Effects of Storage Time on Resin Composite Attachment Characteristics in Aligner Template Trays

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Abstract

Aim: The aim of this study was to evaluate the effect of pre-polymerization storage time on the mechanical and chemical properties of resin composite attachments fabricated using clear aligner template trays. Specifically, the study assessed how storage time influenced the microhardness, ambient light-induced polymerization, and shear bond strength of attachments bonded to enamel under simulated clinical conditions.

Methods: Resin composite attachments were prepared using Tetric EvoCeram nanohybrid composite filled into clear aligner template trays and stored in opaque black retainer cases at 20°C for five time intervals: 1 hour, 1 day, 1 week, 2 weeks, and 1 month. Microhardness of cured samples was measured using a Vickers microhardness indenter. Ambient light-induced polymerization of uncured attachments was evaluated using FT-IR spectroscopy. Shear bond strength of attachments bonded to human premolars was tested using a universal testing machine. A total of 26 samples per time point were tested for microhardness, 28-29 samples for spectroscopy, and 6-8 premolars per time point for shear bond strength. Statistical analysis included ANOVA with post hoc Tukey testing, with significance set at $p<0.05$.

Results: Microhardness values showed significant differences across time points (ANOVA $p=0.0004$), with the greatest hardness at 1 week and significantly lower values at 1 day ($p=0.0002$) and 2 weeks ($p=0.0107$). FT-IR absorbance at 1640 cm^{-1} also differed significantly between certain groups (ANOVA $p < 0.0001$), with the lowest absorbance at 1 day and unexpectedly high absorbance at 1 week, suggesting inconsistent ambient light exposure rather than a linear polymerization trend. Shear bond strength did not differ significantly between time points (ANOVA $p=0.032$), with all groups exceeding clinically acceptable bond strength thresholds.

Conclusions: No clear relationship was found between the pre-polymerization storage time of attachments up to one month and the microhardness, degree of dark curing, and shear bond strength of aligner attachments. Handling time and ambient light exposure may have a greater effect on the mechanical and chemical properties of attachments compared to storage time. Future studies should investigate additional composite materials and environmental controls to refine guidelines for pre-filled aligner storage.

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Introduction

Clear aligner therapy (CAT) has become a widely adopted alternative to fixed orthodontic appliances due to its improved aesthetics, patient comfort, and removability. Since its commercial introduction in the late 1990s, CAT has evolved into a mainstream treatment modality capable of managing a wide range of malocclusions. Central to its success are composite attachment, small, tooth-colored resin shapes bonded to the enamel surface that serve as mechanical aids to enhance the aligner's force delivery, allowing for movement such as extrusion, rotation, and root torque (AlMogbel, 2023).

Attachments are typically formed by filling thermoplastic trays with dental composite, which is then cured directly onto the tooth (Tepedino & Ciavarella, 2023). Light-cured nanohybrid resin composite is an ideal material for this purpose due to its strength, adaptability, and aesthetic properties (Ocak et al., 2025). Clinical workflows for efficient aligner delivery and minimal patient chair time can involve pre-loading the aligner template with composite and storing them for later bonding. Although there have been studies on the effect of aging of aligners (Elshazly et al., 2023) and resin composite (de Lange et al., 1983) there is a lack of research specifically investigating the effects of storage time on the chemical and mechanical integrity of the uncured composite when stored in this manner.

Resin composites are recommended to be stored within a narrow temperature range (El-Maksoud et al., 2024) in the absence of light (Kowalska et al., 2021). Even when stored appropriately, over time resin composite can experience molecular degradation that can compromise the completeness of curing and alter important properties such as hardness, bond strength and degree of polymerization (de Lange et al., 1983). These changes, if present, could affect the performance and reliability of attachments during orthodontic treatment.

The purpose of this study was to investigate the effects of pre-polymerization storage time on resin composite attachments fabricated using clear aligner template trays. Specifically, this research evaluated (1) the surface microhardness of the attachments as a measure of polymerization and mechanical strength, (2) the degree of polymerization via Fourier Transform Infrared (FT-IR) spectroscopy to determine whether ambient curing occurred during storage, and (3) the shear bond strength of attachments to enamel as a measure of adhesive performance.

Understanding these outcomes is essential for developing evidence-based clinical protocols regarding the fabrication, handling, and storage of composite-filled aligner template trays.

Literature Review

Clear Aligner Therapy and Composite Attachments

The invention of clear aligner therapy (CAT) has transformed modern orthodontic treatment. In 1945, Harold D. Kesling introduced a pioneering appliance that would lay the groundwork for modern CAT, the “Tooth Positioning Appliance” (Kau et al., 2023). The tooth positioning device was a custom-made removable rubber appliance that resembled a mouth guard that sought to position teeth without traditional braces. This innovation paved the way for subsequent developments such as clear thermoplastic appliances in the 1950’s. In 1959, Nahoum built upon Kesling’s work by creating the first documented clear aligner appliance using a vacuum-based thermoplastic material. With this new clear aligner appliance, Nahoum made use of auxiliary elements such as acrylic buttons for inter-arch elastics which are used in clear aligner therapy today. Between the 1970’s through the early 1990’s, Ponitz and McNamara made developments on the idea of an “invisible retainer” that could be used for final detailing during the retention phase of treatment. Sheridan further refined this idea in 1993 by reducing the 1mm thickness of the “invisible retainer” made by McNamara down to 0.030mm with the introduction of the “Essix appliance”. With the help of two orthodontists, Align technology (Santa Clara, CA, USA) was founded by Zia Chishti and Kelsey Wirth in 1997. Their revolutionary first patents computerized the traditional analog method of CAT. Instead of sequentially sectioning and repositioning teeth on a dental cast, a computerized method was used to generate a series of clear aligners to move the teeth. This was the first thermoplastic orthodontic appliance to leverage the power of modern CAD/CAM technology. CAT has become an increasingly popular choice for patients seeking an aesthetic option for orthodontic treatment.

A key factor to the efficacy of CAT are the composite attachments which are bonded to teeth to facilitate orthodontic movement. These attachments are small shapes added to the digital tooth models and transferred to the teeth by packing resin composite into the wells of the aligner template and curing it directly onto the tooth surface (AlMogbel, 2023). The composite attachments are crucial for precise force delivery as well as retention of the aligner to the dental arch (AlMogbel, 2023). Filling aligner templates can take over 30 seconds per attachment when using packable composite (Lin et al., 2021). Clear aligner therapy routinely involves preparing ten or more attachments, which adds up to several minutes total per template. If done chairside, this could result in a significant amount of waiting time for the patient. In a busy orthodontic practice that is seeing over 50 patients a day, chair time is a valuable resource that needs to be conserved through efficient workflows. Filling aligner templates in larger batches for the next several CAT deliveries would yield much greater efficiency than filling them one by one as patients come in for their appointments. There is also the issue of last-minute appointment reschedules. If a patient reschedules for several weeks out from their original delivery appointment, it is currently unclear if that filled aligner template needs to be emptied out and refilled at the time of the new appointment or if it can be kept in dark storage for several weeks without sacrificing clinical efficacy of the attachments. This research seeks to shed light on this issue and give clarity on how long filled aligner templates can be kept in storage.

Resin Composite Materials for Orthodontic Attachments

CAT attachments can be made from either packable or flowable resin composite. The decision on which type of resin is used is a choice that considers material handling preferences, clinical efficacy, and specific case requirements. Packable nanohybrid composites carry several mechanical properties that would intuitively be thought of as desirable for long lasting CAT attachments such as increased strength and wear resistance relative to flowable composite. The addition of nanoparticles to dental materials has greatly increased their efficacy when compared to previous generations of packable composite (Bilal & Arjumand, 2019). Earlier iterations of dental resins contained macrofiller particles, but more recent research has demonstrated that adhesive resins ideally should have a mixture of micro and nano-sized particles. The combination of these filler sizes provide properties that are ideal for long-term survival in the oral environment such as high compression strength, flexural strength, and wear resistance (Hegde et al., 2011). Nano-adhesives have been shown to demonstrate comparable shear bond strength when compared to orthodontic adhesives (Bilal & Arjumand, 2019). However, nanohybrid composites show superior Vickers microhardness values (García-Contreras et al., 2015a). Despite the more robust mechanical properties carried by packable composite, research has shown that it does not outperform flowable composite. The preparation time of CAT attachments using flowable composite can be as little as 6-seconds per attachment compared to over 30-seconds with packable composite (Lin et al., 2021). A study conducted to evaluate the success rates of attachments between flowable and packable composite showed that over a 6-month period 75% of flowable attachments had the presence of damage compared to 55.6% in the packable group, however this difference was not proven to be statistically significant indicating that composite type does not affect attachment success (Ahmed et al., 2021). Another study investigated the wear volume of clear aligner attachments via a 24-week simulated insertion/removal period (Roatkanjanaporn et al., 2025). The researchers tested three different composites, two flowable and one packable. They had similar findings to Ahmed et al., observing that there was comparable wear among the three composites. Although packable composite has a higher filler content, this does not appear to give a significant advantage to wear resistance over flowable composite in the context of CAT attachments. These findings from the current literature reinforce the idea that packable and flowable resin composite are both acceptable options for CAT attachments and the decision on which material is used should be based on clinical workflows and provider preferences.

Chemistry of Dental Resin Composite Photopolymerization

Dental resin composites are used widely in restorative dentistry and orthodontics for their excellent aesthetic, mechanical and adhesive properties. The performance and durability of these materials depend fundamentally on their polymerization chemistry, which allows conversion from a moldable state to a hard and durable vitrified form (Ferracane, 2011). There is a general composition to all resin composites such as a radiopaque glass acting as a reinforcing filler, a dimethacrylate polymeric matrix, and photo-initiator chemicals that initiate and propagate the polymerization reaction. There are three phases to polymerization: initiation, propagation and termination (Kowalska et al., 2021). The initiation phase begins with free radicals lengthening the polymer chains within the composite. These free radicals are formed by the photo-initiators via a photochemical reaction when light-cured. The most common photo-initiator is camphorquinone (CQ) which enters an excited state after absorbing blue light (wavelength 400-500nm) and begins to produce free radicals which attack the C=C double bonds within the methacrylate monomers. When a monomer is attacked by a free radical, after the electron transfer has occurred there is a remaining electron that is transferred from the initiating side of the monomer to the opposite terminal which converts the entire monomer molecule itself into a radical. The radical form of the monomer repeats the initiation reaction with another monomer, extending the polymer chain and maintaining a radical on the terminal end which propagates the chain reaction. Two radicals reacting with each other terminates the reaction, leaving some uncured monomers. The proportion of uncured to cured monomers is known as degree of conversion (DC).

Storage Conditions and Polymerization Dynamics

Proper storage conditions of resin composites is necessary to preserve their polymerization efficacy and properties. It is typically recommended that resin composites are stored between 4°C and 20°C (El-Maksoud et al., 2024). It is common practice in orthodontic offices to pre-fill the aligner templates with the resin composite prior to the bonding appointments to facilitate minimal chair time for the patient. These pre-filled aligner templates are often set aside within a dental clinic, potentially subjecting the resin composite to temperatures beyond the recommendation of 20°C depending on the temperature of the office. Research has shown that pre-cure temperature of resin composites when cured by a LED curing unit does not affect the composites hardness and shrinkage (Osternack et al., 2013). However, research has also shown that storage duration greater than 6 months can have a negative effect on the rate of strengthening and ultimate strength which has been attributes to less effective polymerization due to the gradual decomposition of the photo-initiator over time (de Lange et al., 1983).

Microhardness as a Measure of Polymerization

Microhardness is a measure of a materials scratch resistance on a micron scale (Honarvar Nazari et al., 2022). Microhardness is a fundamental property that is necessary for the longevity of resin composite in resisting surface damage from abrasive forces (Berto-Inga et al., 2022). Microhardness testing using the Vickers hardness test is a widely accepted method for indirectly determining the degree of conversion of resin composite (Pirmoradian et al., 2020). Degree of conversion (DC) is a physical parameter that describes the proportion of the remaining aliphatic C=C double bonds in a sample of cured resin composite relative to the total number of aliphatic C=C double bonds in the uncured composite (Galvão et al., 2013). The DC of resin composite can tell us important information about the final physical and mechanical properties of a sample and is most commonly measured using Fourier Transform Infra-red Spectroscopy (FT-IR) (Galvão et al., 2013). Microhardness and DC are intimately linked, with a more completely cured resin sample having a high microhardness value and DC when compared to a more incompletely cured sample. Dental composite with a Vickers microhardness of 50 or beyond is regarded as adequate for clinical use (García-Contreras et al., 2015).

FT-IR Spectroscopy in Dental Materials Research

Fourier-transform infrared (FT-IR) spectroscopy is a critical tool in evaluating the DC in dental composites. Dental composites undergo vitrification via a photopolymerization reaction when exposed to light. The aliphatic C=C double bonds of the methacrylate monomers are broken by the reaction and polymerize into a chain of C-C single bonds, thus decreasing the number of carbon double bonds in the cured state relative to the uncured state (Moraes et al., 2008). The aliphatic C=C double bonds absorb light at the 1640 cm^{-1} wavenumber, and this absorption can be measured using FT-IR (Moraes et al., 2008). If two samples of resin composite have the same starting concentration of aliphatic C=C double bonds and one sample undergoes some degree of light curing, the cured sample should show a decreased absorption at the 1640 cm^{-1} wavenumber ($1/\lambda$) due to a decrease in the C=C double bond concentration. There are two methods for detecting absorption: transmission and reflectance (Moraes et al., 2008).

The oldest and most commonly used method is transmission, where in the detector measures the intensity of the infrared radiation once it has passed through the sample. The reflectance method measures the infrared intensity after it has reflected off the sample. The different reflectance methods available include attenuated total reflectance, specular, and diffuse.

Shear Bond Strength of Orthodontic Attachments

Shear bond strength (SBS) testing is a standard method for evaluating adhesion between orthodontic attachments and enamel surfaces (Finnema et al., 2010). In 1975, Reynolds described the appropriate shear bond strength for an orthodontic bracket to be between 5.9 to 7.8 MPa (Cruz et al., 2021). Adequate SBS ensures the stability of an orthodontic attachment which is especially important during clear aligner therapy where the time between adjustments can be several weeks longer than with fixed appliances. Debonded attachments can severely compromise the efficacy of clear aligner tooth movement, so sufficient SBS is necessary for efficient and predictable treatment. For many years it was believed that exceeding the SBS established by Reynolds more than 40 years ago would run the risk of damage to the enamel, causing fracture or cracks when the attachments were removed (Rüger et al., 2011). Improvements in dental materials and curing methods has allowed the SBS to increase to 14.05±6.52 MPa without risk of irreversibly damaging the enamel upon removal of attachments (Cruz et al., 2021).

Aims and Hypotheses

The general aim of this study was to evaluate the effect of storage time on the mechanical and chemical characteristics of resin composite attachments fabricated using clear aligner template trays. As attachments play a critical role in facilitating complex tooth movements during clear aligner therapy, understanding how storage time influences their integrity is essential for ensuring optimal clinical outcomes.

The specific aims of this study were:

1. To assess the effect of storage time on the microhardness of resin composite attachments fabricated using clear aligner template trays, as an indicator of the degree of polymerization and surface mechanical integrity.
2. To determine the degree of ambient light-induced polymerization of uncured resin composited placed in clear aligner template trays when stored for different periods of time using FT-IR spectroscopy.
3. To evaluate the impact of storage time on the shear bond strength of resin composite attachments bonded to enamel surfaces of human teeth, simulating clinical bonding conditions following delayed polymerization.

Hypotheses

The following null hypotheses were tested:

1. There is no relationship between the microhardness of clear aligner attachments and pre-polymerization storage time.
2. There is no relationship between storage period curing as measured using FT-IR spectroscopy and pre-polymerization storage time.
3. There is no relationship between the shear bond strength of clear aligner attachments bonded to enamel and pre-polymerization storage time.

Methods

Sample Size Selection

Sample size calculations were performed to achieve 80% power for detecting meaningful differences between time points using a two-sample t-test at $\alpha = 0.05$. Preliminary data estimated variability, indicating that at least 26 samples per group were needed to detect an effect size of 0.8 standard deviations. For the shear bond strength test, a convenience sample was used.

Microhardness Testing

To evaluate the effect of storage time on the microhardness of resin composite attachments, samples were prepared and tested at five distinct time intervals: 1 hour, 1 day, 1 week, 2 weeks, and 1 month. Each group consisted of 26 samples (n=26).

A standardized rectangular model, representing an aligner attachment in thickness, was digitally designed using the software 3D-Builder (Microsoft Corporation, Redmond, WA, USA), with dimensions of 7mm x 5mm x 2mm. The design was exported for 3D printing, and the printed model was used to fabricate clear aligner template trays through thermal vacuum forming using Zendura™ FLX (Bay Materials LLC, Fremont, CA, USA), a thermoplastic polyurethane commonly used in the fabrication of clear aligner trays. The thickness of the Zendura™ FLX material was .030” (.76mm). The template trays were filled with Tetric EvoCeram (Ivoclar Vivadent, Schaan, Liechtenstein), a nanohybrid resin composite in shade A1 (Figure 2). Composite was placed into each well using a dental plastic instrument and leveled flush with the surface of the template to ensure consistency with the dimensions of the original digital model. Following placement, the filled templates were immediately stored in opaque black retainer cases to prevent light exposure and stored in a dark incubator at a controlled room temperature of 20°C for the duration of each group’s designated storage time.

After completion of the storage period, the samples were removed from the incubator and light-cured through the top surface of the template using an Ortholux™ Luminous LED Curing Light (3M Oral Care, St. Paul, MN, USA) for a standardized duration of 9 seconds. After polymerization, the composite attachments were removed from the aligner template wells and prepared for microhardness analysis.

Microhardness testing was conducted using a Buehler Micromet® Microhardness Testing Machine (Buehler, Lake Bluff, IL, USA)(Figure 1). A Vickers diamond pyramid indenter was applied to each specimen using a load of 1.961N for a dwell time of 10 seconds. The force was applied to the flat composite surface that was in contact with the base of the aligner template well to simulate the external surface of an attachment. Five indentations were made per sample at non-overlapping regions of the composite surface, and the resulting dimensional measurements were used to calculate the Vickers Hardness Number (VHN). The mean VHN of the five indentations was recorded as the representative value for each sample.

This process was repeated for all specimens across all time points, generating a total of 130 data points (26 samples x 5 time groups) for statistical analysis.

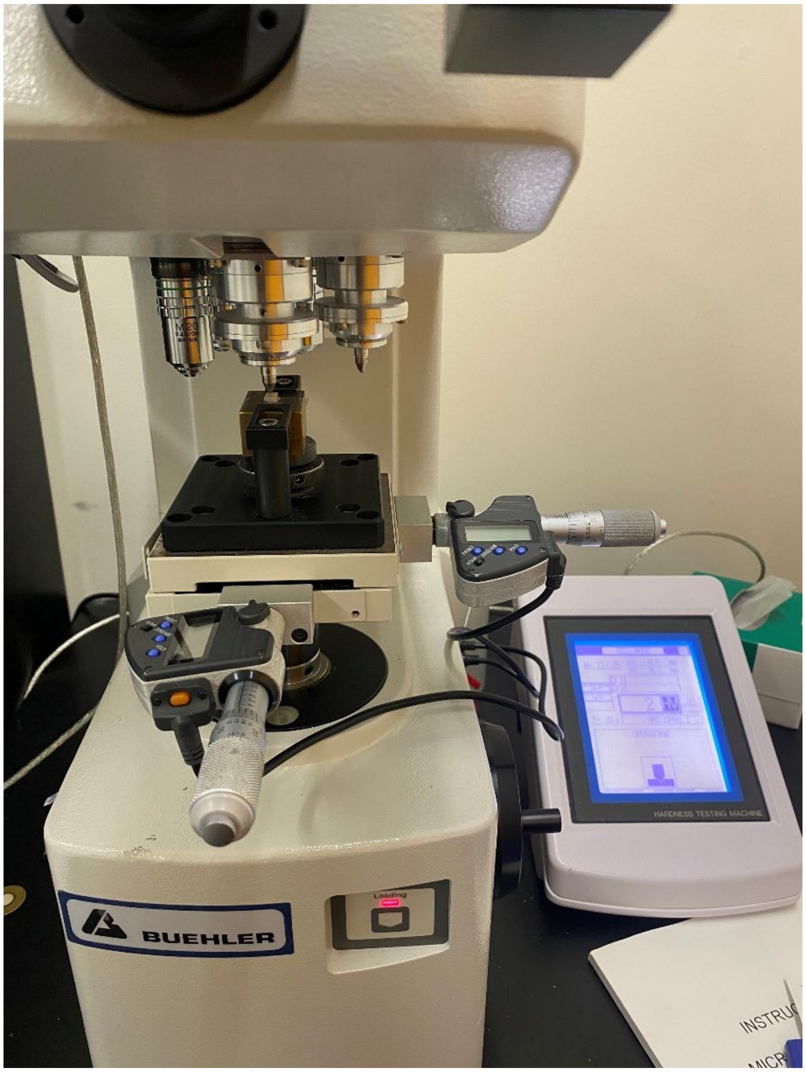

Figure 1: Photo of the Buehler Micromet® Microhardness Testing Machine. An attachment sample can be seen receiving an indentation on the testing platform for microhardness analysis.

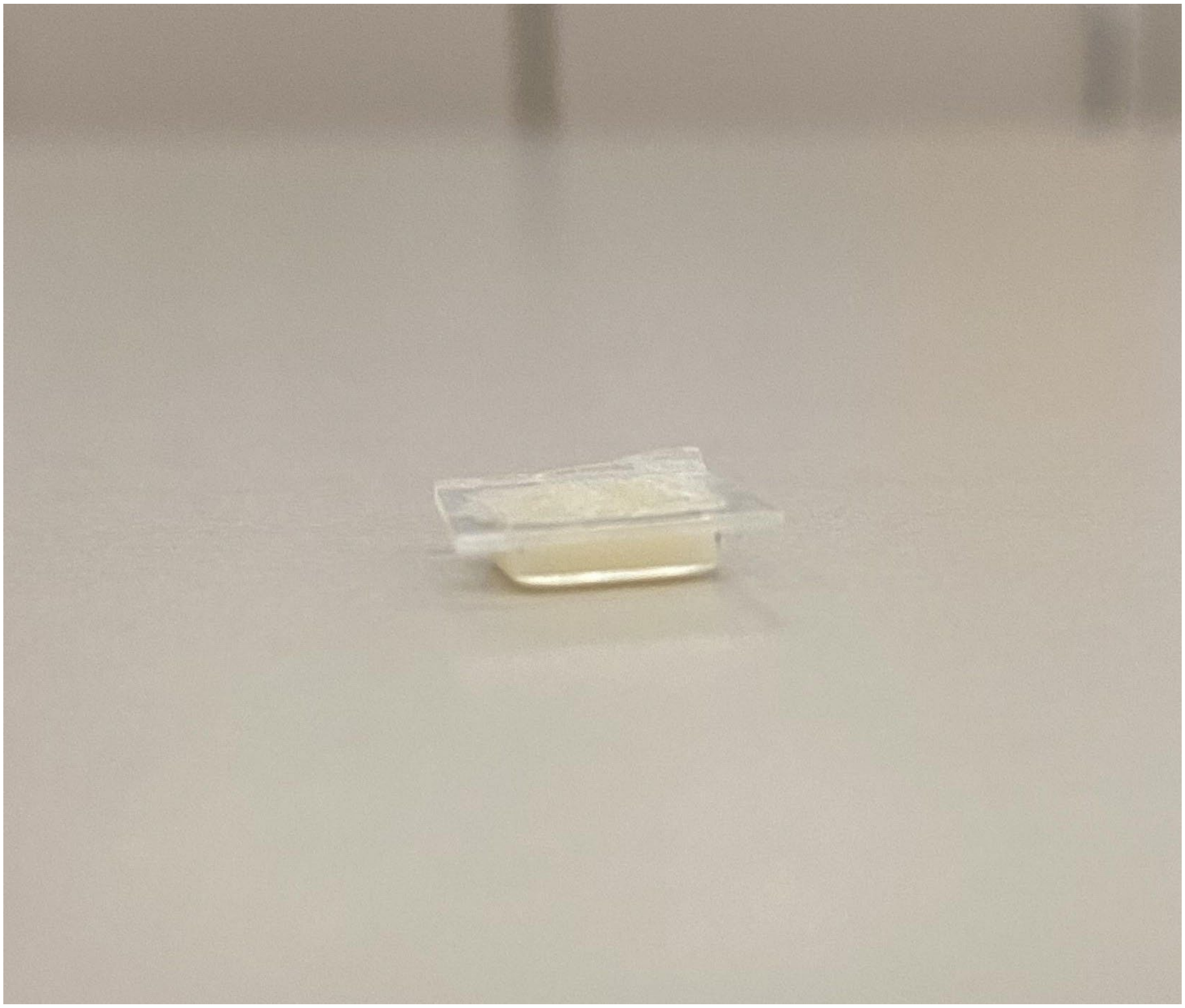

Figure 2: Photo of microhardness template tray filled with composite.

Spectroscopy

To evaluate the degree of polymerization of the resin composite during storage, Fourier-transform infrared (FT-IR) spectroscopy was employed. Samples were tested at five storage intervals: 1 hour, 1 day, 1 week, 2 weeks, 1 month.

The number of samples for each respective time point was as follows: 1 hour (n=28), 1 day (n=28), 1 week (n=27), 2 weeks (n=29), and 1 month (n=29).

Using 3D-Builder (Microsoft Corporation, Redmond, WA, USA), a digital model of a small rectangular attachment measurement 3mm x 1.25mm x 2mm was created. This model was 3D printed to fabricate a master mold, which was then used to form thermoplastic clear aligner templates via thermal vacuum forming using Zendura™ FLX.

Each aligner was filled with Tetric EvoCeram nanohybrid resin composite in shade A1 using a dental plastic instrument. Composite was carefully leveled with the surface of the template to match the dimensions of the digital design. Immediately after placement, filled templates were placed in opaque black retainer cases and stored in a dark temperature-controlled incubator at 20°C for the designated storage time.

After completion of the assigned storage period, samples were removed from the template wells and analyzed using FT-IR spectroscopy. Spectroscopic analysis was conducted using a Thermo Scientific™ Nicolet™ iS50 FT-IR Spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA)(Figure 3). Spectra were initially obtained in Attenuated Total Reflectance (ATR) mode and then subjected to Advanced ATR Correction to convert the spectral output to Transmission mode for accurate quantification of absorbance.

The absorbance value at the wavenumber 1640 cm^{-1}, corresponding to the stretching vibration of aliphatic C=C double bonds, was recorded for each sample. The presence and magnitude of this absorbance peak served as a measure of the extent of polymerization during storage. A decrease in absorbance at 1640 cm^{-1} indicated a reduction in unreacted C=C double bonds, suggesting polymerization had occurred during storage. This procedure was repeated for all samples across all time points, yielding a total of 141 data points for analysis.

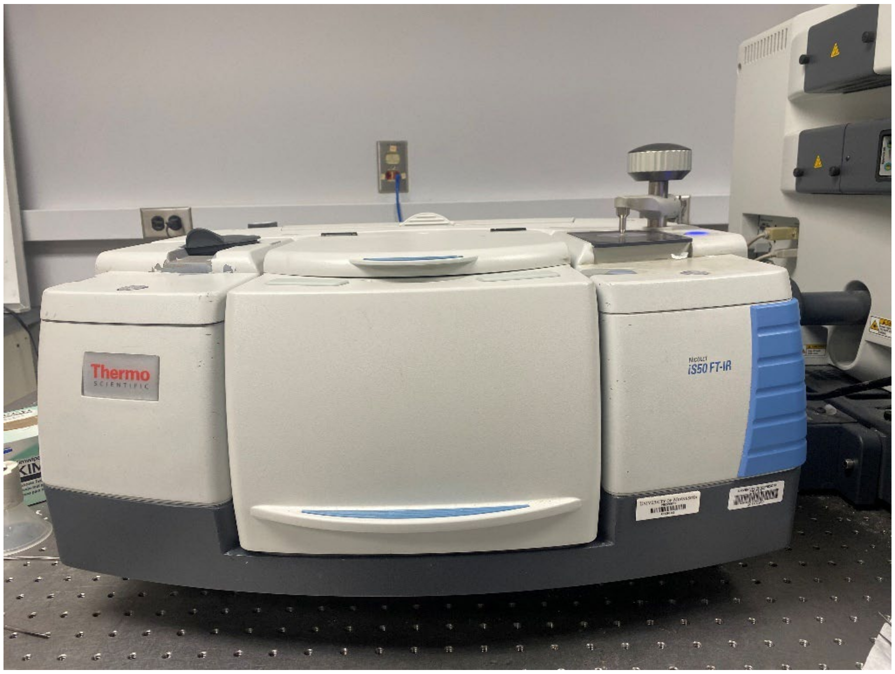

Figure 3: Photo of the Thermo Scientific™ Nicolet™ iS50 FT-IR Spectrometer. A sample of uncured composite can be seen secured under the sample press undergoing spectroscopic analysis.

Shear Bond Strength

To assess the effect of storage time on the shear bond strength of resin composite attachments to enamel, tests were conducted at five storage intervals: 1 hour (n=8), 1 day (n=6), 1 week (n=6), 2 weeks (n=6), and 1 month (n=6).

Human premolar teeth were collected from the University of Minnesota Oral Surgery Department following extraction. Premolars with intact facial enamel surfaces and no gross carious lesions restorations were selected for inclusion. The teeth were stored in 0.1% thymol solution at room temperature for a period not exceeding two months. Research has shown that teeth preserved in 0.1% thymol solution maintain consistent mechanical and chemical enamel properties if stored for two months or less (Aydın et al., 2015). The thymol solution was prepared by dissolving 1g of thymol crystals in 1L of heated distilled water.

Each tooth was mounted in self-curing dental acrylic resin with the crown fully exposed to allow access to the facial surface. After mounting, a digital scan of each tooth was acquired using the iTero Element® Intraoral Scanner (Align Technology, San Jose, CA, USA)(Figure 4). The scanned files were imported into 3D-Builder, where a 3mm x 1.25mm x 2mm rectangular attachment was digitally added to the height of contour of the facial surface of each tooth model to simulate a clear aligner composite attachment (Figure 5).

The modified digital models were then 3D printed to produce physical replicas, and individualized thermal vacuum-formed templates were fabricated from each model using Zendura™ FLX aligner material and trimmed, resulting in custom-fit mini-template trays for each tooth. Each template well was filled with Tetric EvoCeram nanohybrid resin composite in shade A1 using a dental plastic instrument, leveled flush with the template surface, and stored in opaque black retainer cases in a dark, temperature-controlled incubator at 20°C for the designated storage duration.

At the time of testing, the facial surface of each premolar was prepared by etching with 37% phosphoric acid gel for 15 seconds, followed by a water rinse and drying with compressed air until frosting appeared. Assure® Plus (Reliance Orthodontic Products, Itasca, IL, USA) was applied to the etched surface using a microbrush, gently air dispersed, and light-cured for 6 seconds with the Ortholux™ Luminous LED Curing Light.

The corresponding aligner template was retrieved from storage and seated onto the tooth with finger pressure to ensure uniform adaptation and contact between the composite and enamel surface. The same curing light was used to polymerize the composite for 9 seconds through the template. After curing, the template was removed, and any flash was eliminated using a high-speed handpiece with a carbide finishing bur.

Shear bond strength testing was performed using a MTS Criterion Model 41 (MTS Systems Corporation, Eden Prairie, MN, USA). Each specimen was positioned in the universal testing machine, and a vertical metal pin applied a shear force at the interface between the attachment and the tooth (Figure 6). The force required to debond the attachment was recorded in MPa and documented as the maximum shear bond strength value for each sample.

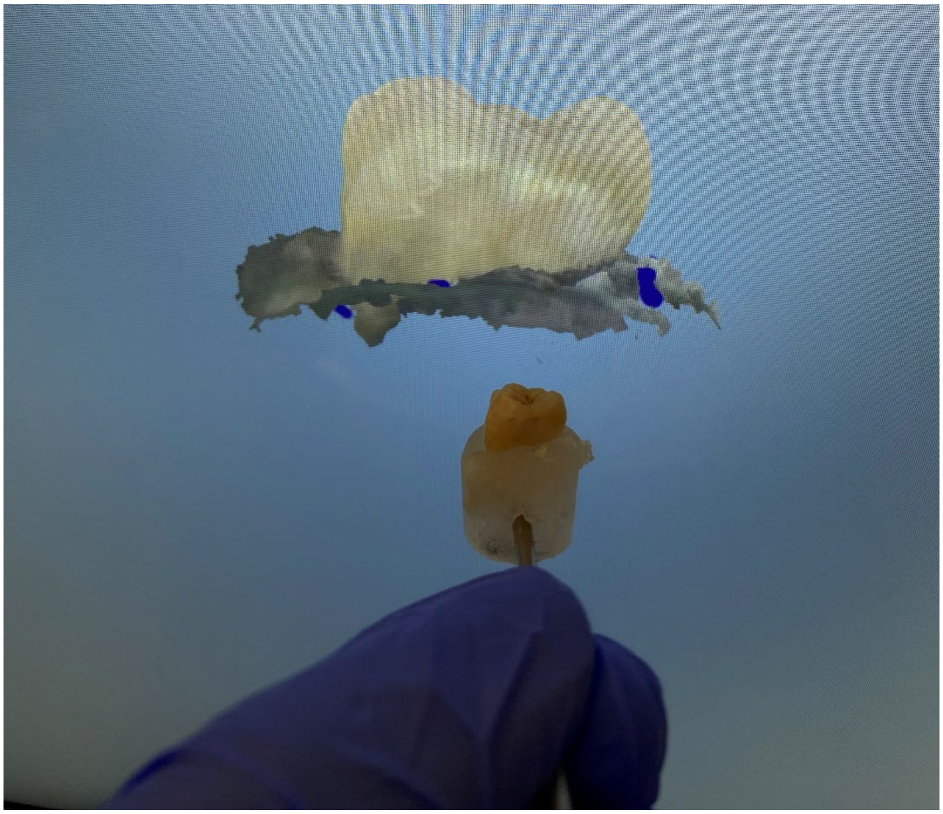

Figure 4: Photo of digital scan of acrylic mounted premolar.

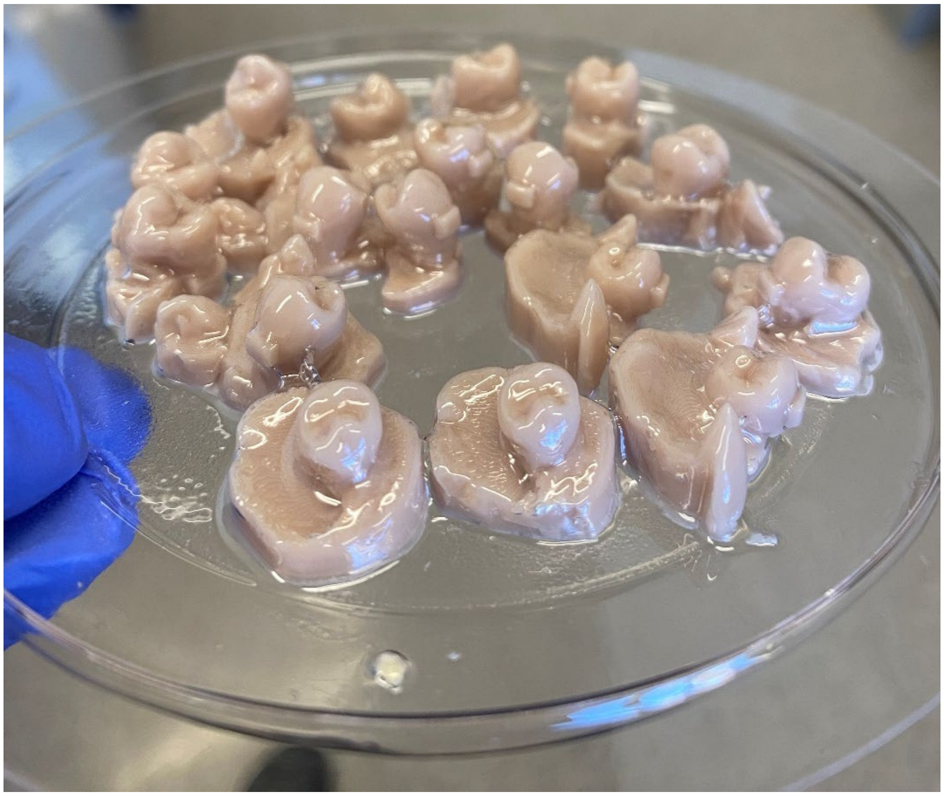

Figure 5: Photo of 3D-printed replicas of mounted premolars with aligner attachments added to the facial height of contour.

Figure 6: Photo of mounted premolar after its aligner attachment has undergone shear bond strength testing.

Statistical Analysis

Descriptive statistics were calculated for each outcome variable and are reported as mean, standard deviation (SD), median, and range. These summary measures were used to characterize the distribution of data within each time point group for all three experiments: microhardness, spectroscopy, and shear bond strength.

To compare means across the five storage time points (1 hour, 1 day, 1 week, 2 weeks, and 1 month), one-way analysis of variance (ANOVA) was performed. When the overall F test from the ANOVA indicated a statistically significant difference among group means, post hoc pairwise comparisons were conducted to identify which specific groups differed. Tukey's method was used to adjust for multiple comparisons and control the family-wise error rate.

A p-value of less than 0.05 was considered statistically significant for all tests. All statistical analyses were performed using SAS software, version 9.4 (SAS Institute Inc., Cary, NC).

Results

Microhardness

The mean, standard deviation, median, and range for microhardness are shown in Table 1. The mean Vicker's microhardness per time point ranged from 34.8 (1 day) to 40.0 (1 week). A significant result after an ANOVA (p-value: 0.0004) prompted a Tukey's post hoc test which showed statistically significant differences in microhardness between 1 day (34.8) vs. 1 week (40.0)($p=0.002$), indicating greater microhardness at the 1 week storage time point. Tukey's post hoc test also showed statistically significant differences in microhardness between 1 week (40.0) vs. 2 weeks (36.2)($p=0.0107$), suggesting reduced microhardness when samples were stored for 2 weeks compared to 1 week. Figure 7 shows a box and whisker plot of the microhardness test results. Despite significant differences between some groups, overall, there was no clear trend in microhardness with increased pre-polymerization storage time of composite attachments.

Storage time	n	Mean	S.D.	Minimum	Median	Maximum	Tukey Grouping*
1 hour	26	37.5	3.9	28.3	37.6	44.6	A, B
1 day	26	34.8	3.2	29.4	34.3	42.9	B
1 week	26	40.0	3.8	33.9	40.7	49.8	A
2 weeks	26	36.2	5.3	28.4	37.1	48.7	B
1 month	26	37.0	4.0	29.8	36.6	46.7	A, B

Table 1: Vicker's microhardness (VHN) of resin composite attachments with different pre-polymerization storage times. *Groups sharing the same letter are not significantly different ($p<0.05$).

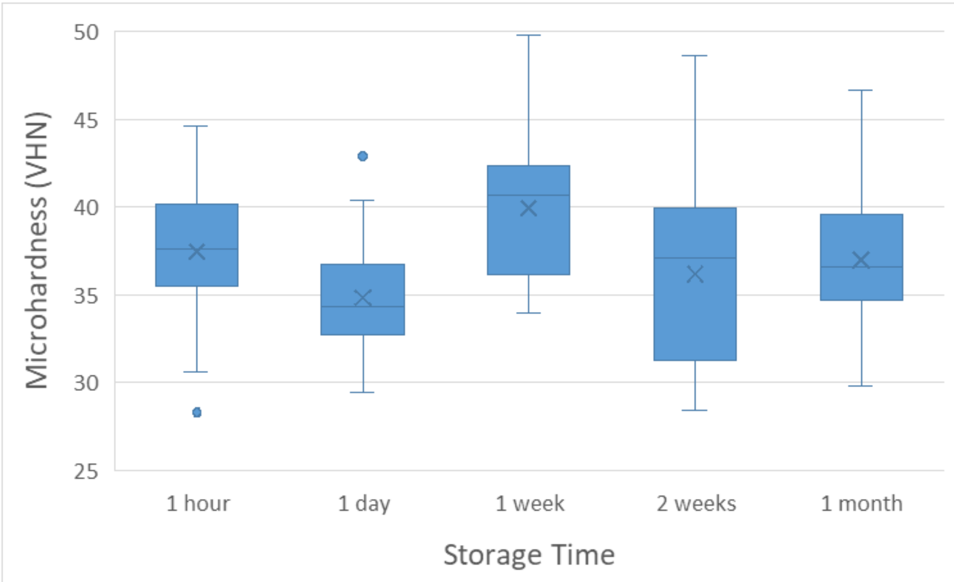

Figure 7: Box and whisker plot of Vicker's microhardness of resin composite attachments with different pre-polymerization storage times. Each box contains the middle 50% of the data. The bottom of the box represents the 25th percentile (Q1); the top edge represents the 75th percentile (Q3). The overall height of the box shows the spread of the central half of the data between Q1-Q3 (IQR). The horizontal line within each box marks the median value. The "X" symbol within each box marks the mean value. On each box the "whiskers" represent 1.5 x IQR from the upper and lower edges of the box. The bottom vertical line represents the smallest value within 1.5 x IQR below Q1 and the top vertical line represents the largest value within 1.5 x IQR above Q3. Any dots outside the vertical lines represent outlier values.

Spectroscopy

The mean, standard deviation, median, and range for absorbance are shown in Table 2. The mean absorbance ranged from 0.056 to 0.064. The standard deviations in all groups were small indicating tight clustering of data. The highest maximum absorbance was 0.088 at 1 week, and the lowest maximum absorbance was 0.059 at 1 day. A significant result after ANOVA (p-value: 0.0004) prompted a Tukey's post hoc test which showed statistically significant differences between 1 hour vs. 1 day, 1 day vs. 1 week, 1 week vs. 2 weeks (Table 2). There was less absorbance in the samples stored for 1 hour (0.062) vs. 1 day (0.056), implying a decrease in the presence of aliphatic C=C double bonds either by ambient light induced polymerization or other molecular changes. There was greater mean absorbance in samples stored for 1 day (0.056) compared to 1 week (0.064). There was less absorbance in samples stored for 1 week compared to those stored for 2 weeks, which again suggests a reduction in the concentration of aliphatic C=C double bonds between the two time points. Figure 8 shows a box and whisker plot of absorbance of uncured composite. Despite statistically significant pairwise differences between individual groups, no clear relationship as noted overall between storage time and absorbance.

Storage time	**n**	**Mean**	**SD**	**Min**	**Median**	**Max**	**Tukey Grouping***
1 hour	28	0.062	0.010	0.054	0.056	0.083	A, B
1 day	28	0.056	0.002	0.052	0.055	0.059	C
1 week	27	0.064	0.010	0.056	0.060	0.088	A
2 weeks	29	0.059	0.002	0.053	0.059	0.065	B, C
1 month	29	0.060	0.003	0.055	0.061	0.065	A, B, C

Table 2: Absorbance at 1640 cm^{-1} wavenumber of uncured resin composite attachments with different storage times. *Groups sharing the same letter are not significantly different ($p<0.05$).

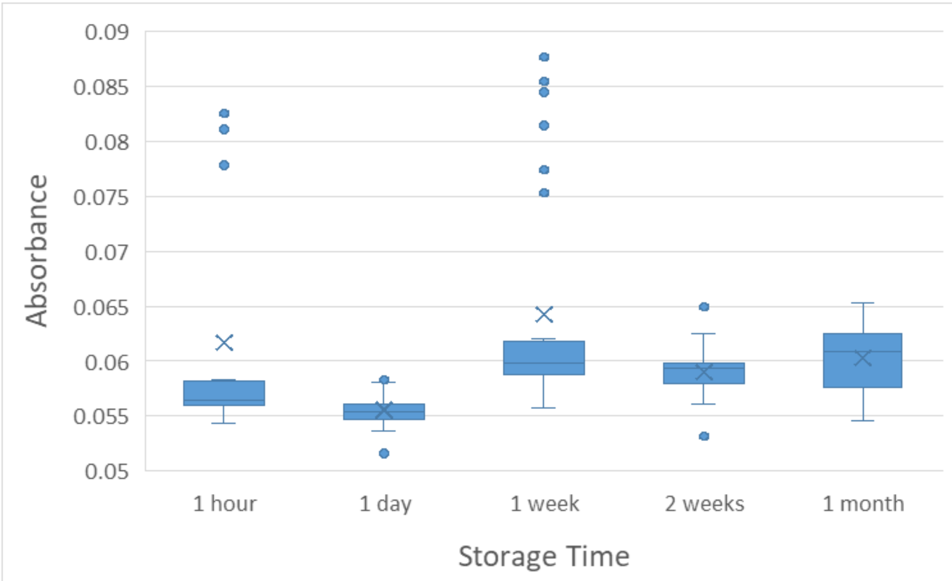

Figure 8: Box and whisker plot of absorbance. Each box contains the middle 50% of the data. The bottom of the box represents the 25th percentile (Q1); the top edge represents the 75th percentile (Q3). The overall height of the box shows the spread of the central half of the data between Q1-Q3 (IQR). The horizontal line within each box marks the median value. The "X" symbol within each box marks the mean value. On each box the "whiskers" represent 1.5 x IQR from the upper and lower edges of the box. The bottom vertical line represents the smallest value within 1.5 x IQR below Q1 and the top vertical line represents the largest value within 1.5 x IQR above Q3. Any dots outside the vertical lines represent outlier values.

Shear Bond Strength

The mean, standard deviation, median and range for shear bond strength are shown in Table 3. The highest mean bond strength was observed at 1 hour (27.0 MPa) while the lowest was at 1 day (13.2 MPa). The standard deviations were relatively large across all the groups with a range of 8.6 MPA to 18.7 MPa, indicating a high degree of variability in shear bond strength measurements. The ANOVA found no statistically significant differences between groups (Table 3). Figure 9 shows a box and whisker plot of shear bond strength. No clear trend or relationship exists between pre-polymerization storage time and shear bond strength of attachments bonded to human teeth.

Storage Time	n	Mean	SD	Minimum	Median	Maximum	Tukey Grouping*
1 hour	8	27.0	13.8	12.4	22.2	49.9	A
1 day	6	13.2	10.3	2.3	11.1	25.9	A
1 week	6	15.7	8.6	2.0	15.4	27.3	A
2 weeks	6	23.8	12.2	10.5	22.2	42.6	A
1 month	6	20.8	18.7	5.1	16.2	55.3	A

Table 3: Analysis of shear bond strength (MPa) across all time points. *Groups sharing the same letter are not significantly different ($p<0.05$).

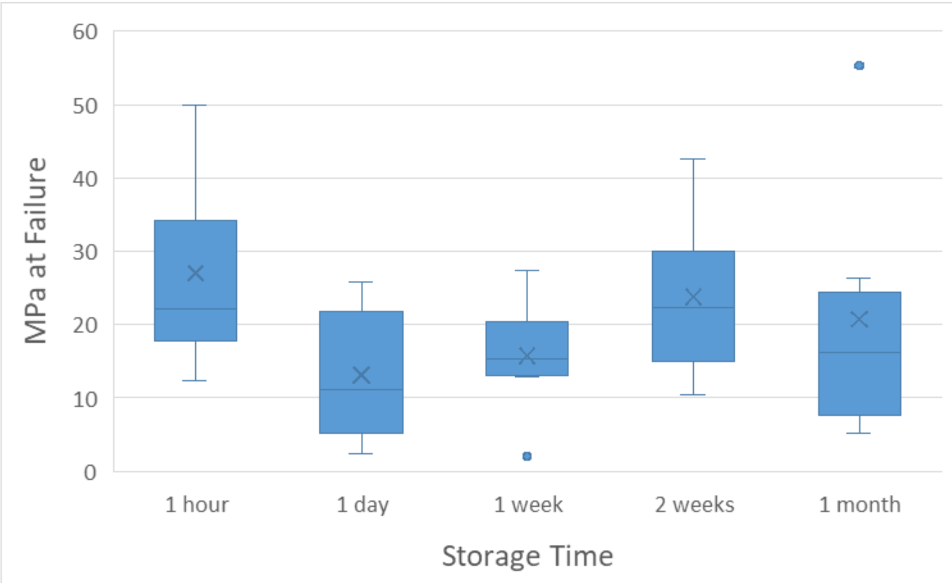

Figure 9: Box and whisker plot of shear bond strength. Each box contains the middle 50% of the data. The bottom of the box represents the 25th percentile (Q1); the top edge represents the 75th percentile (Q3). The overall height of the box shows the spread of the central half of the data between Q1-Q3 (IQR). The horizontal line within each box marks the median value. The "X" symbol within each box marks the mean value. On each box the "whiskers" represent 1.5 x IQR from the upper and lower edges of the box. The bottom vertical line represents the smallest value within 1.5 x IQR below Q1 and the top vertical line represents the largest value within 1.5 x IQR above Q3. Any dots outside the vertical lines represent outlier values.

Discussion

Clear aligner therapy (CAT) has become a cornerstone of modern orthodontics, offering a discreet, removable and patient-friendly alternative to traditional fixed appliances. From its early beginnings in the mid-20th century to its contemporary digital form, CAT has undergone major advancement in design and technology (Kau et al., 2023). A critical element of CAT success is the use of composite attachments. These attachments serve as mechanical anchors for aligners, enhancing their grip on the teeth and enabling complex forces such as extrusion, rotation, and root torque (AlMogbel, 2023). To ensure consistent performance, these attachments must possess optimal mechanical and chemical properties at the time of bonding. This study was conducted to evaluate how pre-polymerization storage time of resin composite attachments influences attachment properties when composite is pre-loaded into aligner template trays, a common practice in many orthodontic clinics aimed at reducing chair time.

Three specific experimental approaches were used to address the central research question. Microhardness was tested to assess the surface mechanical properties of cured composite, which are tightly correlated with degree of polymerization (Pirmoradian et al., 2020). FT-IR spectroscopy was used to quantify chemical changes in the resin composite by measuring residual aliphatic carbon double bonds (C=C), which diminish with polymerization (Moraes et al., 2008). Finally, shear bond strength of composite attachments bonded to human teeth was tested to evaluate whether pre-polymerization storage time affected the strength of the adhesion of the composite to enamel under simulated clinical bonding conditions. Each test provided unique insight into how pre-filled, template-loaded composite attachments behave over storage times ranging from 1 hour to 1 month.

Vickers microhardness testing revealed statistically significant differences among some time points (ANOVA $p=0.0004$), partially rejecting the first null hypothesis. The highest mean microhardness was observed at 1 week, which was significantly greater than at 1 day ($p=0.0002$) and 2 weeks ($p=0.0107$). This result suggests that resin composite stored for 1 week had increased surface hardness compared to that stored for 1 day, possibly due to low-level ambient polymerization or molecular reorganization. However, the reduced microhardness at 2 weeks compared to 1 week implies that prolonged storage may lead to degradation or softening, possibly due to aging or partial decomposition of the photo-initiator system (de Lange et al., 1983). Prior research has reported that the Vickers microhardness value for clinically acceptable resin composite should generally exceed 50 VHN (García-Contreras et al., 2015). The microhardness values in the current study values fell below this threshold, indicating the samples universally did not reach their full polymerization potential when being prepared for microhardness testing, possibly due to under-curing or other inconsistencies in handling. A study conducted by Feinberg et al. also investigated the surface hardness of Tetric EvoCeram that was cured through clear aligner material. Feinberg et al. followed a similar procedure for testing hardness, performing five tests per sample with a minimum of 1mm between indents and the dimensions of the indents being used to calculate a Vickers hardness value (Feinberg et al., 2016). The depth of Feinberg's attachments matches ours at 2mm; however, to cure the composite they used the Bluephase G2 curing light (Ivoclar Vivadent) for 10 seconds. Their curing procedure appears to have achieved a greater degree of polymerization to our study based on the mean hardness value found being 57.0, which better aligns with the recommendations of Garcia-Contreras. Despite increases or decreases in microhardness between time points, the lack of a linear trend correlating to increasing pre-polymerization storage time suggests that significant changes between time points in the current study were due to uncontrolled factors such as unrecorded changes in incubator temperature or inconsistent exposure time to ambient light during handling of the samples, or possibly lack of complete curing.

FT-IR spectroscopy had a similarly curious pattern of results. The absorbance values at 1640 cm^{-1} , indicative of residual aliphatic C=C double bonds, differed significantly across certain time points (ANOVA $p<0.0001$). The lowest absorbance was recorded at 1 day, while 1 week showed the highest absorbance values, including several high outliers. Statistically significant pairwise differences were found between 1 hour vs. 1 day ($p=0.006$), 1 day vs. 1 week ($p<0.0001$), and 1 week vs. 2 weeks ($p=0.0268$). These results reject in part the second null hypothesis that there is no relationship between storage period curing and pre-polymerization storage time. Prior FT-IR studies have shown that a decrease in absorbance at the 1640 cm^{-1} wavenumber indicates that polymerization has occurred due to the consumption of C=C double bonds (Galvão et al., 2013). Interestingly, the fluctuating pattern observed in these results with unexpected high absorbance at 1 week differs from the generally linear decrease reported in fully light-exposed composites (Galvão et al., 2013). Since the photopolymerization reaction of resin composite only consumes C=C double bonds and there is not a chemical pathway for C=C double bonds to be reintroduced, any apparent increases in absorption are likely due to a decreased ambient light exposure during handling relative to other time points. This would preserve a higher concentration of the initial C=C double bonds making it appear that there has been an increase in absorption, when in reality the other time points have seen a decrease in absorption from the potential maximum due to ambient light exposure. As a whole, the results of the current study show no clear trend or relationship between storage time and polymerization during storage. In addition to ambient light exposure during handling, the observed fluctuations may be influenced by variables such as temperature variations, or differences in how the composite settled within the tray.

Shear bond strength testing did not yield statistically significant differences across time points (ANOVA $p=0.32$), accepting the third null hypothesis. Despite numerical trends showing the highest bond strength at 1 hour (mean = 27.0 MPa) and lowest at 1 day (mean = 13.2 MPa), the variability within groups and the limited sample size, constrained by access to extracted premolars, reduced the power to detect a true difference. Notably, the mean shear bond strength values in this study exceeded those established for orthodontic brackets by Reynolds in 1975 (Cruz et al., 2021). These results are consistent with more recent studies showing that modern composites can achieve bond strengths of 14 MPa or higher without risk of damaging enamel (Cruz et al., 2021). Research by Chen performed a similar comparative study of clear aligner shear bond strength between different composite materials. Similar to our study, Chen et al. collected human premolars and bonded clear aligner attachments that were sheared off to give a maximum MPa reading (Chen et al., 2021). They tested two bulk fill composites and one flowable composite, with the results of the average shear bond strength ranging from 15.3 MPa to 23.49 MPa which is a lower range than the bond strengths of our study which ranged from 21.1 MPa to 43.2 MPa. A study performed by Alsaud investigated shear bond strength of clear aligner attachments bonded to ceramic material. In addition to their bonding surface being ceramic instead of enamel, Alsaud's study differed from ours by subjecting the bonded attachments to 10,000 cycles of 5°C to 55°C 30 second baths prior to shear bond testing, simulating the moisture and heat of the oral environment (Alsaud et al., 2022). After thermocycling their aligner attachments were loaded until failure. The mean shear bond strength found by Alsaud with the packable composite they tested was 13.79 MPa, similar to the values established by Cruz and Chen. Kircelli tested the shear bond strength of aligner attachments composed of Tetric EvoCeram bonded to extracted human premolars. Similar to our study, clear aligner attachments of Tetric EvoCeram were bonded to the premolars and mounted in a shear bond testing machine with a blade applying an occluso-gingival force to the attachment until failure (Kircelli et al., 2023). The mean shear bond strength they found was 16.6±3.6 MPa which is consistent with the findings of other studies (Alsaud et al., 2022; Chen et al., 2021; Cruz et al., 2021). From a clinical perspective, our results are promising. Throughout all time points tested, the average shear bond strength of our attachment, even those stored for 1 month prior to bonding, met or exceeded the performance demonstrated by aligner attachments in previous research.

Microhardness and spectroscopy showed parallel findings, particularly in the 1-week group, where both surface hardness and absorbance peaked. This paradoxical relationship, suggesting both high polymerization density (via microhardness) and unpolymerized monomer (via FT-IR), could indicate that polymerization may occur unevenly across the surface and subsurface regions of the material, leaving behind unreacted monomer (Kowalska et al., 2021). Shear bond strength did not show any statistically significant differences between the time points, suggesting that bonding performance may be more robust to subtle chemical and mechanical shifts. Future studies are needed to repeat this test with greater sample sizes to strengthen the evidence of this notion.

Compared to prior investigations, this study was unique in its focus on pre-polymerization storage rather than post-cure performance. Previous studies have focused mainly on the differences between composite types (Ahmed et al., 2021; Lin et al., 2021) or curing protocols (Osternack et al., 2013), while this study examined an understudied clinical question: the effect of storage time on prefilled aligner templates. The implications of this research are particularly relevant for clinicians who load clear aligner trays in advance of bonding appointments. As composite attachments are critical for precise force application and aligner retention, any alteration in their mechanical behavior could influence aligner seating or lead to premature wear. Therefore, while storage time does not appear to have a clear, clinically significant impact on the composite's properties, careful handling and storage of the pre-filled aligners is necessary to ensure consistent performance of the attachments after curing.

Several limitations must be acknowledged. First, the number of extracted teeth available for shear bond strength testing was limited, reducing the power to detect statistically significant differences. The high variability within and between groups also suggests biological variation in enamel surface properties, which could be mitigated in future studies by increasing sample size. Second, while all storage was conducted at 20°C in opaque containers, minor light exposure or temperature fluctuations during handling could not be fully controlled. Future studies should include a more robust temperature, humidity, and light controlled storage environment in addition to methods of further minimizing ambient light exposure while handling samples to precisely isolate the effects of storage duration.

Another limitation is that all testing was performed in vitro, which does not fully replicate intraoral conditions. Clinical factors such as salivary contamination, intraoral temperature, and masticatory forces may interact with composite properties differently than in a laboratory setting. Additionally, the study only tested one composite (Tetric EvoCeram), leaving open the question of whether other popular materials, such as Transbond XT or flowable composites, would show similar or different patterns after extended periods of pre-polymerization storage time in aligner templates.

Several questions remain. For example, what is the maximum allowable storage time before chemical or mechanical properties fall below clinical thresholds? Does the type of composite or aligner material influence the degree of degradation during storage? These questions point to the need for future studies that explore additional composite brands, aligner materials, and curing systems.

In conclusion, this study demonstrated that storage time up to one month does not have a clear effect on the chemical or mechanical properties of pre-loaded resin composite in aligner template trays. Instead, other factors such as handling of the pre-loaded aligner template trays and storage conditions may have a larger impact. These findings offer important insights for clinicians who prepare aligner templates in advance of bonding and reinforce the need for careful handling protocols to preserve composite performance. While longer storage times may not compromise bonding, imprecise handling and storage protocols may introduce variability in material properties that could affect clinical outcomes. Ongoing research is needed to refine best practices and define clear guidelines for storage and handling pre-loaded attachment templates in aligner therapy.

Conclusions

- Microhardness, degree of polymerization via FT-IR spectroscopy and shear bond strength of composite attachments did not demonstrate any clear, linear trends as storage time increased.
- Storing pre-filled aligner attachment template trays for up to one month does not appear to have a significant effect on attachment properties.
- Variation in composite properties and performance across time points may be better explained through differences in ambient light exposure during handling and storage temperature fluctuations.

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