



Comparison of traditional and simplified methods for repairing CAD/CAM feldspathic ceramics

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PURPOSE. To evaluate the adhesion to CAD/CAM feldspathic blocks by failure analysis and shear bond strength test (SBSt) of different restorative systems and different surface treatments, for purpose of moderate chipping repair. **MATERIALS AND METHODS.** A self-adhering flowable composite (Vertise Flow, Kerr) containing bi-functional phosphate monomers and a conventional flowable resin composite (Premise Flow, Kerr) applied with and without adhesive system (Optibond Solo Plus, Kerr) were combined with three different surface treatments (Hydrofluoric Acid Etching, Sandblasting, combination of both) for repairing feldspathic ceramics. Two commercial systems for ceramic repairing were tested as controls (Porcelain Repair Kit, Ultradent, and CoJet System, 3M). SBSt was performed and failure mode was evaluated using a digital microscope. A One-Way ANOVA (Tukey test for post hoc) was applied to the SBSt data and the Fisher's Exact Test was applied to the failure analysis data. **RESULTS.** The use of resin systems containing bi-functional phosphate monomers combined with hydrofluoric acid etching of the ceramic surface gave the highest values in terms of bond strength and of more favorable failure modalities. **CONCLUSION.** The simplified repairing method based on self-adhering flowable resin combined with the use of hydrofluoric acid etching showed high bond strength values and a favorable failure mode. Repairing of ceramic chipping with a self-adhering flowable resin associated with hydrofluoric acid etching showed high bond strength with a less time consuming and technique-sensitive procedure compared to standard procedure. [*J Adv Prosthodont 2017;9:257-64*]

KEYWORDS: Phosphate acid monomers; Ceramics; Surface treatment; Chipping repair; Ceramic repair system

INTRODUCTION

Due to its resistance and stability in the oral environment and superior aesthetics¹, dental ceramic is frequently used for restoring damaged teeth, replacing missing teeth, and improving the esthetics of the natural dentition.² However,

ceramic materials are brittle and may fracture due to fatigue load, improper substructure design, inadequate thickness, micro-porosity, and mismatch of coefficient of thermal expansion between core and veneered ceramic.^{3,4} Ceramic fracture, commonly described as chipping, has been reported as one of the main reasons for restoration failure.^{5,6} Since the 1980s, the development of CAD/CAM technology in dentistry introduced the industrial production of feldspathic blocks. Industrial production is claimed to guarantee an effective reduction of internal porosity for CAD/CAM blocks with respect to pressed and veneered porcelain⁷ limiting the occurrence of chipping failure. However, both for traditionally layered as well as for industrially produced blocks, feldspathic ceramic chipping is still a significant issue. A possible classification and treatment recommendation for a chipped ceramic restoration was published by Heintze and Rousson.⁸ These authors proposed a chipping scale comprising three grades according to the extension of

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chipping and subsequent treatment. “Small veneer chip-pings” were classified as grade 1 and considered treatable with polishing. “Moderate veneer chippings” were classified as grade 2 and considered treatable with a resin composite-based repairing procedure. “Severe chippings” were classified as grade 3, requiring the replacement of the entire restoration. Ceramic repair with the use of composite resin was proposed in 1994 by Burke and Grey,⁹ with the high bond level demonstrated in *in vitro* studies after ceramic surface conditioning. Hydrofluoric acid etching, sandblasting, and chemical processing have been reported as the most effective systems for surface conditioning of the ceramic substrate for resin-based material bonding.¹⁰ The repair of fractured ceramic restorations with resin composite has some advantages, as it preserves the main body of the restoration, avoids remaking the restoration with further reduction of tooth structure, and is an easy, fast, and inexpensive procedure. Disadvantages of the procedure include a possible reduction in the longevity of the repaired restoration compared to the original and the handling difficulties in the operating field.¹¹ This last aspect requires that clinicians should be familiar with the materials and techniques available to repair ceramic restorations.¹² The most common methods for the surface treatment of ceramic after chipping are acid etching, sandblasting with aluminum oxide or sandblasting with silica-coated particles,⁶ used in combination with flowable resin composite. More recently, a new self-adhering flowable resin composite, Vertise Flow (Kerr, Orange, CA, USA), has been introduced to the dental market. According to the manufacturer, this innovative material combines the rheological properties of a flowable resin composite with the adhesive potential of a bonding agent. Therefore, the need for a separate bonding procedure is eliminated, improving the ease of use and reducing overall treatment time.^{13,14} The adhesive potential of Vertise Flow is mediated by a bi-functional phosphate monomer (GPDM). The GPDM can chemically bond to the oxides of the hydrofluoric acid treated ceramic surface on one side and to the resin on the other side.¹⁰ On the basis of this peculiar aspect, it seemed worthwhile to investigate the bonding potential of this new self-adhering flowable material to feldspathic ceramic using different surface treatments compared to conventional systems, in order to investigate its efficacy as a simplified system for feldspathic ceramic chipping repair.

The null hypotheses tested were i) the new self-adhering flowable resin composite can achieve similar shear bond strengths to ceramic repaired with conventional systems; ii) the surface treatments do not influence the repairing ability of the tested ceramic repair systems.

MATERIALS AND METHODS

CAD/CAM ceramic feldspathic blocks for CEREC System (Vitablocs Mark II, Vita Zahnfabrik, Bad Sackingen, Germany 2M2C I-14 #14640) were selected as ceramic substrates.

110 blocks were randomly divided into 11 groups ($n = 10$). A 1-mm thick slice was cut away with a slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) from the surface opposite to the pin retainer in order to expose the internal surface of the ceramic substrate as it occurs with ceramic chipping. The surface treatments and repairing systems tested are reported in Table 1. All the materials were used strictly according to the manufacturers' instructions. Group 10 - Porcelain Repair Kit group [PRK] and group 11 - CoJet + Premise Flow group [CJP] were used as the control groups since they are indicated for the repair of porcelain chipping.

In order to test the materials on a standardized bonding area, an aluminum split mold was used to attach onto the substrate a silicon mold with a 3-mm internal diameter. For all the groups, a 2-mm thick resin build-up was layered and light-cured with a quartz-tungsten-halogen curing device (VIP, Bisco Inc., Schaumburg, IL, USA; 600 mW/cm²). The repairing procedures are reported in table 1. After preparation, the bonded specimens were then left undisturbed for 24 hours in 100% humidity at 37°C prior to the shear bond strength test (SBSt).

The SBSt were performed in a universal testing machine (Triax Digital 50, Controls, Milan, Italy), a shear load was applied in a direction parallel to the bonded interface at a crosshead speed of 0.5 mm/min until failure occurred. The load at failure was recorded in Newtons (N). The diameter of the debonded composite cylinder was measured with a digital caliper (Orteam s.r.l, Milan, Italy). Bond strength was then calculated in Mega Pascals (MPa) dividing the load at failure by the adhesive surface area (in mm²).

Failure mode was separately evaluated by two experienced evaluators (MC and AV) using a digital microscope (Shuttlepix p-400r, Nikon, Tokyo, Japan) at 40× magnification. Failure was classified as: (i) cohesive within the substrate, (ii) mixed (if adhesive and cohesive fractures occurred simultaneously), (iii) adhesive (between repairing material and ceramic), or (iv) cohesive within the repairing system. Where the evaluators disagreed, differences in failure classification were discussed until a final mode was established by consensus.

Two randomly selected specimens per different surface treatment were gold coated and processed for SEM observations (JSM-6060LV, JEOL, Tokyo, Japan) at 1500× magnification in order to visualize the surface morphology.

Shear bond strength data were statistically analyzed. As the data distribution was normal according to the Kolmogorov-Smirnov test and group variances were homogenous according to the Levene test, One-Way Analysis of Variance (ANOVA) was applied, followed by the Tukey test for post hoc comparisons. The Fisher's Exact Test was applied to the failure analysis data, for single comparison between groups, and Fisher's Exact test was again applied for pair wise correlations. In all the analyses the level of significance was set at $P = .05$.

Table 1. Group materials and procedures

Groups	Surface treatment	Adhesive system	Repairing material	Procedure	Manufacturer	Batch
1 – [SAN + VF]	Sandblasting with 100 µm aluminum particles at 4 ATM of pressure for 20 seconds	NO	Vertise Flow - self-adhering flowable resin composite	20 second brushing then layered	Kerr, Orange, CA, USA	Vertise Flow 3412893
2 – [SAN + PF]	Sandblasting	NO	Premise Flow - nano-filled restorative flowable composite	Layered	Kerr	Premise Flow 3183782
3 – [SAN + OP]	Sandblasting	Optibond Solo Plus - single-component total-etch adhesive system	Premise Flow	15 second brushing for optibond then premise layering	Kerr	Optibond Solo Plus LB01754 Premise Flow 3183782
4 – [HFE + VF]	Acid Etching treatment with a 9% hydrofluoric acid gel for 120 seconds†	NO	Vertise Flow	20 second brushing then layered	Kerr	Vertise Flow 3412893
5 – [HFE + PF]	Acid Etching	NO	Premise Flow	Layered	Kerr	Premise Flow 3183782
6 – [HFE + OP]	Acid Etching	Optibond Solo Plus	Premise Flow	15 second brushing for optibond then premise layering	Kerr	Optibond Solo Plus LB01754 Premise Flow 3183782
7 – [SFE + VF]	Sum of treatments; Sandblasting followed by Acid Etch	NO	Vertise Flow	20 second brushing then layered	Kerr	Vertise Flow 3412893
8 – [SFE + PF]	Combination of treatments	NO	Premise Flow	Layered	Kerr	Premise Flow 3183782
9 – [SFE + OP]	Combination of treatments	Optibond Solo Plus	Premise Flow	15 second brushing for optibond then premise layering	Kerr	Optibond Solo Plus LB01754 Premise Flow 3183782
10 – [PRK]	Combination of Treatments	Silane coupling agent followed by Peak Universal Bond	PermaFlo	1 minute silane evaporation, 10 gently brush the peak Universal Bond then PermaFlo layering	Ultradent Product Inc., South Jordan, UT, USA	
11 – [CJP]	Sandblasting with 30 µm alumina particles coated with silica (CoJet® Sand)	ESPE® Silane followed by Visio™ Bond	Premise Flow	5 minutes silane evaporation, air thinned Visio™ Bond and then Premise layering	3M ESPE, St. Paul, MN, USA	CoJet System 68421 Premise Flow 3183782

† Porcelain Etch Gel, Ultradent Product Inc., South Jordan, UT, USA

RESULTS

The SBSt results are summarized in Table 2. Vertise Flow in combination with hydrofluoric acid etching showed the highest result (19.18 ± 2.78 MPa), but its result was not statistically different from those of the other groups with hydrofluoric acid etching, with the exception of Group 5 - hydrofluoric acid etching + Premise Flow [HFE+PF] and of the control group 10 - Porcelain Repair Kit [PRK]. The lowest shear bond strengths were achieved by Group 2 - sandblasting + Premise Flow group [SAN+PF] (2.32 ± 1.84 MPa). No statistically significant differences were found among the three tested ceramic repair systems when the ceramic surface was treated only with sandblasting. In addition no statistically significant differences were found between Group 5 - hydrofluoric acid etch + Premise Flow [HFE+PF] (8.59 ± 3.27 MPa) and the two controls Group 10 - Porcelain Repair Kit [PRK] (12.96 ± 2.56) and Group 11 - CoJet + Premise Flow [CJP] (9.55 ± 4.02 MPa).

The SEM observation of the ceramic surface revealed differences among the tested treatments (Fig. 1). The hydrofluoric acid etching generates regular and deep irregularities on the surface by dissolving the silica amorphous phase of the feldspathic ceramic. When applied after the sandblasting treatment, no morphological difference was observed compared with the ceramic surface treated only by acid etching. The specimens sanblasted with $100 \mu\text{m}$ aluminum particles showed a surface where groves, pits and fissures generated a moderate micromechanical retention for the repairing material. The ceramic surface sandblasted with $30 \mu\text{m}$ alumina particles coated with silica (CoJet Sand) showed the smoothest surface compared with the other ceramic treatments tested.

Analysis of failure showed that, for surface treatment, a significantly higher level of favorable fracture mode was shown where the hydrofluoric acid etching was performed compared to sandblasting and tribochemical coating (Table 3). The Vertise Flow showed the best results among the tested materials, with 60% of ceramic cohesive fractures and only 20% of mixed and adhesive fractures, and with no cohesive restorative material fractures. No statistically significant differences were found among Vertise Flow, Premise Flow, and Optibond + Premise Groups (Table 4). Results in percentages for all the groups are summarized in Fig. 2.

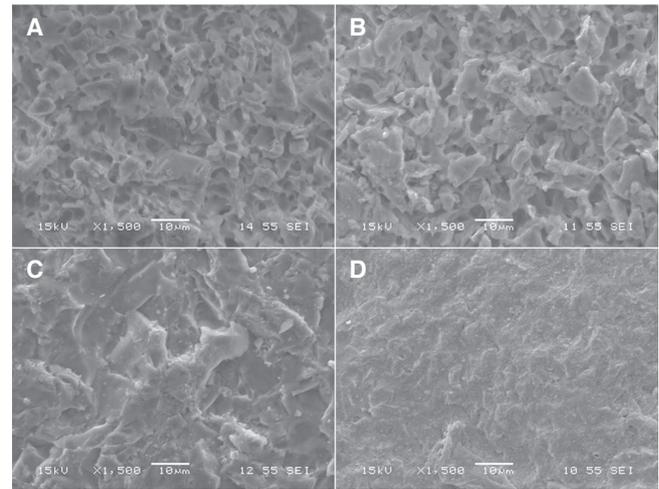


Fig. 1. SEM observation of the ceramic surface treatments. Hydrofluoric acid etching (A), Sandblasting with $100 \mu\text{m}$ Alumina particles followed by hydrofluoric acid etching (B), Sandblasting with $100 \mu\text{m}$ Alumina particles (C) and Tribochemical coating with $30 \mu\text{m}$ Alumina particles coated with silica (D).

Table 2. Shear Bond Strength Test values and statistical analysis results $P = .05$

Surface Treatment - Materials	Mean (MPa)	SD
Hydrofluoric Acid Etch - Vertise Flow ^a	19.18	2.78
Sandblasting + Hydrofluoric Acid Etch - Vertise Flow ^{a,b}	17.60	1.77
Sandblasting + Hydrofluoric Acid Etch - Optibond + Premise Flow ^{a,b,c}	14.70	3.38
Sandblasting + Hydrofluoric Acid Etch - Premise Flow ^{a,b,c}	14.20	2.67
Hydrofluoric Acid Etch - Optibond + Premise Flow ^{a,b,c}	13.99	2.48
Hydrofluoric Acid Etch - Premise Flow ^{b,c}	12.96	2.56
CoJet + Premise Flow ^{c,d}	9.55	4.02
Porcelain Repair kit ^{c,d}	8.59	3.27
Sandblasting - Optibond + Premise flow ^{d,e}	5.36	2.04
Sandblasting - Vertise Flow ^{d,e}	5.35	3.94
Sandblasting - Premise Flow ^e	2.32	1.84

Table 3. Failure mode by surface treatment and Fisher’s Exact Test results $P = .05$

Surface treatment failure analysis (%)				
	Cohesive composite	Adhesive	Mixed Adh/Coh ceramic	Cohesive ceramic
HF Acid Etch ^a	0	0	40	60
Sandblasting + HF Acid Etch ^a	25	5	15	55
Sandblasting ^b	6.7	60	26.6	6.7
Tribochemical Coating – CoJet ^b	0	40	60	0

Table 4. Failure mode by material and Fisher’s Exact Test results $P = .05$

Repairing materials failure analysis (%)				
	Cohesive composite	Adhesive	Mixed Adh/Coh ceramic	Cohesive ceramic
Vertise Flow ^a	0	20	20	60
Premise Flow ^{a,b}	13.3	6.7	40	40
Optbond + Premise Flow ^{a,b}	0	33.3	26.7	40
Porcelain Repair Kit ^b	20	20	60	0
CoJet + Premise Flow ^b	0	40	60	0

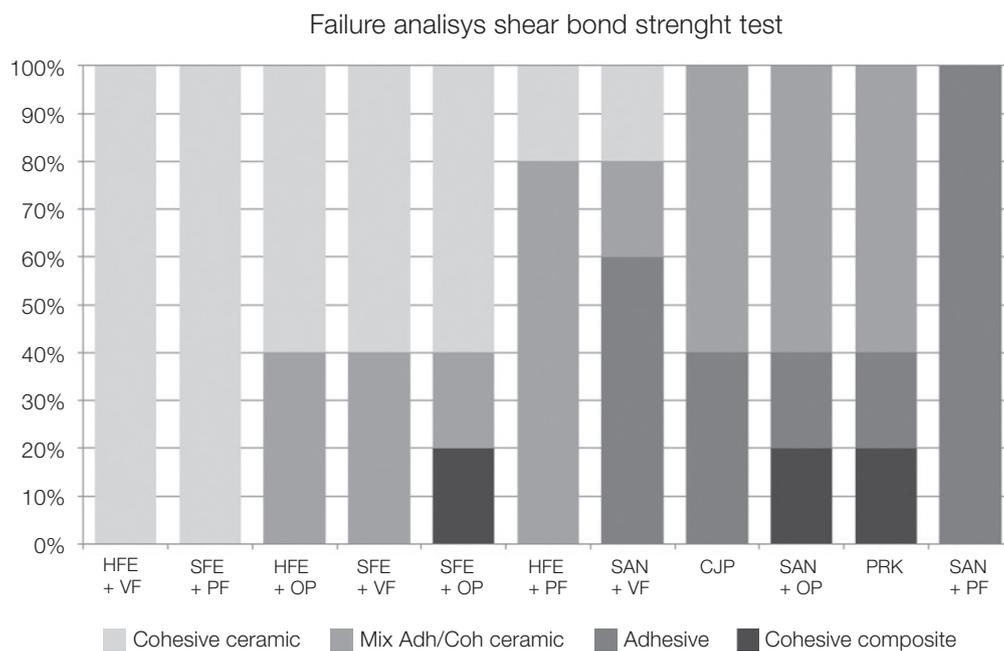


Fig. 2. Failure analysis after shear bond strength test result for groups.

DISCUSSION

Since the new self-adhering flowable resin composite achieved statistically comparable or even higher shear bond strengths to ceramic in respect to the other tested systems, the first null hypotheses was partially rejected. Statistically significant differences were also found among the surface treatments performed. Thus, the second null hypotheses was rejected.

The bond strength between two substrates can be measured *in vitro* using several methods (shear, micro-shear, tensile, micro-tensile). The principle of those tests is to apply a loading force that generates stress at the adhesive interface until specimen failure is observed. However, none of these tests is accepted as a universal method and each of them shows advantages and limitations.

The micro-tensile bond strength (MTBS) test is considered a valuable method because it generates uniform stress distribution across the adhesive interface,¹⁵ limiting the possibility of cohesive failure in the substrate.¹⁶ In spite of those advantages, MTBS is a technique-sensitive method that can present a high frequency of premature failures.¹⁷ Moreover, it can be affected by cutting speed, shape of the sample, and brittleness of the substrate.^{18,19}

Concerning the shear bond strength test (SBSt), it was observed that the critical load recorded could not clearly describe the bond strengths obtained by the different surface treatments at the adhesive interface.²⁰

Della Bona and van Noort,²¹ in their study to compare SBS test with the tensile bond strength test, concluded that the model proposed by the SBS test was too heavily influenced by the nature of the substrate and could not be used to study the real bond strength reached between two substrates. Notwithstanding these limitations, SBS test is considered a common and practical bond test mostly because it avoids the specimen sectioning and trimming steps that can introduce early micro-cracking in brittle substrates.²²⁻²⁴

Recently the SBSt was used, for their similarities with the clinical situations, as *in vitro* model to analyze the performance of ceramic repair systems.²⁵⁻²⁷ In this regard, when the adhesion value of the repairing system exceeds the cohesive value of the feldspathic ceramic, the repairing action can be considered effective, and a higher bond strength value is unserviceable.

Knight *et al.*²³ in 2003 performed a study with SBS and reported that no statistically significant differences were found when testing four resin composite systems for ceramic repair after a hydrofluoric acid etching surface treatment followed by silane application. The study reported that when the surface is acid etched, all the specimens obtained a high level of SBS and cohesive ceramic failure; the study concluded that all the tested systems were valuable for clinical use. Moreover in 2005, van der Vyver *et al.*²⁴ indicated that clinical significance and comparability between repairing systems could be described using SBS because bond strengths that equal or exceed the cohesive strength of the porcelain would be sufficient for ceramic repairs with resin

composite.

Several studies focused on the possibility of conditioning the ceramic surface in order to create a chemical bonding and/or a micro-mechanical retention for resin composite. Goia *et al.*²⁸ in 2006 compared hydrofluoric acid etching with sandblasting of the ceramic substrate in a microtensile test and concluded, in overall accordance with the present study, that bond strength was improved by the use of hydrofluoric acid etching. In 2012, Queiroz *et al.*²⁹ found higher shear bond strengths on ceramic surfaces treated with hydrofluoric acid compared with chemical treatments by different ceramic primers. They concluded that i) the use of primers alone was not sufficient to achieve an adequate bond strength to feldspathic ceramic and ii) acid etching was necessary, in correlation with ceramic primer, to obtain a sufficient bond strength between feldspathic ceramic and composite resin. The use of ceramic primers as coupling agents in order to generate a chemical bonding between resin composites and ceramic has been proposed since 1978 by Newburg and Pameijer,³⁰ but the advantage coming from their use is still controversial.³¹ The most common system marketed for ceramic repair usually combine the effect of micro-retention obtained by sandblasting or acid etching with chemical bonding by the use of silane as a coupling agent.³² Application of silane as a coupling agent to the ceramic substrate surface should provide a chemical covalent and hydrogen bond between the ceramic and the composite resin. Some of the silanes available in the market containing carboxylic acid have been reported to provide clinically adequate bond strengths even without hydrofluoric acid etching. The differences in silane composition and concentration result in different adhesion ability of the substrates.³³ Kussano *et al.*³⁴ focused on the importance of silane application, which they considered to be the main influence on resin ceramic bond strengths – even more important than acid etching or sandblasting. Their results are in overall disagreement with the results of the present study; in the groups where silane coupling agent was applied with different surface treatments, lower shear bond strength was measured when compared with surfaces treated with hydrofluoric acid etching. No evidence has been found yet to show whether the combination of sandblasting and acid etching can provide higher bond strengths than treatment with either procedure used alone. Some studies reported an advantage in the use of both procedures together^{20,21,33,34}, while other studies reported that hydrofluoric acid etching alone can be considered a sufficient treatment.^{15,16} The latter finding is in agreement with the present study, where no statistically significant differences were found in SBS and no differences were observed on the ceramic surface morphology in the SEM analysis between hydrofluoric acid treated and sandblasting plus hydrofluoric acid treated groups.

Kimmich and Stappert¹⁰ in 2013 indicated that the use of resin containing bi-functional phosphate monomers was potentially able to establish a direct chemical bond to ceramic. The GPDM-based Vertise Flow self-adhering resin adhesive system contains a bi-functional phosphate mono-

mer that can act as a coupling agent,³⁵ thereby making unnecessary the sensitive step of intraoral silane application^{10,36} as well as the bonding application when the ceramic surface is treated with hydrofluoric acid. The acid preferentially etches the amorphous glassy phase or the crystalline phase generating unsaturated oxygen bonds, which serve as bonding partners for bi-functional phosphate monomers.^{37,38} This mechanism could explain the comparable or even superior bonding ability of Vertise flow to feldspathic ceramic surfaces treated with hydrofluoric acid versus traditional multi step systems based on adhesive application followed by composite resin layering. Few data are available regarding the ability of Vertise Flow on ceramic bonding. In 2014, Erdemiur *et al.*²⁶ found a low bond strength for Vertise Flow when applied on lithium disilicate reinforced ceramic. It is reasonable to speculate that the high crystalline content, compared to feldspathic ceramic, limits the action of adhesive monomers mostly addressed to the glassy matrix. In the evaluation of the results, it should be noted that the test is not able to simulate clinical loading forces and long-term aging within the oral environment, and this represent a limitation of the *in vitro* study. Likewise, in any bonding process, the bond strength of a ceramic repair system is in fact susceptible to chemical, thermal, and mechanical influences in the oral environment. The present study was performed on industrial feldspathic blocks for the CAD/CAM system. Even if the chemical nature of the ceramic tested is very similar to the veneering feldspathic ceramic used in PFM or PFZ restorations, it would be of interest to perform further studies to investigate the same repairing protocol with veneering ceramic systems.

CONCLUSION

Based on the present study, it can be concluded that for repairing industrially produced feldspathic ceramic blocks for the CEREC CAD/CAM system, acid etching with hydrofluoric acid is sufficient to condition the substrate and that the simultaneous use of sandblasting does not further improve the bond strength. The use of hydrofluoric acid and simplified self-adhering Vertise Flow showed higher or comparable bond strengths to feldspathic ceramic than all the other combinations tested, suggesting the use of this clinically simplified approach for repairing moderate chipping.

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