SHORT THESIS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY (PHD)

# In vitro investigation of universal adhesives and composites for nanohybrid and bulk-fill repair

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## Table of content

# 1. Background of the doctoral thesis

Advancement of adhesive technology and introduction of minimalinvasive concept resulted more conservative treatment modalities in restorative dentistry. In recent years there has been a paradigm shift towards the use of resin-based composites (RBCs) for the direct restoration of both anterior and posterior teeth. RBCs became the most popular restorative material of choice due to the improvements in their compositions and placement techniques resulting a well-balanced tooth-restorative biomechanical unit paralel with strenghtening the remaining tooth structure. However several drawbacks as polymerization shrinkage, limited degree of conversion and limitation in fracture strenght associated with reduced lifespan of resin based restorations in hostile oral environment. Fractures, secondary decay are the two main cause of failures leading to the replacement of restorations in everyday clinical practice. Nowdays a widely accepted protocol is the repair of failing restorations based on the micromechanical roughening of the surface, the presence of unreacted monomers and possibilities of chemical bonding to the exposed filler particles. Due to the diversity of RBCs ideal repair protocol still not exist.

## 1.1 Failure of composite restorations

The minimum intervention approach, the demands of esthetic restorations a shift has been driven that RBCs became the first choice restorative material in most of clinical practices (1–3). Patients' oral environment (4–7) operator skills (8) physical, chemical, mechanical parameters of resin based materials (9–15) the type of adhesives (16,17), size and type the prepared cavity (6,18,19) are the main influencing factors of the restorations' lifespan.
The most common type of failures are microleakage, discoloration followed by secondary decay around the cavity margins and bulk fracture due to the propagation of microcracks (20–22). Follow-up studies reported annual failure rates around 0-7,5 % of composite restorations, 12% of restorations showed increased wear rate after ten years of function and at the same time 50% are being replaced (23,24) representing 50-70% of the activity of general dental practice (25–27) .

## 1.2 Micromechanical and chemical adhesion on the tooth-restoration interface Adhesives

Introduction of resin based materials (28) in a combination with an etching step, and application of low viscosity adhesive material provide the possibility of micromechanical and chemical adhesion towards the tooth structure (29–31) While the protocols and the effectiveness of interaction have been improved by manufacturers and operators the process is still invariably technique sensitive (32).
From the beginning the overall adhesion concept based on the reliable enamel adhesion but on the last ten years turned towards the simplification of clinical steps, besides reducing technique sensitivity, shorten the application time and achieve a well-controlled dentin adhesion (33–37).

## 1.3 Reliability of adhesive systems

The conventional three step etch and rinse approach still considered as the most accurate and durable adhesion, representing the gold standard proved by short and long term results (17). Siplified one or two step adhesives show comparable results in short term with three step adhesives (13,38,39), but their hydrophil nature seems to be one of the major shortcomings in the oral environment (39,40).

Success rate of two-step 10-MDP functional monomer containing adhesives after 8 years (41,42), one step adhesives in short term has been reported to be limited (13). The hydrophilicity of these complex solutions in long term may jeopardize the adhesive interface integrity (39,40).

## 1.4 Concept of composite repair

Failing but still serviceable restorations with fracture, crack formation, discoloration or secondary decay can be extense their longevity by application of repair procedure (43,44). This concept may extend the service time with aproximately 2-7 years (45–48). Refurbishing, repolishing and relayering as an minimally interventive repair concept means a conservative, time and cost effective therapy with lower risk of pulp irritation (49–53)

Basic steps of the intervention is the surface roughening and application of intermedier agent providing a micromechanical and chemical intimate connection between the old and fresh resin based composite (54). The time interval between the placement and the repair of the restoration is seems to be one of the main contributing factor (1,53,55). The 14 days timeframe means immediate repair, when C=C are still available for chemical bonding. In this case the importance of the intermedier layer hypothetically negligible. (55,56). Beyond this period of time the circumstances much more worse, the hydrolysis and degradation of resins should be taken into account (57–59).
The composition of the repaired composite such as the type of resin and filler components is also a critical factor, but unfortunately in most cases these details are unknown and not available for the operators (60–64).
The proper strategy may ensure an effective, durable result in medium and long term (65,66), however data about an universal protocol is still limited.

## 1.5 Objectives

The doctoral thesis gives an overview about the composition of resin based composite materials, adhesive systems and technologies focusing of the background of main clinical failures, and a summary of possibilities of composite repairs highlighting the importance of mechanical surface treatment, quality of surface texture and effectiveness of universal bonding agents.

The primary aim of the first 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 analyze the nature of interfacial failure, using scanning electron microscopy (SEM) and profilometry examinations of failed interfacial surfaces. The primary aim of the second in vitro study was to evaluate the effects of different universal adhesive systems on the repair bond strength of bulk fill composites. The secondary aim was to evaluate differences, if any, in repairability between low and high viscosity bulk fill composites. The tertiary aim was to detect the nature of interfacial failure, using stereo microscopy, profilometry and scanning electron microscopy (SEM) examinations of failed interfacial surfaces.

## 2. Methods and materials

### 2.1 Specimen preparation for shear bond strenght testing

An universal nanohybrid resin composite material (Tetric EvoCeram™, Ivoclar Vivadent, Lichtenstein) was applied in 2mm thickness into the rectangular recess (25mm length x 13mm width x 4mm depth) of the individually fabricated Teflon molds. The layer of resin composite was photo-polymerized in a light oven (Dentacolor XS Kulzer, Germany) for 180 s, operating with a wavelength 320-500 nm (435 nm peak), to achieve maximum polymerization. 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-polymersiation in the light oven for 180 s. Subsequently, with an exception of positive control specimens 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 30 s 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 ± 1.0 °C) for 24 h. 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 resin composite blocks were randomly divided, using randomisation tables, into six equal groups, each of distinct 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 20 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s using the Bluephase 20i (Ivoclar Vivadent, Lichtenstein) hand held LED light cure unit operating at a measured output of 1200 mW/cm2 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 10 s, waited for 10 s, dried with oil-free air/water syringe for 5 s and light cured for 10 s (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 μm $Al_2O_3$ (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-PrepTM, RønvignA/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 and the application of one coating of Gluma Self-Etch adhesive system (Heraeus Kulzer, Hanau, Germany) with circular brushing motion for 20 s, dried for 5 s and photopolymerised for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 5: Air-borne particle abrasion with 50 μm $Al_2O_3$ (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-PrepTM, RønvignA/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 and the application of one coating of Tokuyama Bond Force II adhesive system (Tokuyama Dental, Tokyo, Japan) with circular brushing motion for 10 s, waited for 10 s, dried for 5 s and photopolymerised for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 6: Air-borne particle abrasion with 50 μm $Al_2O_3$ Bego, Bremen, Germany) using an intraoral sandblaster (Dento-PrepTM, RønvignA/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 ± 1.0 °C) for one minute.

## 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 20 s. 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 24 h at 23 ± 1.0 °C room temperature before being subjected to repair bond strength testing.

## 2.4 Specimen preparation for microtensile strenght testing

An incremental layer of 3-4 mm Tetric EvoCeram™ bulk fill (TECBF) (Ivoclar Vivadent, Liechtenstein) was applied to the specimens in half of the individually fabricated Teflon molds (square recess 10 mm length × 10 mm width × 14 mm depth at the centre), whereas SureFil SDR Flow™ bulk fill composite (SDR) (Dentsply Sirona, North Carolina, USA) was applied in similar increments to the remaining half of the molds. The first incremental layer of each bulk fill composite application was photo-polymerized in a polymerization oven (LC-6 Light Oven, Scheu GmbH, Iserlohn, Germany) for 180 seconds prior to the application of the second 3-4mm increment layer of the respective bulk fill composite on the polymerised first layer. The second increment layer of each 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-polymersiation in the light oven for 180 seconds. The light oven was equipped with six fluorescent light tubes (3 UVA and 3 blue light) generating wavelengths between 340 and 420nm, the maximum being 370nm for UV-A and 350-450nm for blue light to achieve maximum polymerization. All molds were incrementally filled and photo-polymerized to a depth of 7mm.
Subsequently, specimens from the TECBF group and specimens from the SDR group were successively removed from the Teflon molds and their top surfaces were consecutively polished with wet 500-grit, 1000-grit and 1200-grit silicon carbide discs using a polishing machine (Struers LaboPol35, Struers A/S, Rodovre, Denmark) at 300rpm for 30s and cleaned for 10min 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 (25 ± 1.0 °C) for 24 h. One group of 15 specimens of each TECBF and SDR served as positive control (PC) groups and the surfaces were not polished.

## 2.5 Surface conditioning methods

The TECBF and the SDR specimens were each randomly divided, using block randomisation, into eight equal sample groups. The eight sample groups of each bulk fill material were further randomly divided into two equal subgroups. Whilst one of the subgroups of each bulk fill material remained polished without any additional surface preparation, the remaining half was subjected to an accelerated ageing in a thermal cycling machine (SD Mechatronik

Thermocycler THE-1100, Germany) for 5000 cycles at 5–55°C with a dwell time of 30s. Subsequently, all non-aged and aged specimens were placed back in their respective Teflon molds and air-dried (25 ± 1.0 °C) for 24 h. The non-aged and aged specimens received the following surface conditioning treatments according to the manufacturers' instructions:

Group 1: (negative non aged TECBF control group): no adhesive system was used between non aged TECBF and TEC.

Group 2: one thin coating of Heliobond adhesive system (Ivoclar Vivadent, Lichtenstein) applied to the non-aged TECBF with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 3: one thin coating of Tokuyama Bond Force II™ adhesive system (Tokuyama Dental, Tokyo, Japan) applied to the non-aged TECBF with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 4: one thin coating of Scotchbond universal adhesive system (3m ESPE, Neuss, Germany) applied to the non-aged TECBF with a disposable applicator and circular brushing motion for 20 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 1A: (negative aged TECBF control group): no adhesive system was used between aged TECBF and TEC.

Group 2A: one thin coating of Heliobond adhesive system (Ivoclar Vivadent, Lichtenstein) applied to the aged TECBF with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 3A: one thin coating of Tokuyama Bond Force II™ adhesive system (Tokuyama Dental, Tokyo, Japan) applied to the aged TECBF with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 4A: one thin coating of Scotchbond universal adhesive system (3m ESPE, Neuss, Germany) applied to the aged TECBF with a disposable applicator and circular brushing motion for 20 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 5: (negative non aged SDR control group): no adhesive system was used between non aged SDR and TEC.

Group 6: one thin coating of Heliobond adhesive system (Ivoclar Vivadent, Lichtenstein) applied to the non-aged SDR with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 7: one thin coating of Tokuyama Bond Force II™ adhesive system (Tokuyama Dental, Tokyo, Japan) applied to the non-aged SDR with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 8: one thin coating of Scotchbond universal adhesive system (3m ESPE, Neuss, Germany) applied to the non-aged SDR with a disposable applicator and circular brushing motion for 20 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group 5A: (negative aged SDR control group): no adhesive system was used between aged SDR and TEC.

Group 6A: one thin coating of Heliobond adhesive system (Ivoclar Vivadent, Lichtenstein) applied to the aged SDR with a disposable applicator and circular brushing motion for 10 s,

waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).
Group 7A: one thin coating of Tokuyama Bond Force II™ adhesive system (Tokuyama Dental, Tokyo, Japan) applied to the aged SDR with a disposable applicator and circular brushing motion for 10 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).
Group 8A: one thin coating of Scotchbond universal adhesive system (3m ESPE, Neuss, Germany) applied to the aged SDR with a disposable applicator and circular brushing motion for 20 s, waited for 10 s, dried with oil-free air/water syringe for 5s and light cured for 10 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).
Subsequently, the Teflon molds in all groups were removed and the specimens were air-dried (25 ± 1.0 °C) for one minute.

## 2.6 Repair resin composite application

The repair resin composite used was the nanohybrid Tetric EvoCeram (Ivoclair Vivadent, Liechtenstein). With the exception of the two positive control groups the repair universal nanohybrid resin composite material was incrementally packed according to manufacturers' instructions into the remaining 7mm height of the 14mm deep central recesses of the Teflon molds and light-cured in a light oven (Dentacolor XS Kulzer, Germany) for 180 s. In the two positive control groups no adhesive was applied and the respective TECBF and SDR bulk fill composites were immediately applied in accordance with manufacturers' instructions to the full 14mm depth of the molds. 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 24 h at 25 ± 1.0 °C room temperature before being subjected to repair bond strength testing.

# 3. Methods

## 3.1 Shear 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.

## 3.2 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.

## 3.3 Profilometry and SEM examination

Another five randomly selected specimens, using a randomisation table, from each test group were examined under three-dimensional high resolution profilometry (Ambios Technology XP-1, Santa Cruz, California, USA) to examine the surface roughness profiles. A Stylus tip radius of 2.0 microns was travelling at a tracing speed of 0.5 mm/s applying a stylus force of 1 mg. 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 analyses statistically using a two-way multivariate analysis of variance (ANOVA) at $\alpha=0.05$.

## 3.4 Statistical analysis

The data were subjected to statistical analysis using a two-way multivariate analysis of variance (ANOVA), two independent sample ttest to analyse the equality of means and the Kolmogorov-Smirnov test at a 95% confidence interval level.

## 3.5 Microtensile bond strength (µTBS) testing

The fabricated block specimens were adhesively secured using a flowable resin composite (M+W Select Permaplast LH viscous flow, M+W Dental, Büdingen, Germany) to a flat polymer support surface prior to being mounted on a hard tissue microtome (Leitz 1600, Leitz, Wetzlar, Germany). Subsequently, the blocks were serially sectioned perpendicular to the interface using the diamond-coated saw (Leitz, Wetzlar, Germany) of the microtome at low speed to obtain sticks with approximate dimensions of 1.0 mm x 1.0 mm x 14 mm. A minimum of 30 sticks were obtained for each group.

All stick specimens were individually mounted on an universal testing machine (Instron 5544, Norwood, Massachusetts, USA) equipped with 2 kN load cell and strength tests were carried out at a 1 mm/min crosshead speed. The applied force (N) was recorded. The µTBS was calculated using the formula: $\sigma=F/A$ where F is the applied force and A is the attached surface area of the samples. The latter was verified using a digital caliper (DC54150, DML digital micrometers, Sheffield, England).

## 3.6 Failure analysis and SEM

Five specimens from each group were randomly selected, using a computer-generated allocation sequence, and their surfaces were examined using a stereo microscope (Olympus SZ61, Tokyo, Japan) at a magnification of 45x to determine their mode of failure. Mode of failure was recorded as 'adhesive' (failure at the adhesive interface), 'cohesive' or 'mixed failure' (more than one type of failure). Subsequently, specimens were examined under SEM. The surfaces of 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.

## 3.7 Profilometry examination

The another five randomly selected specimens, using a randomisation table, from the aged and non-aged test groups were examined under three-dimensional high resolution profilometry

(Ambios Technology XP-1, Santa Cruz, California, USA) to examine the surface roughness profiles. A Stylus tip radius of 2.0 microns was travelling at a tracing speed of 0.5 mm/s applying a stylus force of 1 mg. 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 a significance level of 0.05.

### 3.8 Statistical analysis

The Kolmogorov-Smirnov test was performed to verify normal distribution of bond strength values within the various groups. The Levene's F-test was used for variance homogeneity. The data were normally distributed. Multiple comparison procedures were performed using the two-way ANOVA test followed by Tukey's post-hoc HSD test to determine any significant differences in μTBS between the bulkfill groups, artificial ageing, adhesion protocols, and the occurrence of interaction between these variables in the positive and negative control groups against the experimental repair protocols. The independent t-test was applied to compare the μTBS values for the different bulkfill composites and for the profilometry analyses. The Welch's t-test was applied where different variances were observed in the profilometry groups. The assessment of the equality of variances for the profilometry groups was carried out using Levene's F-test. All statistical tests were carried out at a set significance level of 0.05. Statistical analysis was carried out using IBM SPSS Statistics V22 (Statistical Package for Social Science Inc, Chicago, Illinois, USA).

## 4. Results

### 4.1. Bond strength data of shear bond strenght test

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 ± 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 ± 0.55 MPa) and the use of GSE following sandblasting (4.79 ± 0.54 MPa) ($p=0.061$) or polishing (4.34 ± 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 ± 0.36 MPa) and the positive control group (5.66 ± 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 ($p < 0.01$).

### 4.2. Failure analysis, Profilometry, SEM

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%). 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

## 4.3 Bond strength data of microtensile bond strength test

The bond strength values of specimens treated with adhesive techniques yielded significantly higher bond strength values comparedto the non-aged and aged negative control groups ($p < 0.01$). The Tokuyama Bond Force II™ (TBF II) and Scotchbond™ universal (SBU) adhesive systems consistently resulted in significantly higher bond strength values than the use of the Heliobond™ (HB) adhesive system inall surface conditioned groups irrespective of the type of bulk fill RBC substrate ($p < 0.01$). No significant differences in bond strength values were observed between repaired non-aged TECBF specimens treated with TBF II (42.07 ± 1.76 MPa) and SBU (42.43 ± 1.26 MPa) ($p = 0.519$) and between aged TECBF repair groups of TBF II (39.46 ± 1.81 MPa) and SBU (38.12 ± 1.79 MPa) ($p = 0.051$). There was no significant difference between the non-aged SDR specimens treated with TBF II (46.33± 1.95 MPa) and SBU (46.8 ± 1.43 MPa) ($p = 0.458$), and between aged SDR repair groups conditioned with TBF II (43.27 ± 1.62 MPa) and SBU (43.51 ± 2.17 MPa) ($p = 0.731$). Whilst ageing did not result in a significant difference in the TECBF and SDR groups treated with TBF II and SBU, ageing resulted in significant lower bond strengths values in both Bulk fill groups conditioned with HB (TECBF, $p<0.001$; SDR, $p <0.001$). The Tukey's test did not show statistically significant differences in bond strength between non-aged SDR specimens conditioned with TBF II ($p = 0.12$) and SBU ($p = 0.27$) compared to the positive SDR control group. Marginal significant differences were noted in bond strength ($p<0.05$) and SBU ($p <0.05$) compared to the positive TECBF control group.

## 4.4. Failure analysis, Profilometry, SEM

The surfaces of five randomly selected specimens from each test group and the positive and negative control groups were examined using optical microscopy to investigate the mode of failure and by SEM examination to investigate the surface morphology of the failed surfaces. Stereo microscopy examination showed that positive control specimens of both bulkfill composites showed 100 % cohesive failures, whilst with the exception of the negative non aged SDR group (Group 5) all negative control groups exhibited exclusively adhesive failures. TECBF specimens treated with SBU (Groups 4 and 4A) showed 66.67 % cohesive failures in non-aged specimens, and 53.33 % cohesive failures in aged samples. Of the TECBF specimens treated with the TBFII adhesive system, cohesive failure was observed in 46.67 % in non-aged (Group 3) and 33.33 % in aged (Group 3A) specimens. In the TECBF test groups the greatest cohesive bond strength (66.67 %) was observed in the non-aged SBU group (Group 4) followed by the aged SBU group (53.33 %, Group 4A). The non-aged and aged SDR specimens treated with SBU showed cohesive failures of 53.33 % (Group 8) and 33.33 % (Group 8A) respectively. In the non-aged and aged SDR specimens treated with TBFII cohesive failure was 33.33 % (Group 7) and 40 % (Group 7A) respectively. In both bulkfill groups the use of HB resulted predominantly in adhesive failures. The failure mode of aged TECBF treated with HB (Group 2A) was comparable to the non-aged control groups of TECBF (Groups 1 and 1A). There was strong evidence that the SDR specimens provided a more irregular and rougher surface finish than the TECBF specimens ($p < 0.005$). SEM examinations have confirmed these findings. In contrast to theTECBF groups, no significant difference was found between the aged and non-aged SDR specimens.

## 5. Conclusions

Within the limitations of this study, the following conclusions can be drawn:
1. Nanohybrid composite (TEC) substrate treated with sandblasting yielded statistically higher bond strength values when compared to non-sandblasted polished up to 500 Grit substrate surfaces.
2. The use of sandblasting followed by the application of TBF II containing universal adhesive (TBF II) yielded the statistically highest repair bond strength values, suggesting that this repair protocol may be recommended to achieve the best outcome for nanohybrid composite repairs.
3. The bonding performance observed in nanohybrid composite (TEC) repairs treated with sandblasting and the 10-MDP containing universal adhesive system (TBF II) is comparable to the bond strength values of cohesive composites.
4. Bulk-fill RBC (SDR, TECBF) treated with universal adhesive systems containing a functional monomer 10-MDP (TBF II, SU) yielded statistically higher bond strenght values when compared to non-functional containing adhesive.
5. The viscosity of bulk-fill RBC (SDR, TECBF) does not influence repair bond strenght values, suggesting both low and high viscosity RBCs are amenable to successfull repair using functional monomers to achieve the best outcome.
6.The repair bond strenghts obeserved in bulk-fill RBC (SDR, TECBF) repairs treated with the 10-MDP containing universal adhesive systems (TBFII, SU) is comparable to the bond strenght values of cohesive RBCs.

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## Keywords

resin based composite, bulk-fill composite, universal adhezives, 10-MDP, 4-META, air-abrasion, composite repair

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## List of publications related to the dissertation

1. Blum, I. R., **Martos, R.**, Szalóki, M., Lynch, C. D., Hegedűs, C.: Effects of different surface treatments and adhesive self-etch functional monomers on the repair of bulk fill composites: a randomised controlled study.
   *J. Dent. 108*, 1-9, 2021.
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2. **Martos, R.**, Hegedűs, V., Szalóki, M., Blum, I. R., Lynch, C. D., Hegedűs, C.: 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.
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## List of other publications

3. Szalóki, M., Hegedűs, V., Fodor, T., **Martos, R.**, Radics, T., Hegedűs, C., Dezső, B.: Evaluation of the Effect of the Microscopic Glass Surface Protonation on the Hard Tissue Thin Section Preparation.
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4. Suta, P., Márton, I., D. Tóth, E., **Martos, R.**: Sterilizálási lehetőségek a fogászatban: rugalmas anyagok csírátlanításának korlátai egy eset kapcsán.
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