.96
5	5	8.20	0.51	7.58	8.96

Twenty-five specimens tested for the linear firing shrinkage were used for the flexural strength test. The specimens were ground so as to produce rectangular test pieces of 5 ± 0.25 mm in width, 1 ± 0.1 mm in thickness and 20 mm in length. Opposing faces of the test specimens were flat and parallel to within 0.05 mm. Test specimens were cleaned, ensuring that all traces of grinding debris were removed. Prior to the flexural strength test, all specimens were measured with micrometer (Moore and Wright, Sheffield, UK). The flexural strength of the specimens was tested using a three-point bending test with testing machine (Zwick/Z 020, GmbH and Co., Ulm, Germany) at a cross head speed of 0.5 mm/min. The distance between supports was arranged to be 15 mm. The diameter of each of the bearing surfaces was 1.6 mm. Each rectangular aluminous core porcelain specimen was placed centrally on the bearers of the bending device so that the load was applied to the center of the wide face along a line perpendicular to the long axis of the test specimen until fracture occured. The failure load was recorded in Newton for each test piece and the flexural strength (M), in Newton per square millimeter (MPa), was calculated by the following formula [18]:

$$M = 3Wl/2bd^2$$

where W is the load at failure in Newtons, l is the test span (center to center distance between supports) (mm), b is the width of the specimen (mm), d is the thickness of the specimen (mm).

2.1. Statistical analyses

To determine whether there was a statistically significant difference among the mean linear firing shrinkage and flexural strength values of the five test groups, Kruskal–Wallis statistical test was performed. Statistically significant difference was found at the 95% confidence level ($p < 0.05$). Mann Whitney U statistical test was used to compare the mean values of the five test groups with each other in pairs and to demonstrate which porcelain groups show statistically significant difference.

3. Results

3.1. SEM micrographs of the control and wollastonite added groups

SEM micrograph taken from the specimen of the control group presented evenly distributed Al_2O_3 particles throughout the glassy phase of porcelain (Fig. 1). SEM micrograph of 2% wollastonite added specimen showed Al_2O_3 particles, limited number of pores and needle-like wollastonite materials embedded into the glassy phase of porcelain (Fig. 2). In comparison with the specimen that contains 2% wollastonite, increase in the numbers of wollastonite materials and pores, and the needle-like wollastonite pull-out were prominent in the SEM micrograph of 5% wollastonite added specimen (Fig. 3).

The statistical summary of the measured linear firing shrinkage and flexural strength values for dental aluminous core porcelain are listed in Tables 4 and 5.

3.2. Linear firing shrinkage

Kruskal–Wallis test revealed statistically significant differences among the means of the linear firing shrinkage values for tested groups ($p = 0.007 < 0.05$) (Table 5). According to Mann Whitney U test (Table 6), the mean linear firing shrinkage value obtained for the control group was significantly higher than those obtained for the porcelain groups containing a weight of 2%, 4%, and 5% wollastonite. Although the mean linear firing shrinkage value of control group was higher than that of the group containing a weight of 3% wollastonite, this difference was not statistically significant. The mean value for the group containing a weight of 2% wollastonite was higher than those for groups containing a weight of 3–5% wollastonite but only the difference between the mean values of the groups containing a weight of 2% and 5% wollastonite was statistically significant.

3.3. Flexural strength

With regards to the flexural strength test, no statistically significant difference was found among the five test groups ($p = 0.142 > 0.05$), even though the mean flexural strength values of all wollastonite added groups were higher than the one obtained for the control group. The maximum mean value for the group containing a weight of 2% wollastonite was found as 156.9 MPa. When the mean values of wollastonite added groups were compared, the values decreased as the amount of wollastonite increased for the groups containing 2–4% wollastonite. But the mean value of the group containing 5% wollastonite was higher than the mean values of groups containing 3% or 4% wollastonite.

4. Discussion

The increased application of ceramic restorations and the demand for the long-term clinical performance of conventional ceramic restorations have resulted in the development and introduction of several ceramic restorative materials and techniques. Current research of new dental ceramics has focused on improved strength, fabrication of net-shape restorations with minimal shrinkage and better esthetics than conventional metal-ceramic restorations [19]. In the previous works several techniques have been described to overcome firing shrinkage of the porcelain restorations. One of them is the application of good condensation techniques used during the modelling of dental porcelains. Some authors [20,21] have believed that the firing shrinkage is minimized if the porcelain powder and liquid is well condensed. But others [22,23] have stated that even when porcelain particles are packed under hydraulic pressure, the residual spaces form over 40% of the volume, suggesting that condensation techniques cannot have a significant effect on the firing shrinkage. The other consideration is the application of different sized particles to employ bimodal and trimodal particle distribution, which was suggested by Rasmussen et al. [3]. They concluded that different sized particles which can be obtained from porcelain powders by sedimentation method are very time consuming when obtaining very fine particles and this method might be useful only for obtaining narrow distributions of coarser sized particles. In order to eliminate the sintering shrinkage of the conventional dental porcelains that were obtained by powder/liquid particle processing, the unfired porcelain build-up is generally made overcontoured or oversized. Overcontouring the porcelain build-up may cause considerable uncertainty as to the exact dimensional change that can be expected, particularly when different brands of porcelain and manipulative variables are considered [2]. This method is also used to obtain desired dimensions while fabricating porcelain cores from

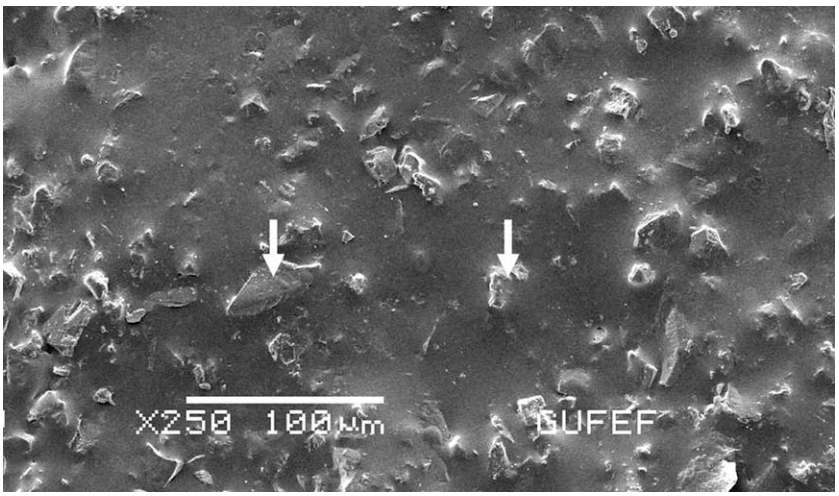


Fig. 1. Scanning electron micrograph of the sintered aluminous porcelain without wollastonite (White arrows show alumina particles).

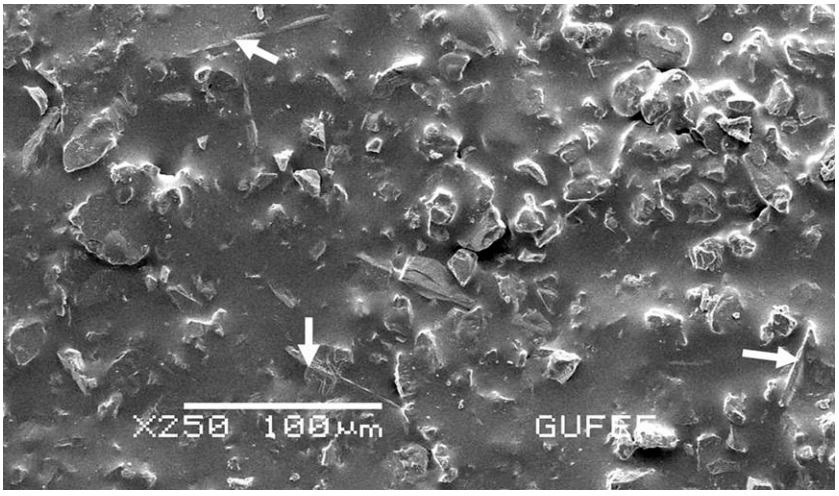


Fig. 2. Scanning electron micrograph of the sintered aluminous porcelain containing 2% wollastonite (White arrows show needle-like wollastonite materials).

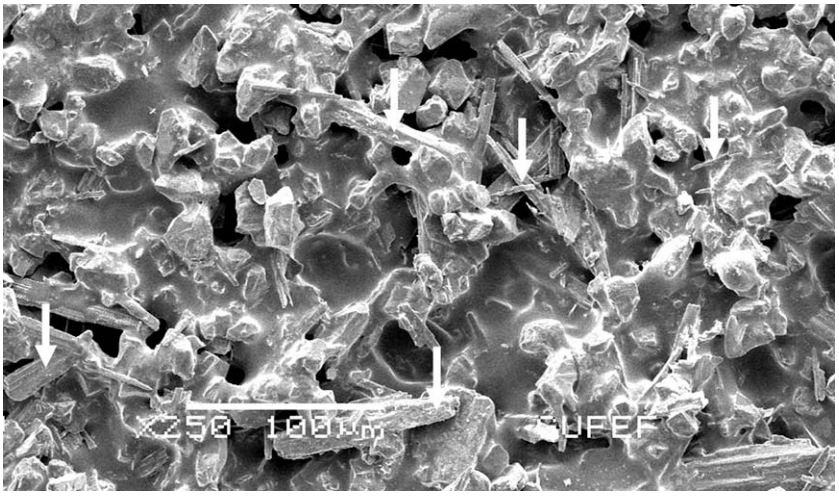


Fig. 3. Scanning electron micrograph of the sintered aluminous porcelain containing 5% wollastonite (White arrows show needle-like wollastonite materials).

pre-sintered porcelain blocks with CAD/CAM systems [5,24]. Pre-sintered porcelain blocks at the green stage requires an enlarged green body to compensate for the sintering shrinkage. The enlargement factor depends on the density of the green compact, the density of the body after sintering and their respective weights. Thus, the precision of the white body directly depends on a constant

Table 4
Statistical summary of the flexural strength values for dental aluminous core porcelain groups.

Wollastonite weight ratio (%)	*n*	Mean value	SD	Minimum	Maximum
0	5	128.42	14.01	105.00	141.00
2	5	156.90	14.41	135.60	173.60
3	5	144.08	12.98	133.70	163.30
4	5	137.64	18.51	116.90	158.80
5	5	147.08	22.24	125.30	184.30

Table 5
Kruskal–Wallis test results for the linear firing shrinkage and the flexural strength values.

	Linear firing shrinkage	Flexural strength
Chi-Square	14.409	6.878
df	4	4
Asymp. sig.	.007	.142

Table 6
Mann Whitney U test results for the linear firing shrinkage values.

Wollastonite weight ratio (%)	0%	2%	3%	4%	5%
0		–[a]		–[a]	–[a]
2					–[a]
3					
4					
5					

[a] Denotes which porcelain groups show statistically significant difference ($p < 0.05$).

quality of the green bodies and the stability of the sintering process. The machining of the restorations from fully sintered blocks eliminates sintering shrinkage but it is quite time consuming and leads to induced microcracks on the material because of the machining process. However, extreme hardness of sintered high-strength ceramics results in high wear of the machining tools [24]. More recently, shrinkage-free $ZrSiO_4$ material has been developed, particularly for the applications where oversize of the mould or machining of the sintered part present severe technical or economical obstacles [25]. Additional to shrinkage-free property of this material, this system requires complicated processing techniques and an expensive CAM milling unit to fabricate the crowns in the green state. In addition the sintering of the green bodies takes much time (approx. 4 h) similar to the other porcelain systems fabricated by CAD/CAM systems [26]. In order to circumvent the drawbacks associated with the methods described above, efforts should be made to develop new strategies that provide significant reduction or elimination of the firing shrinkage of dental porcelain systems. Therefore we strongly believe that it is necessary to test the efficiency of wollastonite on the linear firing shrinkage of dental aluminous core porcelains.

Our study demonstrated that the mean linear firing shrinkage values obtained for porcelain groups with and without wollastonite were within the acceptable limits reported in the standard ANSI/ADA 69 [18] and the addition of wollastonite into dental aluminous core porcelain contributed to a notable reduction in the firing shrinkage. This reduction could be due to the fact that the unmelted acicular wollastonite fibers can preclude the conglomeration of ceramic particles by filling the space between them. Depending on firing temperature wollastonite can maintain its acicular shape within the microstructure, or it can be made to melt and diffuse throughout to form strong, competent grain boundaries. Since all porcelain groups in this study were sintered at 1120 °C which is below the melting point of wollastonite, it is mostly probable that the acicular shaped wollastonite fibers remained unmelted. In comparison with the control group, the linear firing shrinkage values decreased as the amount of wollastonite increased in the porcelain powder. The linear firing shrinkage was reduced by as much as 20% with 5% wollastonite addition. The results of this study are consistent with the results of the research conducted by Robinson and Craig [27], in which similar reduction rates were reported with respect to firing shrinkage when various amounts and grades of wollastonite were added into sanitaryware bodies.

Wollastonite is an extremely interesting material that is used significantly in ceramics such as wall tile and sanitaryware, where it promotes low shrinkage, good strength, low warpage, and fast firing [14,27,28]. Wollastonite is relatively inexpensive, non-carcinogenic and easy to handle and transport [27]. In addition it has a small size allowing for a more homogenous mixture with the porcelain powder. Because of these favourable properties, wollastonite appears to be an appropriate candidate for using with dental porcelain systems.

In the present study, not only the effect of wollastonite on the firing shrinkage but also on the flexural strength, which plays an important role on the long-term clinical performance of dental ceramic restorations, was investigated. In order to determine the mechanical strength of brittle materials, such as porcelain, tensile strength is generally considered as the more meaningful property [29]. The bending or flexural tests are most widely used in the ceramics industry and generally considered as the most satisfactory means of assessing tensile strength [30]. The International Standards Organization (ISO 6872) [31] supports the use of simple three-point bending test as a means of evaluating the strength of dental porcelains. Numerous researchers used three-and-four point bending test methods to test the strength of glass and ceramic materials and the bending test has been reported to be a simple, reliable and sensitive method for testing the relative strength of dental ceramic materials [30,32,33]. For this reason three-point bending test was preferred to test the flexural strength of bar shaped dental aluminous core porcelain system in this study.

In dentistry, the strength of porcelain restorations can be improved either by making the porcelain itself stronger and tougher or by providing the porcelain with a stronger substructure that supports the porcelain. Glassy materials such as dental porcelain can be strengthened by dispersing ceramic crystals of high strength and elasticity in the glass matrix [1]. Spinner and Tefft [34] showed that the inclusion of particles of polycrystalline alumina into a feldspathic glassy matrix led to an increase in the elastic modulus and consequently increased the strength of composite material. In 1965, Mc Lean and Hughes [23] have described a core porcelain that contains up to 50% of fused alumina crystals plus a glass phase. The veneer porcelain that was used in conjunction with this aluminous core was essentially a conventional feldspathic porcelain. In our study, different amounts of wollastonite were added into the porcelain powder and higher mean flexural strength values were obtained for all wollastonite added groups in comparison to the control group. While 119 MPa was found for the control group, the highest mean flexural strength value, which was 156.9 MPa, was found for 2% wollastonite added group. These findings showed that 30% increase in the flexural strength was achieved. The mean value obtained for aluminous core porcelain without wollastonite in the current study is in agreement with the one reported in the previous study [32], in which three-point bending test was carried out. The increase in the flexural strength of wollastonite added aluminous core porcelain groups can be attributed to the needle-like shaped wollastonite fibers that have ability to inhibit the microcrack propagation by bridging the microcracks, as was reported in the investigation of Robinson and Craig [27].

One of the main factors that has to be considered while choosing the crystals to reinforce dental porcelains is the similarity of the thermal expansion of crystals to that of the glass [1,35]. Binns [36]

has described that the strength and elasticity of crystal-glass composites may increase progressively with the proportion of the crystalline phase, if the glass and crystals have similar thermal expansions. In 1970, Jones [37] studied the transverse strength of a feldspathic dental porcelain glass on its own and with inclusions of 10% and 20% of alumina, and 10% of quartz and 10% of fused silica by weight. The author determined a considerable reduction in the flexural strength of the fired dental porcelain bodies containing 10% quartz and 10% fused silica by weight and concluded that the high thermal expansion coefficient of quartz and the low thermal expansion of fused silica were responsible from the lowered strength. The previously reported thermal expansion coefficient of wollastonite is 6.3 over the temperature range 25–300 °C and of aluminous core porcelain is 6.8 over the temperature range 27–327 °C [38,39]. Since wollastonite and aluminous core porcelain seem to have similar thermal expansion coefficients, wollastonite can be considered to be used as a filler in this type of dental porcelain.

In spite of the fact that marked improvement was noted in the flexural strength as wollastonite was added, the highest mean flexural strength value was obtained for the 2% wollastonite added group. As can be seen from the Figs. 2 and 3, increase in the number of wollastonite can result in increase in the number of pores, which can cause reduced flexural strength of 3% or more wollastonite incorporated specimens. On the other hand, Jones [40] reported that the addition of alkali oxides or modifier oxides, such as Na_2O, K_2O, and CaO can lead to decreased strength because of the disruption of Si–O bond linkages. With respect to the flexural strength, the results of this study showed that the most suitable content of wollastonite in dental aluminous core porelain was 2% by weight, whilst, with regards to the sintering shrinkage, the most suitable content of wollastonite in dental aluminous core porcelain was 5% by weight.

We consider that the current preliminary study can be a good starting point for the future studies on improving the sintering shrinkage and strength properties of high technology dental ceramics.

5. Conclusions

Within the limits of this study the following conclusions were drawn:

1. The significant reduction in the linear firing shrinkage of dental aluminous core porcelain was obtained by the incorporation of wollastonite. The more the amount of wollastonite was incorporated into porcelain powder, the smaller the linear firing shrinkage was obtained. The highest reduction (approx. 20%) in the linear firing shrinkage was obtained by the addition of 5% wollastonite.
2. The incorporation of wollastonite increased the flexural strength of dental aluminous core porcelain. The highest increase (30%) in the flexural strength was achieved by the addition of 2% wollastonite.

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