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# Evaluation the Flexural Strength of Emax Cad and E_Max Press After Thermocycling Ageing

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## *Abstract*

***Aim:*** *To investigate the impact of thermocycling ageing on the flexural strength of E-max cad and E-max press*
*The objective of this research is to evaluate the impact of aging via various thermocycling protocols on the flexural strength of a feldspathic ceramic.*

***Material and Methods:*** *Thirty six bars of ceramic, ivoclar e max, with dimensions of 16×4×2 mm were utilized. Bars were distributed randomly into three groups (n=6) that were defined in accordance with the number of thermal cycles (TCy): G0-no TCy; G5000-5000 cycles of TCy, G10000-10 TCy. After aging, samples were subjected to a three-point bending test in a universal test machine with a speed of 1mm/min and a load of 1kgf until the catastrophic failure was reached.*

***Results:*** *Concerning the mechanical test, the following values of mean and standard deviation values (MPa), were obtained: G0cad (612.12± 83.16) ; G5000cad (445.44 ± 34.99); G10000cad (366.34± 41.30) and G0press (534.09±42.83) ;G5000press (428.68±17.40) and G10000press (382±38.49).Variance analysis of was conducted using the regression equation (p=0.387) and it showed that there was a significant correlation between the flexural strength and the thermal cycles number.*

***Conclusion:*** *One can conclude that aging in water per se through various temperature cycles numbers has an effect on the flexural strength of a feldspathic ceramic. The thermocycling of both type of lithium disilicate has negative effect on the flexural strength, Cad ceramic showed more significant decrease in flexural strength than press ceramic which was less negatively affected.*

## Introduction

Due to the growing need for aesthetics in practices of dentistry, systems of (CAD/CAM) have been developed. Moreover, it is vital to regain the semi-natural opalescence, shining and the translucent restoration so as to enhance the natural tooth appearance [1].

The clinical success with long-term related to dental ceramics is directly affected by a number of factors, amongst which are the crystal-based microstructure of the ceramic substance, the materials fabrication- technologies in addition to the clinical environment [2].

Prosthetic dentistry undergoes a great shift from metal ceramic restorations to all-ceramic prostheses, particularly in terms of aesthetic and biocompatibility. Nevertheless, ceramics are deemed brittle and suffers from fracture susceptibility. Hence, developing a hard and esthetic ceramic has been given a considerable attention [3].

The disilicate ceramic made of Zirconia and lithium were compared and it has been demonstrated that zirconia materials are still less efficient from translucency point of view [4].

Esthetics is a personal choice that is impacted by an individual's personality and cultural factors. Looking back in time, people's perceptions of what makes a perfect smile have shifted. The shining, white smile with teeth aligned perfectly is, nowadays, regarded as an essential an inseparable part of human's beautiful appearance. Therefore, the materials that meet these specifications are needed now in the dental treatment [5].

Ceramic glazing is represented by a mixture of a powder and a liquid that are fired on the surfaces of the ceramic and this leads to a glossy surface [6].

## Categorization According to the Processing Technique

There is a technique (more user-friendly) and simple to categorize the ceramics that are utilized in the field of dentistry, which is stood for by the method of processing. So, it is vital to notice that all the substances could be processed through various methods but, generally, they might be categorized as the following:

(1) powder/liquid and systems that are based on glass.
(2) System blocks that are based on glass, which are machinable or pressable.
(3) CAD/CAM or slurry, die-processed [7].

***IPS e. max CAD:*** The I-P-S- e.max CAD "blue block" utilizes a process crystallizing that involves 2 stages and this process utilizes a process that is controlled with double-nucleation, that the lithium-meta-silicate crystals that precipitate throughout step 1 show in. Resultant glass ceramic, shows interesting features of processing in terms of milling and it is inclined to be with "blue color" in this stage, which is dependent on the amount of colorant added. In the step of the second heat treating, which is preformed after the occurrence of the process of milling, the phase of meta-silicate is totally molten and lithium-disilicate becomes crystals. This occurrence of heat-treatment takes place at the temperature degree of (840-850º) centigrade approximately in the furnace of porcelain. This grants the ultimate restoration a glass ceramic with fine grains involving (70%) volume of crystal that incorporates a glass (AlSafar 2023).

***IPS e.max Press:*** The material of I-P-S-E.max Press is made in a similar way to that of I-P-S-E-max (CAD) is produced as far as the formation of the initial ingots of glass is concerned. They are made up of various powders, which are melted and cooled to room temperature to produce these glass ingots. After the formation of the glass, the ingots are nucleated and they are crystallized in one-heat treatment to yield the final ingots. After that, the ingots are pressed at a temperature of approximately 920ºC for a period of 5-15 minutes to and forms a 70% crystalline lithium disilicate restoration [8].

The blocks, which are crystalized partially and, then, utilized to milling in I-P-S- E-Max CAD, comprise forty per cent of crystals of lithium-metasilicate ($Li_2SiO_3$), with a size of 0.2e1.0 mm with platelet shaped, are set in a glassy phase along with lithium disilicate nuclei [9].

The partially-crystalized state, is milled more easily, leads to lower bur wear and high stability in terms of the edge [10].

Again, as milling is complete, the restoration will be subjected to another round of heat treatment. The manufacturer takes into consideration that the material is fully crystallized after being tempered at 850˚C for 20 to 25 minutes in a vacuum [11].

### *Biocompatibility*

The ceramic substances biocompatibility in the environment of the mouth, has been tested by means of employing various methods (e.g., the test of cytotoxicity and the agar-diffusion test) [12].

Additionally, improving those materials within a couple of weeks of aging in addition to the stability (relative) of cytotoxic after-polishing response reveals that these could have a good performance clinically in the long run. moreover, the agar diffusion tests, were conducted based on (ISO 10993-5) stipulations, in which results showed that the E-max substances have been regarded as noncytotoxic [13].

### *Thermocycling*

Thermocycling involves water baths with various temperature degrees throughout multiple cycles. Fisher, et al., demonstrates that the thermal cycles were the reason behind aging and this led to developing a period of clinical service in terms of the materials of restoration. Impacts of thermal-cycling on the materials that involve frequent contractions and/or expansions, which lead to cracks and fractures propagation, retention loss, cracks formation at the interfaces and finally a degradation in restorations. Moreover, water absorption in addition to the hydrolytic deterioration, both exert certain negative impact on the substances [9].

## Material and Methods

### *Materials*

All material used in this study were within expiration date.

### *Samples Preparation*

**Sample preparation of E-max CAD:** For CAD/CAM lithium disilicate partially crystalized blocks HT/A2 B40 were cut using ISOMET 1000 (Buehler, precision saw) with water coolant and with the use of isomet diamond wafering blades in order to get rectangular bars with dimensions of (16 mm×4 mm×1.5 mm). A digital caliper was used after that for making sure of these dimensions. Samples were crystalized in full using Programat P310 (Ivoclar Vivadent) in an attempt to follow the instruction of the manufacturer concerning the firing cycles as the temperature degree 850˚C was the maximum firing degree.

For surface roughness and color, the slices of the ceramic were prepared from a block of e .max CAD/CAM with dimensions of (10×10×1.5 mm) and a shade A2 HTby lowspeed diamond saw-isomet 4000* (*Isomet 4000, Buehler, Lake Bluff, H., USA.) with water-coolant system. The ultimate specimens thickness was asserted by means of a digital caliper for the purpose of standardization.

### *Finishing and Polishing*

All the specimens finishing and polishing were conducted using grit carbide sandpapers #600, #1200, and #2400 under a running water and by means of using the polishing machine type (Phoenix beta) [15].

### *Sample Preparation of Ips E-max Press*

The wax resin samples were designed by making wax model through exo- CAD program with dimensions (16 ×4 mm×1.5 mm). Then the models were printed by 3D printer as shown in figure (3.9) and invest these model by e.max press HT A2 IPS E-max ceramic ingots Ivoclar Vivadent AG, Schaan Liechtenstein) were pressed in the investment mold [16].

The wax resin is designed in exact dimension, spruing and poured into investment ring.
The invesment mixed by weight 125 gr powder and 15 ml of liquid as manufacture instruction using bellavest investment (bego) after mixing in a vacuum for a period of 1 min and mixing rate about (450) rpm mixer to avoid air bubble entrapment.

After pouring the investment, there should be a waiting interval for (15) minutes to have the investment dried, then the ring invesment is put in the furnace to get wax resin elimination under heating degree of 915º C and the heating should be steady for (30) second to ensure the evaporation of the resin. Then, the ring mould was placed in ivoclar furnace (P 5050) to inject the e-max press with 100 grams pressure according to manufacture instructions.

The ingot of L. di-silicate (I-P-S-e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) shade (A2) high-translucency (HT), was chosen and plasticized and, after that, pressed using an alumina plunger (as shown in figure 3.12). Ingots were heat-pressed first into the investment mold. The process was conducted by utilizing a press-furnace with the: 700˚C, 920˚C for press and 25 minutes-period of time (Programat EP3010, Ivoclar Vivadent, Schaan, Liechtenstein).

After injecting e max press in the invested mold, the investment is left to cool and then e max press samples are extracted from the investment mold with the sandblast to remove, cut the investment and extract and clean the e max.

### *Standerdization, Grinding and Polishing of the Sample*
To obtain standard samples, the thickness, length and width of all samples were processed by means of using a grinding machine type (Buehler, phoenix beta, grinding and polisher, German) and with a speed of 600 rpm and 80D -600C-800C-1200 -2400 C abrasive papers of grit silicon carbide under water that is running. The ultimate thickness of every specimen was measured by means of utilizing a manual or digital caliber (1.5 ± 0.03 mm), between grinding and its last stage. Dimensions of the sample was: 16 mm length and 4 mm width according to ISO specification (7268).

### *Thermocycling*
In this study (100) SD-Mevhatronic thermocycler German made has been utilized. Samples have been, all, subjected to thermo-cycling for (5000) cycles for the first group and (10000) cycles for the second group in temperature degrees between 5˚C and 55˚C in deionized water with a dwell time of 30 seconds and transfer time of 10 seconds.

The protocol of thermocycling used in this study was conducted in compliance with the protocol used, where the exposure temperature ranged between 5˚C and 55˚C [17].

The exposure for (10,000) cycles, could be called the cycles of thermal alternation, which occurred in one year. Despite the fact that the thermo-cycling protocol does not resemble the real condition, just as in the mouth environment, but it can utilized for evaluating such materials' behavior when thermal stress is applied to them [18].

### *Flexural Strength Testing*
Thirty six (36) from every group have been tested in a bending assembly with three points, connected to a testing machine (Multitest 2.5-I; Mecmesin, W. Sussex, United Kingdom). Samples were used, until occurrence of fracture took place, and the load used was (1000) N cell, a crosshead speed of 1 mm/min, as it is shown in figure (3.16). The flexural strength (FS) was measured by means of using the equation FS=3Fl/2bd2 and this was conducted in accordance with the standards of ISO 6872-2015; where F: is the fracture loadmrepresented by (N), l: stands for the test span (16 mm), b: stands for the bar width (4 mm). And d: bar thickness (2 mm). After that, the mean of FS (MPa) for every group was determined.

## Results
### *Flexural Strength*
***Normality for tests of flexural strength:*** Values of the flexural strength were input in SPSS package to conduct the statistical analysis in order to obtain the statistical results and to analyze them. The data that was input to Shapiro-wilk normality test and the test results, was distributed parametrically and normally.

***The descriptive statistics of the flexural strength test of lithium disilicate materials with without ageing:*** Table (1) shows the data of flexural strength of the current research and the number of samples, the arithmetic means and the standard deviation before and after thermocycling ageing and demonstrates that empress ceramic, had less value than cad in the control group (0 cycles), which mean that the CAD ceramic has more strength than empress at non aging condition.

This table illustrates that there are significant changes (decrease in flexural strength) throughout the artificial ageing, the significant change include both CAD and press ceramic through both 5000 and 10000 cycles. The CAD ceramic demonstrated more statistically significant decrease of flexural strength after (5000) cycles and the decrease became more at (10000) cycles.

The press ceramic demonstrated a significant decrease in flexural strength after (5000) cycles and further decrease in (10000) cycles but the deterioration was less than the ceramic CAD.

The IPS e max press showed resistance through the thermocycling in comparison to CAD ceramic, but both were significantly changed. The press ceramic shows a better resistance to thermocycling in terms of flexural strength, as shown in table (2) of two-way anova.

| Entry time | Entry temp. | Heating rate | Final temp. | Holding time | Lower table | Start vacuum | Release vacuum |
|---|---|---|---|---|---|---|---|
| 6:00 min | 400C$^0$ | 30C$^0$/min | 845C$^0$ | 10:00min | 700C$^0$ | 550C$^0$ | 845C$^0$ |

**Table 1:** Program parameter of ips e max CAD crystallization.

| Cycles | N | Mean CAD | SD CAD | Mean press | SD press |
|---|---|---|---|---|---|
| Control | 6 | 612.12 | 83.16 | 534.09 | 42.83 |
| 5000 | 6 | 445.44 | 34.99 | 428.68 | 17.40 |
| 10000 | 6 | 366.34 | 41.30 | 382.80 | 38.49 |

**Table 2:** Descriptive statistics for L.di-silicate (press and cad) and the effect of thermocycling ageing.

| Source | DF | Sum Squares | Mean Squares | F Value | Pr>F |
|---|---|---|---|---|---|
| Materials | 1 | 4183.274 | 4183.274 | 2.02 | 0.1651 |
| Thermocycling | 2 | 233429.3 | 116714.6 | 56.49 | <.0001 |
| Materials Thermocycling | 2 | 17643.86 | 8821.928 | 4.27 | 0.0233 |
| Error | 30 | 61985.94 | 2066.198 | - | - |
| Corrected Total | 35 | 317242.3 | - | - | - |

**Table 3:** Two way ANOVA of flexural strength of both ceramic cad and ceramic press thorough the thermocyclic aging.

| Thermocycling Materials | 0 | 5000 | 10000 | |
|---|---|---|---|---|
| **CAD ceramic** | 612.12+83.16 A | 445.44+34.99 C | 366.34+41.30 D | 474.63+118.35 A |
| **Press Ceramic** | 534.09+42.83 B | 428.67+17.40 C | 396.45+20.35 CD | 453.07+66.40 A |
| | 573.10+75.09 A | 437.07+27.76 B | 381.40+34.80 C | |
| The different letter refers to significant differences between the groups at a likelihood of (P<0.05). | | | | |

**Table 4:** Duncan's multiple range test of flextural strength (mpa) of press and CAD ceramic through thermocyclic aging.

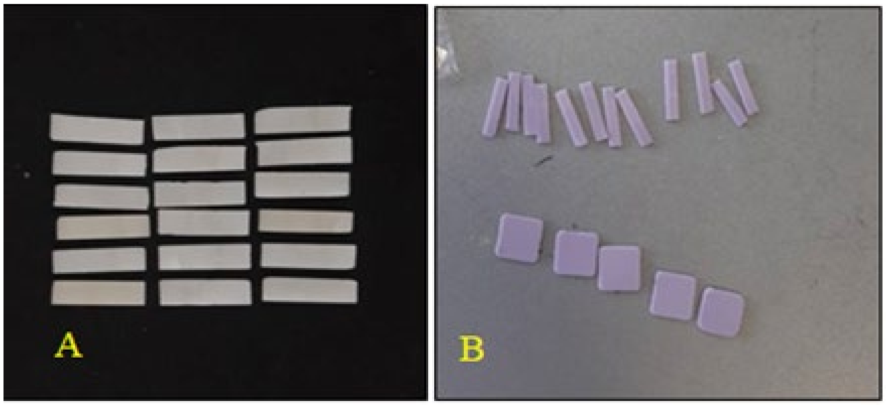


**Figure 1:** (Lithium disilicate) e-max cad. A) Fully crystalline sample; B) Partially crystalline sample.

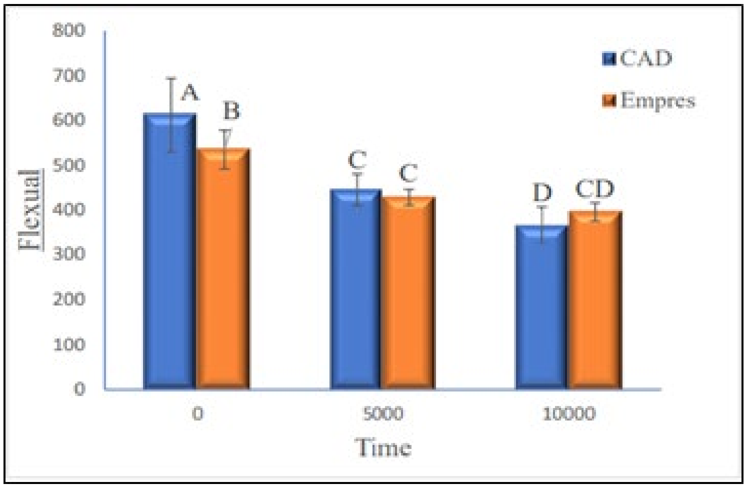


**Figure 2:** Duncan's multiple range test (column graph), which shows the impact of various thermo-cycling ageing on the value of flexural strength of various dental lithium disilicate (press and cad ceramic).

## Discussion

Concerning the thermocycling impact on the lithium disilicate strength degradation, there is lack of data that is badly required. The thermocycling protocol used in the current research was conducted in compliance with its counterpart that was employed in the previous studies, in which exposure temperature degrees were 5ºC to 55ºC [18,19].

There is difference between invitro study and invivo study and one of them is that the saliva pH at night-time is considered as harmful to the loss of glass-based systems mass, as it breaks the framework of the silica glass and this leads to lowering the level of strength. The intraoral loads, which are uncontrolled and alkaline (pH), could be interacting with the defects, thus leads to a decrease of the material flexural strength, which in turn, facilitates the nucleation and crack propagation. (20).

The flexural strength of e max press shows deterioration of both types of lithium disilicate during thermocycling with little advantage to e max press. In the current study, the flexural strength mean value in the control group IPs e max press 534.09 ± 42.83 and 664.36 for CAD. However, it comprises all values of flexural strength mentioned by the manufacturing authority and indicated by others parties (300-400 MPa) [21].

The press lithium disilicate possesses less flexural strength in comparison to CAD lithium disilicate and this is could be due to CAD lithium disilicate, which is more homogenous and has less internal defect as it was manufactured to be in a metasilicate state, and then milled to be fully crystalized in the ultimate state.

Lithium disilicate CAD has more pores in the surface, so that it can be influenced more by theremocycling, particularly in (10000) cycles compared to the e max press.
The final microstructure involves lithium disilicate crystals that are highly interlocked and this results in a multiple crack deflection [22].

Aging specimens, which uses the thermocycling treatment is regarded as a common protocol that is used for evaluating the degradation of dental materials with the course of time. Additionally, When restorative materials are immersed in water, with or without thermocycling, this will results in a slow growth of crack, which could decrease the flexural strength. It was reported that Lithium disilicate is highly susceptible to slow growth of crack after it had been aged by cyclic fatigue [23].

From the Theoretical point of view, the mismatch of thermal expansion between the ceramic crystals and glassy matrix leads to tangential compressive stresses around the crystals and this might be responsible for crack deflection and increase of strength [24].

The microcrack in ceramic, induced by thermal expansion, was reported as an effective method to strengthen the ceramics because there is a significant change in thermocycling for CAD e max with less range value of number for flexural and this may be due to the sample preparation by CAD CAM machine, which could initiate some cracks that can propagate subsequently [24].

De Pinho Barcellos et al., whose results were in agreement with this study, recently found statistically-significant differences in L. di-silicate strength, per ageing type. This might be because lithium disilicate is a bi-phase-substance, the variation temperature resulted in variant thermal contractions of phases and a decline of strength. Additionally, based on several researchers, the ageing protocol, led to extra material damages compared to the protocol related to in-vitro; that show that the thermo-cycling (long lasting) couldn't have been sufficient to inflict a remarkable decrease in strength.

So, variations in mechanical materials characteristics, due to thermo-cycling, are mostly a consequent of the thermal incompatibility of combination of different phases and substances like the zirconia and porcelain [25]. In addition to that, temperature degrees variations and water in the Feldspathic ceramic, which is employed in the current work is insufficient to create morphological changes, or to cause a strength decline in. Hence, thermo-cycling ageing could be a vital provision, just when one assesses various materials as well as various thermal behaviors [26].

L. di-silicate involves (65%) of the "volume fraction of lithium disilicate (Guazzato et al., 2004). The cracks propagate basically to (34%) volume fraction of residual glass (Guazzato et al., 2004) and they deflect when they reach crystals (Apel et al., 2008). Crack deflection demands more energy and results in enhanced strength of the material (Quin, 2007).

The origin of the fracture shows signs of fracture that are less defined, like the compression curl on the opposite side of the sample, or may be less sharp hackle lines and might be resulting from the effect of torsion effect in the final stages of fracture that are related to the characteristic of the bar's clamped-end.

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Dephinho et al., take sem scan that shows compression curls on the opposite side, main origin and a second event possibly indicating torsion, closest view of the compression curls of fracture origin that refers to the fracture origin propagation and the results of the researcher [27] is not in conformity with the ones of this study, as he argues that ceramic is brittle and is not influenced by the thermo-cycling, particularly the (10000) cycles (de Pinho Barcellos et al., 2018).

The researcher [28], is in disagreement with this work, which illustrated that changing the number of thermal cycles to reach (15000) didn't cause significant differences in the flexural strength related to feldspathic ceramic. As for the protocols of aging, only few studies dealt with the potential resistance of ceramics with the course of time.

Ageing by means of thermo-cycling demonstrated that there was no "significant decline in the flexural strength in accordance with the number of the thermal cycles that have been performed. The reason might be because the water (at different temperature degrees) have been utilized as a one factor of aging and the crack increase is connected, as well, to the existence of repeated minor loads.

In addition to that "additional factors impact the final resistance of the ceramic materials, including the size of specimens, the environment of the test, the procedure of polishing, the specimen area" that undergoes tension and the test method [21].

The researcher [29] disagree with this study and states that there was no significant change in flexural strength by repeated pressing. Thiago porto (2018) argues that there is significant effect of thermocycling on the lithium disilicate. Therefore, the hypotheses are rejected, concerning the differences between "the material class flexural strength and flexural modulus in terms of the materials that were presented with respect of the statistical significances when the long-term thermocycling test is applied, all show that flexural strength values for this material are sensitive to thermo-cycling [30].

Shereen Saleem (2020), disagrees with the current study. She states that thermodynamic has no significant effect on the flexural strength and this is may be because the size of crystals and large size crystals strengthen the effect, which result in crack deflection and bridging.

Munir (2023) agree with this study and state that the third hypothesis was also accepted as significant differences were observed among the Bi Flexural Strength values of tested materials with coffee thermocycling. In addition to that, Porto et al., (2019), observed a prominent increase in the flexural strength of L. di-silicate ceramic, after storing in the water for one week [30].

Soliman (2017) agree with this study and states that there is significant deference in flexural strength after thermo-mechanical cycling. Salem and Asaad (2020) Disagree with our study and state that thermodynamic aging did not have effect significantly on the stability of color and flexural strength of the materials tested while it influenced -significantly- the Rosetta SM surface roughness [31].

This could be interpreted by the fact stipulating that lithium disilicate is a glassy material that possesses monocrystalline structure, which has been stabilized completely by means of the crystallization cycle, is not polymorphic in nature, and does not undergo water penetration or LTD (low temperature degradation). Consequently, the artificial aging has no impact on the flexural strength of lithium disilicate ceramic material. Moreover, there is another explanation, which is represented by ability of water to blunt a tip of crack of the glass-ceramic in the moist environment, thus decreases the crack growth. However, this theory is deemed controversial, as even when the blunted cracks are subjected to extending in moist [32].

## Conclusion and Suggestions

### *Conclusion*

Throughout this study, it was concluded that: The ageing processes affected the milled ceramic more than the heat pressed. From both groups, milled and heat-pressed in related to physical properties.

### *Suggestion*

1. Study the effect of 140 000 to 350 000 cycles of thermocycling in physical properties to expect more deterioration.
2. Study other physical properties like bi axial flexural strength, wear resistance compressive strength, tensile strength.
3. Study the effect of thermodynamic aging with the effect of thermocycling ageing.
4. Study the difference with other types of lithium disilicate eg (Sirona Dental Cerec Block, rosetta SM, IPS Empress CAD) and which one is affected more by thermocycling.

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