The Effect of Aging by Storage on the Flexural Strength of Self-cured and Dual-cured Bis-acryl Resins used as Provisional Restoration Materials in Fixed Prosthodontics

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ABSTRACT

Background. Provisional restorative materials require good mechanical properties, such as sufficient flexural strength, to remain functional within the oral cavity. Developments in provisional materials have resulted in the production of bis-acryl resins with varying curing methods. However, their performance in a moist environment remains uncertain.

Objective. This in-vitro study aimed to assess the effect of aging by storage on the flexural strength of self-cured and dual-cured bis-acryl resin materials.

Methods. Self-cured (*Protemp 4*) and dual-cured (*Care C&B*) bis-acryl resins were used in this study. A total of 40 bar-shaped specimens were fabricated and stored in distilled water at 37 ± 1°C. The control group was stored for 1 day (24 hours) while the treatment group was stored for 7 days prior to testing. Flexural strength was determined through a Three-Point Bending Test using a universal testing machine. Paired t-test was used to compare the mean flexural strengths of the bis-acryl resins after 1 day and after 7 days of storage. Independent T-test was used to compare the mean flexural strengths of the self-cured and the dual-cured bis-acryl resin materials within the same storage period.

Results. The results showed that after 7 days of aging by storage in distilled water at 37 ± 1°C, both self-cured and dual-cured bis-acryl resin materials exhibited a statistically significant decline in mean flexural strength (p <0.05). Within the same storage period, the difference in mean flexural strengths of the two materials was not statistically significant (p >0.05).

Conclusion. Within the limitations of the study, it was found that aging by storage significantly decreased the mean flexural strength of the self-cured (*Protemp 4*) and dual-cured (*Care C&B*) bis-acryl resin materials. Future studies simulating actual intraoral conditions are recommended.

Keywords: flexural strength, self-cured, dual-cured

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INTRODUCTION

A provisional restoration is placed after the tooth preparation phase and before delivery of the definitive fixed partial prosthesis.[1] It is necessary for pulpal protection, tooth position maintenance, occlusal function, and intra- and inter-arch relationship preservation.[2] On average, the fabrication of a definitive prosthesis by the dental laboratory takes about 7–10 days during which the provisional restoration must serve its purpose.[3]

One of the mechanical properties that contribute to the success of a provisional restoration is having adequate flexural strength. The flexural strength of a material corresponds to the maximum stress that it can withstand before failure when subjected to a bending load. During mastication, provisional restorations bear heavy occlusal load which may generate deflections in the material. For instance, in a three-unit provisional bridge restoration, the pontic bends towards the edentulous ridge due to masticatory load. This exerts compressive stress on the occlusal surface and tensile stress on the tissue surface, thus producing flexural stresses on the provisional bridge.[4] Therefore, sufficient flexural strength is desired to withstand functional forces without undergoing deformation.[5] At low flexural strengths, the provisional restoration may fracture leading to tooth movement as well as functional and esthetic concerns.[6]

In addition to masticatory forces, the presence of food components, beverages, moisture from saliva, and constant thermal and chemical changes can influence the properties of provisional materials placed inside the mouth.[5,7] To simulate these intraoral conditions and determine how they affect the material's properties, the process of aging may be applied. A simple and commonly used aging technique is aging by storage. This consists of storing the materials in a fluid (usually distilled water or artificial saliva) for a pre-determined period at a temperature of 37°C.[8]

Provisional restoration materials can be divided into two main categories based on their composition: mono-methacrylates or acrylic resins and dimethacryclates or bis-acryl/composite resins. Monomethacrylates include self-cure polymethylmethacrylate (PMMA) while dimethacrylates include bisphenol A-glycidyl dimethacrylate (Bis-GMA) and urethane dimethacrylate (UDMA; light-cured resins).[9]

Compared to PMMA, bis-acryl resins have reduced polymerization shrinkage and heat production due to their divinyl methacrylate monomer and filler particle content.[10,11] In addition, bis-acryl resins are dispensed through automix tips allowing easier manipulation and less air entrapment. Nowadays, bis-acryl resins have different modes of curing available, namely self-cured, light-cured, and dual-cured.[12] This provides an opportunity to select a material based on the clinician's preferred mode of curing.

Recently, a new dual-cured bis-acryl resin material called *Care C&B* has been introduced to the Philippine market. However, limited data has been found regarding its mechanical properties, how it performs in a moist environment, and how it compares to extensively studied provisional materials, such as *Protemp 4* (a self-cured bis-acryl resin). Hence, this study aims to determine the effect of aging by storage on the flexural strength of *Care C&B* dual-cured bis-acryl resin compared with that of *Protemp 4* self-cured bis-acryl resin.

MATERIALS AND METHODS

Study Design and Sample Size

A true experimental study design was conducted to compare the flexural strength of the self-cured and the dual-cured bis-acryl resin materials after storage in distilled water for two different durations. A total of 40 bar-shaped bis-acryl resin specimens were fabricated, consisting of 20 self-cured and 20 dual-cured specimens. The self-cured specimens (n=20) were then randomly assigned to a control group (n=10) and a treatment group (n=10). The same process of randomization was performed on the dual-cured specimens. The sample size was computed using G*Power 3.1.9.7. Statistical Power Analysis through a two-group, two-tailed t-test with a large Cohen's d effect size of 1.33, a statistical power of 0.8, and a desired statistical significance of 0.05.

Study Setting

The study was conducted in the Research Laboratory of the College of Dentistry, University of the Philippines Manila. The laboratory was equipped with the necessary materials; it was well-lit and was air-conditioned. The temperature of the laboratory was maintained at a constant 20°C.

Data Collection

Materials

The manufacturer, composition, mode of curing, shade, and lot number of the two bis-acryl resins used in the study are described in Table 1.

Specimen fabrication

Following the manufacturer's instructions, the catalyst and base pastes were dispensed on a metal mold until the mold was filled (Figures 1A-D). The dimensions of the specimens were 25 mm x 2 mm x 2 mm based on International Standards Organization Specification No. 27.[15] For curing of dual-cured specimens, light curing units (VK-107, Vokodak, China) that produce light with an intensity greater than 1200 mW/cm^2 were used.

Any flash or excess material was carefully removed by gently abrading the specimens with wet silicon carbide abrasive paper (320 grit) until the specified dimensions were reached.[16] While abrading, the specimen's dimensions were frequently measured using a digital caliper (JIGO-150, Jigong, China).[17]

Table 1. Bis-acryl Resins for Provisional Restorations Used in the Study

Material	Manufacturer	Composition	Mode of Curing	Shade	Lot No.
Protemp 4	3M ESPE, Germany	**Base paste:** Ethoxylated bis-phenol A dimethacrylate, Amorphous silica (7631-86-9), surface modified with 2-propenoic acid, methyl-, 3-(trimethoxysilyl)propyl ester (2530-80-0) and phenyltrimethoxy silane (2996-92-1), Reaction Products of 1,6-DiIsocyanatohexane with 2-[(2-Methacryloyl) Ethyl]6-Hydroxyhexanoate and 2-Hydroxyethyl Methacrylate (DESMA), 2-Propenoic acid, 2-methyl-, 3-(trimetoxysilyl)propyl ester, hydrolysis products with silica[13] **Catalyst paste:** 2,2'-[(1-methylethylidene)bis(4,1-phenyleneoxy)] bisethydiacetate, Benzyl-phenyl-barbituric acid, 2-Propenoic acid, 2-methyl-, 3-(trimetoxysilyl)propyl ester, hydrolysis products with silica, (1-methylethylidene)bis(4,1-phenyleneoxy-2,1-ethanediyl)(1-phenylenoxy-2,2'ethoxyethanediyl)bisacetate, tert-Butyl peroxy-3,5,5-trimethylhexanoate[13]	Self-cure	A2	10383474
Care C&B	VERICOM, South Korea	**Base paste:** barium silicate, fumed silica, dimethacrylate stabilizer, photo initiator, pigments[14] **Catalyst paste:** barium silicate, fumed silica, dimethacrylate, catalyst, stabilizer[14]	Dual-cure	A2	TE4201A2

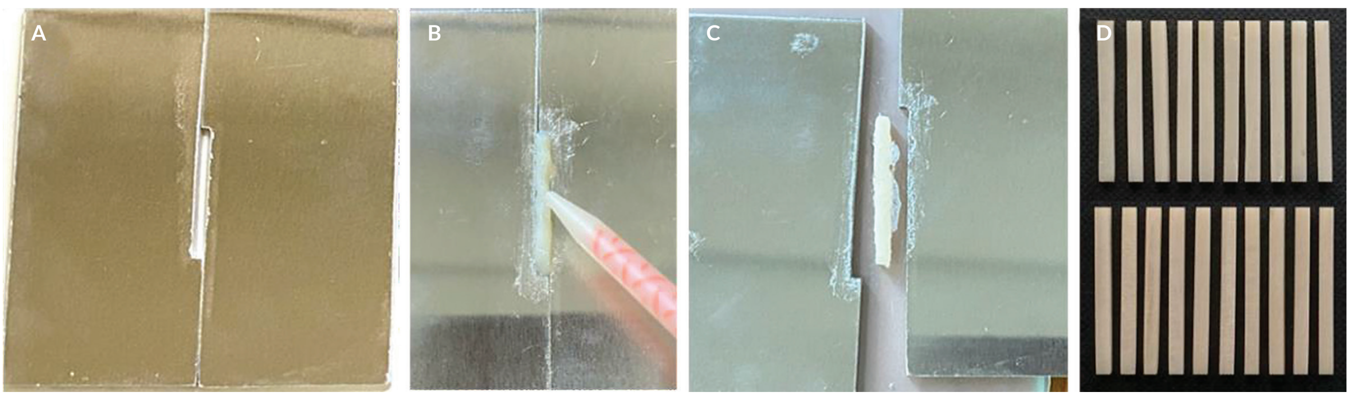

Figure 1. **(A)** The custom metal mold for specimen fabrication, **(B)** Dispensing the bis-acryl resin material, **(C)** A freshly prepared specimen, **(D)** Actual specimens tested in the study.

Aging by Storage

After achieving the specified dimensions, the specimens were stored in distilled water and placed inside an incubator (Freas 815, Precision Scientific Company, USA) with a temperature of 37 ± 1˚C to simulate a moist environment. The control group was stored in distilled water at 37 ± 1˚C for 1 day (24 hours) while the treatment group was stored in distilled water at 37 ± 1˚C for 7 days before testing.[11]

Flexural Strength Testing

After the specified storage durations, specimens were removed from water storage and dried with absorbent paper. Testing commenced immediately. The flexural strength of each specimen was determined by conducting a three-point bending test, using a universal testing machine (AGS-X, Shimadzu, Japan). Each specimen was placed on the jig of the universal testing machine. It was held by two support beams that were 20 mm apart (Figures 2A-D). The crosshead of the machine was activated with a speed of 1.0 mm/min towards the middle of the jig until the specimen fractured.[15]

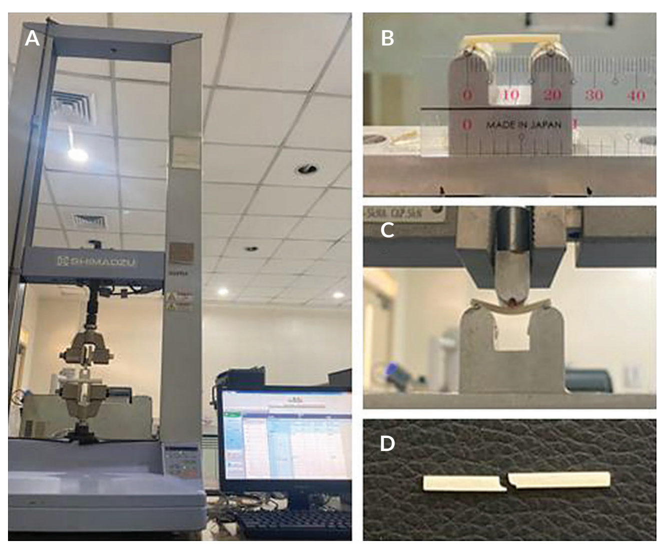

Figure 2. **(A)** Universal Testing Machine used (AGS-X, Shimadzu, Japan), **(B)** Support beams 20 mm apart, **(C)** Three-point bend test; **(D)** A fractured specimen.

Computation of flexural strength

The maximum load obtained (in Newtons) through the 3-point bending test was recorded to compute the material's flexural strength (in Megapascals) using the following formula:[11]

$$\sigma = \frac{3FL}{2bh^2}$$

where σ is the flexural strength, in Megapascals; F is the load, in Newtons, at the fracture point; L is the length of the support span; b is the width, in millimeters, of the specimen; h is the height, in millimeters, of the specimen. Computation was done manually through Microsoft Excel.

Statistical Analysis

Descriptive statistics was used to summarize the data obtained from the flexural strength computation. Shapiro-Wilk test was conducted to determine whether the data follows a normal distribution. Paired t-test was used to compare the mean flexural strengths of the bis-acryl resins after 1 day and after 7 days of storage in distilled water at 37 ± 1°C. Independent t-test was used to compare the mean flexural strengths of the self-cured bis-acryl resin and the dual-cured bis-acryl resin within the same storage period. A p-value of less than 0.05 was considered statistically significant. Microsoft Excel and STATA version 14 were used for data management and analysis, respectively.

Ethical Consideration

Neither human participants nor identifiable human tissue, biological samples, and data were involved in this study. This study was registered at the Research Grants Administration Office of the National Institutes of Health (RGAO-2024-0617) and was granted a certificate of exemption from ethical review by the Research Ethics Board of the University of the Philippines Manila (UPMREB 2024-0412-EX). The study complied with all the relevant institutional and national ethical guidelines.

RESULTS

A total of 40 bis-acryl resin specimens were tested in this study. The mean flexural strengths of the self-cured and the dual-cured bis-acryl resin materials after two different durations of aging by storage are presented and compared in Table 2.

The *Protemp 4* (self-cured) specimens exhibited a statistically significant decline in mean flexural strength after 7 days of water storage at 37 ± 1°C compared to after 1 day of storage (t = 5.6074, p = 0.0003). The same trend was observed in the *Care C&B* (dual-cured) specimens, wherein compared to after 1 day of storage, the mean flexural strength was significantly lower after 7 days of storage (t = 4.9280, p = 0.0008).

Table 2. Mean Flexural Strengths (MPa) ± SD of the Bis-acryl Resins after 1 Day and after 7 Days of Storage

Material	Storage Condition	
	1 day in distilled water at 37 ± 1°C	7 days in distilled water at 37 ± 1°C
Protemp 4 (self-cured)	216.289 ± 18.684[a]	199.190 ± 9.579[b]
Care C&B (dual-cured)	211.561 ± 15.513[a]	203.532 ± 17.1[b]

**Different letters indicate a statistically significant difference (P <0.05).*

The mean flexural strengths of the self-cured and dual-cured materials after being stored for the same duration can also be seen in Table 2. After 1 day of storage, the *Protemp 4* (self-cured) specimens exhibited a higher mean flexural strength than the *Care C&B* (dual-cured) specimens, but the difference was not statistically significant (t = 0.6157, p = 0.546). After 7 days of storage, the *Care C&B* (dual-cured) specimens had a higher mean flexural strength than the *Protemp 4* (self-cured) specimens, but the difference was also not statistically significant (t = -0.7005, p = 0.493).

DISCUSSION

This study aimed to determine the effect of aging by storage on the flexural strength of *Protemp 4* (self-cured) and *Care C&B* (dual-cured) bis-acryl resin materials. The results of this study showed that the flexural strengths of both bis-acryl resin materials are significantly lower after 7 days of water storage at 37 ± 1°C than after 1 day of storage (Table 2).

Previous studies have shown that storing dental resins in water negatively impacts their mechanical properties, including flexural strength.[11,16] This phenomenon can be attributed to hydrolysis, where water infiltrates the resin matrix, leading to stress formation and matrix degradation.[8] The intermolecular forces that hold polymer chains together are diminished.[18] Hydrolysis also leads to detachment of inorganic fillers within the resin due to breakdown of the silane-treated layer.[11] Bis-acryl polymers exhibit high water absorption due to their polarity and high diffusion coefficient.[15] Thus, the greater the amount of water absorbed by the material, the lower the strength.[19] A similar trend was observed when provisional resin materials were stored in artificial saliva.[15]

Contrasting results were found in the study of Kuphasuk et al., where flexural strength testing was performed 30 minutes after mixing at 23°C; after 24 hours of water storage at 37°C; after 7 days of water storage at 37°C; and after 7 days of water storage with and without thermocycling. The bis-acryl resin materials being compared were *Luxatemp Fluorescence* (self-cured), *Protemp 4* (self-cured), and *Integrity* (dual-cured). Results showed that all bis-acryl resins exhibited low flexural strengths 30 minutes after mixing but increased after 7 days of water storage with or without thermocycling.[20]

Kuphasuk et al. explained that low flexural strengths at an early setting stage might be due to 2 factors: stress development and insufficient cross-linking. Development of stress inside the resin during the early polymerization phase makes the materials prone to fracture. However, subjecting the material to conditions with a higher temperature, such as water storage at 37°C and thermocycling reaching up to 55°C, could reduce the stress within the resin through relaxation resulting in a higher flexural strength. Further cross-linking of the polymer chains over time could have also increased the flexural strengths of the materials.[20] Thus, increase in the flexural strength could not be solely attributed to water storage or thermocycling because further cross-linking over time could have also had an effect.

This is the reason why, in the present study, the control group was stored for 1 day (24 hours) before testing to allow sufficient cross-linking of polymers and proper polymerization of the bis-acryl materials. As for the treatment group, a storage period of 7 days was established since provisional restorations commonly remain in the oral cavity for 1 week while the definitive restoration is being fabricated.

The International Organization for Standardization (ISO) 10477 standard has established that a flexural strength of at least 50 MPa is required for polymer-based crown and bridge materials.[21] The results of this study showed that the flexural strengths of both materials exceeded the ISO standard value of 50 MPa, even after 7 days in storage. This implies that both materials retained sufficient flexural strength even after one week in a simulated moist environment. However, it must be noted that this study was conducted in a laboratory with optimal conditions, which is different from a clinical environment.

Provisional materials must possess sufficient flexural strength to survive the deforming effect of functional forces.[9] The importance of having adequate flexural strength is emphasized when parafunctional habits, such as bruxism, exist since these habits exert strain on teeth and prosthetic restorations.[22] Flexural strength is also crucial in long-span provisional restorations, cases with short occluso-gingival pontic and connector height, and when restorations are worn over a long period of time.[23,24]

At low flexural strengths, the provisional restoration may fracture, causing issues with function and esthetics, movement of abutment teeth and their antagonists, development of dental caries, and possible endodontic involvement.[10] All of these can delay the ongoing prosthodontic treatment.[25]

Another factor that affects the flexural strength of a resin material is mode of curing. Self-cured resins are commonly used as provisional materials since they do not require a light curing unit.[26] In addition, sufficient polymerization can occur at deep layers of a self-cured material due to its chemical-cure property.[27] However, a significant drawback is their limited working time after mixing. In contrast, light-cured resins harden on command with a light curing unit, thereby allowing immediate placement of the restoration. The downside is that areas distant from the light source may not fully cure.[28] The latest innovations in provisional resin materials are dual-cured bis-acryl resins. These combine the benefits of both materials: the self-cure property ensures sufficient polymerization in areas with limited light penetration, while its light-cure property provides rapid setting of the outer layers.[29]

The results of this study also showed that there is no significant difference in the flexural strength of *Protemp 4* (self-cured) and *Care C&B* (dual-cured) after being stored for the same duration (Table 2). This is inconsistent with the results of previous studies which have found a notable difference in the flexural strengths between the two materials.

A systematic review and meta-analysis by Astudillo-Rubio et al. compared the flexural strength, fracture toughness, and hardness of dimethacrylates and monomethacrylates used in direct fabrication of provisional restorations. It was found that dual-cured bis-acryl resins exhibit greater flexural strength than self-cured resins due to substantial photo-initiated polymerization. In addition, self-cured bis-acryl materials exhibit higher hardness and flexural strength than light-cured due to higher filler particle content.[30]

Similar findings were obtained in a study by Akiba et al., where the dual-cured provisional resin materials used were *Tempsmart* (GC), *Luxatemp Automix Solar* (DMG), and *Integrity Multi-Cure* (DENTSPLY). As a comparison material, the self-cured bis-acryl provisional resin *Protemp Plus* (3M) was utilized. Specimens which were stored in distilled water at 37°C for 24 hours served as the baseline. The experimental groups underwent either 5,000 cycles or 10,000 cycles at 5°C and 60°C. Results showed that the flexural strengths of the dual-cured specimens were significantly higher than the self-cured specimen, regardless of aging condition.[18]

Resin-based materials have a significantly higher degree of conversion under the dual-cure mode due to the type of resin monomer content.[8] Self-cured bis-acryl resins commonly contain bisphenol a-glycidyl methacrylate (Bis-GMA). Bis-GMA is known to be highly viscous due to its stiff central phenyl ring core. The monomer's high viscosity results in a lesser degree of conversion. Dual-cured bis-acryl resins, on the other hand, commonly contain urethane dimethacrylate (UDMA). UDMA is known to have lower viscosity (100 times less) than Bis-GMA. Thus, resins that contain UDMA attain higher degree of conversion and polymerization rate compared to resins that contain Bis-GMA.[31] This leads to an enhanced structural state in the dual-cured resin, resulting in improved physical characteristics and mechanical performance.[8]

Protemp 4 (self-cured) contains ethoxylated bisphenol a glycol dimethacrylate (Bis-EMA) with a concentration of 45-55 % by weight.[13] Bis-EMA is a hydrophobic counterpart of Bis-GMA and its water sorption is known to be low.[31] *Care C&B* (dual-cured), on the other hand, contains UDMA with a concentration of 15-30%.[32] Therefore, even though

UDMA is responsible for a high degree of conversion that results in high flexural strength, its concentration in *Care C&B* is relatively low.[31] This could be one of the factors why there is no significant difference in the flexural strengths of the two materials after the same storage period.

A recent experimental study by Katayama et al. reported contrasting results, showing that the performance of self-cured resins surpassed that of a dual-cured resin.[16] The self-cured provisional resin materials used in the study were *Luxacrown* (DMG), *Luxatemp Automix Plus* (DMG), *Luxatemp Star* (DMG), and *Protemp 4* (3M) while the dual-cured provisional resin material used was *Tempsmart* (GC). The materials were tested after 24 hours, after storage in distilled water, and after thermal cycling at 5˚C and 55˚C for 10,000 cycles.[16]

After 24 hours at room temperature, *Luxatemp Star* (self-cured) had the highest flexural strength (113.4 MPa). *Tempsmart* (dual-cured) only ranked 4^{th} with a flexural strength of 98.4 MPa. After storage in distilled water at 37˚C for 7 days, *Luxatemp Automix Plus* (self-cured) exhibited the highest flexural strength (102.8 MPa). *Tempsmart* (dual-cured) only ranked 4th with a flexural strength of 80.2 MPa. Lastly, after 10,000 thermal cycles between 5 and 55˚C, *Luxatemp Star* (self-cured) displayed the highest flexural strength (115.5 MPa). Again, *Tempsmart* (dual-cured) only ranked 4^{th} with a flexural strength of 87.9 MPa.[16]

The correlation analyses by Katayama et al. found that flexural strength and inorganic filler content have no significant correlation. However, flexural strength was found to have a positive correlation with elastic modulus and dynamic hardness, and a negative correlation with the maximum wear depth. It was then discussed that the significant difference in flexural strengths of the materials could be related to variations in the number of monomers in every material.[16]

The mean flexural strength value of *Protemp 4* obtained in the study by Katayama et al. was different from the value obtained in the present study. In the study by Katayama et al., a mean flexural strength ± SD of 74.9 ± 8.4 was obtained after storage in distilled water at 37˚C for 7 days.[16] In the present study, a mean flexural strength ± SD of 199.190 ± 9.579 was obtained in the same conditions. The difference may be attributed to dissimilarities in specimen preparation. In the study by Katayama et al., specimens were polished using 1000-grit silicon carbide abrasive paper, then immersed in an ultrasonic bath for 30 minutes.[16] However, in the present study, the specimens were abraded using 320-grit silicon carbide paper, without subsequent ultrasonic cleansing.

Differences in flexural strengths of bis-acryl resin materials could also be attributed to varying filler particle size. Inorganic fillers help to dissipate functional stresses, prevent crack propagation, and improve elastic modulus, strength, and wear resistance of bis-acryl resins.[33] *Care C&B* contains fumed silica fillers with an average particle size of ≤12 nanometers.[14] However, the filler size of *Protemp 4* remains undisclosed by its manufacturer (3M ESPE). In the study by Katayama et al., a scanning electron microscope (SEM) image of *Protemp 4* showed that the material uniformly contained spherical nanofillers, but did not report their exact size.[16] Nanofillers tightly hold monomer and polymer contents together. Due to their small size and spherical shape, homogenous stress distribution occurs within the material, thereby preventing crack propagation. All of these advantages enhance the material's flexural strength.[8] However, a comparison of the actual filler sizes between the two materials cannot be made due to the undisclosed filler size of *Protemp 4*.

A similarity in filler particle type was found between the two materials. Common filler types incorporated in resin materials are silica, ceramic, and quartz.[34] Both *Protemp 4* and *Care C&B* resin materials contain silica fillers (Table 1). *Protemp 4* contains silane-treated silica 20-30 % by weight, according to its manufacturer (3M ESPE).[13] However, the specific amount of fumed silica fillers in *Care C&B* remains undisclosed by its manufacturer (VERICOM). A thorough literature search reveals that no correlational studies on filler type and flexural strength have been done in the past. Previous studies have only focused on filler content, size, and shape. Therefore, it cannot be automatically presumed that having the same filler type affects the flexural strengths of the materials being compared.

Limitations of the Study

The study did not simulate natural oral conditions, in the truest sense. Most studies that investigate the flexural strength of a dental resin material use artificial saliva and perform thermocycling. Majority investigate not only flexural strength, but also fracture toughness, color stability, and other mechanical properties. Similar studies commonly test multiple products, not just two. Some studies have also performed tests to determine filler content, shape, size, and uniformity. However, all of these were not performed in the present study due to lack of access to the said materials and testing machines. Thus, the findings of this study should be limited to the two materials tested and should not be generalized to other self-cured and dual-cured bis-acryl resin materials. Lastly, application of the results to a clinical setting is not advised.

CONCLUSION AND RECOMMENDATIONS

Within the limitations of the study, results show that the mean flexural strengths of *Care C&B* (dual-cured) and *Protemp 4* (self-cured) bis-acryl resin materials are significantly lower after 7 days in water storage at 37 ± 1˚C compared to just 1 day in storage. The results also show that there is no significant difference in the mean flexural strengths of *Care C&B* and *Protemp 4* after the same storage period.

The researchers strongly recommend a follow-up study focusing on the effect of aging by thermocycling on the flexural strength of *Care C&B*, the novel dual-cured bis-acryl resin material tested in the study. Frequent temperature changes occur in the oral cavity when eating and drinking. This can be simulated through aging by thermocycling which subjects specimens to multiple cycles of alternating high and low temperatures.[8] The use of artificial saliva is also suggested to better simulate intraoral conditions.

Lastly, it is also recommended that other physical and mechanical properties of *Care C&B* be investigated, since this study only focused on flexural strength.

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Statement of Authorship

All authors certified fulfillment of ICMJE authorship criteria.

Author Disclosure

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