

Effect of Different Acid Treatments on the Shear Bond Strength of Computer-Aided Design and Manufacturing (CAD-CAM) Ceramics for Dental Applications

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Abstract

The purpose of this in vitro study was to compare the shear bond strengths (SBS) of hybrid ceramic; Vita Enamic (VE), zirconia-reinforced lithium silicate ceramic; Vita Suprinity (VS), feldspathic ceramic; Vita TriLuxe forte (VT), and lithium disilicate ceramic; IPS e.max CAD (IPS). Eighty specimens were prepared from four different CAD-CAM ceramic materials. The CAD-CAM ceramic specimens were randomly divided into two subgroups according to acid types: phosphoric (P) acid and hydrofluoric (HF) acid. Resin cement (Panavia V5; Kuraray, Japan) was applied using a 3 x 3-millimeter mold. All specimens were stored in distilled water at 37 °C for 24 hours. The SBS test was performed using a universal testing machine (0.5 mm/min). Two-way ANOVA was used to detect significant differences among the CAD-CAM ceramic materials and acid types. Subgroup analyses were conducted using Tukey HSD post-hoc test ($p < 0.05$). The lowest SBS value was seen in the VE group (4.5 ± 2.2 MPa) for P acid etching. The highest SBS value was seen in the IPS group (19.5 ± 8.3 MPa) for HF acid etching.

Keywords: Shear bond strength, CAD-CAM ceramics, resin cement.

I. Introduction

Computer-aided design and computer-aided manufacture (CAD-CAM) have been used in dentistry since 1980s. The advent of this technology has put the production of new materials for fixed and removable prosthesis at the top of the agenda. With the development of CAD-CAM ceramic blocks, aesthetic dentistry practices are increasing, and new restoration options are being created¹. The most important advantage of restorations made with CAD-CAM ceramic blocks is that they can be done in one session. In addition, the restorations can be produced in a homogeneous structure as they do not contain any sub-structure material, and cause less erosion in the opposing dentin^{2,3}. Although new blocks continue to be produced everyday, the most frequently used CAD-CAM blocks are feldspathic, composite (hybrid and nano-ceramics), lithium-disilicate-reinforced, leucite-reinforced, and zirconium-reinforced ceramics³⁻⁹.

The success of full ceramic restorations depends on the mechanical strength of the material, as well as the bonding strength of the restoration. For certain types of ceramics, different surface treatment methods are proposed to provide roughness and promote micromechanical retention¹⁰. An increase in restoration-to-tooth connection leads to a reduction in decementation and microleakage³. Differences in SBS values of cements occur due to the different chemical structures of the CAD-CAM ceramic materials. In laminate veneers and endocrown restora-

tions, bonding strength is a key factor¹¹. In previous studies, different surface treatments have been used to show the bond strength of various cements^{8,12-15}. However, new dental materials and cements are being produced everyday.

The purpose of this study was to evaluate the SBS of four different CAD-CAM ceramic blocks when these are etched with different acid agents. The null hypotheses of this study were that (1) the type of material, and (2) the type of acid treatment would not affect the SBS of resin cement to CAD-CAM ceramic blocks.

II. Materials and Methods

In the present study, four different CAD-CAM ceramic materials; VE, VS, VT, and IPS, and two different acid agents [40 % P acid (K-Etchant Gel; Kuraray Noritake Dental, Japan) and 9.5 % HF acid (Porcelain Etchant; Bis-co, Schaumburg, IL, USA)] were tested. Manufacturers and the composition of the materials used in this study are presented in Table 1. The specimens were positioned in a 1.2 x 4-cm polyvinylchloride cylinder and embedded in an acrylic resin (Palapress vario; Heraeus Kulzer, Germany). The specimens were wet-ground on one surface using 600-, 800-, 1000-, and 1200-grit silicon carbide (SiC) paper. The four groups were divided into two subgroups according to type of the etched acid agents ($n = 10$). The first group was etched with P acid for 5 seconds (sec), and the second group was etched with HF acid for 20 sec. Ceramic primer (Clearfil Ceramic Primer Plus; Kuraray, Japan) was applied to the acid treated surface,

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Table 1: Compositions and manufacturers of the materials

Groups	Composition	Manufacturer
VE	14 % polymer, 86 % fine structure feldspar ceramic	VITA Zahnfabrik, H. Rauter GmbH, Bad Säckingen, Germany
VS	ZrO ₂ , SiO ₂ , Li ₂ O	VITA Zahnfabrik, H. Rauter GmbH, Bad Säckingen, Germany
VT	Fine-structure feldspar ceramic SiO ₂ , Al ₂ O ₃ , NaO ₂ , K ₂ O, CaO, TiO ₂	VITA Zahnfabrik, H. Rauter GmbH, Bad Säckingen, Germany
IPS	SiO ₂ , Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅ , and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein
P	40 % phosphoric acid	Kuraray Noritake Dental, Japan
HF	9.5 % hydrofluoric acid	Bisco, Schaumburg, IL, USA
CP	> 80 % ethanol, 10-MDP	Kuraray Noritake Dental, Japan

VE; Vita Enamic, VS; Vita Suprinity, VT; Vita Trilux, and IPS; IPS e.max CAD, P; K-Etchant Gel, HP; Porcelen etchant, CP; Clearfil Ceramic Primer Plus

which was then washed and dried. Resin cement (Panavia V5; Kuraray, Japan) was applied using 3 x 3-mm molds. All specimens were stored in distilled water at 37 °C for 24 h and the SBS test was performed with a computer-supported universal testing device (MTS Criterion® Series 40; USA) and the load was applied at a crosshead speed of 0.5 mm per minute until failure occurred.

In order to see the effect of different acids on the surface of the CAD-CAM ceramic blocks, one specimen from each group was sputter-coated (Bal-Tec SCD 050, Bal-tec AG, Liechtenstein) with a 15-nm layer of gold-palladium (Au-Pd) and imaged using a scanning electron microscope (SEM; LEO Evo 40XVP, Carl Zeiss, Oberkochen, Germany). Images were examined at 20 kV within a magnification range of 100x to 10000x.

Analyses were performed with statistical software (IBM SPSS Statistics v23.0; IBM Corp). The normality of the data distribution was evaluated by the Shapiro-Wilk test. Parameters with normal distribution were analyzed using the two-way ANOVA. To determine differences, the Tukey HSD test was used ($p < 0.05$).

III. Results

Results of the descriptive analyses of the shear bond test are shown in Table 2. In intra-group comparisons, the SBS values of the VE and VS groups with HF acid etching were higher than with the P acid ($p < 0.05$). There were no significant differences between the SBS values of the VT and IPS groups with different acid etching ($p > 0.05$). In the P-acid-etched specimens, the SBS values of the VE group (4.5 ± 2.2 MPa) were significantly lower than the VT (16.9 ± 6.9 MPa) and IPS (16.1 ± 8.8 MPa) groups ($p < 0.05$). In the HF acid group, the SBS values of the VE (11.3 ± 4 MPa) group were significantly lower than the VS (18.3 ± 4.2 MPa) and IPS (19.5 ± 8.3 MPa) groups ($p < 0.05$). Representative SEM micrographs etched with P and HF acid, and VE, VT, VS and IPS surfaces are presented in Figs. 1 (a, b, c, d, e, f, g, and h) respectively. Treatment with HF acid

resulted in increased surface roughness, with irregularities and pores on the treated surfaces of all materials tested.

Table 2: Descriptive statistics and multiple comparison results

Material	Phosphoric acid Mean \pm SD (MPa)	Hydrofluoric acid P* Mean \pm SD (MPa)	P*
Vita Enamic	4.5 ± 2.2^a	11.3 ± 4^a	0.000
Vita Suprinity	9.7 ± 4.4^{ab}	18.3 ± 4.2^b	0.001
Vita Triluxe forte	16.9 ± 6.9^b	15.8 ± 5^{ab}	0.690
IPS e.max CAD	16.1 ± 8.8^b	19.5 ± 8.3^b	0.388

SD: Standard Deviation, MPa: Megapascal. * Two-way anova. Mean values followed by different lowercase letters in the same column show statistical difference ($P < 0.05$)

IV. Discussion

This study was conducted to evaluate the effect of different acid etching agents on the SBS between resin cements and CAD-CAM hybrid ceramic, feldspathic ceramic, zirconia-reinforced ceramic, and lithium disilicate ceramic. The bonding efficacy was evaluated using the SBS test and the effect of etching on the ceramic substrates was evaluated by SEM analysis. The results of the current study revealed that the selected ceramic materials and different acid agents have an influence on the SBS. Therefore, the null hypotheses of this study were rejected.

Adhesion played an important role in the strengthening of all ceramic restorations. Hence, the research on SBS continues to maintain its importance with new production

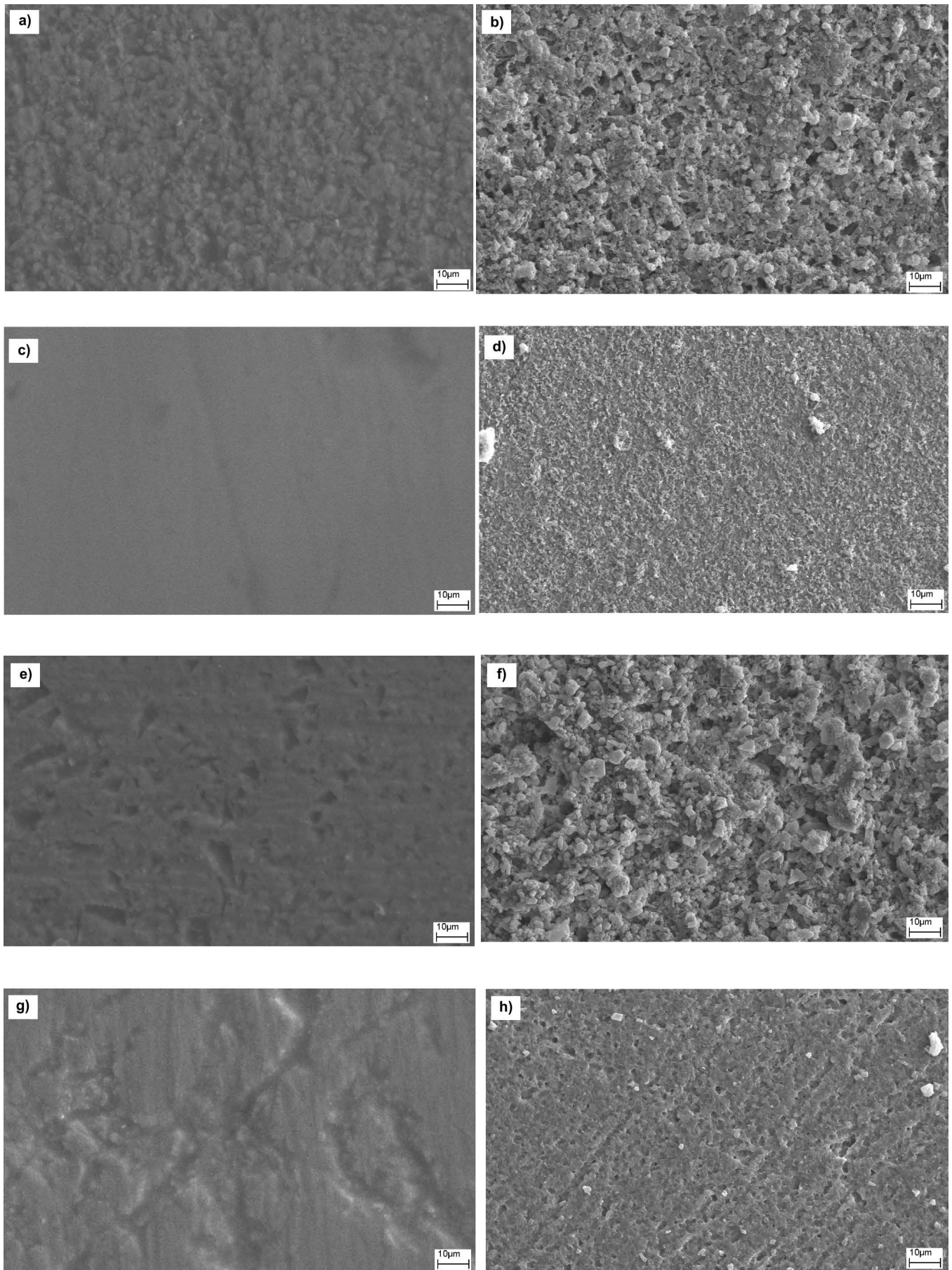


Fig. 1: Representative scanning electron microscopy images, a) VE after 40 % P acid and b) 9.5 % HF acid etching c) VS after 40 % P acid and d) 9.5 % HF acid etching e) VT after 40 % P acid and f) 9.5 % HF acid etching g) IPS after 40 % P acid and h) 9.5 % HF acid etching. (P: Phosphoric, HF: Hydrofluoric)

techniques, materials, and bonding agents. Weak bond strength between restorations and resin cements could lead to frequent decementation and a non-homogeneous distribution of forces that could result in cohesive failure under occlusal forces¹⁶. Strong bond strength between the tooth structure and the surface of the restoration is crucial².

Previous studies of acid etching at different concentrations with different durations have been done^{2,7,10,15}, however, surface treatment protocols for CAD-CAM ceramic blocks are still being worked on. In some studies, the HF acid concentrations were between 4.8 % and 10 %^{1-3,15,17} and the etching time 20 sec¹⁵, 60 sec⁷, and 120 sec³. With P acid concentrations of 40 % the etching time was 5 sec¹ and 60 sec⁷. In this study, 40 % P acid was etched for 5 sec following the recommendations of the manufacturer and 9.5 % HF acid for 20 sec.

In this study higher bonding values were observed in the HF acid etching of the VE group, contrary to Kutalmis et al.⁷ On the other hand, Barutcugil et al.³ found that increasing the etching time did not affect the bonding of VE; applying 10 % HF for 120 sec received identical SBS values in this current study.

Bellan et al.¹⁸ etched VS for 20 sec and VE for 60 sec with 10 % HF and the VS bonding value was 12.8–16.6 MPa, similar to this study, and the VE bonding value was 25.97–32.88 MPa, unlike this study. Compared to the previously mentioned studies, etching VE for 60 sec provided higher bonding. Hu et al.¹⁹ found that the bonding values of the VS group were higher than the VE group, similar this study. The study of Sato et al.²⁰ found similar bond values with 10 % HF acid etching for 20 sec and 40 sec for VS blocks.

Ustun et al.¹ found similar results to the IPS and VS groups in this study; however, the VE bond strength values were different as a result of the current study applying HF acid to the VE group for 20 sec instead of 60.

Mokhtarpour et al.¹⁵ compared HF acid applications and SBS values at different concentrations and at different times. The elongation of the etching time in the IPS group did not cause a significant increase in SBS. Several articles studied the etching protocol of lithium disilicate and extending the etching time was found to affect the flexure strength of the material negatively²¹.

The limitations of this study were related to the insufficient number of parameters which did not compare various resin cement types, acid concentrations, and etching times. The manufacturer did not specify etching time for the HF acid, however, both the etching times and acid concentration in the P acids is based on the manufacturer's recommendations. This study will guide clinicians in terms of bonding values that occur when different acids are used with newly introduced resin cements. Furthermore, the current study has information about the types of acids, acid concentrations, and their etching times to be used in restorations that require more retention.

V. Conclusions

Within the limitations of this study, the following conclusions can be drawn:

1. Applying HF acid increased shear bond strengths of VE and VS compared to P acid etching within the groups.
2. SBS of same acid agents to VE and VS groups were significantly different from other groups.

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