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INFLUENCE OF HYDROFLUORIC ACID CONCENTRATION AND ETCHING TIME ON PULL OFF OF LITHIUM-DISILICATE GLASS ANTERIOR CROWNS

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ABSTRACT

Aim: Is to evaluate the Influence of hydrofluoric acid concentration and etching time on pull off of lithium-disilicate glass anterior crowns.

Material and Methods: forty-five lithium disilicate (IPS e.max cad) full coverage crowns were fabricated on prepared extracted human maxillary central incisors. The crowns (n=45) were divided into three groups (n=15) according to the hydrofluoric acid (HF) concentrations used for the cementation of these crowns to the teeth; I) HF 4.9%, 2) HF 9.6%, 3). HF 11%. Then each group was subdivided into three subgroups (A, B, C) according to etching time (20seconds ,35seconds&60seconds respectively). Each crown was cemented to the corresponding prepared tooth using an adhesive resin cement. Then, the whole crown tooth assembly were subjected to thermocycling (5°C - 55°C,5000 cycles,). A universal testing machine was used to assess pull-off test. Data were collected, tabulated and statistically evaluated.

Results: The results showed a statistically significant difference in the mean values of the pull off test between the groups regarding HF concentration. Group 1 (4.9% hydrofluoric acid) (206.5 ± 13.1) < group 2 (9.5% hydrofluoric acid) (244.1 ± 25.4) < group 3 (11% hydrofluoric acid) (284.4 ± 13.5). For 9.6% and 11% HF acid concentrations, etching durations used in this study do not have a significant effect on the retention of lithium disilicate glass crowns. However, for 4.9% HF the retention means significantly increased with elevating the etching duration.

KEYWORDS: Hydrofluoric acid, Lithium disilicate IPS E.max Cad, Pull-Off Test

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INTRODUCTION

Glass ceramics are frequently used to restore missing, broken, and decayed teeth. The main reasons that encourage the dental practitioners to use them are their ideal features, such as their capacity to adhere to dental structure, best aesthetics, excellent biocompatibility, and coefficient of thermal expansion that is close to that of tooth structure. The excellent optical and satisfactory mechanical qualities allowed the lithium disilicate glass ceramics to have excellent therapeutic results and a long-term high survival rate(1). Numerous factors, including ceramic composition and luting techniques, affect the clinical outcome of ceramic restorations. Combining hydrofluoric acid (HF) etching with a coupling agent (silane) results in the optimum adhesion to lithium disilicate. This procedure has been acknowledged as the most popular surface conditioning for glass ceramics(2).

The strategies for ceramic surface treatment can be classified according to their mode of action; chemical, mechanical, or chemomechanical. The most successful method for glass-ceramic reinforced by lithium crystals is the mechanical surface modification by hydrofluoric acid (HF) etching patterns. HF dissolves glassy matrix and exposes the embedded LD crystal As a result, more surface area is allowed for micromechanical coupling, which enhances the contact between ceramic and resin cement and improves the bonding quality (3).

The action of hydrofluoric acid on the glass ceramic surface is duration and concentration dependent. Most of the glass ceramic manufacturers suggests that the most effective etching protocol for glass ceramics is 4% hydrofluoric acid for 20 seconds

Moreover, many invitro researches followed different etching protocols using multiple hydrofluoric acid concentrations with different etching durations.

As there is no general agreement on the best etching process for glass ceramics, particularly for lithium disilicate glass ceramics, this research was conducted to test the effect of various concentrations of hydrofluoric acid with different etching durations on the pull off of glass ceramic anterior crowns.

The postulated null hypothesis of this study was that neither the hydrofluoric acid concentrations nor the etching times will have a significant effect on pull off of lithium disilicate glass anterior crowns

MATERIALS AND METHODS

Sample size estimation

Calculation of sample size was done by windows software named G-power 3.1.9.4 (Heinrich-Heire, Dusseldorf, Germany) ANOVA F test was used: fixed effect, omnibus and one way. Based on the previous study(4) each subgroup of the three tested groups contained 4 samples at least. An extra sample was added to each subgroup for result confirmation. A beta error ß of up to 20% was acceptable with an 80% study power and an alpha level α set at 5% with a 95% significance level.

Ethical approval

The research protocol was approved by the Research Ethics Committee of Minia University Faculty of Dentistry (RHDIRB2017122004) with protocol number (478/2021) at meeting number (77).

Preparing samples

Forty-five freshly human extracted upper central incisors free from caries, cracks or any coronal defects were used. Teeth were extracted for periodontal causes and the difference in the dimensions of the teeth were not more than ±5%. under running water, the blood and soft tissue adhering to tooth structure were removed. The teeth were cleaned from debris via the use of ultrasonic scaler (Guilin Woodpecker Medical Instrument Co.Ltd. China). Then the teeth were stored in distilled water at room temperature till use.

As a mean of retention, holes (1 mm) in depth were performed in the roots of teeth. Multi-hole Teflon split mold of 2 cm height and 2 cm diameter was used for teeth mounting using chemically cured acrylic resin (Acrestone, Egypt). After mixing the resin according to manufacturer instruction, the acrylic resin was poured into the mold in dough stage. Dental surveyor (NDI. Ney Dental Inc, Bloomfield Connecticut 06002 USA) was used to insert the teeth in the mold parallel to the long axis of the mold so that the level of acrylic resin is with 2 mm below the cemento-enamel junction.

To standardize the reduction for the teeth an addition silicon index was made before preparation. Addition silicon impression material (Zhermack S.p.A. | Via Bovazecchino, Badia Polesine (RO) ITALY) was mixed according to manufacturer instructions and the crowns of the teeth were inserted in the mix 2mm below cemento-enamel junction. Standard teeth preparation was carried out by new selected stones (834A/030& TR-12 & FO-22). Each mounted upper central tooth was seated on dental surveyor that was attached to a low speed handpiece, which was connected to an electric micro motor rotating at 15000 rpm speed. The preparations criteria were 1.5mm axial reduction, 1.5 mm incisal reduction, 1mm thickness shoulder finish line, about 15-20° taper.

Construction of crowns

Using an intraoral scanner (Cerec Omnicam. Dentsply Sirona,Long Island City , New York, USA) an optical impression was taken to the prepared teeth. Then, a specific software (Cerec Premium4.4.4 software) was used to design standardized crown for each corresponding prepared tooth. Each crown was designed with two retentive arms at the incisal one third of the crown to allow for the retention of wires while performing the pull off test using the universal testing machine. Then the data were sent to the milling machine (Cerec MC XL Premium, Dentsply Sirona, Long Island City, New York, USA). All the crowns were fabricated from IPS e.max cad blocks of the same patch no (#K03370) figure (1)

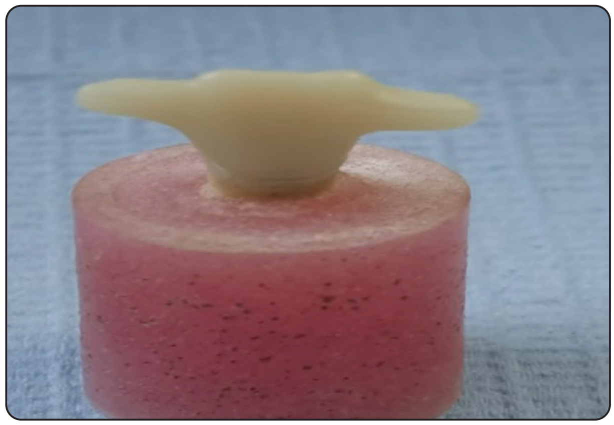

Fig. (1) Completed IPS e.max crown

Grouping of samples

The grouping of samples was carried using blind randomization. Samples were allocated blindly in the groups with the help of a non-dental friend who was asked to distribute the forty-five samples into three groups (n=15) without knowing the aim nor the intervention that will be carried in the samples. After that he was asked to divide each group into three sub groups (n=5) following the same manner.

- Group 1, 4.9% hydrofluoric acid.
- Group 2, 9.6% hydrofluoric acid.
- Group 3, 11% hydrofluoric acid.

Then each group was further subdivided into 3 subgroups (A,B&C) (n=5) according to the application time of hydrofluoric acid for (20sec, 35sec&60sec) respectively.

Teeth preparation for cementation

Each prepared tooth was etched for thirty seconds using 37% phosphoric acid. Then the tooth was rinsed thoroughly with air water spray of

2 bar pressure on a distance of 10cm and dried for 10 seconds. A thin film of a universal adhesive (all bond universal, BISCO, USA) was applied to the prepared tooth and thinned by air pressure. The adhesive will be cured light curing was done by a wireless LED light cure device with wave length 420-480nm and light intensity 1000-1200 m W/cm2 for twenty seconds.

Cementation of crowns

After HF application to the fitting surface of the crowns, each specimen was cemented to its corresponding tooth according to manufacturer's instructions under astatic load with 50 N using an adhesive resin cement (Duo-Link Universal Adhesive Cementation System. Bisco. USA).

Teeth-crowns assembly were stored in distilled water at room temperature till used. Thermocycling (SD Mechatronic Thermocycler. Westerham. Germany) was done for 5000 cycles from 5°to 55°c with 1minute dwell time.

Pull off test

Universal testing machine (INSTRON-CAT. NO:2710-115.USA) was used to establish the pull off testing. The pull off test was done to the cemented crowns along the path of insertion with crosshead speed 0.5mm/minute until disconnection of the two holding devices and debonding done. The maximum pull-off load was recorded in (N). figure (2)

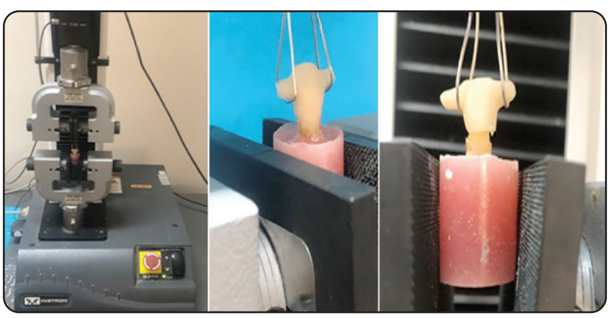

Fig. (2) Specimens in universal testing machine for pull-off test

Statistical analysis

Data were verified, coded and analyzed using SPSS version (21). One-way ANOVA test was calculated to test the mean differences between groups and two-way repeated measure ANOVA (RM-ANOVA) test was calculated to test the mean differences of the data that follow normal distribution and had repeated measures. Post-hoc test was calculated using Tukey's corrections for pairwise comparisons between the two study groups

RESULTS

1. Effect of different hydrofluoric acid concentrations:

There was statistically significant difference between the groups regarding the mean of the pull-off test results ($p < 0.001$). Broadly, mean pull-off test results for group 1 (4.9% hydrofluoric acid) was significantly lower (206.5 ± 13.1) compared with group 2 (9.5% hydrofluoric acid) (244.1 ± 25.4, $p < 0.001$) and group 3 (11% hydrofluoric acid) (284.4 ± 13.5, $p < 0.001$). Also, mean pull-off test results for group 2 (9.5% hydrofluoric acid) was significantly ($p < 0.001$) lower compared with group 3 (11% hydrofluoric acid) table (1)-figure (3).

2. Effect of etching time:

When comparing etching times there was non-statistically significant difference between the groups regarding mean pull-off test results ($p = 0.349$). In other words, mean pull-off test results for group 1 (4.9% hydrofluoric acid) was insignificantly lower (236.6 ± 36.7) compared with group 2 (9.5% hydrofluoric acid) (244.7 ± 35.2, $p = 0.327$) and group 3 (11% hydrofluoric acid) (253.8 ± 38.8, $p = 0.209$). Likely, mean pull-off test results for group 2 (9.5% hydrofluoric acid) was non-significantly ($p = 0.302$) lower compared with group 3 (11% hydrofluoric acid) table (2)-figure (4).

TABLE (1) Effect of Hydrofluoric Acid Concentration on the Pull-off Test Results

	Group (1) (n = 15)	Group (2) (n = 15)	Group (3) (n = 15)	P-value*
		Pull-off Test Result		
• Mean ± SD	206.54 ± 13.1	244.13 ± 25.4	284.42 ± 13.5	
• Median (Range)	204 (187-229)	246 (198-284)	284 (261-312)	< 0.001
P-value**	1 vs. 2 < 0.001	2 vs. 3 < 0.001	1 vs. 3 < 0.001	

ANOVA test was used to compare the mean difference between groups
*****Post-hoc test was used for pairwise comparison with Tukey's correction***

TABLE (2) Effect of Time on the Pull-off Test Results

	T1 (20sec) (n =15)	T2 (35sec) (n = 15)	T3 (60sec) (n = 15)	P-value*
		Pull-off Test Result		
• Mean ± SD	236.59 ± 36.7	244.72 ± 35.2	253.79 ± 83.2	
• Median (Range)	228 (187-294)	245 (196-300)	269 (188-312)	= 0.349
P-value**	1 vs. 2 = 0.327	2 vs. 3 = 0.302	1 vs. 3 = 0.185	

ANOVA test was used to compare the mean difference between groups

*****Post-hoc test was used for pairwise comparison with Tukey's correction***

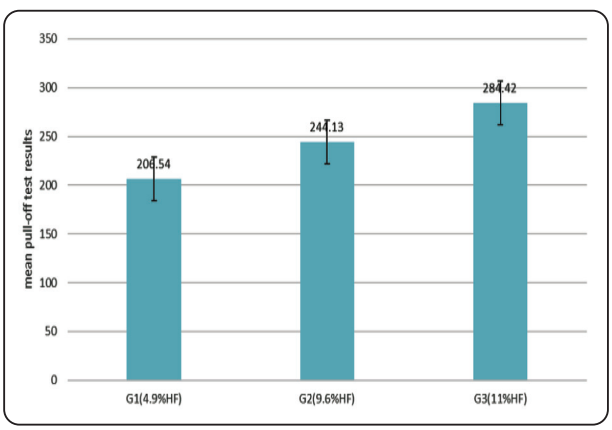

Fig. (3) Bar chart showed the effect of hydrofluoric acid concentration on the pull-off test results

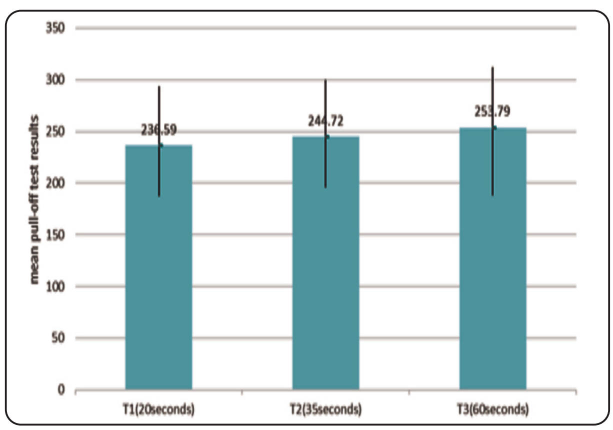

Fig. (4) Bar chart showed the effect of etching time on the pull-off test results

3. Effect of the different hydrofluoric acid concentration on the pull-off test results over time among the studied samples: -

There was significant difference ($p = 0.035$) regarding the interaction between time and hydrofluoric acid concentration on the pull-off test results over time.

Moreover, for 20 seconds time, there was significant difference ($p < 0.001$) in the mean pull-off test results i.e. mean pull-off test results for group 1 was significantly lower (189.9 ± 7.9) compared with group 2 (233.5 ± 25.4, $p < 0.007$) and group 3 (277.4 ± 11.8, $p < 0.001$). Also, mean pull-off test results for group 2 was significantly($p=0.001$) lower compared with group 3.

Regarding 35 seconds time, there was significant difference ($p < 0.001$) in the mean pull-off test results i.e. mean pull-off test results for group 1 was significantly lower (206.1 ± 9.8) compared with group 2 (245.5 ± 20.7, $p = 0.007$) and group 3 (282.5± 12.9, $p < 0.001$). Also, mean pull-off test results for group 2 was significantly ($p = 0.002$) lower compared with group 3.

Regarding 60 seconds time, there was significant difference ($p < 0.001$) in the mean pull-off test results i.e. mean pull-off test results for group 1 was significantly lower (214.7±16.7) compared with group 2 (253.4 ± 30.5, $p = 0.015$) and group 3 (293.3±13.4, $p < 0.001$). Also, mean pull-off test results for group 2 was significantly ($p = 0.012$) lower compared with group 3.

Respecting the difference in each group; for group 1, there was significant ($p=0.021$) steady increase in the mean pull-off test results from 20s to 60s. On the other hand, for groups 2 and 3, non-significant ($p=0.233$ and 153) increase was reported from 20s to 60s. Table (3)-figure (5)

TABLE (3) Result of Effect of Interaction of Conc. and Time on the Pull-off Test Results

	Group 1 (n = 15)	Group 2 (n = 15)	Group 3 (n = 15)	P-value*
T1	189.90 ± 7.9^{a}	233.48 ± 15.7^{a}	277.39 ± 11.8^{a}	< 0.001
• P-value**	1 vs. 2 = 0.007	2 vs. 3 = 0.001	1 vs. 3 < 0.001	
T2	206.07 ± 9.8^{b}	245.54 ± 10.9^{a}	282.54 ± 12.9^{a}	< 0.001
• P-value**	1 vs. 2 = 0.001	2 vs. 3 = 0.002	1 vs. 3 < 0.001	
T3	214.66 ± 16.1^{c}	253.37 ± 19.5^{a}	293.33 ± 13.4^{a}	< 0.001
• P-value**	1 vs. 2 = 0.015	2 vs. 3 = 0.012	1 vs. 3 < 0.001	
P-value***	= 0.021	= 0.233^{NS}	= 0.153^{NS}	= 0.035^{4*}

***** ANOVA test was used to compare the mean difference between groups***
*****Post-hoc test was used for pairwise comparison with Bonferroni correction***
******Repeated Measure ANOVA test was used to compare the mean difference within group***
^{4*}Repeated Measure ANOVA test was used to compare the mean difference between groups over time
Statistically Significant p<0.05 *Statistically Significant
Same letters within the same column means are not significantly differen

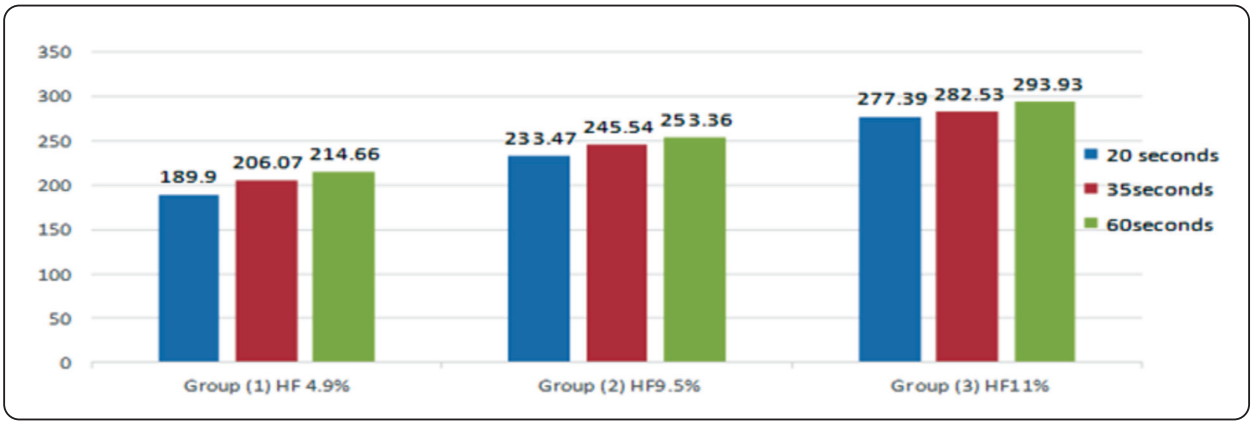

Fig (5) Result of Effect of Interaction of Conc. and Time on the Pull-off Test Result

DISCUSSION

Indirect restorations play a crucial role in tooth restoration. To repair lost or weakened tooth structure and to improve or restore aesthetics, crowns and veneers are applied to teeth for both functional and cosmetic reasons.

The adhesion process of indirect restoration is a critical issue because of multiple steps for the preparation of the tooth surface and the ceramic, being a sensitive technique susceptible to contamination and that also consumes time in clinical practice[5]. The adequate selection of the adhesive system, the application techniques and surface treatment are crucial to achieve strong durable bond [6]. In the current study, we aimed to examine the effect of different concentration of hydrofluoric acid as an effective mean of surface treatment and etching time on retention of adhesively cemented crowns.

Over the last few decades, dental ceramics field has progressed rapidly, both in the proprieties of ceramics and the techniques of manufacturing [7]. Computer-aided design (CAD) and computer-aided manufacturing (CAM) was developed to solve 3 challenges. The first challenge was to ensure adequate strength of the restoration. The second challenge was to create restorations with a natural appearance. The third challenge was to make tooth restoration easier, faster, and more accurate[8].

A machinable ceramic material was selected in this study (IPS e.max cad) A lithium disilicate based ceramic. One of greatest advantage of this material is that it exhibits high levels of biocompatibility and chemical stability to almost all organic and inorganic materials [9].

The clinical success of adhesive ceramic restorations is heavily dependent on luting resin cement and cementation procedures, including ceramic surface treatment. Surface treatment of lithium disilicate is attained by combined hydrofluoric (HF) acid and subsequent silane application. During the cementation procedure, chemical bonds and micromechanical interlocking are formed at the resin-ceramic interface. Micromechanical retention is provided by HF acid etching of the ceramic surface, while chemical coupling is provided by the application of a silane coupling agent. The HF removes the glass matrix creating a rough surface with irregularities within the lithium disilicate for bonding [10]. the increase of HF etching time affected the surface roughness and the flexural strength of a lithium disilicate-based glass ceramic. As hydrofluoric acid is a biohazardous material so the proper handling methods and shorter timing of application is a demand.

Resin cements can be classified into conventional and adhesive resin cement[11]. According to the adhesive system used to the prepared tooth structure before cementation the adhesive resin cement can be categorized into: total-etch, self-etch and self-adhesive resin cements [12].

In this study, we used Duo-Link Universal Adhesive Cementation System (BISCO. USA) it has many advantages as high bond strengths and excellent marginal integrity. As a total-etching system it contains a separate phosphoric acid enamel etching step which improves bonding to tooth structure and allows excellent marginal integrity[13].

Many studies showed that, etch and-rinse cementation technique had the highest SBS to enamel due to its higher etching capability. Etch and rinse technique is preferred for bonding to enamel, since the micromechanical interaction provides a long-lasting bond to enamel[14].

Silane application to the ceramic surface can influence the bonding between the resin cement and the ceramic restoration[15]. This is because of silane facilitate the contact with the ceramic due to bi-functional molecules through additional chemical bonding, besides providing a bond between silica in the ceramic and the organic matrix of the resin cement by way of siloxane bonds[16].

Sound human upper central incisors free from caries were used in current study and were stored in distilled water until use to prevent dryness and crack formation [17]. To mimic clinical situation crowns in this study were cemented to natural teeth not to composite blocks[18]. The teeth were randomly divided to overcome variations in teeth microstructure of enamel and dentin that could result in incorrect results. Holes of 1mm depth were done in the roots of teeth to allow for retention of the teeth in acrylic molds in which the teeth were embedded parallel to the long axis of the tooth[19].

Standard preparation of teeth were done following the guideline of standard anterior glass ceramic crown reduction and to the recommended thickness by the manufacturer regarding the use of this material [20], Then teeth were scanned and crowns were milled using CAD/CAM technology according to manufacturer's instructions.

Crowns were then cemented to their corresponding teeth after they have been treated using Duo-Link universal adhesive cementation system according to manufacture instruction. Cementation was done under static load of 50N for 5minutes for obtaining same cement thickness then stored in labeled containers in distilled water [21].

Crowns are subjected to significant temperature changes during intake of food of various temperatures, chemicals intraorally. The common artificial aging method to simulate thermal oral conditions is thermocycling[22]. In this process ,all specimens were subjected to a standardized and reproducible condition.

Authors in their experimental studies rarely give a thorough explanation for the choice of temperature and time conditions. The varied number of cycles, temperatures, dwell time and intervals between baths hinder in the comparison of results across studies. Consequently, results obtained from thermal cycling are contradictory. Approximately 10,000 thermal cycles correspond to 1 year of clinical function. This estimate is based on the hypothesis that such cycles might occur 20 to 50 times per day[23], however the accepted number of cycles is 5000 cycle as suggested by many authors[24,25]. Thermal cycling (5°C/55°C, 1 min) is the most efficient aging procedure[26]. The aging strategy in the present study consists of thermocycling for 5000 thermal cycles (5ºc/55ºc, 1 min dwell time).

Different types of bond strength tests have been utilized to evaluate the bonding efficiency of lithium disilicate glass ceramics to resin cements. Among these methods are tensile, microtensile, shear, and microshear bond strength tests and pull-off tests. The pull-off test was chosen in the present study because it was the most frequently used bond testing method investigating the adhesion between resin cements and ceramic materials[27, 28]. Pull off test is preferable than bond strength test as it take into consideration the complex geometry of an abutment preparation[4].

The study results rejected the previously postulated null hypothesis as statistically significant higher retentive mean values were recorded for higher concentrations of hydrofluoric acid, however for etching time, the mean values have no significance in all study groups.

This was in agreement with Puppin et al.[1] who reported that there is a clear effect of HF concentrations and etching times on bonding of lithium disilicate glass ceramic to resin cement, increasing HF concentrations showed the highest bonding strength values, regardless of the etching times. This was directly associated with a greater dissolution of the vitreous phase and exposure of lithium disilicate crystals.

This was also accordance other researches, which reported higher retention values with increasing HF concentrations ranging from 2.5% to 15% applied on lithium disilicate at room temperature although, the author suggests to use a concentration range between 7.5% to 10% [29]. The mechanism of action of HF on lithium disilicate was illustrated by the equation

$4HF + SiO2 \rightarrow SiF4 + 2H2O$ [30], glassy matrix is selectively removed because of the affinity of fluoride to silicon is greater than to oxygen, which makes possible the attack of ionized HF to silanol (silicon-oxygen bonds, SiO2) presented in the glass ceramic[31]. So, within limits the more the HF concentration, the higher the content of ionized HF, the greater the glassy matrix removal.

Regarding the evaluation of the influence of the different etching times, the results were higher etching times increased the conditioning ability of HF to lithium disilicate, especially for 4.9% HF concentration. This may be due to that the HF remain in contact for longer periods with the lithium disilicate surface, this will allow more time available for ionized HF to react with silicon (glassy matrix), thus removing more vitreous phase and, consequently, developing more surface irregularities for the mechanical entanglement with the resin cement by the greater exposure of lithium disilicate crystals.

However, the increased etching times did not lead to significantly higher bonding values for 9.6% and 11% HF concentrations. It may be because of that higher HF concentrations do not need extended etching times, as the amount of ionized HF is enough to properly dissolve the glassy matrix of lithium disilicate.

This is in agreement with findings reported in the literature. Kalavacharla et al[32]. examined the shear bond strength in lithium disilicate glass ceramics exposed to different etching protocols and concluded that etching time did not have a significant effect on bond strength for the specimens that were coated with silane while specimens that were not coated with silane exhibited higher bond strength values at higher etchant concentrations and longer etching durations, also Lucas Do et al [33] who found no positive correlation of increasing etching times and bonding strength to lithium disilicate.

Aging weakened the bond strength of resin cement to ceramic, which might be attributed to hydrolytic degradation of the bonding surface as well as thermal fluctuations during the thermocycling process. Many investigations have shown that bond strength decreases following thermocycling or water storage[34].

The mean values of pull off test in our study were lower than the results of other studies not using an aging method as Manal M. A. Madina et al[35] who reported higher mean values of pull off test. This is duo to the specimens are exposed to alterations in temperature and increased water exposure.

Thermal stresses can cause bond failure at the tooth-restorative interface by generating mechanical stresses through variations in the coefficient of thermal expansion[36].

This is in accordance to Amaral et al[37]. who reported that under thermal aging the bond strength is affected by several factors including temperature control, dwell time, and number of cycles in which the latest is the most influential factor. Also Andretta et al.[38] reported that bond strengths after aging were obviously decreased.

CONCLUSION

Within the limitation of this study, the following could be concluded: -

1. The retention of lithium disilicate was affected by the concentration of hydrofluoric acid and 11% hydrofluoric acid concentration showed higher significant mean values than 9.6% and 4.9% concentrations
2. For 9.6% and 11% HF acid concentrations, etching durations used in this study do not have a significant effect on the retention of lithium disilicate glass crowns. However, for 4.9% HF the retention means significantly increased with elevating the etching duration.

RECOMMENDATION

1. As HF acid is a biohazardous material, 20 seconds as a least application time for 9.6% and 11% HF concentrations must be used as higher application times do not produce significant higher retention strengths. However, for 4.9% HF more time is needed (60 seconds) to produce acceptable results.
2. Clinical trials with a controlled standardized study design are recommended to illustrate the clinical long-term performance of the tested material and surface treatment.

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