Effect of different acids surface treatments and thermocycling on shear bond strength of composite resin to feldspathic ceramic

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ABSTRACT

Background: the aim of this study was to evaluate the effect of different surface acids treatments (37%phospjoric acid, 5%hydrofluoric acid, 1.23 acidulated phosphate fluoride) of feldspathic ceramic VITA 3D MASTER , and the effect of thermocycling on shear bond strength using a ceramic repair kit (ivoclar/vivadent).

Material and Methods: sixty Nickel-Chromium metal base plates were prepared(9mm diameter,3mm depth) using lost wax technique, 2mm thick layer of ceramic(VITA 3D MASTER) fused to metal plates, all specimens were embedded in acrylic resin blocks except their examined surfaces and divided into 3 main groups 20 specimens each, Grp A: treatment with 37%phosphoric acid for 2 mins, Grp B: etching with 5% hydrofluoric acid for 2mins, Grp C: etching with 1.23% acidulated phosphate fluoride for 10 mins; monobond-plus, heliobond, resin composite(Tetric EvoCeram) were applied to each specimen according to manufacturer's instruction using transparent split mold(5mm diameter, 4 mm height); specimens were stored in 37°C distilled water for 12 weeks, 10 specimens of each group were subjected to thermocycling between 5 °C and 55 ° C for 800 cycles with 30s dwell time; shear bond strength was determined by a universal testing machine (instron 1122) at a cross head speed 0.5mm/min; One way ANOVA test, LSD test and student-t test were used to analyze shear bond strength.

Results: Mean shear bond strength values for the tested groups were: A1= 11.65±0.68 Mpa, A2=10.88±0.58 Mpa, B1=17.93±0.41 Mpa, B2=17.42±0.35 Mpa, C1=15.17±0.61 Mpa, C2=14.51±0.48 Mpa ; one way ANOVA test showed highly significant difference among groups; LSD test revealed that the use of 5% HF for ceramic surface treatment(GB) was highly significant than the treatment with 37%PA(GA) or 1.23%APF(GC) respectively and the use of 1.23%APF(GC) was highly significant than the use of 37%PA(GA); Student t- test showed a significant difference between subgroups of the same group with and without thermocycling.

Conclusion: ceramic surface treated with 5% HF acid for 2 mins recorded the highest shear bond strength, followed by surface treatment with 1.23% APF for 10 mins, most specimens treated with 5%HF showed cohesive failure with in ceramic while specimens treated with 1.23%AFP showed more (adhesive/cohesive) failure than adhesive or cohesive alone, and specimens treated with 37% PA showed nearly 50:50 adhesive and combination failure, thermocycling reduced the bond strength of each group significantly.

Key words: ceramic repair, different acids treatments, Tetric Evoceram, thermocycling, shear bond strength. (J Bagh Coll Dentistry 2013; 25(1):27-33).

INTRODUCTION

Ceramic and metal-ceramic restorations have been used for several decades by clinicians to provide esthetics and masticatory function[1]. Studies have shown various advantages of the ceramics, like color stability, radiopacity, coefficient of thermal expansion similar to that of dentin, good compressive and abrasive resistance, and esthetics[2,3]. However, dental materials and adhesive interfaces are subjected to stress in the oral environment: masticatory forces, temperatures changes, saliva and pH changes [(4)]. Moreover, trauma and fatigue can cause fracture of the ceramic or destroy the ceramic-metal bond[3] because this restorative material has a low tensile strength and a high modulus of elasticity with a brittle behavior· Problems such as a high treatment cost, possible trauma to the restored tooth, difficulty of removing the restorations, and patient demand for a rapid case resolution, may occasionally delay the replacement of a fractured metal-ceramic restoration [(4)], Intraoral repair of fractured ceramic restorations with composite resin restorative materials presents a substantial challenge for clinicians [(5)], and is also a viable alternative for patients because these restorations are difficult to remove and very expensive to be replaced[3].the type of composite resin also affect its bond strength to ceramic, hybrid type resins at the ceramic interface result in highter bond strength than those of microfilled composites[6] Numerous repair systems are available for recovering of ceramic fractures, the techniques include surface preparation of the ceramics and saline treatment in the bonding procedure[5]. These techniques involve air-particle abrasion of the surface with aluminum oxide, and etching the fractured part with different acids like phosphoric acid (PA), hydrofluoric (HF) acids and acidulated phosphate fluoride(APF) [(7,8,9)]. It has been postulated that acid concentrations and etching times should be adjusted with specific ceramics to optimize bond strenght[10] ,the bond strength of composite resin to aluminous porcelain was found to be inferior to that of feldspathic porcelain, hydrofluoric acid etching time has been reported to range from 60s to 20 mins [(11,12,13)], in addition difference in leucite concentration,

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size/orientation of crystals might affect etching times[14],in the dental literature often the use of 5-10% HF acid has been recommended [(15,16)]. Recent study showed that there is non significant differences between specimens treated with5%HF and 10%,at the same etching time(2mins) [(9)] .APF gel, widely used for in-office fluoride application, consists of sodium fluoride, phosphoric acid, and hydrofluoric acid. It is safe for oral tissue, unlike hydrofluoric acid, which can produce tissue rash and burn [(17)]. One recent study showed that 7-10 mins application of 1.23% APF gel on leucite containing porcelain produced a shear bond strength to composite similar to a 4 mins etch with 9.6%HF [(18)]. It has been found that there is a significant difference between etching with hydrofluoric acid and phosphoric acid 40% for 60 s and advised the use of HF for mechanical retention and silane coupling agents for chemical retention [(19, 20)]. The durability of bond values under the stresses of the oral environment is important for clinical predictability of dental materials. Usually, dental materials are subjected to mechanical, thermal and chemical stresses in the mouth during functions. Thermocycling and water storage *in vitro* is a common way for testing dental materials [(21)].

The purpose of this study is to compare shear bond strength of composite to feldspathic ceramic using different acids surface treatment, 37%phosphoric acid for 2 mins,5% hydrofluoric acid for 2mins and 1.23% acidulated phosphate fluoride for 10 mins, and to evaluate the effect of thermocycling on the bond strength.

MATERIALS AND METHODS

Sixty Nickel-Chromium metal base plates (9mm diameter ,3 mm depth) were fabricated using lost wax technique, and a 2 mm thick layer of ceramic(VITA 3D Master) fused to metal plates, to ensure even surfaces, specimens were wet grounded with a 320,400 and 600 grit silicon carbide cylinders(Mounted stones, American Dent-All Inc, Glendale CA). All specimens were embedded in acrylic resin blocks except their examind surfaces and divided into 3 main groups 20 specimens each, **Grp A**: the surface of the ceramic treated with 37% phosphoric acid(PA) for 2 mins, **Grp B**: the ceramic surface treated with 5% hydrofluoric acid(HF) for 2 mins[9], **Grp C**: the ceramic surface treated with 1.23% acidulated phosphate fluoride(APF) for 10 mins[18], all specimens were rinsed with distilled water for 20s, ultrasonication in water bath for 2 mins and air dried for 30s[18]. For all specimens, following the manufacturer's directions one drop of **Monobond- plus** dispensed on a plastic dish, with the aid of disposable brush the solution applied to ceramic surface, allowed to set for 60s. Subsequently a thin layer of **Heliobond** applied homogenously with the aid of disposable brush, it serves as a bonding agent between the saline and the resin composite, the access material dispersed with oil free air and light cured for 10s. Resin composite **Tetric EvoCeram** (Ivoclar/ vivadent-Liechtenstein) applied to each specimen according to manufacturer's instruction with the use of a plastic transparent split mold (5 mm diameter and 4 mm height) especially designed for this purpose. The composite resin introduced with plastic instrument into the mold and adapted to avoid air entrapment, the material carefully positioned over the ceramic surface and light cured for 20s from each side using **Radii plus LED curing light** (light intensity 1500 mW/cm^2, curing depth 6mm, SDI). All specimens stored in 37C distilled water (DW) for 12 weeks[22], 10 specimens from each group subjected to thermocycling between 5C and 55 C for 800 cycles with 30s dwell time (thermocycling device Alqaisi,Iraq) . Finally we got 6 groups:

GA1: 10 specimens, ceramic surface treated with 37% PA 2mins, stored in distilled water, no thermocycling.
GA2: the same as Grp A1 with thermocycling.
GB1: 10 specimens, ceramic surface treated with 5% HF 2 mins, stored in DW, no thermocycling.
GB2: the same as Grp B1 with thermocycling.
GC1: 10 specimens, ceramic surface treated with 1.23% APF 10mins, stored in DW, no thermocycling.
GC2: the same as Grp C1with thermocycling.

All specimens subjected to a shear load with a universal testing machine (instron 1122, England) with 0.5mm/min cross head speed, a chisel apparatus used to direct a parallel shearing force as close as possible to the composite/ceramic interface, shear load in Newton at the point of failure noted, and calculated in mega Pascal's. Fracture sites examined using stereomicroscope to determine the location and type of failure during debonding[23]. Mode of failure recorded as adhesive (failure at the ceramic-resin interface), cohesive (failure within the ceramic or the composite), or combination (areas of adhesive and cohesive failure). One-way analysis of variance (ANOVA) was performed to test any statistically significant difference among the test groups. Comparison between subgroups before and after thermocycling was performed by the least significant difference (LSD) test.

RESULTS

Mean shear bond strength values, minimum, maximum and standard deviation values of the tested groups are shown in table 2. Tables 3 and 5, showed that ANOVA test's results revealed highly significant differences among non-thermocycled and among thermocycled groups respectively, further analysis using LSD tests was performed as shown in tables(4 and 6) and the results revealed that for both non-thermocycled and thermocycled groups, specimens treated with 5% hydrofluoric acid showed significantly higher values than those treated with 37% phosphoric acid or 1.23% acidulated phosphate fluoride and specimens treated with 1.23%APF showed significantly higher values than those treated with 37% PA. For each acid group, t- test was performed between non-thermocycled and thermocycled subgroups, and the results exhibited that thermocycling had significantly reduced the shear strength values as shown in table 7. Stereomicroscope examination showed that 70% of specimens treated with 5%HF exhibited cohesive failure with in ceramic, 70% of specimens treated with 1.23% APF showed (adhesive/cohesive)failure and 50% of specimens treated with 37% PA exhibited adhesive failure as shown in table 8.

Table 1: Characteristics, composition and manufacturers of the material selected in the study

Materials	Characteristics and composition	LotNo.	Manufacturer
Casting alloy	(Ni 61%, Cr 25.05%, Mo 12.35%, Si 1.80%, Mn 0.03%, C 0.01%)	H09-16	Eisenbacher Dentalwaren ED GmbH-GERMANY
Feldspathic Ceramic VITA 3D MASTER		BVMKSET3D	V ITA Zahnfabrik-Germany
Hydrofluoric acid 5%		90290	India
Phosphoric acid gel 37%		N47997	Ivoclar/ vivadent-Liechtenstein-Germany
Acidulated phosphate fluoride gel 1.23%		24-0867	DEEPAK-USA
Tetric EvoCeram	Light cured nano-hybrid composite, the monomer matrix is composed of dimethacrylates(17-18% weight), the fillers contain barium glass,ytterbium trifuoride, mixed oxide and prepolymers (82-83% W), the particles size between 40nm and 3000 nm with a m ean particle size of 550nm;additional content: additives, catalysts, stabilizers and pigments<1% W.	N36909	Ivoclar/ vivadent-Liechtenstein-Germany
Monobond plus	Saline coupling agent, alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate.	N51095	Ivoclar/vivadent Germany
Heliobond	Light curing bonding agent contains Bis-GMA and triethylene glycol dimethacrylate(99 wt. %), catalysts and stabilizers<1%.	N44963	Ivoclar/vivadent Germany
Radii plus LED curing light	Light intensity 1500 mW/cm^2, curing depth 6mm.		SDI

Table 2: Descriptive Statistics

	N	Minimum	Maximum	Mean	Std. Deviation
a1	10	10.53	12.52	11.659	.68593
a2	10	10.08	11.81	10.8810	.58112
b1	10	17.26	18.43	17.9390	.41924
b2	10	16.90	17.92	17.4290	.35275
c1	10	14.15	16.17	15.1770	.61601
c2	10	13.84	15.22	14.5110	.48425

Table 3: Analysis of variance (ANOVA) test for the effect of acid type on shear strength without thermocycling. (a1,b1,c1)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	198.155	2	99.077	288.523	.000
Within Groups	9.272	27	.343		
Total	207.426	29			

Table 4: Least significant difference (LSD) to compare shear strength values among groups (a1, b1, c1)

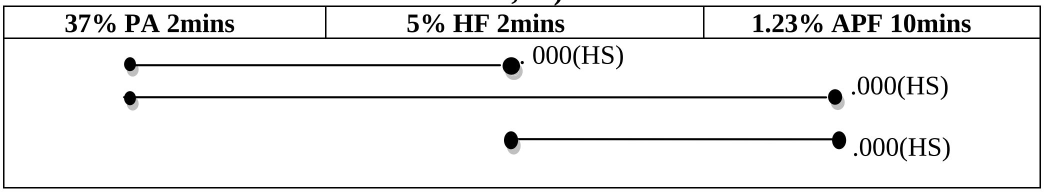

Table 5: Analysis of variance (ANOVA) test for the effect of acid type on shear strength with thermocycling (a2, b2, c2)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	215.226	2	107.613	463.431	.000
Within Groups	6.270	27	.232		
Total	221.496	29			

Table 6: Least significant difference (LSD) to compare shear strength values among groups (a2, b2, c2)

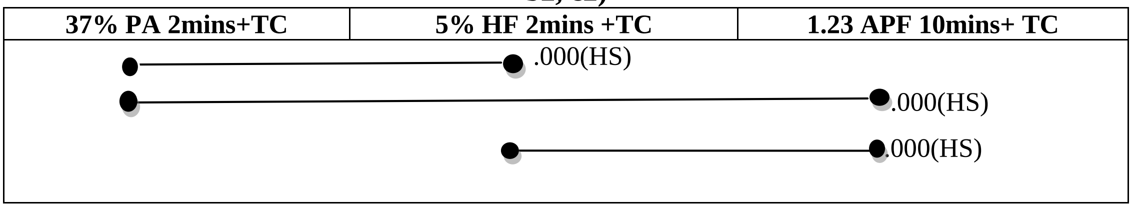

Table 7: T- test for the effect of thermocycling for each acid group

Groups	t-test	p-value	Sig.
A1&A2	2.737	.014	S
B1&B2	2.944	.009	HS
C1&C2	2.686	.016	S

Table 8: Failure modes of the tested groups

Groups	Adhesive	Cohesive with in composite	Cohesive with in ceramic	Adhesive/Cohesive
A1	50%	-------		50%
A2	30%	-------		70%
B1	-------	-------	70%	30%
B2	-------	-------	70%	30%
C1	10%	-------	30%	60%
C2	10%	-------	20%	70%

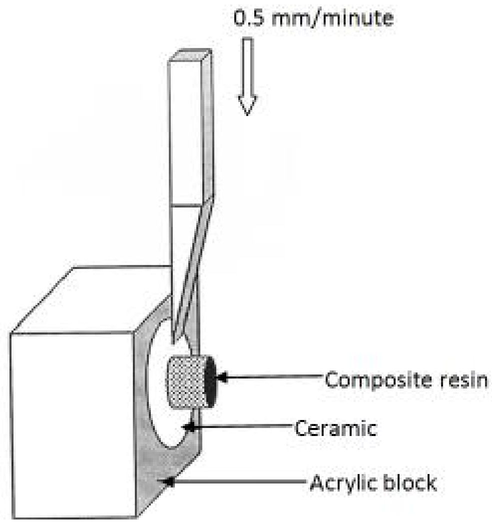

Figure 1: Shear bond strength testing

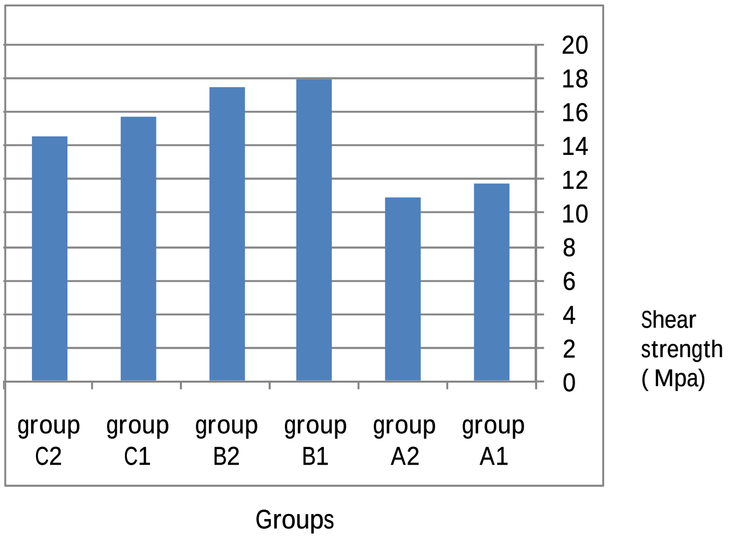

Figure 2: Bar chart shows the differences in mean shear bond strength values (Mpa) among groups.

Figure 3: Cohesive failure within ceramic. Figure 4: Combination failure (adhesive/ cohesive).

Figure 5. Adhesive failure

DISCUSSION

Intraoral repair of fractured porcelain restorations with composite resins presents a substantial challenge for dentists. Multipurpose adhesive systems involve several treatment steps and agents were employed. In this study, LSD test results (table 4 and 6) showed that HF acid group produced statistically highly significant difference compared to APF and PA groups ; this was in agreement with other studies which showed that chemical etching with HF acid dissolves the glassy matrix selectively , the acidic ions of hydrofluoric acid penetrates into the Si-O framework creating ten thousands microporosities/mm^2 in a honey comb appearance to facilitate adhesion of composite resin to the porous surface of ceramic [24, 25]. Etching with APF even with prolonged time results in very shallow etching patterns when compared to HF etching for much shorter time periods [26, 27]. Regarding phosphoric acid it has a minimal effect on the ceramic surface and the bond strength mainly came from the effect of silane coupling agent which has the potential to react with the hydroxyl (-OH) groups present on the surface of porcelain via hydrogen bonding and then through a condensation polymerization (loss of water) reaction [28,29]. In general the reduced shear bond strength values compared to other study done by (AL-Taie L.A., Mohammed S.A. 2010) [9] could be attributed to prolonged water storage and thermo cycling , the hydrolytic degradation happens mainly because of accumulation of water between the filler-matrix that promotes the displacement of inorganic particles or due to the development of superficial flaws related to pre-existent corrosive processes, it is believed that water sorption causes resin softening by swelling of the polymer network and decreasing of the frictional forces between the polymeric chains [22,30,31], exposing the specimens to thermocycling speeds up the diffusion of water in between the composite resin or ceramic, changing the temperature creates stress at the interface of the two materials because of different coefficients of thermal expansion accelerating their structural weakness, promoting union flaws, water storage and thermocycling are detrimental to the silane-ceramic bond as well [32, 33], another factor that might have contributed to the decrease in adhesive resistance values was the sample dimension that had a small area, receiving larger influence of thermal cycling effects on its surface [34]. With the result of this study, it can be concluded that surface treatment of felspathic ceramic with hydrofluoric acid gives the best repair bond strength, a second choice but inferior bond strength is the treatment with acidulated phosphate fluoride for a longer time. Prolonged water storage time has an obvious effect in reducing the bond strength in relation to other studies; subgroups submitted to thermocycling have a significant decrease in bond strength in relation to other subgroups that have not been submitted to thermocycling; other studies are required to evaluate the effect of different surface treatment on other types of ceramics.

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