Author’s response to reviews

Title: HF etching of CAD/CAM materials: Influence of HF concentration and etching time on shear bond strength

Authors:

Antonio Straface (antonio.straface@stud.unibas.ch)
Lena Rupp (lena.rupp@stud.unibas.ch)
Aiste Gintaute (aiste.gintaute@unibas.ch)
Jens Fischer (jens.fischer@unibas.ch)
Nicola Zitzmann (n.zitzmann@unibas.ch)
Nadja Rohr (nadja.rohr@unibas.ch)

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Author’s response to reviews:

Dear Editor

We would like to thank the reviewers for their efforts regarding the review of our manuscript. We have considered their recommendations and made several amendments to deal with the suggestions made. We have included a detailed point-by-point reply to the comments and the respective changes are highlighted yellow in the main document.

Reviewer #1: Language review - excellent

From a purely scientific point of view, whilst I have no expertise in this area, it looks an excellent paper, well researched and well written

Thank you very much for your comment.

Reviewer #2: Interesting paper of a well-studied topic but using novel and current materials, reason why this work may be of interest to the readership interested in CAD/CAM dental ceramics. However, some points should be addressed and explained:

* Page 3, phrase between lines 57 to 59, not quite precise as micromechanical retention achieved by HF etching is not directly related to chemical bonding, such approach depends entirely on silanization.
Thank you, this is of course correct. We have clarified the sentence as follows: “To achieve micromechanical interlocking with the resin composite cement, the surface of silicate ceramics has to be roughened [27-30]. The recommended procedure comprises etching with 5% hydrofluoric acid (HF) and application of silane coupling agent to additionally achieve a chemical bond [31-36].”

* Page 3, phrase between lines 59 and 61 should be clarified, as the recommended surface treatment regarding HF concentration is true only for lithium disilicate ceramic, other silicate ceramics are recommended to be etched with 10% HF according to manufacturers.

Thank you for this comment, however, according to the manufacturer VITA etching with HF 5% for 60s is indeed recommended for the other ceramics used in the present study. The recommendations for different ceramics vary among the manufacturers and countries, in which the respective ceramics are available, hence we left the range of the recommended treatment for all ceramics between 5% to 10%.

* Please clarify in the introduction the background, correlation and justification to measure the diametral tensile strength of resin cements involved in this study, because until the objective, nothing referred to such variable before.

Thank you very much for pointing this out. The following justification for measuring the diametral tensile strength was added to the introduction: ”Resin composite cements are brittle materials and therefore susceptible to tensile loading rather than to compressive stress [55-57]. Since adhesion of cements to ceramics is commonly tested using a shear bond strength test design, it would be of interest to analyze the effect of the cement´s indirect tensile strength on shear bond strength.”

* Page 5, lines 12-14, please state the crystallization parameters employed on the ceramics needing this process.

The crystallization process was performed according to the recommendations of the manufacturers as follows: “For EC temperature increase was 30°C/min for 15min up to the crystallizing temperature of 850°C which was held for 10min. Cooling temperature was 680°C. For VS temperature increase was 55°C/min to 840°C for 8min with cooling at 680°C.”

* Why using 30 seconds as an etching time instead of 20 seconds, as it is the recommended time for lithium disilicate ceramic?

The reason for using 30 s etching time was the standardization of the etching time for all tested ceramics. Additionally, the same etching times were used in previous publications of our group and allowed for comparison of the values.

Since there was overall no statistical difference between 15s and 30s for lithium disilicate ceramics, it was decided not to additionally test for 20s as it is suggested by the manufacturer.
Page 5, lines 52-55, please add some references supporting the described procedure of placing the shearing chisel 2mm away from the bonding interface, as this is not the conventional procedure, and please clarify this procedure with further explanations.

The reference was added to the Methods section and further explanations were added as follows: “The distance of 2mm was chosen to prevent extensive cohesive failures by increasing the leverage effect.”

Additionally, we have provided further explanations in the Discussion section: “The distance of 2mm was selected in the present protocol because less crucial cohesive material fractures were observed when specimens were sheared with 2mm distance due to the increased leverage effect leading to fractures at lower forces [30, 33].”

Page 6, statistical analysis: please report "p" values and each test used to prove parametric analysis assumptions (normality and homoscedasticity).

The name of the normality test was added with the used significance value to the Methods. “All data was tested for normal distribution using Shapiro-Wilk test (StatPlus Pro, v6.1.25, AnalystSoft; Walnut, CA, USA) (p<0.05). To analyze diametral tensile strength one-way ANOVA was applied followed by Fisher LSD test to investigate differences between resin composite cements (p<0.05). For SBS data one-way ANOVA was performed for each cement to test the influence of etching time. Three-way ANOVA was applied for each etching time to test for effects of the factors substrate, HF concentration, and cement. Post-hoc Fisher LSD test was performed to determine differences within the subgroups (p<0.05).”

When study design is performed, the researcher should not manipulate the definition of factors and decide which are going to be considered and which will not. So, even that the factor HF concentration resulted not statistically significant it should not have been eliminated as a factor, as the confection of the groups was conceived taking this factor into account. The same for the first attempt of eliminating the material factor from the ANOVA analysis. This should have been analyzed before performing the study, because the correct form is to consider all factors, in this case 4 and consequently not practical at all.

Thank you for this valuable comment. We corrected the applied statistical methods accordingly and did not pool the data for analysis to avoid false negative results. We now performed one-way ANOVA on each cement/substrate/HF combination to test the effect of etching time. Three-way ANOVA was performed on each etching time to test the effect of substrate, HF and cement. The statistical results are now displayed in the Result section and Table 2.

It was not described in the methods section the procedure used to analyze surface morphology or failure pattern but results from both aspects were reported in the results section.

The methodology used to analyze the failure patterns was added to the Methods section: “Failure patterns were classified visually as either cohesive failure in the substrate, adhesive failure,
mixed or cohesive failure in the cement. Images of those typical failure patterns were obtained with scanning electron microscopy (ESEM XL30, Philips, Eindhoven, the Netherlands).”

* Third hypothesis says: "lower diametral tensile strengths of resin composite cements results in lower shear bond strength and cohesive fractures within the cement", but it was not stated in the methods section nor in the results section if a statistical correlation test was performed or how a correlation between both variables was tested to accept or reject this hypothesis.

Yes, we agree with the reviewer, a hypothesis requires a statistical analysis. However, statistical validity of comparing 4 indirect tensile strength values to the immense amount of SBS data is questionable, therefore it was decided to remove the third hypothesis.

* Discussion part should have less comparison between the current outcomes and previous results and should have more explanations of the current results. The explanation regarding the shear test using 2 mm is not convincing, as the proper procedure is to apply the load at the very interface in order to avoid combination of difference forces or vectors, as it is happening in the present work, and so the applied force is not pure shear.

Yes, you are right, when the force is applied with a 2mm distance the force is not pure shear. However pure shear is also not the case during the “normal” shear test, because the increased tension and compression lead to non-uniform stress distributions. Additionally, the shear test has proven to be reliable for assessing differences in bonding performance as long as no fractures occur within the substrate material. To prevent extensive cohesive fractures, the distance of 2mm was chosen to increase the leverage. This detail was also added to the discussion: “The distance of 2mm was selected in the present protocol because less crucial cohesive material fractures were observed when specimens were sheared with 2mm distance due to the increased leverage effect leading to fractures at lower forces [30, 33].

A more important issue than avoiding cohesive failures, is the protection and delimitation of the bonding area, taking into account that luting materials could went out from the acrylic cylinders as no method to seal this area was described on the methods section.

These details were added to the Methods section: “An acrylic cylinder with an inner diameter of 2.9mm, outer diameter of 4.1mm, and height of 4mm was tightened in a custom made device onto the substrate surface to avoid leaking of the cement.”

* A new paragraph should be added stating the disadvantages or problems associated to the (macro) shear test and the limitations of this study.

The following paragraph was added to the Discussion section as recommended by the Reviewer:

“The applied shear bond strength design can be considered a comparable method to the ISO 29022 shear test, although the SBS values obtained with the present design were generally lower than those generated with the ISO test [50]. SBS testing is a valuable method to assess bonding
performance between interfaces as long as failures occur at the interface with no fractures of the substrate. As soon as cohesive fractures are involved the test method has been criticized as unreliable [50, 65, 66].”

Some problems pointed before are reflected on the conclusion as it states that: “Within the limitations of this study the recommended surface pretreatment of VM, VE, EC and VS is HF etching with concentrations of 5% or 9% for 30s to 60s”. This may lead to a confusion to the reader because the recommended time of etching lithium disilicate is 20 seconds, but the current conclusion recommends an etching time of 30 seconds or above indiscriminately of the material, being this not correct as in this paper authors did not include 20 seconds as one of the tested times, so that action excluded this time from the analysis.

Thank you for this comment. The recommendation for the etching time in the discussion was changed to 15s to 60s and the 20s recommended by the manufacturer were additionally mentioned in the Discussion. “In the present study differing etching patterns were observed for EC between 15s and 30s that did not affect SBS values. For practical reasons and to ensure complete etching of the substrate with sufficient dissolution of the glassy matrix the authors recommend an etching time with HF for longer than 15s and up to 60s as the manufacturer also recommends an etching time of 20s.”

Reviewer #3: Dear Authors and Editors,

This is a very interesting manuscript that will be of great interest to readers.

I had some difficult to understand the results because table and figure captions have missing statistics.

In general, the manuscript contains new and significant information, the Abstract (Summary) clearly and accurately describes the content, the methods are described comprehensively and the interpretations and conclusions are justified by the results. The language is acceptable as well.

I recommend minor revision as follows:

Page 4 - First phrase: "…and results in an increased ceramic surface." It should include the word area after surface to elucidate.

Thank you, the term “area” was added after surface: “The application of HF acid reacts with silicate, which leads to the removal of the glass phase and results in an increased ceramic surface area [37-39]”

At the phrase: "It has been demonstrated that self-adhesive cements are suitable for bonding to dentin, but considered inferior in comparison with the etch-and-rinse technique to enamel substrate [5, 51-53]." Recent studies show that such inefficiency of universal adhesives is relative and depends mainly on how strong the adhesives are. In vivo mild adhesives are as
durable as etch-and-rinse technique to enamel especially when it is dry. I recommend not to generalize.

Thank you for this comment. We referred to self-adhesive cements, in which no additional primer is applied to bond to the tooth substrate and did not address universal adhesives. Self-adhesive cements are inferior to adhesive cements where an additional one-/or multi-step primer is applied due to the low viscosity of the primer being able to penetrate the substrate. We clarified the sentence as follows: “It has been demonstrated that self-adhesive cements are suitable for bonding to dentin, while bonding to enamel substrate is considered inferior in comparison with the etch-and-rinse or self-etch adhesive techniques, in which the applied primer allow further micromechanical interlocking [5, 51-54].”

Methods

I recommend to describe the materials in their full name in the first time they appear in the text and not only in the abstract "(Vitablocs Mark II, Vita, [VM]; Vita Enamic, Vita, [VE]; e.max CAD, Ivoclar Vivadent, [EC]; Vita Suprinity PC, Vita, [VS])."

The names of the materials were already given in full in the Methods section, but we also added the abbreviations accordingly: “Shear bond strength (SBS) [30, 33, 50, 58, 59] of two adhesive (Panavia V5 [PV5], Kuraray Noritake; Vita Adiva F-Cem [VAF], Vita) and two self-adhesive (RelyX Unicem 2 Automix [RUN], 3M Espe; Vita Adiva S-Cem [VAS], Vita) cements to four different CAD/CAM materials (Vitablocs Mark II [VM], Vita; Vita Enamic [VE], Vita; e.max CAD [EC], Ivoclar Vivadent; Vita Suprinity PC [VS], Vita) was measured (Tab. 1).”

The text describes that the authors used the primers indicated by the manufacturers but it does not mention them in the methodology. This is confusing because the information is only in the figure 1 captions

Page 5 Line 11 and 12.

Yes, thank you for bringing up this point. We have added the full names of the primers and the application technique in the Methods section: “The primer used in combination with PV5 was Clearfil Ceramic Primer plus [CCPP] (Kuraray Noritake). For both VAF and VAS Vita Adiva Ceramic Primer [VACP] (Vita) was used and to RUN RelyX Ceramic Primer [RXCP] (3M Espe) was assigned. The respective primers were applied on the substrate surfaces with a microbrush for 20s and dried with oil-free air.”

Did these data come from ref 54? Its is not clear. It is important to write the units as nm or µm and to describe the method used to measure it.

Yes, thank you for this comment, we have added the units: “The substrate slices were then grinded (SiC paper grit P180, Struers, Baltrup, Denmark) to attain a similar roughness as it is given by a CAD/CAM milling machine (Ra=1.88µm for VM and VE, 2.71µm for EC, 2.52µm for VS after crystallization) [60].”
The SBS (σ) test is well known and its methodology should be referenced. Are its dimensions in accordance with any ISO or ASTM standards? The same to Diametral tensile strength test.

The applied SBS methodology was referenced. The dimensions of the applied test for the contact area are 2.9mm surrounded with an acrylic cylinder, for ISO 29022 it is 2.4mm. The SBS values of the applied test design are generally lower than those obtained with the ISO 29022 test, the results however correlate linearly for adhesive fractures as shown by Hu et al. in Dent Mater 2016;32:223-32. The diametral tensile strength test was also referenced in the Methods section:

“Shear bond strength (SBS) [30, 33, 50, 58, 59] of two adhesive (Panavia V5 [PV5], Kuraray Noritake; Vita Adiva F-Cem [VAF], Vita) and two self-adhesive (RelyX Unicem 2 Automix [RUN], 3M Espe; Vita Adiva S-Cem [VAS], Vita) cements to four different CAD/CAM materials (Vitablocs Mark II [VM], Vita; Vita Enamic [VE], Vita; e.max CAD [EC], Ivoclar Vivadent; Vita Suprinity PC [VS], Vita) was measured (Tab. 1).”

“Diametral tensile strength of all 4 resin composite cements was measured.”

Additionally we discussed the ISO test in the Discussion section:

“The applied shear bond strength design can be considered a comparable method to the ISO 29022 shear test, although the SBS values obtained with the present design were generally lower than those generated with the ISO test [50]. SBS testing is a valuable method to assess bonding performance between interfaces as long as failures occur at the interface with no fractures of the substrate. As soon as cohesive fractures are involved, the test method has been criticized as unreliable [50, 65, 66].”

In table 2 and 3, the authors should have put the statistics.

Statistical analysis was added to Table 2 and 3 using superscript letters.

Table 1. The authors should have put the source of information

The information for the table was taken from the SDS sheets of the products provided by the manufacturers. This information was added to the legend. “Table 1 Investigated materials with composition as provided in the safety data sheets of the products.”

I recommend the authors to make a table with fracture data (percentage of each type of failure found) for each group and to include a diagram or photo of examples of cohesive, adhesive fracture.

I hope I have helped.
Thanks a lot for your comments. A Figure 3 was provided in addition displaying each failure type. Due to the extensive data (144 groups and 4 possible failure types) the authors described the general failure patterns within the text.