

Shear Bond Strength of Three Porcelain Repair Systems on Porcelain in Dry and Aged Conditions

Gülce Subaşı* and Özgür İnan

Department of Prosthodontics, Faculty of Dentistry, Selcuk University, Konya, Turkey

Abstract

Objective: This study compared the durability of bond strength of a resin composite to feldspathic ceramic by using three porcelain repair systems.

Methods: Sixty ceramic blocks (Vitablocks Mark II) were divided into three groups, according to the repair method (CO [Cojet repair kit], CL [Clearfil repair kit], and UL [Ultradent repair kit]). Composite resin was photo-polymerized on each ceramic. Half of the specimens were submitted to the shear bond strength test after 24 hours of water storage or thermal cycling. The bond strength data was analyzed by Weibull analysis and Wald tests ($p=0.05$).

Results: The characteristic bond strength values (σ_0) of repair systems in dry conditions were 5.823, 6.512 and 6.867 MPa and after aging conditions these were 4.112, 3.935 and 4.210 MPa for CO, CL and UL, respectively. Wald test results revealed that there were significant differences among groups in characteristic bond strength (σ_0) ($p<0.001$).

Conclusion: Thermal cycling had a significant effect on the bond strength of three repair kits and bond strength results decreased after thermal cycling.

Keywords: Bond strength; Porcelain repair systems; Thermal cycling

Introduction

Increased patient demand for aesthetic and metal-free restorations has resulted in the progress of all-ceramic restorations. All-ceramic restorations can be created in many ways using different materials. Computer-aided design and manufacturing (CAD/CAM) systems have dramatically enhanced dentistry by providing high-quality restorations [1].

Vita Mark II (VITA Zahnfabrik, Bad Sackingen, Germany) is a machinable feldspathic porcelain that was introduced in 1991 for the CEREC 1 (Siemens AG, Bensheim, Germany) system [2]. It can be used in inlays, onlays, veneers, and anterior crowns [1,2]. Bindl and Mörmann [3] reported that clinically bonded Mark II CAD/CAM-generated partial crowns had shown excellent results up to 5 years after placement. They [4] also evaluated the survival analysis of CAD/CAM-generated monoceramic Mark II crowns for fracture over a period of 2–5 years. The cumulative survival rate regarding fracture of the crown was 94.4% for Mark II monoceramic crowns. Although these restorations demonstrate aesthetic results, they are susceptible to fractures. Previous clinical studies on glass-ceramic inlays, or laminates [5,6], reported chippings of the veneering ceramics. Since such ceramics are cemented adhesively, their removal for indirect repairs without creating any damage either to tooth structure or to the restoration itself is difficult. In addition, cost of the treatment can prevent renewal of the restoration. Therefore, depending on the size of the fracture in the veneering material, intraoral repair methods using resin composites and adequate surface treatments may be indicated [7,8].

The clinical success of the porcelain repair systems depends on the integrity of the bond between porcelain and resin composite. The bond is achieved either by chemical or mechanical methods. Due to insufficient bonding characteristics of the chemical agents, the porcelain surfaces must be physically altered when these agents are used.

Various methods have been reported to repair fractured porcelain with composite. These methods include sandblasting with aluminum

oxide (Al_2O_3) particles, roughening with a diamond bur, etching porcelain surfaces with hydrofluoric acid or acidulated phosphate fluoride gel, and a tribochemically silica-coating system. These methods create micro-mechanical retention on the porcelain surfaces [7-9]. Chemical retention can be provided by using silane-coupling agents [10,11]. Silane-coupling agents can be used in combination with the previous surface alteration methods [7,9,12,13]. Silane is a dual functional monomer consisting of a silanol group that reacts with the ceramic surface and contains a methacrylate group that co-polymerizes with the resin matrix of the composite. The specific silane typically used in dentistry for both intraoral repair and treatment of ceramic restorations before placement is 3-methacryloxypropyltrimethoxysilane (γ -MPS) [10,11].

The purpose of this in vitro study was to compare the shear bond strength of composite resin that had been bonded to a feldspathic porcelain in dry and aged conditions by using three commercially different porcelain repair systems. The tested null hypothesis was that the repair bond strength would decrease after aging conditions.

Materials and Methods

Specimen preparation

The materials used in this study are presented in Table 1. Sixty blocks of feldspathic ceramic (Vita Blocs Mark II) (12 mm x 10 mm x 2 mm) were prepared using a slow-speed diamond saw (Isomet 1000

*Corresponding author: Gülce Subaşı, Department of Prosthodontics, Faculty of Dentistry, Selcuk University, Konya, Turkey, Tel: +903322231186; Fax: +903322410062; E-mail: gulce2subasi@yahoo.co.uk

Received October 26, 2011; Accepted January 30, 2012; Published February 02, 2012

Citation: Subaşı G, İnan Ö (2012) Shear Bond Strength of Three Porcelain Repair Systems on Porcelain in Dry and Aged Conditions. Dentistry 2:119. doi:10.4172/2161-1122.1000119

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Precision Saw; Buehler Ltd, Lake Bluff, Illinois, USA) under water-cooling. The ceramic surfaces were polished with 1000-grit silicon carbide paper under water-cooling and then ultrasonically cleaned (BioSonic JR; Whaledent, New Jersey, USA) in distilled water for 5 minutes. Ceramic specimens were embedded in self-cure acrylic resin (Meliodent; Heraeus Kulzer, Hanau, Germany), leaving one surface of the porcelain uncovered. Samples were then randomly divided into 3 groups (n = 20), depending on the repair method to be employed. In all groups, repair methods were constructed according to the manufacturer's instructions.

Repair methods

Cojet Repair Kit (CO): Ceramic surfaces were air-abraded using an intraoral device (Cojet) that was filled with 30-µm alumina particles coated with silica (Cojet Sand) from a distance of approximately 10 mm and at a pressure of 3 bar for 15 seconds. Following surface conditioning, the sand particle remnants were gently air blown. The conditioned surfaces were then coated with γ-MPS silane (Espe Sil), rested for 5 minutes, and air-dried. Then, an adhesive resin (Visio Bond) was applied using a microbrush, and was air thinned and photopolymerized for 20 seconds.

Clearfil Repair (CL): The ceramic surfaces were air-abraded with the same device (Cojet) that was filled with 50-µm Al₂O₃ particles (Korox, Bego, Bremen, Germany) from a distance of approximately 10 mm at a pressure of 3 bar for 15 seconds. Forty percent phosphoric acid (H₃PO₄) (K-Etchant Gel) was applied to the surface for 5 seconds, rinsed, and dried thoroughly. Clearfil SE Bond Primer and Clearfil

Porcelain Bond Activator were mixed at a 1:1 ratio, waited for 5 seconds, and then applied on the ceramic surface for 5 seconds and air thinned.

Ultradent (UL): The same porcelain-conditioning procedure as the CL group was applied to this group. Thereafter, the porcelain surfaces were etched with 9% buffered hydrofluoric acid (HF) (Ultradent Porcelain Etch) for 1 minute. The acid neutralizer (EtchArrest) was blended into the etchant until the yellow color of the etchant was no longer seen. They were then rinsed with water and dried with air. A silane agent (Ultradent Silane) was applied to the ceramic surface for 60 seconds, evaporated for 1 minute, and dried with air. Thereafter, a puddle coat of bonding resin (PQ1 resin) was firmly scrubbed into the silanated porcelain surface. Resin was thinned with light application of dry air to the point of losing milky appearance, then light cured for 20 seconds.

In all groups, composite resin (Filtek Z250) was used for repairing the porcelain surfaces. Composite resin was packed with a hand instrument to the treated porcelain surfaces by using a special mould, which had a radius of 5mm and a height of 2 mm. Composite resin was light polymerized for 40 seconds. A bluephase light-curing unit (Ivoclar; Vivadent, Schaan, Liechstein) was used at a distance of 0mm with an intensity of 450 mW/cm². The ceramic-composite repaired-specimens groups were then randomly divided into two subgroups according to the storage conditions.

Storage conditions

Non-thermal cycling (NTC): In this group, the specimens were

Brand	Manufacturer	Chemical composition	Batch Number
VITABLOCS Mark II for CEREC/inLab (2M1C 112)	VITA Zahnfabrik, Bad Säckingen, Germany	Mixture of feldspathic crystalline particles embedded in a glassy matrix Vol % ≈30	15670
Cojet Repair Kit (CO)			
CoJet Sand	3M ESPE AG, Seefeld, Germany	Aluminum trioxide particles coated with silica, particles size 30µm.	359747
ESPE-Sil	3M ESPE AG, Seefeld, Germany	3-MPS silane (3-methacryloyloxypropyltrimethoxy silane) , ethanol	381242
Visio Bond	3M ESPE AG, Seefeld, Germany	Bisacrylate, aminodiol methacrylate, camphor quinone, benzyl dimethyl ketale, stabilizers	302239
Clearfill Repair Kit (CL)			
K-Etchant Gel	Kuraray Medical Inc, Okayama, Japan	40% H ₃ PO ₄	00462B
Clearfill SE Bond Primer	Kuraray Medical Inc, Okayama, Japan	HEMA, water, hydrophilic dimethacrylate, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), camphorquinone, tertiary amine	00942A
Clearfill Porcelain Bond Activator	Kuraray Medical Inc, Okayama, Japan	BisphenolA polyethoxydimethacrylate, 3-methacryloyloxypropyltrimethoxy silane.	00241A
Ultradent Repair Kit (UL)			
Ultradent Porcelain Etch	Ultradent Products Inc, USA	%9 buffered HF acid	C101
EtchArrest	Ultradent Products Inc, USA	Calcium carbonate %4.5, sodium bicarbonate % ~10	W051
Ultradent Silane	Ultradent Products Inc, USA	Methacryloyloxy propyl trimethoxy silane %15, isopropyl alcohol %92	0091
PQ1 Single syringe bonding agent	Ultradent Products Inc, USA	Light cured bonding agent with ethyl alcohol solvent carrier. PQ1 is 40% filled and contains FluorUtite™, nature's high fluoride sustained release mineral.	X091
Filtek Z250 (shade C2)	3M ESPE, St Paul, Minn., U.S.A.	Bisphenol A diglycidylmethacrylate (BIS-GMA), urethane dimethacrylate (UDMA) and ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA) resins	7WA

Table 1: Materials used in this study.

submitted to the shear test after being stored in distilled water at 37°C for 24 hours.

Thermal cycling (TC): The specimens were stored in distilled water at 37°C for 24 hours and then submitted to the thermal cycling (5000 cycles, between 5°C and 55°C, with a dwell time of 20 seconds at each temperature, and a transfer time from one bath to the others of 10 seconds) (Custom-made thermal cycling machine, Nova Ticaret, Konya, Turkey).

Thus, considering the repair methods (3 groups) and storage conditions (2 groups), 6 testing groups were obtained, yielding 10 specimens in each group (n = 10).

Shear bond strength test

Shear testing of all groups was performed on a universal testing machine (TSTM 02500; Elista Ltd., Istanbul, Turkey) at a crosshead speed of 0.5 mm/min. A knife-edge blade apparatus was used to direct a parallel shearing force as close as possible to the interface of the ceramic and the composite cylinder. The shear debonding forces were recorded in N. The failure loads (N) were divided by the bonding areas (mm²), and then the shear debonding forces were converted into MPa.

Scanning electron microscopy (SEM) and failure type analysis

The effects of the surface treatments and different etchants on the feldspathic porcelain surfaces were examined in a SEM (LEO 440; Zeiss, Cambridge, UK) at 1.50 KX magnification.

After shear-bond testing, fracture surfaces of all specimens were analyzed both visually and by a stereomicroscope (Leica MZ16; Leica Microsystems, Switzerland) at 10X magnification. Failure types were categorized as adhesive between ceramic and composite (ADHES), and cohesive failure (COHES) and cohesive failure of the ceramic accompanied by adhesive failure at the interface (MIX). For failure type analysis, a specimen that demonstrated a mixed failure type was further selected for SEM at 45X magnification.

Statistical analysis

Due to the strength data of ceramic materials, which usually shows an asymmetrical distribution, the variability of the shear strength

values was analyzed by using two-parameter cumulative Weibull distribution functions. The description of Weibull distribution is given in the formula [14]:

$$P(\sigma) = 1 - \exp[-(\sigma/\sigma_0)^m],$$

where P is the probability of failure, r is the shear strength at a given P, σ_0 is the characteristic Weibull parameter at the fracture probability of 63.2%, and m is the Weibull modulus.

The maximum likelihood method was preferable to least squares method for estimating the Weibull parameters. Wald tests and 95% confidence intervals were used for comparisons of the scales and the location parameters of Weibull distribution. Also, the Wald test is performed for equal shapes or scales. All results were evaluated according to the 0.05 significance level.

For failure type analysis, due to the fact that the expected number of observations in some cells was less than 5, chi-square analysis was not applied.

Results

Shear bond strength results

The Weibull analysis results were presented in Table 2 and Figure 1. Table 2 showed the Weibull analysis and Wald test results for comparisons of six groups, which included the Weibull modulus (m), characteristic strength (σ_0), standard errors of m and σ_0 , 95% confidence intervals of both m and σ_0 , and the other σ_0 parameter. Figure 1 showed the fracture probability of the bond between the ceramic material and resin composite with different shear stress levels.

The characteristic strength (σ_0) values (MPa) and 95% confidence intervals (MPa) of repair groups in dry and aged conditions were as follows: CO-NTC (5.823) (5.342–6.347); CO-TC (4.112) (3.810–4.438); CL-NTC (6.512) (6.020–7.043); CL-TC (3.935) (3.576–4.330); UL-NTC (6.867) (6.151–7.667); UL-TC (4.210) (3.910–4.532).

Table 2 showed the Weibull statistics and Wald test results of 6 groups. According to the Wald tests, there were no significant differences among groups with related m parameter (P = 0.871). However, there were significant differences among groups with related

MATERIAL	Shape (m)	Std. Error	95% CI (MPa)	Scale (σ_0) (MPa)	Std. Error	95%CI (MPa)	$\sigma_{0.05}$	Mean	SD
CO-NTC	7.651	1.757	4.878,12.001	5.823 (b)	0.256	5.342,6.347	3.949	5.498	0.748
CO-TC	8.579	2.154	5.244,14.036	4.112 (a)	0.160	3.810,4.438	2.908	3.884	0.548
CL-NTC	8.338	2.022	5.183,13.412	6.512 (b)	0.260	6.020,7.043	4.653	6.150	0.888
CL-TC	6.821	1.786	4.083,11.396	3.935 (a)	0.192	3.576,4.330	2.196	3.664	0.688
UL-NTC	5.957	1.480	3.660,9.696	6.867 (b)	0.385	6.151,7.667	4.170	6.365	1.258
UL-TC	8.808	2.262	5.323,14.574	4.210 (a)	0.158	3.910,4.532	3.004	3.975	0.607
Chi-square (Df=5)	1.838			157.525					
p-value	0.871			<0.001					

*Same superscript letters in columns show no differences.

Table 2: Results of Weibull analysis, Wald tests and mean shear bond strength results (MPa).

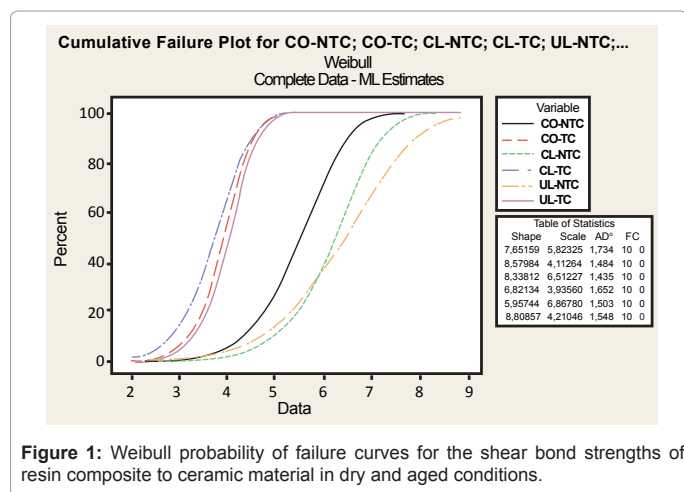


Figure 1: Weibull probability of failure curves for the shear bond strengths of resin composite to ceramic material in dry and aged conditions.

shear-bond strength data (σ_0) ($p < 0.001$) (Table 2). According to the 95% confidence interval results, thermal cycling significantly affected the bond strength results. After the aging condition, bond strength results decreased with respect to the non-thermal cycling groups. In dry and aged conditions, there were no statistically significant differences among CO, CL, and UL shear bond strength (σ_0). However, in each condition, UL revealed the higher bond-strength values.

The mean and standard deviation of shear bond strength values of composite bonded to the ceramic are also shown in Table 2. The mean shear bond strength in MPa ranking from highest to lowest were as follows: UL-NTC (6.365 ± 1.258); CL-NTC (6.15 ± 0.888); CO-NTC (5.498 ± 0.748); UL-TC (3.975 ± 0.607); CO-TC (3.884 ± 0.548); CL-TC (3.664 ± 0.688).

Topographic analysis

The effects of the surface treatments on Cerec porcelain were demonstrated in Figure 2. Figure 2A showed the effect of airborne particle abrasion with 50- μm Al_2O_3 particles, and Figure 2B showed the silica coating with 30- μm SiO_2 particles. Air abrasion of feldspathic ceramic surface produced a rougher surface than the silica-coated surface. The sand particle remnants could be seen in each figure. In addition, the effects of different etchants on the airborne-abraded feldspathic porcelain were shown in Figure 3. Figure 3A showed the effects of 40% H_3PO_4 , and Figure 3B showed the 9% buffered HF acid on the porcelain surface. In both Figures 3A and 3B, honeycomb-like surfaces could be seen. However, 9% buffered HF acid seemed more effective than the 40% H_3PO_4 on the ceramic surface.

Failure types

Table 3 showed the failure types of ceramic repair materials in dry and aged conditions. In dry conditions, specimens treated with CO mainly demonstrated a mix failure type (80%). In the CL group, mainly cohesive failure (70%) was observed, and in the UL group, cohesive (50%) and mix (50%) failures were observed. When observations were evaluated within failure type, visual and stereomicroscopic analysis demonstrated almost all mix and cohesive failures in dry conditions after all repair methods.

After aging conditions, all repair methods predominantly demonstrated a mixed failure type (90%, 80%, and 70% for CO, CL, and UL, respectively). When evaluated within failure type, mix failures (80%) were seen, too.

Discussion

Porcelain can be repaired using porcelain repair systems without removal of the prosthesis. A direct repair option may be practical for

			FAILURE TYPE				
			ADHESIVE	COHESIVE	MIX	Total	
NTC	MATERIAL	CO	Count	0	2	8	10
		% within MATERIAL		,0%	20,0%	80,0%	100,0%
			% within FAILURETYPE	,0%	14,3%	53,3%	33,3%
	CL	Count	1	7	2	10	
		% within MATERIAL	10,0%	70%	20%	100,0%	
			% within FAILURETYPE	100%	50%	13,3%	33,3%
UL	Count	0	5	5	10		
	% within MATERIAL	,0%	50%	50%	100,0%		
		% within FAILURETYPE	,0%	35,7%	33,3%	33,3%	
Total	MATERIAL	Count	1	14	15	30	
		% within MATERIAL	3,3%	46,7%	50%	100%	
			% within FAILURETYPE	100%	100%	100%	100%
	TC	MATERIAL	CO	Count	1	0	9
% within MATERIAL			10,0%	,0%	90,0%	100,0%	
			% within FAILURETYPE	100%	,0%	37,5%	33,3%
CL		Count	0	2	8	10	
		% within MATERIAL	,0%	20%	80,0%	100,0%	
			% within FAILURETYPE	,0%	40,0%	33,3%	33,3%
UL	Count	0	3	7	10		
	% within MATERIAL	,0%	30,0%	70,0%	100,0%		
		% within FAILURETYPE	,0%	60,0%	29,2%	33,3%	
Total	MATERIAL	Count	1	5	24	30	
		% within MATERIAL	3,3%	16,7%	80,0%	100,0%	
			% within FAILURETYPE	100,0%	100,0%	100,0%	100,0%

Table 3: Failure types of ceramic repair kits in dry and aged conditions.

both the clinicians and the patients as an interim solution. This *in vitro* investigation compared the shear bond strengths of three different porcelain repair systems in dry and aged conditions. In the present study, three repair kits were evaluated according to the manufacturer's instructions.

Some authors [15-17] reported that a combination of airborne particle abrasion with Al_2O_3 and etching with HF acid yielded higher bond strength values to silica-based ceramics (feldspathic, Empress I, lithium disilicate-based ceramics), and others [18,19] reported that high-strength ceramics (alumina, zirconia ceramics) treated with a silica-coating technique yielded the highest bond strength values to a composite resin. In this study feldspathic porcelain was used for repairing. The systems used in this study relied on chemical adhesion (silane-coupling agents) as well as on mechanical retention (roughened surfaces). Tribochemical silica coating (in CO group) and air-abrasion methods (in CL and UL groups) were used to obtain mechanical retention on porcelain surfaces prior to porcelain repairing. SEM examinations of different pretreatments revealed that airborne-abraded porcelain surfaces were better than silica-coated surfaces (Figures 2A and 2B).

It is known that airborne particle abrasion and HF selectively

dissolve the weaker glass phase and create retentive surfaces. The porous irregular surface facilitates the penetration of resin into the microretentions of the treated ceramic surfaces [12]. In our study, both CL and UL repair kits were based on acid applications. While CL repair was based on 40% H_3PO_4 acid, UL repair was based on 9% buffered HF acid. In addition, SEM photomicrographs of different etchants on Cerec porcelain revealed that 9%-buffered HF acid demonstrated more microretentive surfaces than the 40% H_3PO_4 acid sample (Figures 3A and 3B). However, the application times of these acids were different from each other. In this study, although the surface roughness of the substrate after the roughening methods was not evaluated, it could be anticipated that airborne particle abrasion with HF acid application in the UL repair group better served the bond-strength results. In our study, this fact could be interpreted by evaluating the SEM micrographs and bond-strength data (Figure 3 and Table 2).

The distribution of data obtained from the strength test related to porcelains is not normal [20,21]. Because ceramics and dental composites exhibit a brittle fracture mode, expressing the mechanical behavior of a brittle material as a single mean value may not sufficient to indicate the true strength and reliability of the bond strength. The Weibull analysis of bond strength data, which related the probability

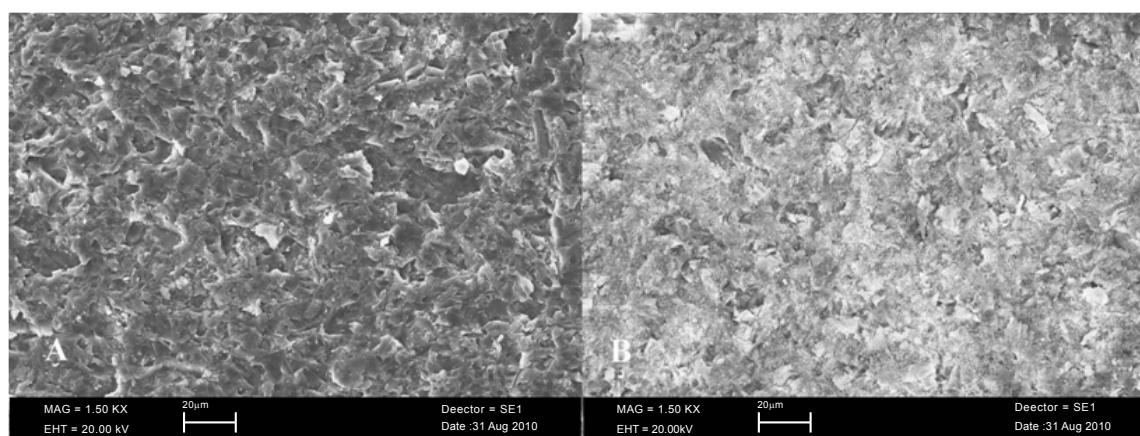


Figure 2: SEM micrographs of conditioned ceramic surfaces. (A) Airborne particle abrasion with 50µm Al_2O_3 particles. (B) Silica coating with 30µm SiO_2 particles (original magnification 1.50 KX).

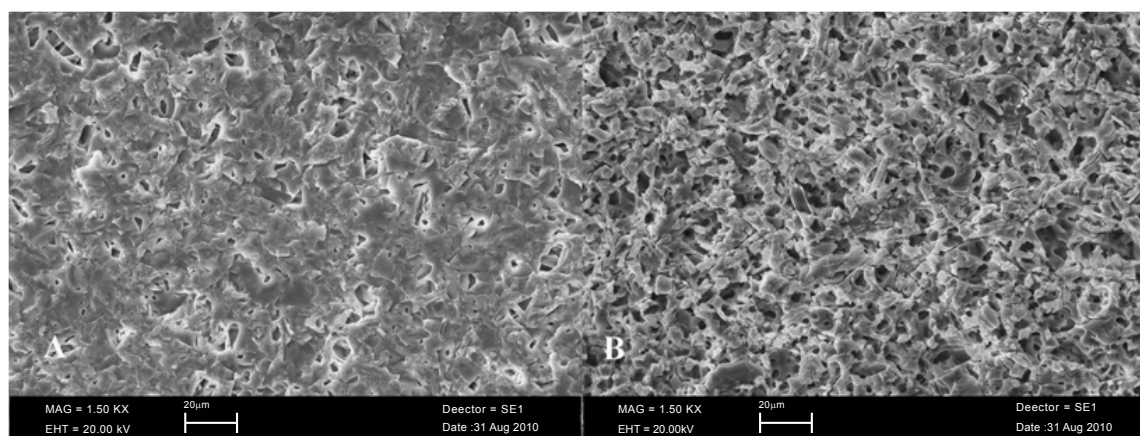


Figure 3: SEM micrographs of different etchants on 50µm airborne particle abraded feldspathic porcelain surface. (A) Etching with 40% H_3PO_4 acid. (B) Etching with 9% buffered HF acid (original magnification 1.50 KX).

of failure at a given stress level, is a more powerful method than using only the mean value [22]. In addition, the failure probability of materials at any stress level could also be predicted. Due to these reasons, Weibull analysis was used for the statistical analysis to evaluate the bond-strength data.

In dry conditions, according to Wald tests and 95% confidence intervals results, there were no significant differences among the three repair kits' characteristic bond strength values (σ_0). However, UL seemed to reveal the highest characteristic bond-strength value.

Van der Vyver et al. [23] compared the shear-bond strength of five porcelain repair systems on Cerec porcelain. They found the best results with the Ultradent repair kit. Similarly, in our study, Ultradent seemed to reveal the highest characteristic bond-strength (σ_0) data in dry and aged conditions.

Thermocycling and water storage in vitro is a common method of testing dental materials to establish their suitability for in vivo use [24,25]. It has been proven that water, owing to its small molecular size and high molar concentration, can penetrate into nanometer-size free-volume spaces between polymer chains that are capable of hydrogen bonding, resulting in a decrease in thermal stability [26]. In this study, the decrease in bond strength for all repair systems from dry to aged conditions could be explained by the water uptake between the interlocking areas. Similar to these studies [24,25], in our study, according to Wald tests and 95% confidence intervals, characteristic bond strength (σ_0) significantly decreased after thermal cycling ($P < 0.001$) (Table 2). Therefore, these results verified the hypothesis. When three repair kits were compared with each other in aged conditions, no statistically significant differences were found. However, thought not found to be statistically significant, Ultradent repair seemed to have the highest characteristic bond-strength (σ_0) value.

The quality of the bond should not be evaluated based on bond-strength data alone. The mode of failure could provide important information about the bond-strength results. In dry conditions, almost all mix and cohesive failures were seen. However, for all three repair systems, mixed failure types increased after aging conditions. This might be due to the chemical bond between the ceramic and composite system and the effect of the aging condition. These findings were consistent with previous studies [16,24].

Many methods of measuring the in vitro bond strength of porcelain repair systems have been described. These include shear [27-29], microtensile [24,25], and microshear [16] bond-strength tests. One of the most commonly employed is the shear bond-strength test [30]. Although it was reported that this test could create non-uniform stress distribution at the bonding interface, this test is often used because of simplicity and ease of application [31]. Therefore, this test method was performed in this study.

This study had some limitations. In this study, the application time and concentrations of acids were different. However, this study was conducted according to the manufacturer's instructions. In further studies, these factors should be controlled. In addition, thermal cycling was used as an aging procedure. To better simulate a clinical environment, thermal cycling should be combined with cyclic fatigue. The *in vivo* bonding ability of these systems should also be tested.

The findings of this study should be carefully considered for clinical

application, and bond-strength durability of these repair systems should also be evaluated after long-term aging procedures. In addition, though not found statistically significant, combining acid etching and silane application is an appropriate selection for feldspathic porcelain repairing. However, further investigations are required to know which conditioning methods play the crucial role in long-term adhesion of porcelain-composite bonding.

Within the limitations of this study, the following conclusions were drawn:

1. Thermal cycling had a significant effect on the bond strength of three repair kits. Characteristic shear-bond strengths (σ_0) of three repair kits decreased after thermal cycling.
2. When bond-strength results and failure types were evaluated together, each of three systems could be recommended. However, though not found statistically significant, the UL repair kit seemed to show a higher characteristic shear-bond strength (σ_0) value than the other repair kits.

Acknowledgements

The authors thank Assistant Professor Mustafa Semiz (Department of Statistics, Selcuk University) for the statistical analysis and Dr.Ihsan Aksit (Erciyes University Technology Research and Application Center) for the scanning electron microscopy analysis.

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