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Effects of Artificial Aging of Direct Resin Nano-Hybrid Composite on Mean Bond Strength Values for Veneer Ceramic Samples

Authors' Contribution:
Study Design A
Data Collection B
Statistical Analysis C
Data Interpretation D
Manuscript Preparation E
Literature Search F
Funds Collection G

ABEFG 1 **Hafiz Adawi**
ABDEG 1 **Kulashekar Nandalur Reddy**
ACDEG 1 **Khurshid Mattoo**
BCEF 2 **Naif Najmi**
BCEF 3 **Majid Arishi**
BCEF 4 **Abdullah Ageeli**
BCEF 5 **Abdullah Bahri**
ADEG 6 **Shafait Ullah Khateeb**
ADEG 6 **Shan Sainudeen**
ADEFG 6 **Suheel Manzoor Baba**
CDEG 7 **Mashail M.M. Hamid**
CDEG 7 **Shahi Jahan Shah**
CDEF 8 **Sumaya Yousuf Jeri**

1 Department of Prosthetic Dental Sciences, College of Dentistry, Jazan University, Jazan, Saudi Arabia
2 General Dentists, Jazi Dental Clinics, Jazan, Saudi Arabia
3 Resident (Prosthodontics), Armed Forces Hospital (Southern Region), Khamis Mushit, Saudi Arabia
4 General Dentists, RAM Dental Clinics, Al-Jubail, Saudi Arabia
5 Resident, Saudi Board (Restorative Dentistry), Jazan Dental Centre, Jazan Health Cluster, Jazan, Saudi Arabia
6 Department of Restorative Dental Sciences, College of Dentistry, King Khalid University, Abha, Saudi Arabia
7 Department of Oral and Maxillofacial Surgery, College of Dentistry, King Khalid University, Abha, Saudi Arabia
8 Department of Orthodontics, Laseran Medical Clinics, Abha, Saudi Arabia

Corresponding Authors: Nandalur Kulashekar Reddy, e-mail: kreddy@jazanu.edu.sa, Khurshid Mattoo, e-mail: drkamattoo@rediffmail.com
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Background: This study investigates the effect of artificial aging of direct resin nanohybrid composites on mean bond strength values for veneer ceramic samples.

Material/Methods: Ninety direct nanohybrid composite resin (Tetric N-Ceram) cylindrical discs were divided into 5 groups (n=18 each) based on aging cycles (thermocycling), as follows: TC=no aging (control), T1=850, T3=2500, T6=5000, and T12=10000 cycles, representing 1, 3, 6, and 12 months of clinical usage, respectively. Lithium disilicate glass ceramic (IPS e.max Press) cylindrical discs were cemented to resin discs using resin cement (Variolink N) after surface treatments (ceramic etching, silaning, composite abrasion). Differences in means between subgroups were calculated using one-way ANOVA, followed by the Tukey honestly significant differences post hoc test. Differences were considered statistically significant with a *P* value ≤0.05.

Results: The highest SBS between ceramic and aged composite was observed at 1 month (m=20.35) but did not differ significantly from the control group (m=20.97). For all other subgroups (3, 6, 12 months) SBS was significantly less than that of the control (*P*≤0.05). At 1 and 3 months, cohesive failures were more common, whereas adhesive failures were more common in 6- and 12-month-old composites.

Conclusions: SBS of aged composites was less than that of non-aged composites, with SBS decreasing proportionally as the resin aged. When cementing a ceramic restoration over existing composite restorations, those older than 1 month should be removed and replaced with new ones.

Keywords: **Composite Dental Resin • Core Paste Resin Luting Agent • Foundation Composite Resin • Optimal Pressed Ceramic • Tooth Crown**

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Introduction

The conceptualization of dental caries disease has evolved to be seen more as a noncommunicable disease that is linked to lifestyle and behavior [1]. The clinical presentation of the disease has changed dramatically from earlier higher prevalence rates in pits, fissures, and posterior teeth to smooth surfaces and anterior teeth [2], thereby leading to the development of interventional concepts, such as minimally invasive dentistry, in which adhesive-bonded restorations form a core treatment option [3]. With advantages such as marginal sealing, decreased cement solubility, aesthetic compatibility, and economic feasibility [4], such restorations can range from simple inlay, onlay, laminates, and crowns to complex occlusal veneers of the posterior teeth, whose retentive ability is not based mainly on mechanical principles, making their cementation (adhesive) vital for extended restoration durability [5]. Long-term successful cementation depends on many external factors (occlusion, anterior guidance, and parafunction) that are not related to material composition and properties [6]. A few adhesive treatment options, including laminates, veneers, and crowns, are specifically indicated in clinical scenarios in which existing composite resin restorations are present and have aged (exposed to oral conditions, discolored, biofilm deposited, abraded, eroded, or worn) [7]. Veneers can be either of resin or ceramic, are usually very thin, and may or may not cover the incisal areas. In many instances, a definitive restoration cannot be provided unless the foundation is appropriately built [8]. Existing foundation resin restorations are also difficult to remove due to the difficulty in discerning color between composite and natural tooth structure [9], increasing the clinical chances of depleting the natural tooth. Other factors, such as replacement costs, compromising or weakening existing natural teeth, or causing pulpal threats, preclude the choice of replacing an existing restoration with a new one. Simultaneously, clinical failures of laminates and crowns have been attributed to defects in underlying/existing previous restorations [7,10]. Despite the known disadvantages of old composite resin restorations, studies show a considerably higher percentage of aged resin restorations being encountered while a definitive restoration is being cemented [11].

Adhesive restoration can be made from restorative materials, such as composite resin (direct and indirect), alloys (base metal or noble metal), or porcelain (ceramics) [12]. Nano-filled resin composites contain a blend of small (nanometer)-sized particles dispersed into larger secondary resin particles, while nanohybrid resins use a different approach of combining micrometer-sized and nanometer-sized fillers [3,11]. Adhesive ceramics originated in 1959, when lithium disilicate (Li2Si2) glass ceramics were discovered in the form of a binary glass ceramic system after precipitation in glass (silver acting as a nucleating agent for crystallization) [13]. Ivoclar Vivadent has to date introduced 2 lithium disilicate-based glass ceramics, IPS Empress II (pressable) in 1998, and IPS e.max Press (castable) in 2001, with improved mechanical and optical properties [14]. High flexural strength and lifelike translucency make them better treatment options than earlier castable (Dicor and Mirage) and present day leucite ceramics in treatment options such as veneers and laminates, in which a thin surface of the natural tooth is restored [15,16]. IPS e.max Press is a crystalline-dominated, pressable lithium disilicate-based ceramic produced using bulk casting methods [17]. Sluggish controlled cooling after melting minimizes internal defects, thereby improving optical and mechanical properties. Restorations made from IPS require cementation by resin cements, which, when bonded to the underlying substrate (natural tooth or foundation restorations), increase restoration fracture resistance, surface and marginal adaptation, and retention [18]. However, the interface between the ceramic (glass) and the resin (plastic) cement requires mechanical (sand blasting or chemical etching [8%-10% hydrofluoric acid]) and chemical (silane coupling agent application) surface treatment [4,11,19]. Together, they thus provide a combination of micromechanical (honeycomb-like) and chemical bonds at the interface between the ceramic and the resin cement. The bond strength and adhesive failures of resin cements to pressable ceramics have been widely studied [20] using different bond strength tests, including push-out tests, tensile and shear bond strengths, and their respective microforms (micro tensile and micro shear) [21].

Ceramic restorations, when luted to composite resin restorations using resin cement, have comparatively greater fracture resistance than other restorative materials [22]. Chen et al investigated the bond strength of feldspathic porcelain (VMK 68) to direct composite restorative resin (Clearfil APX) using different hydrofluoric acid etchant concentrations and different etching times and found that lower etchant concentrations (2.5%) produced greater bond strengths than higher concentrations (5%) [23]. The use of silane coupling agents has also been found to improve the bond strength of resin composites to various types of porcelain [24,25]. Kilnic et al [26] assessed the SBS of resin composites (Filtek Z550) for different types of aged and non-aged ceramics (nanoceramic, resin ceramic, feldspathic, and lithium disilicate). Results showed material and surface treatment types significantly changed SBS [26]. Makishi et al investigated the SBS of 2 multimode adhesives (Scotchbond Universal and All-Bond Universal) on 1-year-aged indirect resin composites and IPS e.max Press and reported higher SBS after 24 h for indirect resin composites than for 1 year and did not find any significant difference between the 2 adhesives used after 1 year [27]. One of the reasons for decreased SBS in indirect resin composites is the use of air abrasion [28], which promotes water absorption into the primed layer. Studies investigating the adhesion of 2 composite resin layers for repair have found that the adhesion that is generally

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achieved in the presence of an oxygen-inhibited layer of unpolymerized resin is absent in aged composite resin [11,29], which can be improved by various surface treatments, such as roughening, etching, airborne particle abrasion, or using silanes/intermediate resins [29,30]. Recent studies on the surface conditioning of polymeric materials have favored airborne particle abrasion (silica-coated alumina particles) with silanization, to produce a more effective bond than acid etching and silanization [31]. The monomeric ends within silane molecules interact with the methacrylate of adhesive resins by free radical polymerization [32]. The protocol of aging for composite resin research has been generally performed through thermocycling for 5000 to 10000 cycles, which equals 6 to 12 months of clinical usage [33]. These time periods do not actually reflect the clinical scenario. In many cases, including complete occlusal rehabilitations and implant supported restorations, there are short aging periods, within 1 to 3 months, between placement of foundation restoration and cementation of ceramic restoration. In other cases, the composite restoration may be further exposed to oral aging, because the definitive restoration needed to be repeated or did not fit.

To the best of our knowledge, there are studies that focus on the influence of resin adhesion to new or fresh polymerized composite resin [26,28,34]. However, studies investigating adhesion to aged composite resin are limited to composite repair [10,30,35] and feldspathic porcelain [36]. Also, no studies have investigated the short-term aging cycles (1 and 3 months), which broaden the clinical spectrum encountered by practitioners. Therefore, in this study, we aimed to evaluate the effects of various aging (thermocycling) cycles of resin nanohybrid composites on mean bond strength values for ceramic (lithium disilicate pressed) samples. We hypothesize that since aged composites will not present a full array of free surface radicals, the adhesion of a pressable ceramic through resin cement will yield inferior bond strengths to that of aged composites. Alternately, the null hypothesis states that there is no difference in bond strength after composite resin undergoes various aging cycles.

Material and Methods

Ethics

This study received its ethics clearance from the concerned Ethics Committee of the College of Dentistry, Jazan University, via approval number CODJU-21151. This in vitro experimental research study was part of the requirement that was conducted by a group of intern students under the direct supervision of staff of the Department of Prosthetic Dental Sciences during the academic year 2022-2023.

Study Design

This study followed a comparative approach between control and experimental groups, with the control group serving as the baseline and experimental groups serving as the test groups. The independent variables for the study were the materials (pressable ceramic, composite resin, and resin cement) and thermocycling (aging cycles of 1, 3, 6, and 12 months), while the dependent variables were the SBS and adhesive failure analysis. **Figure 1** represents the study flowchart, highlighting the sequence and the concerned study variables. Operators who performed testing were blinded to specimen identification and research outcomes.

Operational Definitions [37]

The term cementation has been operationally defined as the process of attaching parts by means of a dental cement, in this case, a resin cement. Cohesive failure is a type of bond failure within a dental material as a result of tension or shearing forces, while adhesive failure is a type of bond failure that takes place at the interface between 2 materials due to shearing or tensile forces. The adhesive can be applied partially or completely to one or both of the substrates, depending on the type of bond failure. Castable ceramic for dental applications refers to a form of glass ceramic that has restorative characteristics and can be cast using the lost wax technique.

Sample Estimation, Preparation, and Grouping

Sample Size

The study conducted by comparing 5 groups (1 control and 4 experimental). The total number of specimens for the study and the number of these specimens in each group were statistically estimated using software (Nquery, Version 7, Informer Technologies, USA) using the formula N=2σ2×(Zα+Zβ) 2/2 [38]. The calculated samples for the total study came out to be 90 specimens, with each group having a minimum of 18 samples (derivation standards of type 1 error rate a=0.05, effect size D2=0.28, and study power assumption 80%), which were guided by earlier similar studies [19,24]. Compensation for faulty sample loss was compensated by keeping 2 additional samples for each subgroup that would replace the defective ones.

Specimen Preparation

Materials used with their respective brands, manufacturers, batch numbers, chemical composition, and working characteristics are listed in **Table 1**. The study was sequenced as the preparation of resin composite specimens followed by their respective aging cycles, and in the concluding stage, the ceramic specimens were prepared and adhesively bonded to aged composite specimens after respective surface modifications.

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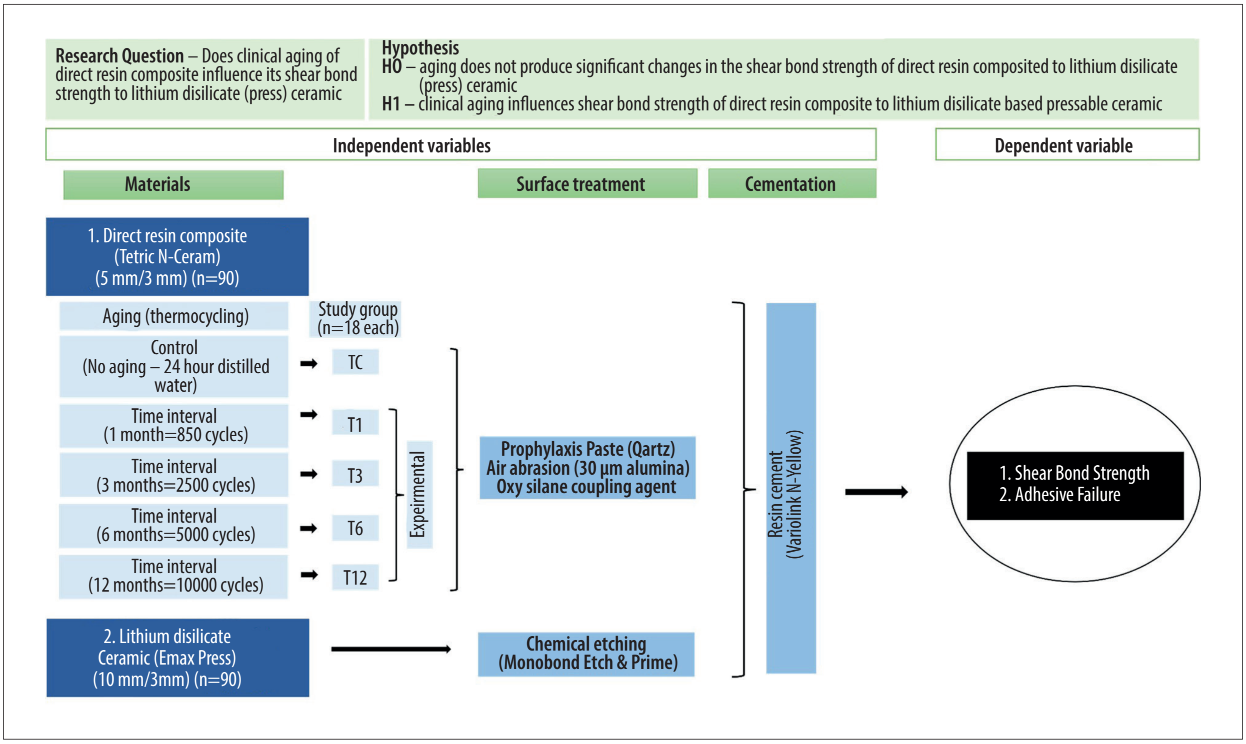

Figure 1. Flow chart showing study design, variables, and study groups. *Compiled Figure created using MS PowerPoint, version 20H2 (OS build 19042,1466), windows 11 Pro, Microsoft corporation).*

For the composite resin specimen (n=90), the nanohybrid composite resin (Tetric N-Ceram, Ivoclar Vivodent, Switzerland) that would represent the existing aged restorations under the ceramic restoration was prepared by packing the specimens into a cylinder-shaped polyethylene mold with a height of 3 mm and a diameter of 5 mm. Incremental photopolymerization was conducted by a halogen unit (Demetron LC, Kerr; Orange, CA, USA; intensity 1200 MW/cm^2; duration, 40 s; and distance, 2 mm), as per manufacturer recommendations. To standardize the light intensity, verification using a radiometer (Demetron LC, Kerr) was performed after preparing every 10 specimens. To protect the formation of an oxygen-inhibited surface layer, a clean, dry glass slab was used to create a smooth surface on each specimen. Once polymerization was accomplished, a total of 90 test specimens were removed and distributed into 5 different groups (1 control and 4 experimental) that were based on the time duration (T) of aging (thermocycling). The specimens in the control group (TC) were placed in distilled water for 24 h at a controlled body temperature (37°C) before subjecting them to testing. The remaining experimental groups T1, T3, T6, and T12 represented aging of 1 month (850 cycles), 3 months (2500 cycles), 6 months (5000 cycles), and 12 months (10000 cycles), respectively. Aging was conducted in a thermocycle bath (Mechatronik, Bayern, Germany), which circulated the samples with alternate immersions in warm and cold bath temperatures (5 to 55 °C), with a dwell time of 5 s. The thermocycling was representative of clinical usage, as indicated in previous studies [11,32].

For the IPS e.max Press (LT) specimens (n=90), a total of 90 ceramic specimens with diameter of 10 mm and thickness at 3 mm were manufactured to facilitate an accurate point of testing apparatus. Specific wax pattern discs were obtained using modeling base plate wax. Each wax disc was then sprued with 3 mm sprue wax (Bego, Germany), and 3 such sprued wax patterns were placed on the IPS muffle (Ivoclar Vivadent, Liechtenstein), followed by investing in manufacturer-recommended investment material (IPS PressVest Premium, Ivoclar Vivadent). After setting up the investment, the ring base and gauge were removed, and burnout was conducted in an automatic furnace (Ney, US Dental) to eliminate wax at the recommended temperature of 900°C (1650°F). The obtained mold was then placed in the porcelain furnace (EP 3000, Ivoclar Vivadent), where lithium disilicate glass ceramic ingots (IPS e.max Press LT) of one particular shade (A1) were heat-pressed. The pressed mold was then cooled slowly at room temperature for 1 h, and the mold was divested (4 bar pressure, 110 µm alumina particles). Each specimen, after divesting, was placed in hydrofluoric acid (1% Invex liquid, Ivoclar Vivadent) for 10 min, followed by water wash, air drying, and air abrasion (110 µm alumina, 2 bar pressure) to remove the reaction layer, as per the manufacturer's recommendations. The discs were then separated from the sprues, and each specimen was examined for surface defects.

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Table 1. List of materials, instrumentation and manufacturer specifications.

S. No.	Materials	Manufacturer	Specifications/features
1.	**Tetric N-Ceram**	Ivoclar Vivodent, Liechtenstein, Switzerland	• Lot number: 200WJ4 (Shade A1) • Light-cure, radiopaque nano-hybrid composite, nano-optimised filler (16 shades) • Resin matrix: Bis-GMA, Bis-EMA, and urethane dimethacrylate monomer (UDMA), light initiator Ivocerin • Fillers: Barium aluminum silicate glass (two particle sizes), filler content: 61% (volume), and 17% polymer fillers, or "isofiller" • Cylinder-shaped specimens (5 mm diameter × 3 mm height)
2.	**IPS e.max Press (LT)**	Ivoclar Vivadent, Schaan, Liechtenstein	• Lot number – 010-040; Monochromatic - Low translucent (LT), (4 Bleach BL, 16 A–D) • Indications: ceramic Veneers (≥0.3 mm) Occlusal veneers (≥1.0 mm), Inlays/onlays, partial crowns, full crowns (≥1 mm), 3 unit anterior/ posterior bridge, implant superstructure and hybrid abutments • SiO2. Li2O. K2O. MgO. ZnO. Al2O3. P2O5 • Discs – 10mm diameter and 3mm height
3.	**Variolink N**	Ivoclar Vivadent, Schaan, Liechtenstein	• Lot number: Z04S53; dual-/light-curing, esthetic luting for ceramics (Base/Catalyst); 4 shades (Yellow, White, Transparent, Bleach XL) and one Clear (light-curing only) • Working time: About 3.5 min. at 37°C/99°F; Mixing ratio: 1: 1 • Monomer matrix: BisphenolA-glycidyl methacrylate, urethane dimethacrylate, triethylene glycol dimethacrylate. • Inorganic fillers: Barium glass,ytterbium trifluoride, Ba-Alfluorosilicate glass, and spheroid mixed oxide. Additional contents: initiators, stabilizers, and pigment • Particle size is 0.04-0.2 m, with a mean particle size of 0.1 m
4.	**Monobond Etch & Prime**	Ivoclar Vivadent AG, Schaan, Liechtenstein	• Application time: 20+40 seconds, rinsing time: 15 seconds, and drying time in water/oil-free air: 10 seconds • Alcoholic-aqueous solution of ammonium polyfluoride, silane methacrylate and colourant
5.	**Thermocycling machine**	Model 1100, SD Mechatronik, Bayern, Germany	• Alternate immersion in warm followed by cold liquid simulates high temperature changes • Warm bath temperature: 25°C to 55°C • Cold bath temperature: 5°C to 15° C • Exposure time – adjustable per bath from 0 to 999 seconds

C – centigrade; mm – millimeter; rpm – rotations per minute; ° – degrees for temperature; m –microns.

Surface Modifications and Cementation

For the resin composite specimens, the surface to be bonded was prepared using an abrasive paste (Qartz Prophylaxis Paste) that simulates the clinical application of oral prophylaxis. The samples were then air abraded with an intraoral air abrasion device (Dento-Prep, Daugaard, Denmark), which uses 30 µm particles of alumina that are coated with silica. The device was kept at a distance of 10 mm for 4 s at a pressure setting of 2.5 bar [39]. Surfaces were cleaned with air, followed by the application of an oxysilane coupling agent (3-methacryloxypropyl trimethoxysilane, ESPE-Sil, 3M ESPE) for 5 min, before the application of the priming agent (Monobond N), as recommended for the resin cement (Variolink N). The primer was left to react for a period of 60 s. For ceramic discs, Monobond Etch & Prime (an alcoholic-aqueous solution of ammonium polyfluoride) was applied for a period of 60 s (applied for 20 s, followed by shaking, and then left for another 40 s), followed by drying with oil- or water-free dry air (10 s) using low pressure. Before cementation, the ceramic specimens were applied with the silane coupling agent and allowed to react for 60 s, which is essentially a step of the final cementation with the resin cement.

For cementation, the 2 conditioned substrates of aged resin composite and ceramic were bonded to the resin cement (Variolink N, Ivoclar Vivadent), as per the manufacturer's instructions and recommendations. A customized aligning device allowed both specimens to be aligned so that surfaces contacted evenly under the constant load (750 g). This ensured

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Table 2. Comparative differences in mean shear bond strength (MPa) values between different types of aged direct resin composite groups (Tetric N-Ceram) and lithium disilicate ceramic (IPS e.max Press).

Groups	N	Mean	SD	df	ANOVA test	
					F statistic	P-value
TC	16	20.97	1.40	4	13.55	0.00001
T1	16	20.35	1.26	4		
T3	16	19.11	1.28	4		
T6	16	18.90	1.73	4		
T12	16	18.27	1.44	4		

Mpa – mega pascals; N – number of specimens; SD – standard deviation; df – degree of freedom; T – time interval; C – control (no aging, 24 hours storage in distilled water at 37 degrees centigrade); 1, 3, 6, 12 – number of months; 1 – 850 cycles equivalent to 1 month; 3 – 2500 cycles equivalent to 3 months; 6 – 5000 cycles equivalent to 6 months; 12 – 10000 cycles equivalent to 12 months of clinical use. Test employed; one way analysis of variance (ANOVA). Statistical significance: All differences at various time intervals in each group were considered to be statistically significant if the probable P value was ≤0.05

that the thickness of resin cement would be uniform for all specimens in each group. Excess resin cement was removed with a microbrush, followed by photopolymerization (40 s) in each direction from a distance of 2 mm. After cementation, an oxygen-inhibiting gel was applied to the free surface, which was kept on the specimens for 5 min. The specimens were then washed, rinsed, and dried. The specimens from all groups were then stored in distilled water before undergoing shear bond testing. The cemented specimens before testing for bond strength were embedded in a hard polyethylene ring (diameter of 2 cm and height of 1 cm) using auto polymerizing polymethylmethacrylate (quick repair). One surface of the specimen was thus embedded within the acrylic, while the other side was uncovered for testing.

Measures, Data Collection, and Interpretation

All embedded specimens were tested for SBS by mounting them in a jig of a universal testing machine (Instron Corp, Canton, MA, USA), using a shear force on the interface between the 2 cemented specimens until failure occurred, which falls as per standard ISO regulations (PN-EN ISO 29022: 2013-10). The force was applied at a crosshead speed of 1 mm/min, while the analysis of stress and strain and the failure load were recorded automatically within the machine software. The load required to debond each specimen was measured in newtons, and the bond strength was then represented in megapascals (by dividing the load by the brackets' mean surface area).

For adhesive failure analysis, the failure sites were examined by 2 independent and calibrated reviewers who were blinded to the study outcome and the specimen samples. All observations were made under an optical microscope (magnification ×20; Amscope, USA). The review consisted of a visual microscopic inspection as well as a digital image of the failed surface, using ImageJ software. Five different types of failures were identified (prefailure, substrate failure, mixed failure, cohesive failure, and adhesive failure). Depending upon the amount of adhesive left over the surface of the specimen, the failure was categorized as no adhesive left (adhesive failure), adhesive left partially (mixed), and complete adhesive left (cohesive).

Statistical Analysis

After entering the obtained data into Microsoft Excel sheets, correction, refinement, and coding were performed before analysis was conducted in SPSS version 22.0 software (IBM Corp, Armonk, NY, USA) using a desktop computer (Lenovo, CT55AG7) through Windows 10 Pro. The mean shear bond strength values and their standard deviations were derived. Differences in means between the experimental and control groups were subjected to one-way analysis of variance test (ANOVA), with bond strength being dependent and aging being independent variables. For differences between pairs (multiple pairwise) of group means, a post hoc Tukey honestly significant difference test was used. All differences were considered significant statistically if the *P* value was either equal to or less than 0.05 ($P \leq 0.05$). Types of failures were evaluated in terms of frequency distribution (percentage).

Results

SBS Between Aged Resin and Pressable Ceramic

Table 2 presents the mean SBS values of the groups investigated in this study. The highest SBS was observed in specimens

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Table 3. Tukey honestly significant difference post hoc pairwise comparison showing significance of Differences between pairs of group means for types of aged direct resin composite groups (Tetric N-Ceram) and lithium disilicate ceramic (IPS e.max Press).

Groups	Shear bond strength (Mpa)				
	TC	T1	T3	T6	T12
TC		0.623	1.869	2.072	2.704
		0.591	0.000*	0.000*	0.000*
T1	0.623		1.246	1.449	2.081
	0.591		0.040*	0.011*	0.000*
T3	1.869	1.246		0.203	0.835
	0.000*	0.040*		0.989	0.302
T6	2.072	1.449	0.203		0.632
	0.000*	0.011*	0.989		0.578
T12	2.704	2.081	0.835	0.632	
	0.000*	0.000*	0.302	0.578	

Mpa – mega pascals; T – time interval; C – control (no aging, 24 hours storage in distilled water at 37 degrees centigrade); 1, 3, 6, 12 – number of months; 1 – 850 cycles equivalent to 1 month; 3 – 2500 cycles equivalent to 3 months; 6 – 5000 cycles equivalent to 6 months; 12 – 10000 cycles equivalent to 12 months of clinical use. Test employed; Tukey HSD (Honestly Significant Difference) Post Hoc test. Statistical significance: All differences at various time intervals between pairs of group means considered to be statistically significant if the probable P value was ≤0.05

that belonged to the control group (m=20.97), while the lowest SBS was observed in the T12 group (m=18.27), indicating that the SBS showed a time-dependent decline in the aged composite resin. Among the 4 experimental groups, the highest SBS was observed in specimens that were aged for the least amount of time (1 month), while the greatest decrease in SBS was observed in specimens aged 12 months. One-way ANOVA showed that the differences between the groups from the control were statistically significant ($P\leq0.05$). The post hoc pairwise group tests are presented in **Table 3**. All subgroups except T1 were found to differ significantly from the control group, with differences between 24 h (control) and 1 month (T1) being not significant ($P\leq0.05$). Although there was a continuous reduction in SBS at succeeding time intervals (T3, T6, and T12), the differences between T3 and T6, T3 and T12, and T6 and T12 were not found to be significant, indicating that most of the reduction in SBS occurred during the first 3 months of aging. The clinical application of these findings is that a composite resin restoration that is older than 3 months will have a significant reduction in SBS and should be either replaced or other means of surface modification must be investigated that will enhance the SBS.

Adhesive Failure Analysis

The frequency distribution of different failures observed in the specimens of each subgroup is presented in **Figure 2**. More cohesive failures were observed in specimens aged between 1 and 3 months, while more adhesive failures were observed in specimens that were aged between 6 and 12 months. Other types of failures (substrate, mixed, and prefailure) occurred with less frequency, with all of them occurring in the samples that were aged up to 3 months.

Discussion

In this study, we intended to compare the adhesion bonding strength of 4 different aged composite resin restorative materials with pressable ceramic when bonded with a manufacturer-recommended resin cement. The main finding of the study, when applied clinically, implies that those composite restorations older than 1 month in the oral cavity, if bonded with resin cement, will have significantly less SBS, which may affect their long-term retention and resistance ability. Another clinically significant finding was that composite restorations that are more than 3 months old did not differ in producing any changes from those that are 6 or 12 months old in reducing bond strength. In other words, the changes that bring about the reduction in SBS of resin composite to ceramic occur within the first 3 months, bringing maximum alterations in the adhesion complex. The changes that occur after 6 months are not clinically significant, as there is less deterioration in the bond strength between ceramic and resin composites. The clinical

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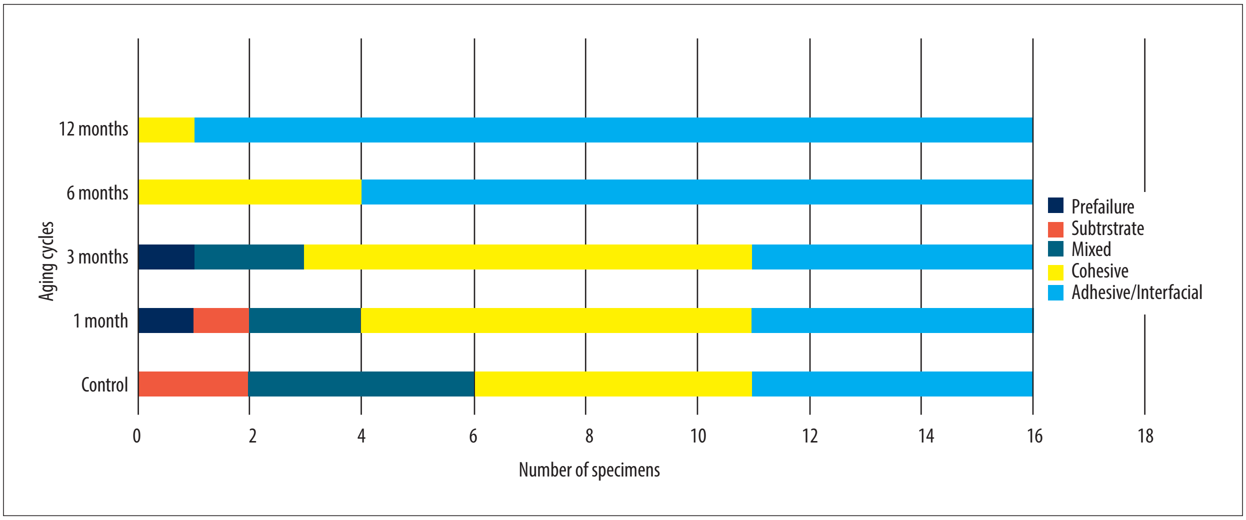

Figure 2. Comparative distribution (number) of various failure modes observed between different types of aged direct resin composite groups (Tetric N-Ceram) and lithium disilicate ceramic (IPS e.max Press). *Compiled Figure created using MS MS Word, version 20H2 (OS build 19042,1466), windows 11 Pro, Microsoft corporation).*

interpretation of adhesive failure analysis implies that as the underlying composite resin ages, the failure type will shift from cohesive to adhesive.

A single restorative resin, pressable ceramic, and resin cement were considered for investigation, since it was necessary to first establish that aged restorations made up of composite resin can be detrimental in terms of adhesive bonding to pressable ceramic. The incidence of encountering an existing composite restoration during the cementation of a ceramic restoration has been reported to be as high as 60% [40]. The resin chosen for this study has low shrinkage and stresses, both of which have been reported to improve the subsurface characteristics of composite resins [41]. Our choice of IPS e.max Press was based on its wider range of applications (cutback, layering, and monolithic full contoured) in partial and complete fixed prosthodontics, primarily due to its high flexural strength (470 MPa) [14,22], fracture toughness (3 MPa) [25,26], and high cumulative survival rates (97.4% for 5 years and 94.8% for 8 years) [18]. This study differs mainly in the aging protocol when compared with other studies, in which aging was performed after cementation of the 2 different specimens. The earliest physical and optical changes observed in composite restoration have been reported to be within a year; the time period can further decrease if patients consume colored foods and drinks [26,32]. Therefore, the time intervals we chose were well within the first year of composite restoration placement, as restorations older than 1 year are easily discerned and are generally replaced due to the risk of secondary caries under the restoration.

The results from this study showed that the highest bond strength among various experimental groups was obtained between the IPS e.max Press cemented with resin cement and the resin composite aged for 1 month, which was the control group (20.97 MPa), and it was not significantly different from the control group. Subsequent aging (3, 6, and 12 months), however, had lower bond strengths that significantly differed from the control group. Dieckmann et al, while investigating the reparability between fresh and aged composites subjected to mechanical pretreatment and thermocycling (6 months), found aged composite surfaces had significantly less repair bond strength than immediately repaired resins [35]. Cotes et al investigated surface treatment (sandblasting) and aging (5000 and 12000 cycles) influences between core build-up composite and luting agent and found microtensile bond strengths were significantly reduced in aged samples [42]. The decreased bond strength of the resin adhesive interface was attributed to hybrid layer hydrolysis, in which water acts as a plasticizer between adhesive polymer chains, such as a molecular lubricant, in turn causing mechanical wear of the exposed adhesive [43,44]. Such hydrolytic degradation has also been reported to result in color and translucency changes in a composite restoration [44]. Chemically, the compositions of the resin composite and the resin cement are alike; therefore, after cementation, there is a chemical interaction between the two [45]. However, in aged restoration, the surface of the composite is completely cured, contaminated, and inactive (fresh composite is soft unless cured). To improve adhesion between the aged composite restoration and the new resin layer, it is imperative to remove or alter the surface layer of the aged composite. Studies on the repair of composites have found that adhesion improves through acid treatment [32], air/alumina oxide abrasion [11], diamond bur grinding [46], silicatization and silanization, and pumice cleaning [47]. The adhesion of restorations such as a crown or a veneer, on the

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contrary, is dependent primarily on the features of the preparation, such as preparation taper [48], without which the factors that adhere the crown to the underlying tooth can only play a secondary role [49].

Composite restorations generally fail due to the degradation of the polymeric matrix and its silanized filler particles [50]. Certain chemicals used routinely in dental practice, such as bleach, alter surface properties, including roughness and color [51]. The degradation can be due to multiple reasons, among which hydrolysis and temperature changes are the main causes that can be related to the aging process considered in our study. Hydrolytic degradation begins with swelling of the matrix, which results in saturation of the polymer, which in turn reduces free radicals present on the surface [52]. This non-availability of free radicals in aged composites results in a compromised reaction with resin cement [53]. Hydrolytic and thermal variations together also cause surface deterioration (roughness), resulting in the failure of filler and matrix interfacial bonds [42]. Since the polymeric matrix is most vulnerable to temperature changes, temperature changes produce contraction and expansion stresses, which in turn create gaps along the cement interface. Once there is a gap, the water further seeps in along with the oral bacteria, which later cause enzymatic degradation of the composite matrix from inside [54]. The reduction of composite resin bond strength in oral conditions has been estimated to be between 20% and 80% of the original strength [55]. Some studies have defended the viability of adhesion between new and aged resin composites. They state that the adhesive is more important than the composite. However, they also state that adhesive alone may not be chemically compatible with the fillers of aged resin that have been exposed due to water sorption [56]. The highest bond strength obtained in this study between pressable ceramic and non-aged composite resin at baseline was 20.97 Mpa, which is lower than that obtained by Gresnigt et al [39] (non-aged Empress-Variolink 22.0), with the differences being due to the difference of materials. The ceramic used in their study was IPS Empress II, and the aged composite used was Estenia, which is an indirect composite used for laminates. High bond strengths are due to more durable siloxane bonds between the filler and the matrix of indirect composites. Unpolymerized resin left in indirect composites (urethane tetramethcyclate) in deeper areas forms copolymers with the methacrylate of the silane, resulting in a stronger bond [57]. In another study, by Kumbuloglu et al [58], resin cement (Variolink II) was bonded to lithium disilicate glass ceramic (Empress II) with 6000 thermocycles. The bond strength obtained was 23.2 MPa, which is higher than that obtained in the present study. Other related studies have also reported higher bond strength when lithium disilicate was adhered with resin cement to composite resin [14,21,24,31]. These studies, however, performed thermocycling after cementation to examine the effect of aging on the bond strength. Most of the differences in the results of these studies can be traced to differences in the aging method or aging cycle. In other studies, the differences may be due to the surface treatment given to the ceramic. In heat-pressed glass ceramics, air abrasion with alumina oxide has been reported to decrease bond strength, as compared with surface etching [59]. The type of ceramic used also influences the SBS outcome. Drumond et al, while comparing pressed and CAD/CAM lithium disilicate ceramics and their relative resin cement conversions, found CAD/CAM ceramic to have a higher microshear bond strength than the pressed ceramic, which was due to the degree of conversion allowed by the CAD/CAM ceramic [60].

The quality of adhesion between 2 chemically dissimilar materials is also interpreted through the types of failures. In the present study, all subgroups, after 3 months of aging, showed a decrease in the SBS that was significant compared with the control group but not significant between time intervals 3, 6, and 12 months. Five types of failures were observed (**Figure 2**) in all subgroups, with more adhesive failures taking place as the aging period of resin composites increased. Cohesive failures have been considered reliable adhesions. The higher frequency of cohesive failures was observed in the 1- and 3-month subgroups, indicating that adhesion can be considered reliable. A similar pattern has been observed in other studies that have investigated ceramic-composite bonding with resin cement. Adhesive failures associated with resin cement have been attributed to the hydrolysis of the polymer matrix and the wear of the filler. Dual-cured resin cement was used in the present study, which contains a photoinitiator and an amine accelerator to attenuate polymerization where light cannot reach [61]. The self-cure action, however, is also dependent upon the degree of light received for activation [62]. SBS is also increased if the filler size in the cement is large [63], as observed in the comparison of resin cements of different filler particle sizes. Other factors that have been reported to influence the SBS of resin cement in a ceramic are organic monomer viscosity, amount of initiator [64], ceramic, crystal size, quantity, and distribution [65]. The bond between ceramic and resin cement is between their inorganic (ceramic silicon oxide) and organic components (cement) [66]. Silane coupling agent application enhances bonding to ceramic by providing a chemical covalent and hydrogen bond of resin cement to the ceramic [66]. Irrespective of the treatment used, most dental ceramics have the general tendency to fail due to clinical factors rather than factors related to material choice or type of surface treatment used [67].

Strengths and Limitations of the Study

This study is perhaps the first to investigate the effect of precementation aging of resin composites on the SBS of a pressable ceramic using resin cement. The study highlights the

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significance of aging restoration earlier than the routinely investigated 6 months in most research on SBS composites. The study has, however, limitations in that we investigated only 1 composite type, 1 ceramic type, and 1 resin cement. We also did not investigate different surface treatments that have been shown to improve bond strength, as mentioned in the literature.

Conclusions

Within the scope and limitations of the present study, it can be concluded that the aging of composite resin decreases its SBS to pressed lithium disilicate glass ceramic (IPS e.max Press). The decrease in SBS is greater as the resin composite ages. Composites that have aged for 3 months do not show any significant changes in bond strength, compared with those that have aged for 6 or 12 months. Clinically, the results of the study indicate that resin restorations older than 1 month should be removed before being cemented to a pressed ceramic with resin cement. Clinicians should also be aware that all laboratory procedures should be done within the stipulated 1-month timeframe so that the ceramic restoration is cemented to the composite that has not become old. Further studies are recommended to investigate the role of various surface treatments on the SBS of aged composite to ceramic restoration.

Department and Institution Where Work Was Done

Department of Prosthetic Dental Sciences, College of Dentistry, Jazan University, Jazan, Saudi Arabia.

Declaration of Figures' Authenticity

All figures submitted have been created by the authors, who confirm that the images are original with no duplication and have not been previously published in whole or in part.

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