M D J

An evaluation of the effects of different pH levels and multiple firing temperatures on hardness of ceramic

Dr. Zahraa Nazar Al-Wahab, B.D.S., M.Sc.

Abstract

This study was conducted to evaluate the effect of three different media (artificial saliva, acidic and alkaline) and the effect of five times of multiple firing procedure on hardness number of IPS e - max pressable ceramic .

Seventy discs shaped specimens were fabricated according to the manufacturer's instructions. The specimens were randomly divided into seven groups. Three for different media and four for multiple firing procedure (ten specimens for each group).

The groups were subdivided as follows: According to different media:

Group 1: specimens will be left in artificial saliva (Neutral media) for 10 days at 37c° in an incubator,

Group2: specimens will be left in an acidic media for 10 days at 37c° in an incubator,

Group 3: specimens will be left in an alkaline media for 10 days at 37c° in an incubator.

According to multiple firing procedures

Group 1: specimens will be exposed to firing one time,

Group 2: specimens will be exposed to firing 3 times,

Group 3: specimens will be exposed 5 times to firing procedures,

Group 4: these specimens will be exposed to firing for 7 times.

Then each group will be subjected to hardness test using Shore D hardness tester. The statistical analysis of the data of the tested groups of different media revealed that the high hardness values was in the alkaline media, while the lowest hardness values was in the acidic media. The highest hardness values of the firing groups were found in the group of 7 times firing, while the lowest hardness values were found in the group 3 times firing and the results were statistically non significant.

As a conclusion, an alkaline media increase the surface hardness of the IPS e – max ceramic, and multiple firing did not affect the surface hardness of the e –max ceramic.

Key words: multiple firing, surface hardness, IPS e – max.

Introduction

Advanced progress in technology and research of new dental materials has resulted in an increased number of all-ceramic systems. Several processing techniques are available for fabricating all-ceramic restoration: sintering, heat pressing, infiltration, casting and machining. Recently, IPS e-max is an innovative all-ceramic system which covers the entire all-ceramics indication range from thin veneers to 10 units FPDs. IPS e-max delivers high strength and high esthetic materials for the press and the CAD/CAM technologies. IPs e-max Press (Ivoclar Vivadent) consists of a lithium-disilicate pressed glass ceramic, but its physical properties and translucency are improved through different firing processes compared to IPs Empress 2. Emax press is a pressed glass-ceramic ingot (lithium disilicate crystals). The lithium disilicate crystals prevent the propagation of micro cracks and contribute to the esthetic translucency of the Ips e.max press restoration.[1] Variations in pH, solution chemistry; wear and mechanical load make the oral cavity a complex environment. Consequences of ceramic degradation include coarseness of the exposed surface, which promotes plaque accumulation, promotion of wear to the antagonist teeth or restorative materials and change to the color of dental ceramics, thereby compromising the aesthetic appearance of ceramic restorations [2].

It has been demonstrated that the microstructure, and consequently the mechanical properties of a glass-ceramic can be modified by varying the thermal treatment to which it is submitted5. For instance, it has been shown that the final crystal growth took place during the pressing and firing steps of E1 processing method13. One practical aspect of processing that might play a role is the fact that the temperature in the press furnace may oscillate after repeated use [3]. The purpose of this study was to evaluate the hardness of ceramic with different media and to evaluate hardness of ceramic with different firing temperatures.

Materials and methods

Seventy disc shaped wax specimens were fabricated from a sheet of modeling base plate wax (2 mm in thickness). Then punched with copper ring (10 mm in diameter) to produce the specimens. In order to invest the wax pattern, the investment powder and the special liquid (IPS Press Vest for different Ivoclar Vivadent press ceramic, Ivoclar, Vivadent AG, FL-9494 Schaan Liechtenstein, Germany) were mixed according to the manufacturer's instructions. The mix then poured inside the silicone ring, and the ring gauge positioned on the silicone ring with a hinged movement, and the investment allowed to set.

Then, the ring gauge and ring base removed with a turning movement. The investment ring was pushed out of the IPS Silicone ring carefully, and the disc shaped specimens were burned out by electrical furnace. The investment ring removed from the preheating furnace immediately after completion of the burnout procedure. The IPS e.max Press ingot placed into the hot investment ring, and the IPS Alox Plunger coated with IPS Alox Plunger Separator, and then the IPS Alox Plunger placed into the hot investment ring. The pressing of ceramic started in porcelain furnace (Computarized porcelain furnace for pressable ceramic (Programat EP 3000, Ivoclar,Vivadent, Germany). At the end of the press cycle, The investment ring was placed on a cooling grid and allowed to cool.

Then the specimens were divested using sandblast machine with 50µm particles and a pressure of 3 bars. The sprues were separated from the specimens using diamond cutting wheel saw.

Preparation of solutions of different pH values:

Three solutions with pH values of 3.5, 7.0, and 10.0 were prepared as described by Pinto et al., 2008. [4] For the neutral solution of pH 7.0, artificial saliva of the following composition was thus prepared: 100 mL of

KH_2PO_4 (2.5mM), 100 mL of Na_2HPO_4 (2.4 mM), 100 mL of $KHCO_3$(1.50 mM), 100 mL of NaCl (1.0 mM), 100 mL of $MgCl_2$ (0.15 mM), 100 mL of $CaCl_2$ (1.5 mM), and 6 mL of citric acid (0.002 mM). The pH of neutral artificial saliva solution was lowered to 3.5 by adding HCl, and raised to 10.0 by adding NaOH.

Sample grouping: Thirty samples were divided as follows :According to different media into 3 group (ten specimens for each):

Group 1: specimens will be left in artificial saliva (Neutral media) for 10 days at 37c° in an incubator

Group2: specimens will be left in an acidic media for 10 days at 37c° in an incubator,

Group 3: specimens will be left in an alkaline media for 10 days at 37c° in an incubator.

The other forty samples were divided into 4 groups according to multiple firing procedures (ten specimens for each):

Group 1: specimens will be exposed to firing one time only.

Group 2: specimens will be exposed to firing 3 times.

Group 3: specimens will be exposed 5 times to firing procedures.

Group 4: these specimens will be exposed to firing for 7 times.

Then the specimens were subjected to Shore D hardness tester.

The data obtained of shore D hardness number were analyzed by One – Way ANOVA test followed by LSD test.

Results

The mean Shore D Hardness Number, Standard Deviation (SD), minimum, maximum values for storage media groups are illustrated in (Table 1). One – Way ANOVA test was done for estimation of any significance among these groups in (Table 2) and LSD test (least significant difference test) was followed to estimate the source of significance in (Table 3).

The mean Shore D Hardness Number (in MPa), Standard Deviation (SD), minimum, maximum values for multiple firing groups are illustrated in (Table 4). One – Way ANOVA test was done for estimation of any significance difference among multiple firing groups in Table 5 and LSD test (least significant difference test) was followed to estimate the source of significance in (Table 6).

Discussion

Basically the chemical durability of dental ceramics is good, but it may be influenced by many factors such as composition and microstructure of the ceramic materials, the chemical character of the ceramic materials, the chemical character of the erosive or acidic agents, the exposure time and the temperature [5]. The normal pH of saliva is 6.8–7.2, but it can be easily lowered or raised by the consumption of food and drink. For example, each time a carbohydrate- rich food is ingested, organic acids are produced by dental plaque, lowering the pH of the oral cavity to an acidic pH of about 4.516). However, acidic products such as lemonade and soft drinks lower oral pH and create an acidic environment in the mouth without bacterial involvement [6].

Hardness is considered an important property when comparing restorative materials. It is a measure of the resistance to permanent surface indentation or penetration. The significance of measuring hardness in dental material is that it delineates the abrasiveness of a material to which the natural dentition may be submitted [7].

Effect of storage solutions

The results of the present study showed a reduction in microhardness of ceramic following storage in acidic solution and this reduction is statistically significant when comparing between acidic solution and artificial saliva. There is a statistically significant difference when comparing microhardness of ceramic following storage in acidic solution and alkaline solution. But there is no significant difference when comparing microhardness of ceramic following storage in artificial saliva and alkaline solution. This result is because of the degradation of dental ceramics occurs when ceramics are exposed to aqueous solutions or erosive agents. This phenomenon takes place as a result of firstly selective leaching of alkaline ions [2]**.** Alkaline metal ions are far less stable in the glass phase than in the crystalline phase [8] and some alkaline ions are leached after exposure to acidic solutions [6]**.** Secondly is the dissolution of ceramic silicate network (Si-O-Si). These mechanisms are controlled by the diffusion of hydrogen ions from an aqueous solution into the ceramic and loss of alkali ions from the ceramic surface into an aqueous solution to maintain an electrical neutrality [2]. These results are in agreement with [2].

Effect of multiple firing

In this study, there is no significant difference when comparing among microhardness of ceramic with multiple firing. This result could be attributed to the crystalline structure of IPS e – max press ceramic. It was produced by controlled crystallization method, in which the crystalline phase (Lithium disilicate $Li_2Si_2O_5$) are nucleated and grown in glass by means of heat treatment. These elongated interlocked crystals form 65% of the microstructure of glass ceramic [3]. IPS e.max lithium disilicate is composed of quartz, lithium dioxide, phosphor oxide, alumina, potassium oxide, and other components. This composition produces a highly thermal, shock-resistant glass ceramic [9]**.**

The thermal expansion mismatch between lithium disilicate crystals and glassy matrix is likely to result in tangential compressive stresses around the crystals, potentially responsible for crack deflection and strength increase. The interlocked microstructure and layered crystals are also likely to contribute to strengthening since the crack propagation is easy along the cleavage planes, but more difficult across the planes, leading to multiple crack deflections due to an array of crystal orientations. The higher resistance to crack propagation is in the direction perpendicular to crystal alignment [10]**.** This also could be attributed to the technology of fabrication; the e – max press is softened by heat in the pressing step, then subjected to a removal of the reaction layer then subjected to heat treatment.[4]. These results are in agreement with Albakry et al.,[7], Albakry et al., [11], Ritter and Rego [9] and Mohsen [1].

As a conclusion: the placement of IPS e – max press ceramic in an alkaline solution will increase its surface hardness clearly and the acidic solution will decrease the surface hardness of IPS e-max press ceramic. The multiple firing of IPS e – max press ceramic in furnace will affect its surface hardness and decrease it, although this reduction was statistically non significant

References

1- Mohsen C. Corrosion effect on the flexural strength & micro-hardness of ips e-max ceramics. Open Journal of Stomatolog, 2011; 1:29-35.

2- Kukiattrakoon B, Hengtrakool C, Kedjarune – Leggat U. Chemical durability and microhardness of dental ceramics immersed in acidic agents. Acta Odontologica Scandinavica, 2010; 68: 1–10.
3- Gonzaga C C, Cesara PF, Okadaa C Y, Fredericcib C, Netob F B, Yoshimura H N. Mechanical Properties and Porosity of Dental Glass-Ceramics Hot-Pressed at Different Temperatures. Materials Research 2008 ;11 :(3)301-306.
4- Pinto MM, Cesar PF, Rosa V, Yoshimura HN. Influence of pH on slow crack growth of dental porcelains. Dent Mater 2008; 24: 814-823.
5- Milleding P, Haraldsson C, Karlsson S. Ion leaching from dental ceramics during static in vitro corrosion testing. J Biomed Mater Res 2002; 61:541- 50.
6- Turp V, Tuncell B, Sen D, Goller G. Evaluation of hardness and fracture toughness, coupled with microstructural analysis, of zirconia ceramics stored in environments with different pH values. Dental Materials Journal 2012; 31(6): 891–902.
7- Albakry, M., Guazzato, M. and Swain, M.V. Fracture toughness and hardness evaluation of three pressable all-ceramic dental materials. Journal of Dentistry 2003; 31:181-188.
8- Anusavice KJ. Degradability of dental ceramics. Adv Dent Res 1992;6:82-9.
9- Ritter R.G. and Rego N.A. Materials consideration for using lithium disilicate as a thin veneer option. Journal of Cosmetic Dentistry 2009; 25(3): 111 – 117.
10- Denry I and Holloway JA. Ceramics for Dental Applications: A Review. Materials 2010; 3:351-368.
11- Albakry M, Guazzato M, Swain MV. Effect of sandblasting, grinding, polishing and glazing on the flexural strength of two pressable all-ceramic dental materials. Journal of Dentistry 2004; 32, 91–99.

Table (1) Mean, SD, Min, Max of Shore D Hardness in MPa for storage media groups

	Control (Neutral)	**Acidic**	**Alkaline**
Mean	97.2	95.56	97.92
SD	1.3755	1.390324	0.969536
SE	0.616816	0.623464	0.434769
Min	95.8	93.9	95.6
Max	99.6	97.4	98

Table (2) One – way ANOVA test for estimation of any significant difference among groups of storage media

	F-test	**P-value**
Between control and Acidic, Alkaline	4.605	0.033 Significant

*P<0.05

Table (3) Least Significant Difference LSD for groups of storage media

	Mean difference	**P-value**	**Sig**
Control&Acidic	**1.640**	**0.049**	**S**
Control& Alkaline	**-0.720**	**0.384**	**NS**
Acidic&Alkaline	**-2.36**	**0.012**	**S**

***p<0.05 Significant **P>0.05 Non significant**

Table (4) Mean, SD, Min, Max of Shore D Hardness in MPa

	Control(1F)	3F	5F	7F
Mean	96.62	95.28	96.68	97.4
SD	3.531572	1.207063	1.810249	1.407125
SE	1.583665	0.541284	0.811771	0.630998
Min	91	93.7	94.3	96
Max	99.4	96.4	98.4	99

Table (5) Estimation of any Significance among multiple firing groups

	F-test	**P-value**
Between control and 3F,5F,7F	**0.815**	**0.504** **Non-significant**

*P>0.05

Table (6) LSD Test (Least Significance Test) for Estimation the Source of Significance among multiple firing groups

	Mean difference	**P-value**	**Sig**
Control&3F	**0.720**	**0.610**	**NS**
Control& 5F	**2.120**	**0.145**	**NS**
Control&7F	**0.780**	**0.581**	**NS**
3F&5F	**1.40**	**0.327**	**NS**
3F&7F	**0.060**	**0.966**	**NS**
5F&7F	**-1.34**	**0.348**	**NS**

***P>0.05 Non significant**