Evaluation of Different pH Solutions on the Mechanical Behaviour of Two Hot Pressed Dental Ceramics

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Abstract: From all the materials that are used in modern dentistry to restore the natural dentition, ceramics have the best features to mimic the tooth structure. Although they are aesthetically superior, their main disadvantages are brittleness and low fracture resistance. Some processes used in manufacturing the ceramic, as well as the environment, can affect its structure and it’s mechanical properties. The aim of this study is to illustrate how the surface roughness and hardness, of one feldspathic glass ceramic and a lithium disilicate glass ceramic, are influenced by the surface processing (glazing and polishing) and after immersion in solutions with different pH and temperatures (tea, carbonated acidic beverage and distilled water) for a given amount of time. The research goals are calculating the Ra parameter and the Vickers microhardness of the tested ceramic disc-shaped samples. This study results showed that the surface treatments (glazing and polishing) affected the Ra parameter and the Vickers microhardness. The glazed samples from both types of ceramic proved to have better hardness. From the selected immersion solutions, the tea affected the most the roughness and especially affected the polished surfaces of the tested ceramics.

1. Introduction

Dental ceramics have been used extensively as a restorative material for all kinds of dental restorations, because they provide good aesthetical and mechanical properties. In order to be successful a material has to resist against the intrinsic characteristic of the material and the environment to which it is exposed [1]. Oral cavity is a complex environment in which the ceramic is in contact with saliva first of all and with other different beverages and foods, that have different pH and different temperatures. The pH found in the oral cavity ranges from 1 to 10 [2]. The pH of the ingested beverages ranges from 3 to 8 and the ones that have low pH values are carbonated acidic beverages.

Another important aspect is the temperature found in the oral cavity that varies from 0 °C to 67 °C. Some in vitro studies showed that increased temperature is accelerating the reactions between the liquids and the ceramics surface [3,4,5]. These factors are known to influence the mechanical
properties, such as surface roughness and microhardness of the dental restorations [6]. The surface roughness has been shown to influence the long term prognosis of the restoration by decreasing its flexural strength [7]. Another aspect that affects the surface roughness is the preparation of the surfaces. Polishing and glazing the ceramic surfaces to a high surface lustre are able to reduce surface roughness, fracture risk and staining [8,9]. Glazing of ceramic restorations is a routine laboratory procedure and it is demonstrated that it is a way of strengthening the glass [10]. Surface roughness can be calculated in terms of average roughness $[Ra]$ and the maximum roughness $[Rz]$, using a profilometer [11]. Vicker’s microhardness is based on indentation toughness measurement and the results are obtained by measuring the cracks that emanate from the vertices of the Vickers indentation [12,13].

This study aimed to investigate the influence of the pH and temperature of different solutions on the mechanical properties of pressed dental ceramics after different surface treatments.

2. Material and methods
Two commercially available hot pressed dental ceramic materials were used in the present study: a feldspathic glass ceramic and a lithium disilicate glass ceramic (Table 1).

| Ceramic system | Shade | Manufacturer | Type | Composition |
|----------------|-------|--------------|------|-------------|
| Vita PM 9      | A2    | Vita Zahnfabrik H. Rauter Gmbh & Co KG | Feldspathic glass ceramic | Feldspar with aluminium oxide |
|                | Medium Translucency | | | 57-80% wt% SiO$_2$, 11-19 wt% Li$_2$O and other oxides such as K$_2$O, MgO, ZnO, Al$_2$O$_3$, P$_2$O$_5$. |
| IPS e.max Press | A2    | Ivoclar Vivadent | Lithium disilicate glass ceramic | |
|                | Medium Translucency | Schaan, Lichtenstein. | | |

Each ceramic disk has 2 surfaces, resulting 48 surfaces. 24 were glazed and 24 were polished. First of all the samples were cleaned ultrasonically. All the surfaces were prepared using abrasive paper (220, 320, 500, 800, 1000, 2000-grit silicon carbide papers) and diamond paste with 40 µm particles (Zirkopol; Feguramed, Germany) for 60 seconds. After this step half of the surfaces were glazed. The glaze was applied using manufacturer’s instructions for each material (Table 3,4). Two layers of glaze were applied to simulate fabrication processes of the restoration.

The specimens of each material (n=12) were randomly divided into three groups (n=4) according to the immersion solution: Tea (Black Tea, Vallery)(pH=7), carbonated acidic beverages (Coca-Cola) (pH=2.5), Distilled Water (D)( pH=7) as the control group. The beverages were chosen according to their pH and temperature. The selected time was 24 hours that simulates one year and a half clinical use.
Table 2. Parameters used for the investment material.

| Parameter                      | Value       |
|--------------------------------|-------------|
| Setting time after investing   | 20-30 minutes |
| Insertion temperature          | 850°C       |
| Holding time                   | 60 minutes  |
| Final temperature              | 850°C       |

Table 3. Pressing programs according to each manufacturer.

| Type of ceramic      | Starting temperature °C | Vacuum level hPa | Heat rate °C/ min | Press temperature °C | Hold time minutes | Press time minutes |
|----------------------|--------------------------|------------------|-------------------|-----------------------|------------------|-------------------|
| Vita PM 9            | 700                      | 47               | 50                | 1000                  | 20:00            | 10:00             |
| IPS e.max Press      | 700                      | 45               | 60                | 930                   | 25:00            | 05:00             |

The tea was prepared using 1g of tea for 100 g of water. The specimens were immersed in a closed individual vial containing 20 ml of each solution. The vials were kept in the incubator for 24 hours at temperature of 37 °C for distilled water, 55°C for tea and at 5°C temperature for carbonated acidic beverage.

Table 4. Specific glaze for each ceramic.

| Type of ceramic | Powder                          | Liquid            |
|-----------------|---------------------------------|-------------------|
| Vita PM 9       | Vita Akzent plus glaze LT powder 5g | Vita Akzent Liquid |
| IPS e.max Press | IPS e.max Ceram Glaze Powder 5 gr Ivoclar | IPS Inline System Glaze Liquid 15ml Ivoclar |

Table 5. Ceramic programs for glazing.

| Type of ceramic | High temperature °C | Low temperature °C | Predry minutes | Preheat minutes | Vacuum level hPa | Heating minutes | Vacuum hold minutes | Firing time minutes |
|-----------------|----------------------|---------------------|----------------|-----------------|------------------|-----------------|---------------------|---------------------|
| Vita PM 9       | 820                  | 500                 | 00:00          | 04:00           | 0                | 04:00           | 00:00               | 01:30               |
| IPS e.max Press | 820                  | 403                 | 00:00          | 06:00           | 50               | 07:00           | 07:00               | 02:00               |

Microhardness of the samples was measured using a DM 8 / DM 2 (Yang Yi Technology Co., Lt, Tainan City 70960, Taiwan) micro-hardness tester. A load from 1 kgf was applied for 10 seconds with a pyramidal indenter. After removing the indenter, the indentation dimensions were microscopically recorded at a 40x magnification. Five indentations were made on each specimen on the glazes and polished surfaces post immersion.

The roughness of both surfaces glazed and polished was measured using a profilometer (SJ 201, Mitotoyo, Japan, Kawasaki). The values of interest were Ra and Rz, Ra meaning arithmetical mean roughness and Rz meaning the maximum roughness of the analysed surface. These roughness parameters were measured after immersing of the samples in specific solutions.
3. Results

3.1 Microhardness

For the glazed specimens that were immersed in distilled water, the Vickers microhardness of the ceramics ranged from 9.56 GPa to 11.80 GPa for lithium disilicate ceramic and for the feldspathic glass ceramic from 8.60 GPa to 9.02 GPa.

For the polished specimens immersed in distilled water, Vickers microhardness ranged from 8.30 GPa to 9.10 GPa for the lithium disilicate ceramic and from 7.30 GPa to 8.30 GPa for the feldspathic ceramic.

After immersing the glazed samples in tea Vickers microhardness ranged from 9.45 GPa to 10.24 GPa for lithium disilicate and from 8.30 GPa to 8.90 GPa for the feldspathic ceramic (Figure 1). Immersing the samples in carbonated acidic beverages proved to change the microhardness parameter and ranged from 7.50 GPa to 9.2 GPa for the feldspathic ceramic for the glazed samples and from 6.67 GPa to 7.50 GPa for the polished samples. The samples made from lithium disilicate proved to have a microhardness from 9 GPa to 10.07 GPa the glazed samples and from 7.60 to 8.90 GPa the polished samples.

The immersion solutions and the temperature do not influence so much the Vickers microhardness. The important factor that makes a difference is the surface processing method. The glazed samples proved to have higher microhardness values. The means are the same (p>0.05) (ANOVA test) meaning that are not noticeable differences (Table 6).

| Table 6. Microhardness (Pa) of the feldspathic and lithium disilicate ceramic. |
|-----------------|--------------------|-----------------|-----------------|--------------------|
|                  | Feldspathic ceramic | Lithium disilicate |
|                  | Glazed             | Polished         | Glazed          | Polished          |
| Distilled water  | 8.6—9.02GPa        | 7.3—8.3 GPa      | 9.56—11.80GPa   | 8.3—9.1GPa        |
| Tea              | 8.3—8.9GPa         | 7.1—8.1GPa       | 9.45—10.24GPa   | 8.4GPa            |
| Carbonated acidic beverage | 7.5—9.2GPa | 6.67—7.50GPa | 9—10.3 | 8.9GPa |

Figure 1. Vickers microhardness of the tested samples.

3.2 Roughness

Ra values of the glazed specimens immersed in distilled water were 0.14 µm for the glazed feldspathic glass ceramic and 0.20 µm for the polished samples. For the lithium disilicate glazed samples the values were 0.1 µm and for the polished samples 0.09 µm.
After immersion in tea, Ra values of the glazed samples from feldspathic glass ceramic were higher (0.19) than for the glazed samples from lithium disilicate ceramic (0.10 µm). For the polished feldspathic samples the Ra values 0.30 µm and for the polished lithium disilicate 0.08 µm.

After immersion in carbonated acidic beverage the Ra values were for the glazed samples 0.10 µm for feldspathic and for lithium disilicate 0.18 µm. For the polished samples 0.08 µm for feldspathic and for the lithium disilicate 0.111 µm. The differences between the samples are not significant.

The smoothest obtained surface was the glazed surface from the lithium disilicate glass ceramic after distilled water immersion (Ra=0.02) and roughest obtained surface was the polished feldspathic surface after tea immersion (Ra=0.38).

The maximum obtained roughness (Rz) was 2.31 µm for the polished feldspathic samples that were immersed in tea (Figure 2, 3)( Table7, 8).

The ANOVA test showed that there were no noticeable differences between the values(p>0.05), means were the same.

**Table 7.** Ra parameter for the tested samples.

|                | Feldspathic ceramic | Lithium disilicate ceramic |
|----------------|---------------------|-----------------------------|
|                | Glazed | Polished | Glazed | Polished |
| Distilled water| 0.14   | 0.2      | 0.1    | 0.09     |
| Tea            | 0.19   | 0.30     | 0.10   | 0.08     |
| Carbonated acidic beverages | 0.10 | 0.08 | 0.18 | 0.111 |

**Figure 2.** Ra parameter values for the tested samples.

**Table 8.** Rz parameter values after imersing the samples in distilled water.

|                | Feldspathic ceramic | Lithium disilicate ceramic |
|----------------|---------------------|-----------------------------|
|                | Glazed | Polished | Glazed | Polished |
| Distilled water| 1.09   | 2.053    | 1.375 | 0.513    |
| Tea            | 1.72   | 2.31     | 0.715 | 0.585    |
| Carbonated acidic beverages | 0.847 | 1.55 | 0.578 | 0.936 |
4. Discussion
The results of this test revealed that the surface roughness of the samples immersed in distilled water are the lowest compared to the samples immersed in other liquids. However, the Vickers microhardness proved to be slightly influenced by the immersion solutions, both by the temperature and the pH. Several in vitro studies [14,15] show that the ceramics are susceptible to corrosion in range of oral fluid pH. The dissolution potentials of ceramics in acidic and alkaline liquids such as carbonated acidic beverages [15] and fruit juices differ considerably based on the patients saliva and diet [16]. Moreover, ceramic corrosion can affect the fracture strength of the materials and can increase their roughness [16]. It is known that patients experience discomfort, when the value of Ra is more than 0.50 µ [17]. All the registered roughness values after immersion in distilled water, tea and carbonated acidic beverages where lower than this value. Previous in vivo studies proved that clinical degradation and roughening of ceramic surfaces appear after 1-2 years after cementing them [18, 19, 20], this is why this study period was 1 year and a half. In this study the Ra parameter slightly increase after the period, but the Rz parameter doubled in tea for feldspathic ceramics. Indentation hardness is a predictor of the wear resistance of an material. The thickness of the samples used in this study was 1.5 mm and simulated the clinical practice. An increase in Vickers microhardness represents an increase in resistance to abrasion for the ceramic surfaces. On the basis of the results found here, it can be concluded that the high temperature of the tea produced a small decrease of the microhardness experienced better on the polished samples of both types of ceramics. The glaze layer protected both of the ceramics and the values of Vickers microhardness were higher than the polished samples. The glaze protected the ceramic samples against low and high temperatures.

5. Conclusions
Within the limitation of this study some conclusions can be drawn:
1. The pH and the temperature have no influence on the mechanical behaviour on the two types of ceramics at the chosen time.
2. The mechanical behaviour and the roughness is influenced by the surface protocols (glazing and polishing) and by the type of ceramic (feldspathic and lithium disilicate).
3. The values of both roughness and microhardness proved that the feldspathic ceramic should be glazed rather than polished.
6. References

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