

Evaluation of the Structural Changes in Porcelain at Different Firing Temperature

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Abstract:

Purpose: To study the structural changes occurring in the dental porcelain mass fired at various firing temperatures using scanning electron microscopy (SEM) and X-ray diffraction (XRD) spectroscopy. Also, additional tests, namely compressive strength, abrasion resistance analysis, and the amount of oxides released, were conducted at different firing temperatures.

Materials and Methods: Six groups (40 specimens in each group) of porcelain mass were prepared. The dimensions and weight of all the specimens were kept constant. The specimens were then heat treated at different firing temperatures (660°C, 760°C, 860°C, 900°C, 960°C, 990°C). Half of the specimens of each group were subjected to a compressive strength test on a universal testing machine and then finely ground using an electrochemical grinder to prepare for XRD analysis. The other half of the specimens was weighed to analyze the amount of oxides released after each

firing cycle. Following this, the specimens underwent an abrasion resistance test on a Nanovea Tribometer. The unaltered surface was scanned using SEM. The data (numerical and graphical) for all the tests were recorded and analyzed using one-way ANOVA and post hoc Tukey test.

Results: The specimens fired at 900°C exhibited superior compressive strength and abrasion resistance. The quantity of oxides released by the specimens fired at 900°C was the least compared to specimens heat treated at the other firing temperatures. XRD analysis proved that the oxide released by the porcelain mass was calcium aluminum chromium oxide. Also, the fewer peaks obtained in the XRD graphs of specimens fired at 900°C signified lesser porosities in the porcelain specimens. SEM analysis depicted a homogeneous mass of porcelain at 900°C.

Conclusion: All the above findings validate the objective of studying the physical and internal structural changes of dental porcelain when subjected to an increasing firing temperature gradient. The specimens fired at 900°C exhibited superior strength and abrasion resistance. SEM analysis depicted a homogeneous mass of dental porcelain, implying that firing was complete at 900°C.

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Introduction

Dental porcelain contains metallic and non-metallic elements—aluminum and oxygen (alumina, Al₂O₃), calcium and oxygen (calcia, CaO), and silicon and nitrogen (silicon nitride, Si₃N₄). [1] The molecules of this material are held together by inter-atomic bonding, which is both ionic and covalent. [2-4] Alumina was the first sintered industrial ceramic to be used in prosthodontics. The desirable properties are achieved through a heat treatment process called firing.^{5,6} The optimum firing temperature to which dental porcelains are heated is 960°C. It has been frequently observed clinically that on application of masticatory stresses, the porcelain layer on fixed dental prostheses (FDPs)

gets fragmented, resulting in prosthesis failure. [7-11] The condensed ceramic is dried before it is fired so that the remaining water is eliminated. This improves the green strength of porcelain. [12-18]

As the ceramic mass is heated across a temperature gradient, the physical structure undergoes continuous transitions. [19-21]

Initially the feldspathic grain boundaries melt and this leads to the merging of grain boundaries among the neighbouring particles. With rise in firing temperature, the larger grains receive alumina to form an amorphous phase intermeshed with unreacted silica particles. Once the optimum firing

temperature is attained, a homogeneous phase is formed, which imparts strength to the final prosthesis against masticatory stresses generated in the oral cavity by oral musculature and the hard tissue. Several studies have been conducted to observe the changes at the junction between porcelain and metal, [3,4,6,7] and also in a mass of zirconia. [22-25,28,30,31] The present study attempted to observe these transitions within the ceramic mass at different firing temperatures, by X-ray diffraction spectroscopy (XRD), and scanning electron microscopy (SEM) and to investigate a method to ensure long-term integrity of the ceramic prostheses under functional stresses in the oral cavity.

Materials and Methods

Specimen Preparation: Six groups (40 specimens in each group; 12 mm long, 8 mm wide, 4 mm thick) of porcelain mass were prepared by addition of modeling liquid to ceramic powder (powder-liquid ratio as prescribed by the manufacturer, Ceramco3 porcelain system; Dentsply, York, PA). The width and thickness of the specimens were slightly altered from ANSI/ADA specification no. 69 to provide for complete movement of the tribometer indenter and obviate fracture during abrasion testing of the specimens. Apart from these two dimensions, all other ANSI/ADA specification guidelines were followed to ensure standardization.

The specimens were repeatedly and completely dried with high absorbent tissue paper and compacted with a plastic compacter. Each specimen was weighed on a high precision chemical balance (Mettler-Toldeo, Columbus, OH). The weight of each specimen was kept constant at 10.25 g.

Heat Treatment (firing)

Specimens of each group were fired through different temperature gradients (660°C, 760°C, 860°C, 900°C, 960°C, 990°C) and bench cooled for 28 to 30 minutes, as recommended by the manufacturer. The recommended firing cycle given by the manufacturer was 975°C for the opaque layer, 960°C for opaceous dentin, 935°C for the final margin, 945°C for glaze, and 940°C for add-on. Through the course of this study, structural changes occurring at each firing temperature would be investigated and correlated with the mechanical properties at those firing temperatures to better understand the firing cycle. This could propagate new ideas to enhance the mechanical properties and thus ensure long-term integrity of the FDPs, thereby improving patient satisfaction.

Post Heat-Treatment (Firing) Weight Estimation

After being subjected to a specific temperature gradient, specimens of each of the six groups were individually weighed once again, and the data recorded.

Compressive Strength Test

Twenty specimens of each group were subjected, one at a time, to a compressive strength test on a universal testing machine (Instron, Norwood, MA) using standard experimental procedures to suit the dimensions of the specimens. [27,29,32,33,35-44] Compressive strength was computed from the fracture load value using Partner Environment Software (Compression/flexural suite W- 3410-102). [26] This procedure was repeated for all the groups, and the data recorded.

X-Ray Diffraction Spectroscopy

The fractured specimens of each group were finely ground (using an electrochemical grinder) and studied under XRD. The

20 fractured specimens of each group (used earlier for the compressive strength test) were finely ground in an electrochemical grinder (Everite, Philadelphia, PA). An electrochemical grinder was used to avoid the chemical alteration of the specimens caused by heat generated in a mechanical grinder. The finely ground mass of dental porcelain of each specimen of all the experimental groups was obtained and then placed on the platform of the XRD machine, one at a time, and tightly packed manually (loose powders would give poor intensities).

The finely ground specimens of porcelain (fired to different temperature gradients) were analyzed by XRD (RigakuUltima IV, Tokyo, Japan). In this study, XRD analysis produced a graphical representation, which typified each element detected in the specimen at that particular time and firing temperature. The peaks obtained in the XRD graph depended on a number of factors including wavelength distribution, crystalline size, and optical systems. Results were presented as peak positions at 2θ and X-ray counts (intensity) in the form of an x-y plot. The number of X-ray counts at which the specimen generated an energy beam at a given wavelength ($\text{Cu K } \alpha = 1.5418\text{\AA}$) was a prime factor in determining the oxide present. The scanning 2θ range was kept between 20° and 140° and the step size at 0.05° . Once, the 2θ range is set, the collimator rotates at a predefined rate to scan the different regions of the specimen being scanned (there is no such rate of scanning as seen in other scientific tools like the tribometer or the universal testing machine).

The XRD equipment applied the fundamental parameter (FP) method to calculate the peak by the convolutions of the curve in the XRD graphs. The XRD equipment used the PDXL software, which is a one-stop complete function powder diffraction analysis software. This software uses the Crystallography Open Database (COD) and the public-domain database of crystal structures to perform automatic phase detection.

Abrasion Resistance Test: The remaining 20 specimens of each group were subjected to an abrasion resistance test on a tribometer (Nanovea, Irvine, CA). The indenter tip (having a diameter of 1 mm) of the tribometer was set to rotate on the surface of the porcelain specimen with a 10 N force for 10 minutes and a rotating diameter of 6 mm. Weight loss in the porcelain specimen during the

experiment was calculated and interpreted as a function of abrasion resistance.

Scanning Electron Microscopy

The intact (unabraded) surfaces of these 20 specimens were prepared to be assessed under SEM (S3700; Hitachi, Tokyo,

Table 1: One-way ANOVA of mean compressive strength for the porcelain specimens

Firing temp	N	Mean (MPa)	Std. deviation (SD)	p value
660oC	20	67.55	4.893	<0.0001
760oC	20	84.5	5.472	<0.0001
860oC	20	104.25	4.494	<0.0001
900Oc	20	109.9	4.701	<0.0001
960oC	20	105.8	4.384	<0.0001
990oC	20	101.9	4.678	<0.0001

Table 2: One-way ANOVA analysis of amount of oxides released at different firing temperatures

Groups	n	Mean (g)	Std. deviation	p value
Group 1 - 660oC	20	0.629	0.050	<0.0001
Group 2 - 760oC	20	0.548	0.047	<0.0001
Group 3 - 860oC	20	0.353	0.031	<0.0001
Group 4 - 900oC	20	0.141	0.030	<0.0001
Group 5 - 960oC	20	0.220	0.033	<0.0001
Group 6 - 990oC	20	0.316	0.032	<0.0001

Japan) with a 1000× magnification.^{19,21} Data and images were procured and recorded.

Statistical Analysis: Data were summarized as mean ± SD (standard deviation) Groups were compared by one-way ANOVA, and the significance of mean difference between the groups was done by Tukey's post hoc test. A 2-tailed $p < 0.05$ was considered significant.

Results

Compressive Strength

The mean compressive strength of porcelain specimens are listed in Table 1. The compressive strength of the porcelain specimens escalated until 900°C and showed a downward trend thereafter. One-way ANOVA showed a significant difference between the six experimental groups ($p < 0.0001$). A Tukey test revealed very little difference between specimens fired at 860°C and 900°C. Moderate changes were observed when specimens fired at 860°C were compared with those fired 960°C and 990°C and when specimens fired at 960°C were compared with those fired at 900°C and 990°C. All other intergroup comparisons using post hoc Tukey's test revealed significant differences in compressive strength values. Therefore, specimens fired at 900°C exhibited maximum compressive strength among all the study groups.

X-Ray Diffraction Spectroscopy

XRD can be done on powdered as well as solid specimens, but the results have been found to be more precise in powdered specimens.¹⁵ In this

study, 20 specimens of each group were triturated using an electrochemical grinder and then manually packed tightly on the holder. The holder was tilted at a 45° angle. Copper was chosen as the material for X-ray generation at a diffraction angle of 2θ . The experimental setup adopted in this test was not specific to the given specimen; rather, it is a standard norm for performing any XRD evaluation. Various peaks observed in the XRD graphs were different for all six temperature gradient groups in this study. The peak intensities, measured in Angstrom units, corresponded to different elements that were the characteristic "fingerprint" of the specimen at that particular phase. The various elements present in the porcelain mass (present as oxides) were silica (SiO₂), alumina (Al₂O₃), boric oxide (B₂O₃), potash (K₂O), and soda (Na₂O). The peak positions were determined by the shape, size, and symmetry in the graph. The shapes and width of the peaks were determined by many factors from the diffractometer and the specimen investigated. The peak arose in the graph when the X-ray beam struck a porous area, whereas the valley between the peaks signified dense porcelain mass in that region. The height of peak signified the size of the porosity. The diffraction peaks became wider as the size of the crystallites reduced. As compared to other firing temperatures, the height of the peaks and the frequency of the peaks were less in the XRD analysis of porcelain specimens fired at 900°C. The peaks obtained in the XRD graph depended on a number of factors like wavelength distribution, crystalline size, and optical systems. The PDXL software of the XRD equipment used the

Crystallographic Open Database (COD) and public domain database to automatically detect the phase of oxides released by the specimen. The energy beams were directed on the specimen at 2θ , and the reflected beams at different degrees represented on the x-axis of the graph were seen in the form of peaks at specific wavelengths defined in Angstrom units. The PDXL software automatic scan detected a constant release of calcium aluminum chromium oxide at all peaks. It was evident in all the XRD graphs that only this oxide was released from all the test specimens at different wavelengths.

Thus, it could be inferred that oxides released were the least in specimens fired at 900°C. This implied that those specimens had minimum porosity and superior strength compared to specimens of all the other groups.

Estimation of Quantity of Oxides Produced

A high precision chemical balance (Mettler-Toledo) was used to measure the weight of specimens of the different groups. Specimens (20) of each group were weighed before heating (average weight 10.25 g) and then after the heat treatment through a specified temperature gradient (firing). Water quickly evaporated as the temperature reached 400°C, leaving behind a dehydrated porcelain mass. A decrease in specimen weight was observed at the end of each firing cycle. The mean decreases in weight are reported in Table 2. A significant finding using one-way ANOVA was that, at 900°C, the mean decrease in weight was minimum. The weight loss of the specimens correlated with the amount of oxides released during the firing cycle. A Tukey test revealed very little change in the amount of oxides released when specimens fired at 900°C and 960°C were compared.

In contrast, a moderate change was observed when specimens fired at 660°C, and 760°C; 760°C and 860°C; 960°C and 990°C were analyzed. Statistical analysis revealed that weight reduction in specimens was least in specimens fired at 900°C. It was inferred that the quantity of oxides released through this temperature gradient was minimum. Figures 1–3 show the fewest peaks (marked by peak values) for the specimens fired at 900°C compared to XRD representations

of the specimens fired at 860°C and 960°C. The quantity and frequency of oxides released denote the porosity in the material. Thus, dental porcelain fired at 900°C exhibited the least porosity, indicating that the quantity of oxides released was minimal, among all the specimens, evident in the XRD graphs.

Abrasion Resistance

Twenty specimens from each of the six groups were tested by a Nanoveatrimeter. The pointed indenter was set to abrade the surface of the porcelain

specimen for 10 minutes with a 10 N load. Abrasion resistance was measured in terms of weight loss in the specimen during the experiment using a digital weight balance (Mettler-Toledo). The data were analyzed using one-way ANOVA. The statistical analysis of the groups showed a significant decrease in weight of the specimens fired at 900°C ($p < 0.0001$). The mean changes in specimen weight are reported in Table 3. Initially, at 660°C and 760°C, the grains of porcelain did not blend with each other, forming a weak, non-homogenized mass. At 860°C, the grains merged partially, whereas at 900°C, the grain boundaries merged to form a completely homogenous single phase. Beyond 960°C, the homogeneous mass got over-desiccated, leading to deteriorated abrasion resistance (Table 3). The mean change in weight was found to be the least in the specimens fired at 900°C, indicating superior abrasion resistance. Tukey's test revealed moderate differences when specimens fired at 860°C and 990°C were compared, whereas significant differences were observed on all the other intergroup comparisons (Table 4).

Scanning Electron Microscopy

The analysis of SEM images showed that at 660°C, the porcelain powder particles started becoming visible. As the firing temperature was raised to 760°C, the edges of the particles started blending with each other. When the firing temperature was raised to 860°C, the particles coalesced to form a homogeneous mix leaving behind a few particles. At 900°C, a decrease in the granular form was observed in the porcelain mass. But beyond 900°C, that is at 960°C and 990°C, the porcelain mass was fragmented, and components were burnt out (Si, Al, Ca, Mg). Representative images of each group are presented in Figure 4. The leaching out of the elements from the porcelain mass brought about structural disintegration. Essentially, the brittleness of dental porcelain decreased while approaching 900°C and then increased subsequently beyond that. The porcelain mass fired at 960°C showed cracks. These cracks in the porcelain mass progressed to cause fragmentation, as the firing temperature was raised to 990°C (Fig 4). This behavior of porcelain on increasing the firing temperature was evident from the SEM images. On analyzing and comparing results for compressive strength, abrasion resistance, SEM, and XRD, it could be concluded that dental porcelain is most stable at 900°C with better compressive strength and abrasion resistance than specimens fired at 660°C, 760°C, 860°C, 960°C, and 990°C. Also, the porcelain mass was most homogenous at this temperature and the fewest oxides were produced (by weight).

Discussion

Dental porcelain has been a material of research interest for the last few decades. Many research

groups have conducted tests to study strength, fracture toughness, chemical properties, etc.^{2,3} The intent of this study was to observe the physical and chemical changes occurring within the ceramic mass at different firing temperatures.

In this study, Ceramco3 porcelain system (essentially feldspathic porcelain) was used because of certain advanced features. Though this system has the same basic composition as any other dental porcelain (as per the U.S. Patent publication no. WO1997030678 A1 i.e., SiO₂: 40% to 65%, Al₂O₃: 12%, Na₂O: 6% to 12%, K₂O: 5.5% to 12.5%, Li₂O: 1% to 3%, CaO: 0.8% to 2.5%, B₂O₃: 0.5% to 4%, CeO₂: 0.1% to 0.8%, P₂O₅: 0.01% to 3%), Ceramco3 has a high potassium content, which is much more stable than low potassium feldspar. This composition imparts greater stability that allows for better manipulation independent of operator efficiency. This system provides superior esthetics. The Ceramco3 system works well with modeling liquid or water and uses a customized firing process.

Proper mixing of Ceramco3 powder and liquid produces a consistency of porcelain, which is not only thick and easy to build up but also helps control shrinkage. The manufacturer recommends a customized firing temperature range of 935 to 960°C, which gives the laboratory technician enough leeway to fire add-on layers and enhances their long-term functional integrity. Colorants are added to help identify the placement of each layer of the build-up for natural variation and greater accuracy. In this study, a wide range temperature

gradient could be adopted due to the advantage of having a customizable firing schedule recommended by the manufacturer.

The physical properties were assessed by performing tests for compressive strength and abrasion resistance, whereas chemical properties were investigated through XRD. The microstructural changes in the porcelain mass were assessed by SEM. Specimens prepared were thicker and wider than specified in ANSI/ADA specification no. 69 to ensure complete movement of the Tribometer indenter and integrity of the specimens during abrasion resistance testing.

The weight of the porcelain specimens was recorded after each firing cycle. Water was constant by weight/volume ration all specimens and subsequently evaporated just above 100°C.

Therefore, the decrease in the weight of the specimens from 660 to 990°C corresponded to the quantity of oxides released during each firing cycle. At 900°C, the mean quantity of oxides released was 0.15 g, which was the lowest of the groups. The fewer oxides released, the lower the porosity would be, leading to a stronger homogenized mass. A Tukey test revealed very little change in the amount of oxides released when specimens fired at 900°C and 960°C were compared, whereas a moderate change was observed when specimens fired at 660°C and 760°C; 760°C and 860°C; and 960°C and 990°C were analyzed.

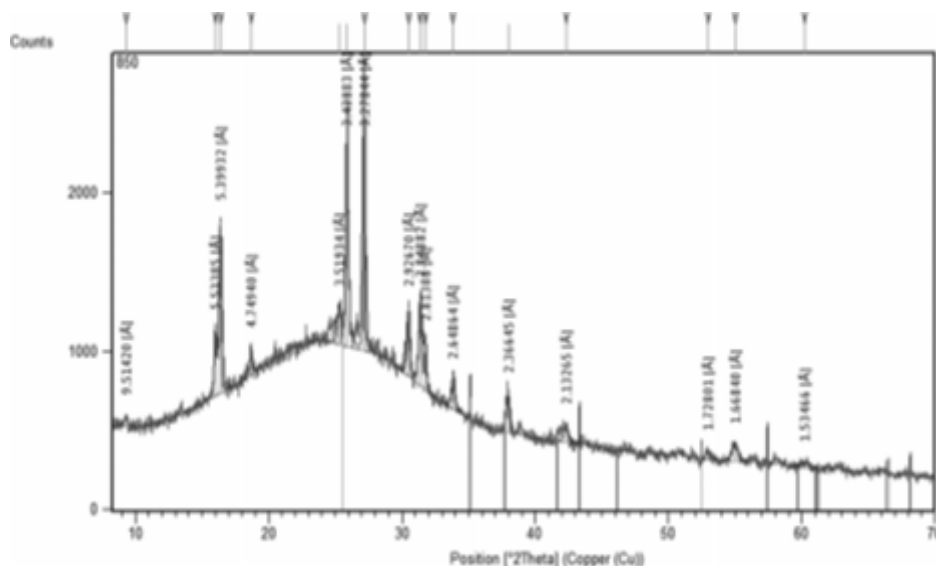


Figure 1 XRD graph of the porcelain specimen at 860°C.

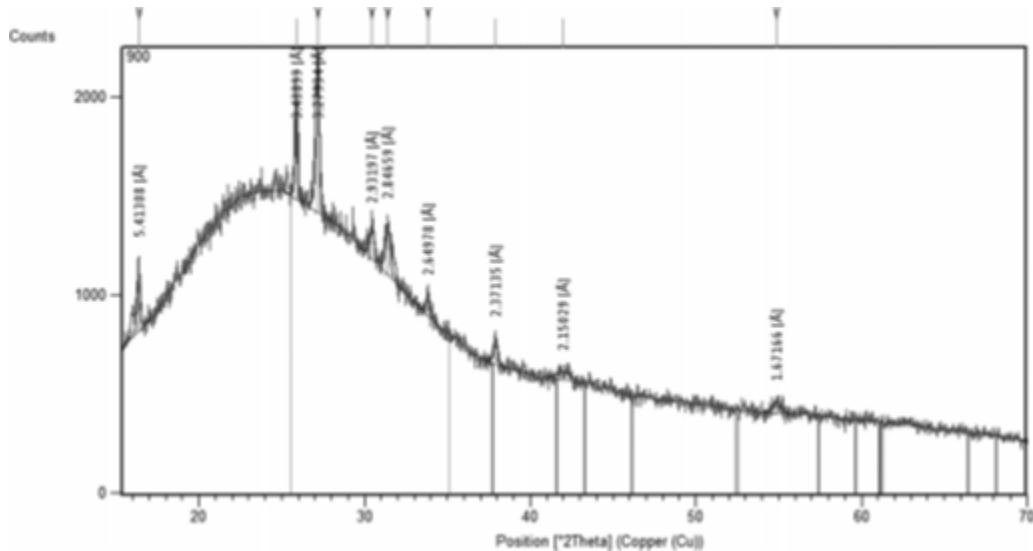


Figure 2 XRD graph of the porcelain specimen at 900°C.

The compressive strength was found to be superior for the porcelain specimens fired at 900°C (109.9 MPa) and showed a decrease beyond 900°C (105.8 MPa at 960°C; 101.9 MPa at 990°C). This indicated that the grains of porcelain merged homogeneously until 900°C and following that, the components got over-desiccated and friable, leading to decreased compressive strength. A Tukey test revealed very little difference between specimens fired at 860°C and 900°C.

Moderate changes were observed when specimens fired at 860°C were compared with those fired at 960°C and 990°C and when specimens fired at 960°C were compared with those fired at 900°C and 990°C. All other intergroup comparisons using post hoc Tukey’s test revealed significant differences in compressive strength values. Diffraction spectroscopy was performed on the finely ground specimens following strength test, to elucidate and quantify the amount of oxides produced. Oxides

were identified depending on the peaks obtained in diffraction spectroscopy. The various elements present in the porcelain mass (present as oxides) were silica (SiO₂), alumina (Al₂O₃), boric oxide (B₂O₃), potash (K₂O), and soda (Na₂O). These peaks depicted the quantum of energy released, each a typical connotation for a particular element. Significant differences were found when all other specimen

groups were compared using a post hoc Tukey test. The peaks obtained in the XRD graph depended on a number of factors like wavelength distribution, crystalline size, and optical systems. The XRD equipment used the PDXL software, which was preloaded with crystallographic open database and public domain database to auto-detect calcium aluminum chromium oxide as the oxide being released from the specimens of all the six temperature gradient groups.

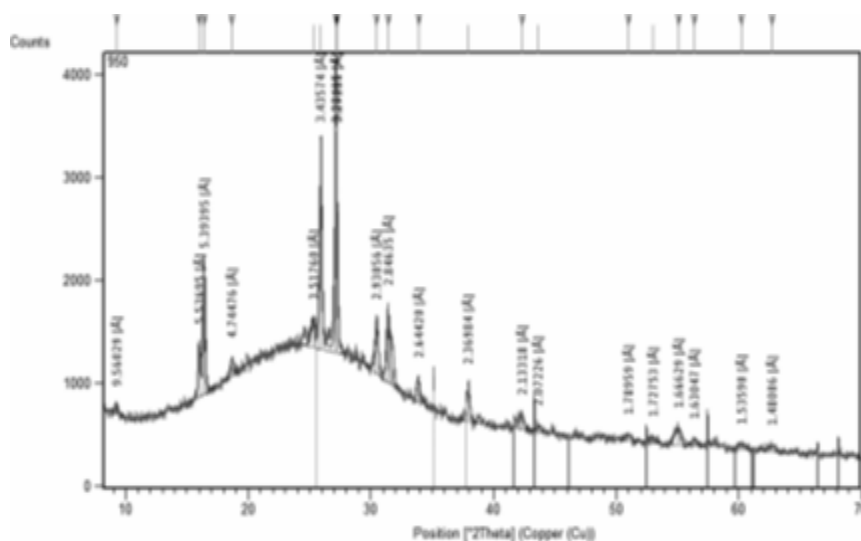


Figure 3 XRD graph of the porcelain specimen at 960°C.

Twenty specimens from each group were subjected to a tribology test to evaluate their abrasion resistance. Tribology is defined as the science and engineering of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication, and wear. A pointed indenter as used to abrade the surface of the porcelain specimens under a 10 N constant load for 10 minutes. The weight loss of the porcelain specimens was recorded to be interpreted as resistance to wear.

The lesser the weight loss, the better the abrasion resistance. Results indicated that porcelain specimens fired at 900°C showed the least weight loss and maximum abrasion resistance, and this was statistically significant when compared with other groups ($p < 0.0001$). Tukey's test revealed moderate differences when specimens were fired at 860°C and 990°C, whereas significant differences were observed in all other intergroup comparisons. The assessment of mechanical properties of dental ceramics was in agreement with the earlier work of Denry et al, [19,20] who stated that microcracks developed during porcelain firing played a significant role in dental restoration success.

The intact surfaces of the porcelain specimens (20) were observed under a scanning electron microscope to comprehend the behaviour of these specimens subjected to different temperature gradients during the previously performed experiments.

Initially, until 860°C, non-homogenized grains of porcelain were visible, and contributed to the lower compressive strength and abrasion resistance. At higher firing temperatures, the grains coalesced, first along the boundaries and then throughout the body. Porcelain specimens fired at 900°C displayed homogeneity, which explains the superior abrasion resistance and better compressive strength. Beyond that, at 960°C and 990°C, the porcelain mass got over-desiccated, leading to leaching out of the constituents, and thus, weakening the mass. Guzzato et al [26] studied the microstructure of alumina and zirconia glass-infiltrated dental ceramics. Certain findings observed in their study were confirmed in this study: micro-cracks were observed in the ceramic mass, heat-treated to 960°C.

Micro-cracks occurring internally in the fired porcelain were believed to play a vital role in deflecting a crack and dispersing the energy. This in turn increased the fracture toughness of the ceramic under observation. On the contrary, if many microcracks were present in a given area, they could coalesce to form a single large crack. Many workers have studied the changes occurring along the interface of metal and ceramic. Saini et al [34] studied the change at the interface of porcelain and

nickel chromium alloy using SEM and XRD. The present study attempted to observe the transitions in the internal structure of porcelain mass, dependent on an upward-progressing temperature gradient.

The optimum firing temperature for dental porcelain is 960°C. In this study, it was observed that at 900°C, the quantity of oxides released by porcelain was the least, compared to other firing temperatures. This showed that dental porcelain was least porous when fired at 900°C, and that it had superior strength and fracture resistance. The oxide released was calcium aluminum chromium oxide as characteristically connoted in the energy release graphs. The leaching out of this oxide led to a decreased strength of the porcelain mass and poor abrasion resistance. Also, SEM studies clarified that porcelain blended more homogeneously at 900°C, compared to other firing temperatures. Internal composition, density, and strength were essential to ensure long-term integrity of ceramic prostheses under

Study limitations

The present study dealt with structural changes within the porcelain mass. Interaction with the metal alloy during the firing cycle would have an impact on the micro-structural changes at the porcelain/metal alloy interface. This aspect has not been dealt with in the present study. Moreover, this was an in vitro study, and the effects of the dynamic oral environment (saliva, etc.) on the long-term integrity of fixed ceramometal restorations warrants further research.

Conclusion

The results clearly showed superior abrasion resistance and compressive strength values for those specimens fired at 900°C.

SEM indicated that dental porcelain displayed optimum homogeneity at 900°C. XRD revealed the amount of oxides released from the specimens at different temperature gradients. The oxide released was calcium aluminum chromium oxide, as characteristically connoted in the energy release graphs. The fewest oxides were released at 900°C. The more oxides released, the greater the degree of porosity in the porcelain mass. The results of this study strongly indicate that porcelain fired at 900°C displayed superior compressive strength, greater abrasion resistance, optimum homogeneity, and lower porosity. These findings provide information on structural changes within the porcelain mass subjected to a temperature gradient and also provide an insight into methods to ensure long-term

integrity of FDPs under functional stresses in the oral cavity.

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