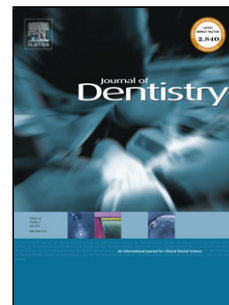


## Accepted Manuscript

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**A randomised controlled study on the effects of different surface treatments and adhesive self-etch functional monomers on the immediate repair bond strength and integrity of the repaired resin composite interface using 3D surface profilometry and scanning electron microscopy**

**Short title: Repair bond strength and integrity of repaired resin composite**

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**Abstract**

**Objectives:** To evaluate the effects of different surface conditioning methods on the immediate repair bond strength and integrity of the repaired composite interface.

**Methods:** One hundred and five resin composite blocks made of a nanohybrid resin composite were randomly assigned to one of the following surface conditioning groups (n=15/group): Group 1: Gluma Self Etch™ adhesive system, Group 2: Tokuyama Bond Force II™ adhesive system, Group 3: non-roughened and non-conditioned surfaces, Group 4: sandblasting and Gluma Self Etch™, Group 5: sandblasting and Tokuyama Bond Force II™, Group 6: sandblasting only. A positive control group was also used. Resin composite identical to the substrate was applied and the repaired specimens were subjected to shear bond strength (SBS) testing. Representative samples from all groups were subjected to scanning electron microscopy and surface profilometry to determine their mode of failure. The data were analysed statistically using Analysis of Variance (ANOVA) and two independent sample t test ( $\alpha=0.05$ ).

**Results:** The mean SBS of all test groups ranged between 1.92 and 5.40 MPa and varied with the degree of composite surface roughness and the type of adhesive system employed. Significantly highest SBS values ( $5.40 \pm 0.36$  MPa) were obtained in Group 5 ( $p=0.017$ ) which were comparable to the coherent strength of the resin composite in the positive control group ( $p>0.05$ ).

**Conclusions:** Under the tested conditions, significantly greater SBS of repaired resin composite was achieved when the substrate surface was conditioned by sandblasting followed by the application of the Tokuyama Bond Force II™ adhesive system.

**Clinical significance:** Effecting a repair of a nanohybrid composite restoration with sandblasting and the application of TBF II would seem to enhance the interfacial bond strength and integrity of the repaired resin composite interface. Clinical trials are necessary to determine the usefulness of this technique.

**Keywords:** Nanohybrid composite repair, Minimally invasive dentistry, Shear bond strength

**Introduction:**

Resin based composites (hereon = composites) are the restorative materials of choice for the restoration of anterior and posterior teeth.<sup>1</sup> However, in common with all dental restorations placed into the hostile oral environment, resin composites commonly suffer deterioration and degradation in clinical service over time.<sup>2,3</sup> The annual failure rates of anterior and posterior composite restorations have been reported to vary between 1% and 4%.<sup>4-7</sup> The replacement of failing restorations has been reported to constitute up to 60 percent of all activity performed in general dental practice.<sup>8</sup>

It is widely accepted that replacement of failing restorations, particularly those with localised defects, may be considered as excessively interventional as most of the restorations may be clinically and radiographically free of failures.<sup>9-11</sup> Furthermore, restoration replacement invariably leads to an acceleration of the downward spiral of the tooth with its associated consequences, including unnecessary sacrifice of healthy tooth structure in locations distant from the site of the deteriorating restoration. Additional risks associated with restoration replacement include progressive cavity enlargement and weakening of the restored tooth, repeated insults to the pulp with increased risk of irreversible pulpal damage and misuse of patients' time and financial resources.<sup>2,3,11,12</sup>

While some failing restorations inevitably require replacement, it is suggested that many deteriorating, yet serviceable, restorations may be given extended longevity through repair procedures (i.e. partial replacement of a restoration that presents no clinical or radiographic evidence of failure), provided the repaired restoration satisfies the necessary clinical requirements<sup>13,14</sup> thereby also extending the longevity of the restored tooth. Advancements in adhesive technology have led to the notion of restoration repair becoming an intrinsic part of dental undergraduate curricula worldwide<sup>15-20</sup> and it is inherent in the modern concept of minimally invasive dentistry.<sup>21</sup>

Despite a myriad of *in vitro* reports investigating various mechanical and chemical surface treatment approaches to improve the repair potential of composites,<sup>22-29</sup> no gold standard protocol exists for treating the aged composite substrate prior to repair. Whilst the conventional three-step etch-and-rinse approach may still be considered the gold standard of adhesive bonding,<sup>30,31</sup> the use of all-in-one one bottle self-etch adhesive systems has increased in use and popularity among clinicians in recent years<sup>32,33</sup> as adhesion and priming takes place at the same time, and no rinsing is required. In this

procedure, the clinical application time is shortened, and technique sensitivity is reduced, resulting in improved user friendliness.

Self-etch adhesives contain specific acidic functional monomers which enhance the performance of adhesion.<sup>34</sup> The functional monomers help conditioning the substrate surface, increase monomer penetration,<sup>35</sup> and also improve chemical adhesion.<sup>36</sup>

Although many reports exist on the repair of hybrid and microhybrid composites using three-step and two-step adhesive strategies,<sup>37</sup> limited data exists, to date, regarding the best protocol for performing a repair using nanohybrid resin composites and self-etch adhesive bonding systems.

The primary aim of this *in vitro* study was to evaluate the effects of different surface treatment and conditioning methods on the repair bond strength of a nanohybrid resin composite material. The secondary aim was to the nature of interfacial failure, using scanning electron microscopy (SEM) and profilometry examinations of failed interfacial surfaces. The null hypothesis tested was that there was no statistically significant difference in repair bond strengths between the various repair protocols.

## 2. Methods and materials

### 2.1 Specimen preparation

One hundred and five custom made Teflon mold retention bases were fabricated containing a rectangular recess (25mm length x 13mm width x 4mm depth) with a cylindrical form of 2mm diameter and 2mm depth at the centre of the recess (Fig.1).

A universal nanohybrid resin composite material (Tetric EvoCeram™, Ivoclar Vivadent, Lichtenstein) was applied in 2mm thickness into the rectangular recess of the Teflon molds. The layer of resin composite was photo-polymerized in a light oven (Dentacolor XS Kulzer, Germany) for 180 seconds, operating with a wavelength of 435nm, to achieve maximum polymerisation. An increment layer of 2mm of the same resin composite was applied on the polymerised first layer. The increment layer specimen was covered with a glass slide to achieve a flat smooth surface and prevent the formation of an oxygen inhibition layer, prior to photo-polymerisation in the light oven for 180 seconds. Subsequently, ninety resin composite blocks were removed from the Teflon molds and their top surfaces were polished with a wet 500-grit silicon carbide disc using a polishing machine (Struers LaboPol35, Struers A/S, Rodovre, Denmark) at 300 rpm for 30s and cleaned for 10 min in an ultrasonic bath (Quantrex 90 WT, L&R Manufacturing Inc., Kearner, NJ, USA) containing deionized water to eliminate possible

contamination. All surface polished resin composite specimens were placed back into the Teflon molds and air-dried ( $23 \pm 1.0^\circ\text{C}$ ) for 24 hours. One group of 15 specimens served as a positive control and the resin composite surfaces were not polished.

## 2.2 Surface conditioning methods

The surface polished ninety resin composite blocks were randomly divided, using randomisation tables, into six equal groups, each of 15 specimens to receive the following surface conditioning treatments according to the manufacturers' instructions:

*Group 1:* one coating of Gluma Self-Etch™ adhesive system (Heraeus Kulzer, Hanau, Germany) applied with a disposable applicator and circular brushing motion for 20s, dried with oil-free air/water syringe for 5s and light cured for 20s using the Bluephase 20i (Ivoclar Vivadent, Lichtenstein) hand held LED light cure unit operating at a measured output of  $1000 \text{ mW/cm}^2$  intensity.

*Group 2:* one coating of Tokuyama Bond Force II™ adhesive bottle system (Tokuyama Dental, Tokyo, Japan) applied with a disposable applicator and circular brushing motion for 10s, waited for 10s, dried with oil-free air/water syringe for 5s and light cured for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

*Group 3 (negative control group):* No air abrasion and no chemical surface conditioning was used.

*Group 4:* Air-borne particle abrasion with  $50\mu\text{m Al}_2\text{O}_3$  (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-Prep™, RønviignA/S, Daugaard, Denmark) from a distance of 10mm at a pressure of 2.5 bar for 10 s followed by washing (10s) and drying (10s) with air/water syringe and the application of one coating of Gluma Self-Etch adhesive system (Heraeus Kulzer, Hanau, Germany) with circular brushing motion for 20s, dried for 5s and photopolymerised for 20s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

*Group 5:* Air-borne particle abrasion with  $50\mu\text{m Al}_2\text{O}_3$  (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-Prep™, RønviignA/S, Daugaard, Denmark) from a distance of 10mm at a pressure of 2.5 bar for 10 s followed by washing (10s) and drying (10s) with air water syringe and the application of one coating of Tokuyama Bond Force II adhesive system (Tokuyama Dental, Tokyo, Japan) with circular brushing motion for 10s, waited for 10s, dried for 5s and photopolymerised for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

*Group 6:* Air-borne particle abrasion with 50 $\mu$ m Al<sub>2</sub>O<sub>3</sub> (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-Prep<sup>TM</sup>, RønvingA/S, Daugaard, Denmark) from a distance of 10mm at a pressure of 2.5 bar for 10 s followed by washing (10 s) and drying (10 s) with air/water syringe.

Subsequently, the Teflon molds in all six groups were removed and the specimens were air-dried (23  $\pm$  1.0°C) for one minute. The description, composition and manufacturers of the adhesive materials used in this study are summarised in Table 1. Details of the surface conditioning procedure used for each group are summarised in Table 2.

### 2.3 Repair resin composite application

The repair resin composite was identical in type and brand to the substrate resin composite material. The base of the Teflon molds had a prepared cylindrical recess of 2mm diameter and 2mm depth which was used for the resin composite application procedure. In the six test groups and the positive control group, the universal nanohybrid resin composite material was packed using a flat plastic instrument into the cylindrical recess and light cured (Bluephase 20i, Ivoclar Vivadent, Lichtenstein) for 20s. In the positive control group, the repair resin composite was immediately applied on the prepared fresh resin composite substrate. All surface treatment and resin composite application procedures were performed by a single experienced operator in accordance with the manufacturers' instructions.

Subsequently, the Teflon molds were removed and all specimens were stored for 24h at 23  $\pm$  1.0°C room temperature before being subjected to repair bond strength testing.

### 2.4 Bond strength testing

All specimens were individually mounted on a universal testing machine (Instron, Norwood, Massachusetts, USA) and subjected to shear bond strength (SBS) testing travelling at a crosshead speed of 0.5 mm/minute. The shear force was applied until failure occurred. For calculation of the SBS results the applied force was recorded and compression load at break divided by the contact area of cylindrical repair. The data were subjected to statistical analysis using a two-way multivariate analysis of variance (ANOVA), two independent sample t test to analyse the equality of means and the Kolmogorov-Smirnov test at a 95% confidence interval level.

## 2.5 Failure analysis

Five specimens were randomly selected, using a computer generated allocation sequence, and their surfaces were examined under optical microscopy (Olympus SZ61, Tokyo, Japan) at 45x magnification. Mode of failure was recorded as *adhesive* – failure at the substrate-repair resin interface, *cohesive* – failure within the resin substrate or within the repair composite, or *mixed* – areas of adhesive and cohesive failure. Subsequently, these specimens were examined under SEM. The specimens were sputter-coated with a 50 nm gold layer (Bio-Rad SEM Sputter Coating Unit PS3, Microscience Division, West Chester, USA) to aid conductivity and examined using a Hitachi S-4300 SEM (Hitachi Science Systems, Ltd., Tokyo, Japan) at accelerating operating voltages of 5 and 15 kV in the secondary electron mode for taking high-resolution electron micrographs.

The failed surfaces of another five randomly selected specimens, using a randomisation table, from each test group and the PC group were examined under three-dimensional high resolution profilometry (Ambios Technology XP-1, Santa Cruz, California, USA) to examine the surface roughness profiles at the failed surfaces. A Stylus tip radius of 2.0 microns was travelling at a tracing speed of 0.5 mm/s applying a stylus force of 1mg. The arithmetical mean deviation of profile (Ra), root mean square deviation of profile (Rq), maximum depth of profile peak (Rp) and maximum depth of profile valley (Rv) amplitude parameters were recorded and determined using three dimensional profilometry (Ambios Technology Inc. software, Santa Cruz, California, USA). The data was analysed statistically using a two-way multivariate analysis of variance (ANOVA) at  $\alpha=0.05$ .

## 3. Results

### 3.1 Bond strength

The results of the shear bond strength tests are presented in Table 2.

Surface roughening with alumina sandblasting yielded significantly higher repair bond strength compared to no surface modification in the negative control group ( $p<0.01$ ).

The bond strength values of specimens treated with adhesive techniques presented significantly higher bond strength values compared to where surface polishing alone ( $p=0.02$ ) and sandblasting ( $p=0.03$ ) was used. The Surface conditioning with alumina sandblasting and the use of TBF II resulted in significantly higher bond strength values



( $5.40 \pm 0.36$  MPa) than all other surface conditioning methods ( $p=0.017$ ). No significant difference in bond strength values was noted between the use of TBF II without sandblasting ( $4.71 \pm 0.55$  MPa) and the use of GSE following sandblasting ( $4.79 \pm 0.54$  MPa) ( $p=0.061$ ) or polishing ( $4.34 \pm 0.48$  MPa) ( $p=0.082$ ).

There was no significant difference between the specimens prepared with sandblasting and the TBF II adhesive system ( $5.40 \pm 0.36$  MPa) and the positive control group ( $5.66 \pm 0.49$  MPa) ( $p=0.094$ ). With the exception of the use of sandblasting and TBF II, the positive control group presented significantly higher bond strength values compared to all surface conditioning methods ( $p<0.01$ ).

### 3.2 Failure analysis

The surfaces of five randomly selected specimens from each test group were examined using optical microscopy to investigate the mode of failure and by SEM examination to investigate the surface morphology of the failed surfaces.

Optical microscopy examination showed that polished specimens had significantly more adhesive failures than sandblasted surfaces ( $p=0.001$ ). Specimens treated with polishing and GSE (Group 1) showed 100% adhesive failures, whereas those treated with polishing and TBF II (Group 2) showed 73% adhesive failure. In contrast, the sandblasted surfaces conditioned with TBF II (Group 5) showed mostly cohesive failures (80%), while the sandblasted surfaces conditioned with GSE (Group 4) showed predominantly adhesive failures (60%). A summary of the findings is shown in Fig. 2.

The profilometric findings are presented in Table 3. These results revealed that there were significant differences between the sandblasted and polished groups in respect to surface roughness values and all other amplitude parameters tested. There was strong evidence that the sandblasted specimens provided a more irregular and rougher surface finish than the polishing technique ( $p=0.0001$ ). SEM examinations have confirmed these findings as shown in Fig. 3a-d.

## Discussion

Notwithstanding recent developments in adhesive technology and composite material science, failure of composite restorations, notably by fracture and localised secondary caries remains a problem in clinical practice<sup>3</sup> and with the globally expanding use of

adhesive and composite systems,<sup>38-42</sup> the need for composite restoration repair will increase. This is particularly supported by longitudinal studies which have shown that composite repairs can extend the longevity of dental restorations.<sup>43-47</sup> However, there is still some debate as to the best repair protocol and it is therefore presently difficult for clinicians to select a repair protocol to achieve the best clinical outcome. Evidence about how best to perform a state-of-the-art composite repair using self-etch one step adhesive systems, which are increasingly used in clinical practice owing to their reduced chair time and simplicity of use is scarce.

The present study was undertaken to evaluate the effect of different surface treatments and self-etch one step adhesive systems on the bond strength and integrity of the repaired composite interface. Whilst it is recognised that *in vitro* findings cannot be directly translated to the *in vivo* situation they are a useful predictor of the potential clinical performance of materials and techniques, which can be useful in making decisions about whether to perform this procedure.

Mechanical surface treatments have the ultimate goal to improve micromechanical interlocking between the aged composite substrate and the repair composite, whereas adhesive systems are applied in an endeavor to improve chemical bonding between composites at the adhesive repair interface.<sup>48</sup> Several studies evaluated physical, chemical, and physico-chemical treatments of the repaired composite surface seeking improved repair bond strengths.<sup>22-29</sup> However, the large variation of materials, techniques, and testing methods used in laboratory investigations for assessing surface treatments of composites for repair procedures makes it difficult to make direct comparisons between studies. The 10-MDP (Tokuyama Bond Force II™ - TBF II) and 4-META (Gluma Self Etch™ - GSE) functional monomer-based adhesives were used in the present study as these adhesive systems have shown superior results when chemically interacting with hard tooth tissues.<sup>34,49-51</sup>

In this study, a shear bond test was used, as tensile forces occur close to the force application area, which may affect the substrate more than the adhesive interface itself.<sup>52</sup> The microtensile test, suggested by Sano et al.<sup>53</sup> assesses the bond strength of specimens with reduced areas of adhesive joints where fractures occur at the adhesive interface. At around the same time, Sau et al.<sup>54</sup> reported that loading the specimens under shear could be considered clinically more relevant than flexural or tensile loading, since it produces elements of shear, tensile and compressive stresses that often

occur during chewing. For this reason, a shear bond test was considered appropriate for this study.

The findings of the present study show that the mean shear bond strengths for the repair protocols tested ranged from 4.33 MPa (Group 1) to 5.42 MPa (Group 5) with the mean repair bond strength observed for sandblasting followed by the use of the TBF II adhesive system (Group 5) being significantly higher than observed with all other repair protocols. Thus, the null hypothesis was rejected. The shear bond strengths in all adhesive test groups were significantly higher than in the negative control groups, where only mechanical surface treatment was used. The negative control groups served as a baseline for all experimental groups and consistently yielded the lowest shear bond strength values. It is noteworthy that the shear bond strength values observed in this study were lower than those observed for the repair of nanohybrid and nanofilled composites by Rinastiti et al.<sup>55</sup> As previously mentioned, it is difficult, however, to make direct comparisons with the results of other studies given differences in the methodologies used.

The findings of this study suggest that it is necessary to achieve a microretentive composite surface to facilitate effective composite repair. In contrast to the findings of Wendler and coworkers,<sup>28</sup> the superior performance of sandblasting of the substrate surface observed in the present study is in agreement with previous reports from similar studies, using various types of composite substrates.<sup>24,25,27,55,56</sup> This may be attributed to the altered surface characteristics of the composite substrate, which, subsequent to sandblasting, is covered in a layer of small alumina particles, providing both micromechanical retention and sites for chemical adhesion.<sup>57</sup> Following the application of an adhesive bonding system to this layer a chemical bonding occurs with the adhesive resin and the sandblasted composite surface.<sup>58</sup>

In this study, sandblasting of the composite substrate and the use of the self-reinforcing adhesive 10-MDP yielded significantly higher mean repair composite interface bond strength and resulted in more cohesive failures, compared to all other repair protocols used in the study, which showed predominantly adhesive interfacial failures. This finding is in agreement with the findings of other researchers who attribute the higher bonding effectiveness to the more intense and stable chemical bonding of 10-MDP and to its higher etching potential, a combination other functional monomers appear to

lack.<sup>49,50</sup> It has been reported that the presence of non-converted C=C double bonds in the treated substrate layer plays a key role for the adhesion of the repair composite.<sup>28</sup> Furthermore, the use of 10-MDP functional monomer has been shown to increase repair bond strength, which may be attributable to its thixotropic property of deeper infiltration into microretentions created by sandblasting, but also by improved direct chemical interaction with the unreacted C=C double bonds in the composite substrate.<sup>28</sup> In addition, the rate of copolymerization of the repair composite with these unreacted double bond groups has been reported to positively affect the repair bond strength.<sup>28,54</sup> It has also been reported that the solvents used in adhesives influence the wetting ability and bond strength of adhesives.<sup>59</sup> The solvent of 4-META containing GSE is acetone, whereas that of 10-MDP containing TBF II is ethanol.<sup>60</sup> The solution of ethanol and 10-MDP has a better wetting ability than the solution of acetone and 4-META and a higher wetting ability can increase the adhesive substrate surface area.<sup>59</sup> Thus, the improved wetting ability of the ethanol/10-MDP solution may also be attributable to the higher repair bond strength values observed with the TBF II adhesive applied on the sandblasted substrate.

The lack of statistical difference in repair bond strengths between the polished + GSE (Group 1), polished + TBF II (Group 2) and the polished + sandblasted + GSE (Group 4) repair protocols, and the statistically significantly superior results observed with the polished + sandblasted + TBF II (Group 5) repair protocol suggest that composite repair is mostly influenced by chemical rather than mechanical bonding and that the selection of the appropriate adhesive system is essential in achieving the best outcome for composite repairs.

A positive control group was included in the present study, as a reference of cohesive strength representing the optimal repair bond strength.<sup>37</sup> Unlike specimens conditioned with the 4-META containing GSE adhesive, the repair bond strength values of specimens treated with sandblasting of the polished substrate and application of the 10-MDP containing TBF II adhesive system were comparable to the cohesive bond strength of the positive control group, as no significant difference in bond strength was observed between these groups. Thus, the view that bond strengths achieved with composite repair cannot be comparable to the inherent strength of composite is challenged by the findings of this study.

Although no predictions can be made in respect of clinical longevity of repairs affected using the protocols used in this study, the sandblasting and application of TBF II was considered to provide the most favourable results in terms of repair bond strength and the predominantly observed cohesive mode of failure of tested specimens.

The findings of the present study provide opportunity to rationalise the selection of repair protocols for the repair of nanohybrid composite restorations. Although no predictions can be made in respect of clinical longevity of repairs affected using the protocols used in this study, the sandblasting and application of TBF II was considered to provide the most favourable results for the repair of localised defects in composite restorations. It is considered that the findings of this study should be of immediate practical relevance in clinical practice when managing failing composite restorations, particularly where self-etch approach is commonly used and the defect is localised and accessible.

## **Conclusions**

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

1. Composite substrates treated with sandblasting yielded statistically higher bond strength values when compared to non-sandblasted polished substrate surfaces.
2. The use of sandblasting followed by the application of TBF II yielded the statistically highest repair bond strength values, suggesting that this repair protocol may be recommended to achieve the best outcome for composite repairs. Clinical trials involving the implementation of this technique are indicated to determine the clinical usefulness of this technique.
3. The bonding performance observed in composite repairs treated with sandblasting and the 10-MDP containing TBF II adhesive system is comparable to the bond strength values of cohesive composites.

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## References

1. Eltahlah D, Lynch CD, Chadwick BL, Blum IR, Wilson NHF. An update on the reasons for placement and replacement of direct restorations. *J Dent.* 2018; **72**:1-7
2. Blum IR. The management of failing direct composite restorations: replace or repair? In: Lynch CD, Brunton PA, Wilson NHF, editors. *Successful posterior composites*. London: Quintessence Publishing Company; 2008. p. 101–13.
3. Blum IR, Lynch CD, Wilson NH. Factors influencing repair of dental restorations with resin composite. *Clin Cosmet Investig Dent.* 2014; **17**; 6:81-87.
4. Baldissera RA,CorreaMB,SchuchHS,CollaresK,NascimentoGG,JardimPS, et al. Are there universal restorative composites for anterior and posterior teeth? *J Dent* 2013; **41**: 1027–35.

5. Da Rosa Rodolpho PA, Donassollo TA, Cenci MS, LoguercioAD, Moraes RR, Bronkhorst EM, et al. 22-Year clinical evaluation of the performance of two posterior composites with different filler characteristics. *Dent Mater* 2011; **27**:955–63.
6. Demarco FF, Collares K, Coelho-de-Souza FH, Correa MB, Cenci MS, Moraes RR, et al. Anterior composite restorations: A systematic review on long-term survival and reasons for failure. *Dent Mater* 2015; **31**:1214–24.
7. Opdam NJ, Bronkhorst EM, Loomans BA, Huysmans MC. 12-year survival of composite vs. amalgam restorations. *J Dent Res.* 2010; **89**:1063-7.
8. Mjör IA, Shen C, Eliasson ST, Richter S. Placement and replacement of restorations in general dental practice in Iceland. *Oper Dent.* 2002; **27**:17–123.
9. Blum IR, Schriever A, Heidemann D, Mjör IA, Wilson NH. The repair of direct composite restorations: an international survey of the teaching of operative techniques and materials. *Eur J Dent Educ.* 2003; **7**:41-8.
10. Gordan VV, Mjör IA, Blum IR, Wilson NHF. Teaching students the repair of resin based composite restorations: a survey of North American dental schools. *J Am Dent Assoc.* 2003; **134**:317–23.
11. Blum IR, Lynch CD. Repair versus replacement of defective direct dental restoration in posterior teeth of adults. *Prim Dent J.* 2014;**3**: 62–7.
12. Kanzow P, Wiegand A, Schwendicke F. Cost-effectiveness of repairing versus replacing composite or amalgam restorations. *J Dent.* 2016; **54**:41-47
13. Deligeorgi V, Mjör IA, Wilson NHF. An overview of reasons for the placement and replacement of restorations. *Prim Dent Care;* 2001;**8**: 5-11
14. Blum IR, Newton JT, Wilson NH. A cohort investigation of the changes in vocational dental practitioners' views on repairing defective direct composite restorations. *Br Dent J.* 2005; **Suppl**:27-30.
15. CD. Lynch, IR. Blum, KB. Frazier, LD. Haisch, NHF Wilson, Repair or replacement of defective direct resin-based composite restorations: Contemporary teaching in U.S. and Canadian dental schools, *J Am Dent Assoc.* 2012;**143**:157-163.

16. IR. Blum, CD. Lynch, NHF. Wilson, Teaching of direct composite restoration repair in undergraduate dental schools in the United Kingdom and Ireland, *Eur J Dent Edu.* 2012;**16**: e53-58.
17. IR. Blum, CD. Lynch, NHF. Wilson, Teaching of the repair of defective composite restorations in Scandinavian dental schools, *J Oral Rehab.* 2012; **39**: 210-216.
18. CD. Lynch, M Hayashi, LL. Seow, IR. Blum, NHF. Wilson, The management of defective resin composite restorations: current trends in dental school teaching in Japan, *Oper Dent.* 2013; **38**:497-504.
19. Brunton PA, Ghazali A, Tarif ZH, Loch C, Lynch C, Wilson NHF, Blum IR. Repair vs replacement of direct composite restorations: a survey of teaching and operative techniques in Oceania. *J Dent.* 2017; **59**:62–7.
20. Kanzow P, Wiegand A., Wilson NHF. Lynch CD., Blum IR. Contemporary teaching of restoration repair at dental schools in Germany – Close to universality and consistency. *J Dent.* 2018; **75**: 121–4
21. Blum IR, Özcan M. Reparative Dentistry: Possibilities and Limitations. *Curr Oral Health Rep.* 2018; **5**:264-269
22. Kupiec, K.A.; Barkmeier, W.W. Laboratory evaluation of surface treatments for composite repair. *Oper. Dent.* 1996; **21**: 59–62.
23. Brosh, T.; Pilo, R.; Bichacho, N.; Blutstein, R. Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. *J. Prosthet. Dent.* 1997; **77**: 122–126.
24. Bouschlicher MR, Reindhardt JW, Vargas MA. Surface treatment techniques for resin composite repair. *American Journal of Dentistry* 2006; **10**: 279–83
25. Özcan, M.; Barbosa, S.H.; Melo, R.M.; Galhano, G.A.; Bottino, M.A. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. *Dent. Mater.* 2007; **23**: 1276–1282
26. Fawzy, A.S.; El-Askary, F.S.; Amer, M.A. Effect of surface treatments on the tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite. *J. Dent.* 2008; **36**: 969–976.
27. Costa, T.R.; Ferreira, S.Q.; Klein-Junior, C.A.; Loguercio, A.D.; Reis, A.



- Durability of surface treatments and intermediate agents used for repair of a polished composite. *Oper. Dent.* 2010; **35**: 231–237.
28. Wendler M, Belli R, Panzer R, Skibbe D, Petschelt A, Lohbauer U. Repair Bond Strength of Aged Resin Composite after Different Surface and Bonding Treatments. *Materials (Basel)*. 2016 Jul 7;9(7). pii: E547 doi: 10.3390/ma9070547
29. Ritter AV, Sulaiman TA, Altitnichi A, Bair E, Baratto-Filho F, Gonzaga CC, Correr GM. Composite-composite Adhesion as a Function of Adhesive-composite Material and Surface Treatment. *Oper Dent.* 2018 Nov 16. doi: 10.2341/18-037-L. [Epub ahead of print]
30. Rosa WL, Piva E, Silva AF. Bond strength of universal adhesives: A systematic review and meta-analysis. *J Dent.* 2015;**43**:765-76.
31. Cuevas-Suárez CE, da Rosa WLO, Lund RG, da Silva AF, Piva E. Bonding Performance of Universal Adhesives: An Updated Systematic Review and Meta-Analysis. *J Adhes Dent.* 2019;**21**:7-26.
32. Reis A, Leite TM, Matte K, Michels R, Amaral RC, Geraldeli S, Loguercio AD. Improving clinical retention of one-step self-etching adhesive systems with an additional hydrophobic adhesive layer. *J Am Dent Assoc.* 2009; **140**:877-85.
33. Chen C, Niu LN, Xie H, Zhang ZY, Zhou LQ, Jiao K, Chen JH, Pashley DH, Tay FR. Bonding of universal adhesives to dentine--Old wine in new bottles? *J Dent.* 2015; **43**:525-36.
34. Turp V, Sen D, Tucelli B, Oczan M. Adhesion of 10-MDP containing resin cements to dentin with and without the etch- and-rinse technique. *J Adv Prosthodont* 2013; **5**:226-33
35. van Landuyt KL, Snauwaert J, de Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, van Meerbeek B. Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials* 2007; **28**:3757-85.
36. van Landuyt KL, Yoshida Y, Hirata I, Snauwaert J, de Munck J, Okazaki M, Suzuki K, Lambrechts P, van Meerbeek B. Influence of the chemical structure of functional monomers on their adhesive performance. *J Dent Res* 2008; **87**:757-61.
37. Loomans BA<sup>1</sup>, Cardoso MV, Roeters FJ, Opdam NJ, De Munck J, Huysmans MC, Van Meerbeek B. Is there one optimal repair technique for all composites? *Dent Mater.* 2011;**27**:701-9.

38. Lynch CD, Frazier KB, McConnell RJ, Blum IR, Wilson NH. State-of-the-art techniques in operative dentistry: contemporary teaching of posterior composites in UK and Irish dental schools. *Br Dent J.* 2010; **209**:129-36.
39. Lynch CD, Frazier KB, McConnell RJ, Blum IR, Wilson NH. Minimally invasive management of dental caries: contemporary teaching of posterior resin-based composite placement in U.S. and Canadian dental schools. *J Am Dent Assoc.* 2011; **142**:612-20.
40. Wilson NH, Lynch CD. The teaching of posterior resin composites: planning for the future based on 25 years of research. *J Dent.* 2014;**42**:503-16.
41. Loch C, Liaw Y, Metussin AP, Lynch C, Wilson N, Blum IR, Brunton PA, The teaching of posterior composites: A survey of dental schools in Oceania, *J Dent.* 2019; <https://doi.org/10.1016/j.jdent.2019.01.005>
42. Blum IR, Wilson NHF. An end to linings under posterior composites? *J Am Dent Assoc.* 2018; **149**:209-213.
43. Fernández E, Martín J, Vildósola P, Oliveira Junior OB, Gordan V, Mjör I, Bersezio C, Estay J, de Andrade MF, Moncada G. Can repair increase the longevity of composite resins? Results of a 10-year clinical trial. *J Dent.* 2015;**43**:279-86.
44. Moncada G, Martin J, Fernandez E, Hempe MC, Mjör IA, Gordan VV. Sealing, refurbishment and repair of Class I and Class II defective restorations: a three-year clinical trial. *J Am Dent Assoc.* 2009; **140**:425–32.
45. Moncada G, Vildosola P, Fernandez E, Estay J, de Oliveira Junior OB, de Andrade MF, et al. Longitudinal results of a 10-year clinical trial of repair of amalgam restorations. *Oper Dent.* 2015;**40**:34–43.
46. Opdam NJ, Bronkhorst EM, Loomans BA, Huysmans MC. Longevity of repaired restorations: a practice based study. *J Dent* 2012; **40**:829–35.
47. Estay J, Martín J, Viera V, Valdivieso J, Bersezio C, Vildosola P, et al. 12 years of repair of amalgam and composite resins: a clinical study. *Oper Dent.* 2018; **43**:12–21.
48. Valente LL, Sarkis-Onofre R, Gonçalves AP, Fernández E, Loomans B, Moraes RR. Repair bond strength of dental composites: systematic review and meta-analysis *Int J Adhes and Adhesives* 2016;**69**:15-26

49. Wang R, Shi Y, Li T, Pan Y, Cui Y, Xia W. Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin. *J Dent*. 2017;**62**:72-80.
50. Yoshihara K, Hayakawa S, Nagaoka N, Okihara T, Yoshida Y, Van Meerbeek B. Etching Efficacy of Self-Etching Functional Monomers. *J Dent Res*. 2018; **97**:1010-1016
51. Hosoya Y, Tay FR. Bonding ability of 4-META self-etching primer used with 4-META/MMA-TBB resin to enamel and dentine: primary vs permanent teeth. *J Dent*. 2014;**42**:425-31
52. da Silva EM, Poskus LT, Guimaraes JGA. Influence of light- polymerization modes on the degree of conversion and mechanical properties of resin composites: a comparative analysis between a hybrid and a nanofilled composite. *Oper Dent* 2008; **33**:287–93
53. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength – evaluation of a micro-tensile bond test. *Dent Mater* 1994; **10**:236–40.
54. Sau, C.W.; Oh, G.S.; Koh, H.; Chee, C.S.; Lim, C.C. Shear bond strength of repaired composite resins using a hybrid composite resin. *Oper. Dent*. 1999; **24**:156–161.
55. Rinastiti M, Özcan, Siswomihardjo W, Busscher HJ. Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. *J Dent*. 2010;**38**:29-38. doi: 10.1016/j.jdent.2009.08.009.
56. Kaneko M, Caldas RA, Feitosa VP, Xediek Consani RL, Schneider LF, Bacchi A. Influence of surface treatments to repair recent fillings of silorane-and methacrylate-based composites. *J Conserv Dent*. 2015;**18**:242-6.
57. Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. *Journal of Prosthet Dent* 1995; **73**:240–9.
58. Özcan M., Corazza PH, Marocho SM, Barbarosa SH, Bottino MA. Repair bond strength of micrhybrid, nanohybrid and nanofilled resin composites: effect of substrate resin type, surface conditioning and ageing, *Clin Oral Invest*. 2013; **17**:1751-1758.

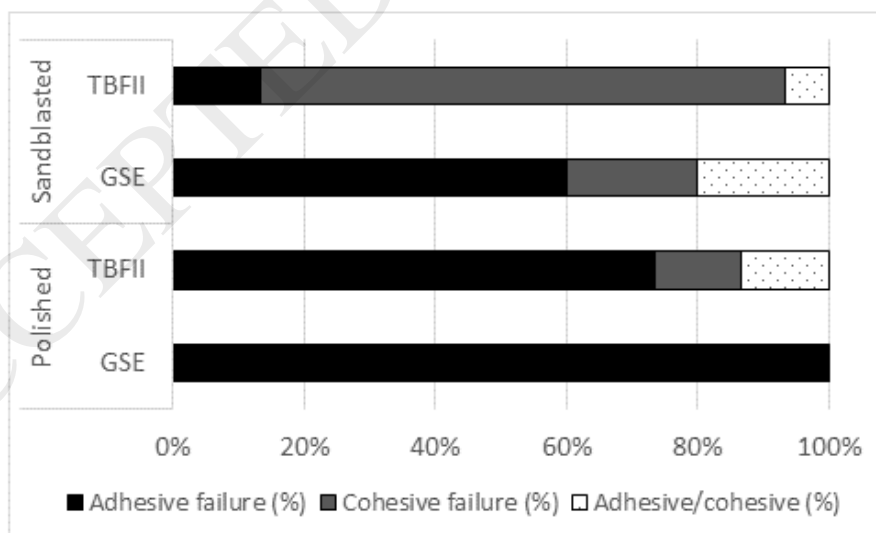
59. Ye Q., Park J, Parthasarathy R, Pamatmat F, Misra A, Laurence JS, Spencer MP, Quantitative analysis of aqueous phase composition of model dentin adhesives experiencing phase separation, *J Biomed Mater Res B Appl Biomater.* 2012;**100**: 1086–1092.

60. Gajewski VE, Pfeifer CS, Fróes-Salgado NR, Boaro LC, Braga RR. Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. *Braz Dent J.* 2012; **23**:508-14.

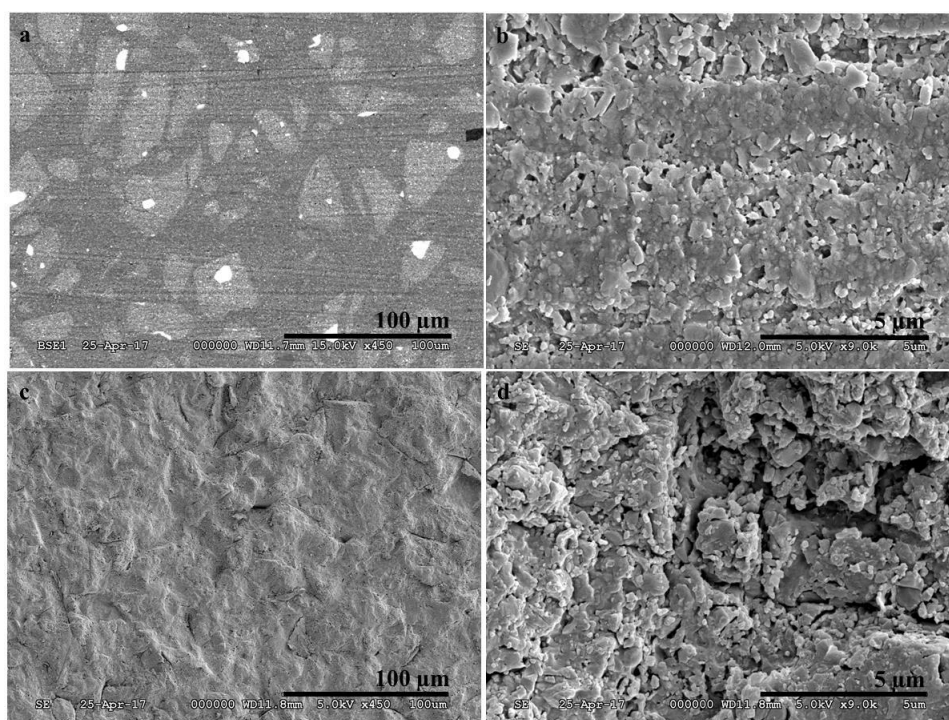
## Figures



**Fig.1** Custom design Teflon mold



**Fig. 2.** Failure mode (%) of the investigated composite samples.



**Fig. 3a-d.** SEM images of polished (a. and b.) and sandblasted (c. and d.) TEC composite surfaces.

**Table 1.** Composite and adhesive materials used in the study

Materials used in this study				
Code	Resin based material	Manufacturer	Components	LOT
TEC	Tetric EvoCeram resin composite	Ivoclar Vivadent Schaan Liechtenstein	<b>Resin:</b> Bis-GMA (Bisphenol A-diglycidyl dimethacrylate), UDMA (Urethane dimethacrylate), Bis-EMA (Ethoxylated bisphenol A dimethacrylate) (17-18% weight) and CQ +Lucirin TPO (2,4,6-Trimethylbenzoyldiphenylphosphine oxide) photoinitiators, stabilisers	V03784 V16179
			<b>Filler:</b> 48.5 m/m% Barium aluminium silicate glass fillers with size is between 40-3000 nm mean particle size of 550 nm 34 m/m% grinded prepolymers with fillers, ytterbium trifluoride, mixed oxide (macro-filler dimension)	
GSE	Gluma Self Etch adhesive system	Kulzer Hanau, Germany	4-META (4-methacryloyloxyethyltrimellitate anhydride), UDMA HEMA (2-hydroxyethyl methacrylate), acetone	010504

TBF II	Tokuyama Bond Force II adhesive system	Tokuyama Dental, Tokyo, Japan	Self-reinforcing 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), Bis-GMA, TEGDMA, HEMA, alcohol, water	027
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Table 2. Comparison of mean repair bond strengths between repair protocols

	Surface conditioning method	Mean SBS (MPa)	95% confidence intervals (MPa)	Statistical groupings
Group 1	Polished+GSE	4.3	3.7-5.0	a,b,d
Group 2	Polished+ TBF II	4.7	4.2-5.3	a,b,d
Group 3 (negative control A)	Polished	1.9	1.5-2.3	c
Group 4	Polished+SB+GSE	4.8	4.2-5.3	a,b,d
Group 5	Polished+SB+ TBF II	5.4	5.0-5.8	e,g,
Group 6 (negative control B)	Polished+SB	3.6	2.8-4.4	f
Positive control group	Repair TEC composite immediately applied	5.7	5.2-6.2	e,g

Lower case letters indicate statistically homogeneous groups. If two data sets share the same letter, they do not differ to a statistically significant degree ( $\alpha=0.05$ ).

**Table 3.** Measured amplitude parameters of surface roughness analysis according to applied surface treatment.

	Ra (nm)	T- test Sig. (2-tailed)	Rq (nm)	T- test Sig. (2-tailed)	Rp (nm)	T- test Sig. (2-tailed)	Rv (nm)	T- test Sig. (2-tailed)
TEC polished	307.59±5.98	0.000	410.85±14.93	0.000	1030.44±30.97	0.027	-1783.70±138.81	0.005
TEC sandblasted	142.40±15.15		179.24±15.15		664.41±102.00		-679.33±19.4	