

ORIGINAL RESEARCH ARTICLE

Evaluation of repairability of different resin composites after pH and thermal cycling

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Abstract

Purpose: The purpose of this study was to investigate the effect of different aging methods on repair bond strength of different types of composite resins.

Materials and Methods: Thirty resin composite samples (5mm×5mm×4mm) were built up with a nanohybrid resin composite and a bulk-fill resin composite by using a silicone matrix (n=30). Indirect resin composite samples were obtained with similar dimensions from prefabricated blocks by a low-speed saw. All the samples were air abraded with 50µm aluminum oxide particles. Specimens were then repaired with a nanohybrid resin composite using two step adhesive system. All these specimens were then randomly divided into three groups for aging process (pH cycle, thermal cycle and control group) and tested for shear bond strength. Results were recorded in MPa. Additionally, fractured specimens were examined under stereomicroscope to determine the mode of failure. The data was statistically analyzed using two-way ANOVA and Bonferroni correction test (p<0.05).

Results: Statistically significant effect was found on the bond strength values of the aging method and the restorative material (p<0.05). Thermal cycle applied samples showed significantly lower bond strength values than both pH cycle and control group samples (p<0.05). Regardless of the aging method, the overall bond strength of bulk fill resin composite is significantly higher than bond strength of indirect resin composite (p<0.05).

Conclusions: Thermal cycle application is an effective aging method and air abrasion has different effects on repair bond strength of the restorative materials according to the content of the materials. In this in vitro study, nanohybrid resin composite and bulk-fill resin composite showed better repairability.

Key words: Air abrasion; Bulk-fill resin composite; Indirect resin composite; pH cycle; Thermal cycle

Introduction

The use of composite resins in restorative dentistry has become a popular practice with the developments in the mechanical and physical properties of adhesive technologies, polymerization systems and resin systems. Although newly developed composite resins are superior to previous generations in terms of wear resistance and color stability, main deficiencies such as polymerization shrinkage which is 1.5% to 3% of the total material volume still remain as a major problem.^{1,2} Shrinkage causes stresses that can go beyond the adhesive and cohesive forces of restorative materials.² The mass to be polymerized in the posterior cavities is so large that shrinkage forces, despite particularly careful application, produce cavities and marginal defects in the cervical margins localized to the dentin.³ This causes microleakage which can subsequently produce secondary caries, pulpal inflammation, postoperative sensitivity and marginal discoloration.⁴

Incremental technique is highly advised to reduce shrinkage stress.⁵ In this approach, multiple composite resin layers with thickness of 2 mm of the prepared cavity for its restoration and light irradiation time to each layer, which can vary between 20 seconds and 40 seconds. This technique takes time and requires attention especially in the posterior region, may increase possible gaps between the layers and the risk of interlayer contamination due to the increase in the time required for the restoration.^{6,7}

Recently, bulk-fill composite resin materials used in layers of 4–5 mm have entered restorative dentistry applications in order to simplify the application procedure of composite resins, shorten the application time, minimize the negative effects of polymerization-induced shrinkage stress and improve light penetration.^{8–10} Low polymerization shrinkage and greater depth of polymerization of bulk fill composite resins are achieved by optimization of the initiator system (new polymerization initiators or higher concentrations of conventional polymerization initiators), modifications of the

filler system (larger fillers or more transparent fillers) or the inclusion of different chemicals in its content.¹¹

It can be said that the treatment approaches applied with indirect composite resin restorations for larger restorations such as inlay, onlay and overlay in teeth with excessive material loss are better alternative treatment options compared to direct composite resin restorations. In order to enhance both their physical and mechanical properties, first and second generation indirect composite resins were developed in the 1960s and 1990s. The composition of first generation indirect composite resin was similar to that of the direct composite resins. In 2nd generation indirect composite resins, it has been tried to increase the mechanical strength of these materials and to decrease the polymerization shrinkage by increasing the micro-hybrid filler content and decreasing the amount of organic matrix.¹² Indirect composite resin restorations are used to overcome the problems like polymerization shrinkage, insufficient mechanical properties of materials and wear resistance seen in direct composite resin restorations. At the same time, indirect composite resin restorations have less postoperative sensitivity, more appropriate interproximal contact, better anatomical morphology, more accurate marginal integrity, better polishability, better color stability than direct composite resin restorations. Even in the deepest parts of the cavity, fully polymerized restorations with longer clinical life can be obtained.^{3,12–16}

Although significant improvements in physical and mechanical properties have been achieved with such materials and good clinical performance has been reported over the last 10–20 years, the biggest problems are still related to wear, discoloration or fractures over time.^{16,17} In most situation, repair of composite resins with direct composite resins is preferred because it is less costly, less time-consuming, less post-sensitive and more conservative compared to complete replacement of the restoration.^{17–19}

Many surface conditioning methods depends on chemical, physical or physico-chemical adhesion principles have been developed for the bonding of composite resins to surfaces other than dental tissue. In physical shaping methods, surface roughening is performed using diamond burs, air abrasion lasers, acidified phosphate fluoride hydrofluoric acid and phosphoric acid, while chemical shaping methods require the use of silane and/or adhesive resins.^{18–23}

Hydrofluoric acid increases the surface roughness by dissolving the filler particles of the old resin composite. It has been shown that silanization and sandblasting increase the bond strength of the repaired composite without etching with hydrofluoric acid, avoiding the use of hazardous and highly corrosive hydrofluoric acid.^{16,24} Surface roughening with phosphoric acid or hydrofluoric acid alone was not found sufficient to create an effective bond in the repaired indirect composite.²⁵

Artificial aging methods such as thermal cycle, soaking in water, immersion in citric acid used in *in vitro* studies to imitate the oral environment has an important role in repairing composite resins.^{26–29} Temperature changes in the thermal cycle not only weaken the physicochemical properties of composite resins, but also reduce the amount of unreacted double bonds within the composite or on the composite surface, that can affect the composite-composite repair strength.³⁰ There is insufficient knowledge about the effect of pH cycling on the bond strength and the degradation of the bonding agent on dental tissue, but there is no study about the repair bond strength of composite resins.^{31–33}

Therefore, the purpose of this study is to investigate the effects of pH and thermal cycling on the repair bond strength of different air-abraded composite resins. The null hypotheses of present study were that pH and thermal cycling would show similar effect on the repair bond strength of different air-abraded composite resins.

Material and Methods

The study design and the materials used in the study are both shown in Table 1 and in Figure 1.

Preparation of samples

30 composite resin samples were prepared from A2 color nanohybrid composite resin (Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) using 4 mm deep silicone matrix according to the manufacturer's instructions. Each layer of the composite resin placed with the layering method in 2 mm thickness was cured with a 20 sec light device (Elipar Freelight 2, 3M ESPE, Germany). 30 composite resin samples were prepared from IVA colored bulk fill composite resin (Tetric N-Ceram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein) in a single layer of 4 mm thickness and cured with a 20 sec LED light device. To obtain a whole polymerization, additionally 20 seconds of light was applied to the surfaces of the composite resins removed from the silicone matrix in contact with the matrix. 30 indirect composite resin samples (Tetric CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut with a low speed saw (Microcut Precision Cutter 201, Metkon, TURKEY) of the same dimensions and obtained from prefabricated blocks. After that, samples were kept in distilled water at 37°C for 1 week. The surfaces of the composite samples obtained were grinded under water with silicon carbide grinding paper (320-grit) and then ultrasonically cleaned for 3 minutes using distilled water.

Air abrasion application

Air abrasion was applied with 50 μ m Al₂O₃ particles at a 90° contact angle at a pressure of 2.5 bar for 15 seconds at a distance of 10 mm from the sample surface of each sample.

Restoration of the specimens

A two-stage self-etch adhesive system (Clearfil SE Bond, Kuraray, Okayama, Japan) was applied to the surfaces of all samples. The primer of the adhesive system was applied to the surface with an applicator for 20 seconds, after that air-dried for 5 seconds. Then the adhesive was applied and thinned with air and light-cured for 10 seconds.

In order to distinguish between the newly made repair material and the filling material, the nanohybrid resin composite (Clearfil Majesty Posterior) in a different shade (A3 shade) was placed in cylindrical transparent matrices with a diameter of 3.5 mm with 2 mm thickness incremental technique, and each layer of the placed resin composite was light-cured for 20 s with the LED light curing unit.

Aging of the specimens

In order to apply the aging procedure, the prepared composite samples were divided into three equal groups with 10 repaired composite samples in each group. While pH cycle was applied to one of the groups and thermal cycle was applied to the other, no aging procedure was performed to the last group to form the control group (Figure 1).

Preparation of pH cycle solutions and pH cycling

The specimens to be applied pH cycling model that is used by Vieira et al.³⁴ for the cariogenic challenge were immersed in remineralizing solution [1.5 mmol/L Ca(NO₃)₂·4H₂O, 0.9 mmol/L Na₂HPO₄·2H₂O, 150 mmol/L KCl, 0.02 mol/L Tris buffer, 0.05 ppm

F, pH 7.0] for 18 h (30 mL per specimen) and in demineralizing solution [2.0 mmol/L Ca(NO₃)₂·4H₂O, 2.0 mmol/L Na₂HPO₄·2H₂O, 75 mmol/L acetate buffer, 0.04 ppm F, pH 4.7] for 6 h (30 mL per specimen) at 37°C. These applications were continued for 5 days. After this procedure, specimens were kept in remineralizing solution two days more. The solutions were renewed daily.³⁴ (Figure 2).

Thermal cycle application

Specimens in this group was subjected to the thermal aging in distilled water between 5°C and 55°C for 500 cycles with a dwell time of 30s and a transfer time of 5s.

Shear bond strength test

Shear bond strength was measured with a universal test device (Mod Dental, Esetron Smart Robototechnologies, Ankara, Turkey). Before connecting the samples to the test device, necessary data such as the maximum load (500N), restoration surface area (9.62 mm²) and the crosshead speed (0.5 mm/min) were entered into the software. SBS values were recorded automatically in megapascal (MPa).

The failure modes were observed under a stereomicroscope at ×20 magnification (Olympus SZ61; Olympus Optical Co, Tokyo, Japan). Failure modes were categorized as adhesive failure between the restorative material and the repair material, cohesive failure in the restorative material, cohesive failure in the repair material and mixed failure.

Statistical analysis

IBM SPSS Statistics 26.0 (IBM Corp. Released 2019. IBM SPSS Statistics for Windows, Version 26.0. Armonk, NY: IBM Corp.) and MS-Excel 2016 programs were used for statistical analysis and calculations. Statistical hypotheses were evaluated by taking the Type-I error level as $\alpha=0.05$.

The conformity of the variables in the study to the normal distribution was evaluated graphically and with the Shapiro-Wilk's test. Mean ± Standard Deviation (SD) was used to display the descriptive statistics of the variables determined to have a normal distribution. Two-way ANOVA was used to assess the effect of the interaction of aging methods (thermal cycle, pH cycle, and control group) and restorative materials on bond strength values. One-way ANOVA was performed to evaluate the difference between restorative materials and aging methods (Thermal cycle, pH cycle and control group) and between aging methods (Thermal cycle, pH cycle and control group) for each restorative material (Tetric N-Ceram Bulkfill, Tetric N-Ceram and Tetric CAD). In case of a difference between them, Bonferroni corrected pairwise comparison results were analyzed.

Results

Statistically significant effect on the bond strength values of aging method ($p < 0.001$) and restorative material ($p = 0.026$), but the interaction of the aging method and restorative material has no statistically significant effect on bond strength values were found ($p = 0.531$) and two-way ANOVA results are also shown in Table 2.

Regardless of the materials used, the total thermal cycle applied samples showed significantly lower bond strength values than both pH cycle ($p = 0.010$) and control group samples ($p < 0.001$) (Table 3). Regardless of the aging method (thermal cycle, pH cycle and control group), the overall bond strength of Tetric N-Ceram Bulk Fill is statistically similar to bond strength of Tetric N-Ceram ($p > 0.05$), but significantly higher than bond strength of Tetric CAD ($p = 0.045$). Bond strength values of Tetric N-Ceram are statistically similar to

both Tetric N-Ceram Bulk Fill and Tetric CAD ($p > 0.05$) (Table 4).

When the aging methods for each material are examined, the bond strength of Tetric N-Ceram Bulk Fill in the thermal cycle group showed significantly lower values than the control group ($p = 0.032$). The bond strength values of Tetric N-Ceram in the thermal cycle group are significantly lower than both the pH cycle ($p = 0.019$) and control groups ($p = 0.003$). For Tetric CAD, no statistically significant difference between aging methods was found ($p > 0.05$) (Table 5).

It was seen that the highest cohesive failure in the restorative material was in the Tetric N-Ceram control group, the highest adhesive failure in the pH and thermal cycled Tetric CAD restorative material group, the highest mixed failures in the pH and thermal cycled Tetric N-Ceram Bulk Fill material and the thermal cycled Tetric CAD restorative material group (Figure 3).

Discussion

In the present study, the effect of different aging methods on repair bond strength of different types of composite resins was analyzed. The null hypotheses of this study were rejected because thermal cycling had a significant effect on the repair bond strength of different air-abraded composite resins that wasn't similar to pH cycling.

Composite resins subjected to the oral conditions reach the highest water saturation and age due to softening of the matrix, formation of microcracks, deterioration of the resin, and dissolution of the filler-matrix interface bonds. Thus, the free radical activity finishes and the oxygen inhibition layer disappears.³⁵ Higher conversion rate of indirect composite resin may also compromise the repair procedure.³⁶ Therefore, the old composite must be activated chemically or physicochemically. Successful bonding between the new and old composite resin can be achieved by three mechanisms: chemical bond with the organic matrix and chemical bond with the exposed filler particles, and micromechanical bonding to treated surface.¹² Chemical bond with the organic matrix is based on unreacted C=C double bonds that remained on the surface of the old composite resin.³⁷ Additionally, it had been reported that wettability of adhesive and surface energy increase with the application of roughening methods.^{17,18,38} For this purpose, there are many studies suggesting the use of air abrasion when repairing of indirect and direct composites.^{12,17,18,23,37-42} In our study, air abrasion was applied to sample surfaces in order to increase surface roughness of the composites, increase surface energy and wettability of the adhesive, and provide micromechanical bonding.

It is stated that composite resins containing high organic matrix content are more susceptible to the air abrasion technique compared to composites with a high filler content, and this effect is influenced by the content of the resin material (for example, alumina filler particles and/or zirconia versus glass) or the condition of the composite resins (new or old).²³ In our study, statistically significant difference was found between the materials used in terms of shear bond strength, regardless of pH and thermal cycle application. Unlike other materials, Tetric N-Ceram has a higher filler ratio, Tetric N-Ceram Bulk Fill contains a germanium-based photoinitiator (Ivocerin) that produces at least two free radicals to initiate the polymerization initiation process, and is more transparent (15%), providing more light penetration and greater depth of polymerization, and the fact that Tetric CAD contains TEGDMA apart from Bis-GMA Bis-EMA UDMA are the differences between the materials. We believe that these differences in material content cause the materials to be affected in different ways by air abrasion application.

Composite resin failures requiring repair often occur when the composite resin ages in medium or long-term oral environments.¹⁰ In laboratory studies, aging methods such as thermal cycle, immersion in water and immersion in citric acid were used to age composite resin materials to mimic the oral environment.^{23,26-29} In addi-

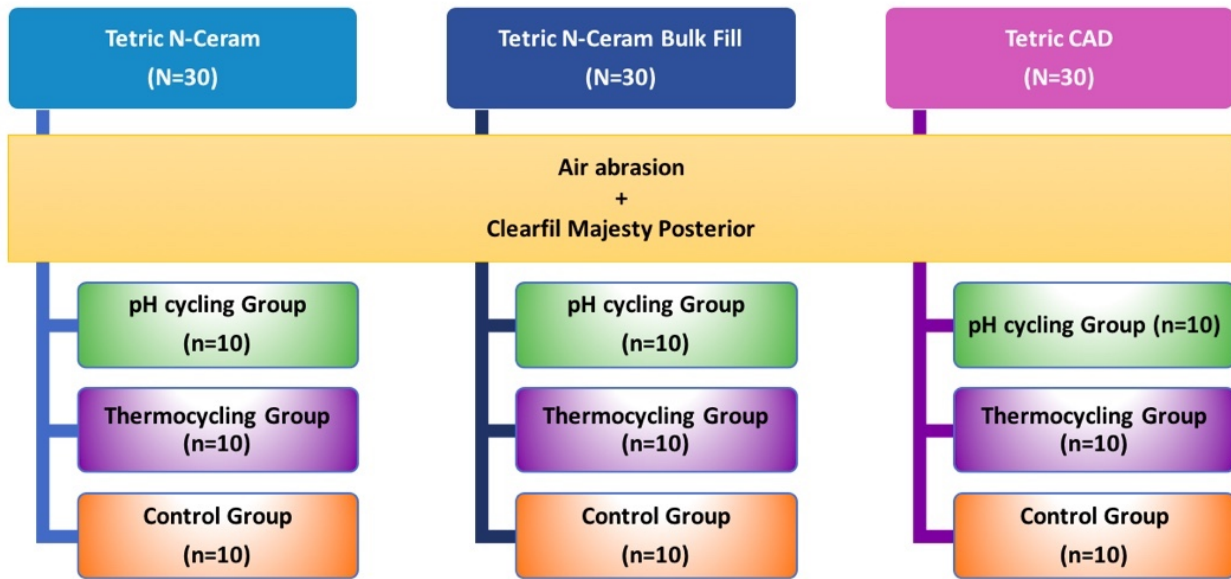


Figure 1. Preparation of the samples and procedures applied in the study.

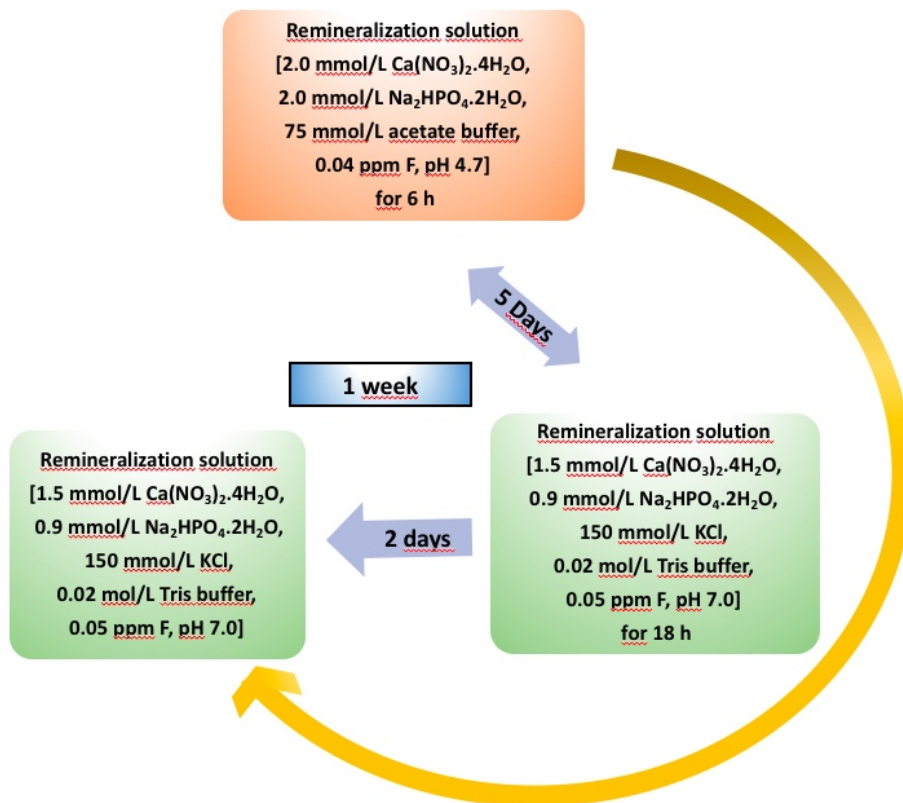


Figure 2. Application time and ingredients of the solutions used in the study.

tion to weaken the physicochemical properties of composite resins, temperature changes in the thermal cycle can also reduce the number of unreacted double bonds by creating thermal stress within the composite (filler-matrix interface) or on the composite surface, which is affecting the bond strength of composite-composite repair.³⁵ In our study, 500 thermal cycles were applied, and it was observed that the materials to which the pH cycle was applied were significantly lower than the repair shear bond strengths and had a

negative effect on the shear bond strength in accordance with other studies.^{23,43} This can be explained by the decrease in bond stability at the adhesive interface between direct and indirect restorations and repair materials due to continuous stress on bonding interfaces caused by temperature changes. Since there is insufficient information about the effect of pH cycling models on the bond strength and the destruction of the bonding agent, which have been successfully used to evaluate artificial caries formation or remineraliza-

Table 1. Materials used in the study and their contents.

Material Brands	Manufacturer	Lot Number	Composition
Tetric N-Ceram (A2) Nanohybrid resin composite	Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany	X55312	ethoxylated Bis-EMA, UDMA, Bis-GMA 18.8 wt% Ytterbium trifluoride, Barium glass filler, Mixed oxide 63.5 wt%, Polymer 17.0 wt%, Catalysts, Additives, Pigments and Stabilizers 0.7 wt% Total filler: 80–81 wt%, 55–57 vol%
Tetric N-Ceram Bulk Fill (IVA) Nanohybrid resin composite	Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany	V34862	UDMA, Bis-GMA, Bis-EMA, 21.0% Ytterbium trifluoride, Barium glass filler 17.0% Mixed oxide 61.0% Initiators (Ivocerin), Stabilizers, Pigments, Additive 1.0% Total filler: 75–77 wt%, 53–55 vol%
Tetric CAD (MT A2/C14) Nanohybrid CAD/CAM resin composite block	Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany	X46886	Barium glass filler 64.0 wt % Silicon dioxide 7.1 wt% Bis-EMA, TEGDMA, Bis-GMA, UDMA 28.4 wt% Additives & Pigments 0.5 wt% Total filler: 71.1 wt%, 51 vol%
Clearfil SE Bond	Kuraray, Tokyo, Japan	000261	Primer: HEMA, Hydrophilic, MDP Dimethacrylate, water, Camphorquinone Adhesive: HEMA, MDP, Bis-GMA, N, N diethanol p-toluidine, Hydrophobic Dimethacrylate, Silanated colloidal silica, Camphorquinone
Clearfil Majesty Posterior (A3) Nanohybrid resin composite	Kuraray, Tokyo, Japan	790044	Surface treated alumina micro-filler, Glass ceramics, Silica filler Bis-GMA, Hydrophobic aromatic dimethacrylate, TEGDMA Total filler: 92 wt%, 82 vol%

Abbreviations: Bis-GMA: Bisphenol A-glycidyl methacrylate, Bis-EMA: ethoxylated bisphenol-A dimethacrylate, UDMA: Urethane dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, MDP: 10-methacryloyloxy-decyl-dihydrogen-phosphate, TEGDMA: Triethylene glycol dimethacrylate.

Table 2. Two-way ANOVA results (MPa)

Variation source	Mean of squares	F	p
Aging method	57.746	8.567	<0.001
Restorative material	25.634	3.803	0.026
Aging method * Restorative material	5.362	0.796	0.531

Table 3. The effect of aging methods on the shear bond strength.

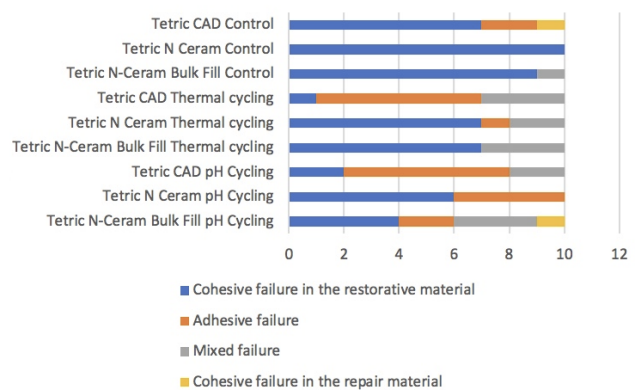
	MPa (Mean ± SD)	F	p
pH cycle	23.89 ± 2.45a		
Thermal Cycle	21.82 ± 2.73b	8.120	0.001
Control	24.45 ± 2.80a		

Abbreviations: MPa: megapascal, SD: standard deviation Values with different superscripts indicate the difference between groups. p value was considered significant at the 0.05 level.

Table 4. The effect of restorative material on the shear bond strength.

Restorative material	MPa (Mean ± SD)	F	p
Tetric	24.15 ± 2.41a		
N-Ceram Bulk Fill			
Tetric N-Ceram	23.65 ± 2.90a,b	3.266	0.043
Tetric CAD	22.36 ± 3.06b		

Abbreviations: MPa: megapascal, SD: standard deviation Values with different superscripts indicate the difference between groups. * p value was considered significant at the 0.05 level.

Distribution of failure modes

Figure 3. Distribution of failure modes.

tion,^{31–33} there is no study about the repair bond strength of resin composites, as they may have different effects on the degradation of composite resins. Therefore, we used the pH cycle as a second aging method in our study. Regardless of the material used in total, the shear bond strength values of the thermal cycle applied samples were significantly lower than the pH cycle applied and control group samples. The pH cycle model used in our study was applied for 1 week. We think that the negative effects of the demineralization solution with a pH of 4.7 on the composite materials and adhesive interface connections in the short-term applied pH cycle model are neutralized with the pH of 7 of the remineralization solution, so that it does not affect repair bond strength of materials as much as thermal cycle used in our study. We believe that a clearer effect will emerge when the long-term pH cycling model is applied.

Table 5. Shear bond strength of restorative materials according to aging methods

Restorative material	Aging method	MPa (Mean ± SD)	F	P
Tetric N-Ceram Bulk Fill	pH cycle	24.49 ± 1.83ac	3.935	0.032
	Thermal Cycle	22.64 ± 2.21a		
	Control	25.33 ± 2.50bc		
Tetric N-Ceram	pH cycle	24.42 ± 1.74b	7.825	0.002
	Thermal Cycle	21.26 ± 2.51a		
	Control	25.27 ± 2.80b		
Tetric	pH cycle	22.76 ± 3.29a	0.493	0.616
CAD	Thermal Cycle	21.56 ± 3.43a		
	Control	22.76 ± 2.54a		

Abbreviations: MPa: megapascal, SD: standard deviation Values with different superscripts indicate the difference between groups in each restorative material. * p value was considered significant at the 0.05 level.

In our study, although statistically significant difference between pH and thermal cycle for Tetric N-Ceram ($p < 0.05$) was found, there were no statistically significant differences observed for other materials ($p > 0.05$). When compared to the control group, it was seen that thermal cycle applications caused statistically lower bond strength values in both Tetric N-Ceram Bulk Fill and Tetric N-Ceram samples. We think that Tetric N-Ceram, which has a higher filler content than other materials, may have been more affected by the thermal cycle application and more filler particles may have been separated from its organic matrix than other materials. The possible effects of the thermal cycle on Tetric N-Ceram may be in the form of swelling of the organic matrix in the composite due to water absorption, microcracks formation with sudden temperature changes, resin destruction and separation of the filler particles by breaking the silane layer on the filler particles surface or the organic matrix-filler interface of the composite.³⁵

It has been shown that composite repair shear bond strength values greater than 18 MPa or between 20–25 MPa are clinically acceptable against occlusal loads.^{44–46} All experimental groups in our study reached clinically acceptable bond strength values higher than 20 MPa against occlusal loads.

In our study, the fact that the highest adhesive failure was observed in pH and thermal cycle applications only in the Tetric CAD restorative material group, this material, unlike other materials, contains TEGDMA, which is a hydrophilic monomer, and causes more water absorption than other materials and the aging methods used in this study we think that it causes stress on the adhesive bond interface in restorative material group.³⁵ Therefore, we can say that additional applications such as silica coating systems or silane application are needed to prevent adhesive failure and to provide cohesive failure in this material.¹⁶

Conclusion

As a result of this study, it was seen that thermal cycle application is an effective aging method and air abrasion has different effects on repair bond strength of the restorative materials according to content of the materials. Short time application of pH cycle as an aging method can be considered as a limitation of our study. Therefore, we believe that the pH cycle should be applied for longer periods in order to clarify the effects of the restorative materials on the repair bond strength.

Author Contributions

Concept: G.D.A., B.Y., Design: : G.D.A., B.Y., Data Collection or Processing: : G.D.A., B.Y., E.B., Analysis or Interpretation: G.D.A., B.Y., E.B., Literature Search: G.D.A., B.Y., Writing: G.D.A.

Conflict of Interest

Authors declare that they have no conflict of interest.

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