Effect of 180 Days of Water Storage on the Transverse Strength of Acetal Resin Denture Base Material

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Abstract

Purpose: Acetal resin has been used as an alternative denture base and clasp material since 1986. The manufacturers claim that acetal resin has superior physical properties when compared to conventional denture base acrylic resins. Limited information is available about transverse strengths of acetal resin. The purpose of this investigation was to compare transverse strengths of pink and white acetal resins to transverse strengths of conventional heat-polymerized polymethylmethacrylate (PMMA) resin in increasing durations of water storage.

Materials and Methods: A transverse strength test was performed in accordance with International Standards Organization (ISO) specification No 1567. Twenty 65 × 10 × 2.5 mm^3 specimens of each resin were prepared; five specimens of each resin group were subjected to three-point bending test after 50 hours, 30 days, 60 days, and 180 days of water storage in distilled water at 37°C. Experimental groups' transverse strengths were compared by three-way ANOVA and Duncan's multiple range tests.

Results: Transverse strength of PMMA denture base material was found to be in accordance with the requirements of ISO specification No 1567. Transverse strengths of white and pink acetal resin could not be calculated in this study, as white and pink acetal resin specimens did not break at the maximum applied force in the three-point bending test. Flexural strength of acetal resin was found to be within the ISO specification limits. As the water storage time increased, the deflection values of PMMA showed no significant difference ($p > 0.05$). Both the white and pink acetal resin showed significant increase in deflection as the water storage time was increased from 50 hours to 180 days ($p < 0.05$).

Conclusion: The results of this study indicated that transverse strength values of PMMA were within the ISO specification limit. Water storage time (50 hours, 30, 60, and 180 days) had no statistically significant effect on the transverse strength and deflection of PMMA. Acetal resin suffered from permanent deformation, but did not break in the three-point bending test. Acetal resin showed significant increase in deflection as the water storage time was increased from 50 hours to 180 days. All materials tested demonstrated deflection values in compliance with ISO specification No 1567.

The material most commonly used in construction of dentures is the acrylic resin polymethyl methacrylate (PMMA). Despite its popularity, the material, although adequate in satisfying esthetic demands, is far from ideal in fulfilling the mechanical requirements of dentures. The fracture of acrylic resin dentures may result from impact failure, (i.e. dropping the denture accidentally) or from fatigue failure caused by flexing under masticatory forces.[1-3] One of the most common causes for breakage of dentures is fatigue (namely, continued flexing of the base during function, which leads to crack development). Midline fracture of the denture base is a flexural fatigue failure resulting from cyclic deformation of the base during function. This fracture stems from initiation and propagation of a crack, and it requires the presence of a stress raiser or localized stress.[4] This is reflected in the unresolved problem of denture fracture and accompanying costs to effect repair.[5] Clinically, denture fracture is a significant problem, since it will cause denture failure in a short time. To overcome these shortcomings, various

modifications of PMMA have been tested to improve the existing material; these modifications include chemical modification to produce high impact strength resins,[6,7] mechanical reinforcement through the inclusion of glass fibers,[8-10] carbon fibers,[11] aramid fibers,[4,12] continuous PMMA fibers,[13] and polyethylene fibers.[7] Experiments with alternative polymers have also been conducted, but these polymers failed to produce dentures of greater accuracy or better performance.[7]

Acetal resin, an injection-molded resin, has been introduced as an alternative to conventional PMMA. Acetal resins are formed by the polymerization of formaldehyde. The homopolymer, polyoxymethylene (POM) is a chain of alternating methyl groups linked by an oxygen molecule. Acetal resins are hard, tough, rigid materials, which show a low coefficient of friction and have high resistance to fatigue. In industry, they are used at sites where wear of components is a problem.[14] The material has been shown to have good biocompatibility, and this has fostered its use in total hip replacement[15] and as an artificial heart valve occluder.[16] It has been used to form a stress-absorbing component in a dental implant system (IMZ).[17] The possible use of polyacetal resins as denture base materials was mentioned by Smith.[18] It has been used as a denture base and denture clasp material since 1986.[19] Because of its biocompatibility, it was considered as a framework material for removable partial dentures for patients with allergic reactions to Co-Cr framework.[14] The manufacturers claim that acetal resins have superior physical properties when compared to conventional denture base acrylic resins. It is reported to have a sufficiently high resilience and modulus of elasticity,[14] however, Teoh[20] suggests that a fluid environment (saline) may deleteriously affect its stress resistance and have implications for the use of POM in situations where contact stresses may be high.

Clinically, a resin material exhibiting a lower transverse strength may be more prone to fracture during function as a denture base than would a resin with a higher transverse strength.[1] Previous studies have shown a decrease in transverse strengths for PMMA denture base resins after water sorption has occurred.[1-21] The influence of water sorption on the flexural strength of denture base polymers was reported by Takahashi et al.[6] They found that denture base polymers of different monomer compositions and polymerization activator systems have different resistances to the influence of water.[9] The flexure test method measures behavior of materials subjected to simple beam loading. International Standards Organization (ISO) specification No 1567 for denture base resins specifies transverse deformation limits from 1 to 2.5 mm for a force of 15 to 35 N and 2 to 5 mm for a force of 15 to 50 N; fracture strength must be more than 55 N.[21] Because water acts as a plasticizer for resins and decreases mechanical properties such as hardness and fatigue limit, it is important to determine transverse strength values of denture base resins after water storage.[9]

Although extensive work has been performed to determine the properties of a variety of materials used for denture base materials,[1-13] little is known about how acetal resin functions in this application. The aim of this study was to compare the transverse strengths of pink and white acetal resins to one conventional heat-polymerized PMMA resin after 50 hours, 30 days, 60 days, and 180 days of water storage.

Materials and methods

White and pink acetal resins (Polyoxymethylene, Batch no: 00553, 00502, Pressing Dental, San Marino, Italy) and one conventional heat-cured PMMA resin (Meliodent, Batch no: 106, Heraeus Kulzer GmbH & Co. KG, Hanau, Germany) were evaluated in this investigation. Twenty $65 \times 10 \times 2.5$ mm^3 specimens of each resin were prepared using a stainless steel mold mounted with type IV dental stone (Cere Rock, GC Germany GmbH, München, Germany) in a denture flask. In accordance with the manufacturer's instructions, 23.4 g : 10 ml powder-to-liquid ratio was used for heat-polymerized PMMA. The resin was polymerized by immersion in boiling water. The specimens were cooled at room temperature for 30 minutes and then kept in tap water for 15 minutes.

Pink and white acetal resin specimens were prepared in accordance with manufacturer's instructions. Both acetal resin materials have the same structure, but pink acetal resin has color fibers in its structure. Acrylic fibers are added to the white acetal resin material, and they amalgamate with the matrix. First the stainless steel mold was placed at a distance of approximately 2.5 cm from the injection opening of the special flask (Muffle-Type 100, Pressing Dental) with type IV dental stone (Marble Stone, Pressing Dental). One cylinder of the acetal resin was placed into the injection tube, and subsequently the tube was placed in the injection machine (J-100, Pressing Dental). The flask was placed into the injection machine (J-100, Pressing Dental) with parameters set as follows: preinjection time with the material maintained at 220°C (melting temperature) for 20 minutes; postinjection time with the temperature maintained at 220°C for 3 minutes and injection pressure of 4 bar. At the end of the process, the flask was removed from the initial position, and the pattern was deflasked. The pattern was polished with rubber points (Pressing Dental) using polishing paste for acetal and acrylic resins (Universal polish, Pressing Dental). After being processed, the specimens were abraded on both sides with 600-grit silicone carbide papers (Struers waterproof silicone carbide, Struers Scientific, Copenhagen, Denmark) to a final thickness of 0.5 ± 0.1 mm, determined with a micrometer set at 0 to 25 mm: 0.001 mm (Mitutoyo Ltd, Singapore).

Test specimens were identified by placing a series of identification notches signifying the type of material and specimen number. All specimens were placed in distilled water and stored in the oven at $37°C \pm 1°C$. Five specimens of each material were subjected to three-point bending test after 50 hours, 30, 60, and 180 days of water storage with the Instron Universal Testing Machine (Model No. 1195, Instron Corporation, Canton, MA) at a crosshead speed of 5 mm/min. The specimens were placed on jigs, 50 mm apart with their ends fixed so that rotation or any other movements at the support were eliminated. All testing was performed at room temperature ($22°C \pm 1°C$). The specimens were exposed to 3- and 5-kg load at the center, and deflection values were recorded. Deflection measurements were made directly from crosshead movement on the testing machine and not from direct measurements with strain gauges on the specimens. They were then loaded until fracture occurred. The maximum load required to fracture the specimens in each treatment was recorded, along with the maximum deformation at the point of load application.

Table 1 Comparison of the performance of materials in three-point bending tests with requirements of the ISO specification

Test condition (ISO standard)	Testing time	White acetal resin*	Pink acetal resin*	PMMA
3 kg deflection (mm)	50 hours	2.00 ± 0.09 a, A	2.00 ± 0.2 a, A	1.90 ± 0.2 a, B
(12.5 mm)	30 days	1.94 ± 0.1 a, A	2.02 ± 0.16 a, A	1.93 ± 0.1 a, A
	60 days	1.99 ± 0.08 a, A	1.94 ± 0.08 a, A	1.99 ± 0.09 a, A
	180 days	1.95 ± 0.1 a, A	1.97 ± 0.09 a, A	1.97 ± 0.1a, A
5 kg deflection (mm)	50 hours	3.59 ± 0.06 a, A	3.57 ± 0.04 a, A	3.35 ± 0.01 a, B
(25 mm)	30 days	3.61 ± 0.04 ab, A	3.59 ± 0.07 ab, A	3.34 ± 0.11 a, B
	60 days	3.65 ± 0.06 b, A	3.62 ± 0.03 bc, A	3.39 ± 0.10 a, B
	180 days	3.67 ± 0.03 c, A	3.70 ± 0.05 c, A	3.35 ± 0.05 a, B
Fracture load (N)	50 hours	117.6 ± 5.07 a, A	120.7 ± 6.93 a, A	71.5 ± 2.68 a, B
(>55 N)**	30 days	122.5 ± 7.52 a, A	122.6 ± 6.05 a, A	68.6 ± 3.46 a, B
	60 days	121.5 ± 5.74 a, A	124.5 ± 4.87 a, A	66.6 ± 2.68 a, B
	180 days	118.6 ± 5.94 a, A	123.5 ± 4.08 a, A	69.5 ± 2.19 b, B

*Specimens did not fracture, and all measurements made at point of maximum loading.

**ISO specification for heat-processed resin.

All statistical analyses were performed at $p < 0.05$ using multiple range tests. Small letters are used for comparisons within columns among times for a particular test type. Capital letters are used for comparisons among materials across rows within a particular test type.

The transverse strength of each specimen was calculated with the following formula

$$S = 3PL/2WT^2$$

Where S = the transverse strength, P = the fracture load, L = the distance between the supports (50 mm), W = the width of the specimen (10 mm), and T = the thickness of the specimen (2.5 mm).

Mean values of ultimate flexural strength within the material combination group were compared in the first instance with three-way ANOVA, followed by Duncan's multiple range tests. P values less than 0.05 were assessed as statistically significant.

Table 2 Comparison of mean deflection for each resin group using three-way ANOVA

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected model	76.099	23	3.309	632.078	0.000
Intercept	905.082	1	905.082	172904.232	0.000
Material	0.512	2	0.256	48.877	0.000
Load	74.924	1	74.924	14313.193	0.000
Time	3.900E-03	3	1.300E-03	0.248	0.862
Material * load	0.460	2	0.230	43.947	0.000
Material * time	2.816E-02	6	4.693E-03	0.897	0.501
Load * time	7.367E-02	3	2.456E-02	4.691	0.004
Material * load * time	9.814E-02	6	1.636E-02	3.125	0.008
Error	0.503	96	5.235E-03		
Total	981.683	120			
Corrected total	76.602	119			

$R^2 = 0.993$ (adjusted $R^2 = 0.992$).

Material: white acetal resin, pink acetal resin, PMMA; Load: 3 kg, 5 kg; Time: 50 hours, 30 days, 60 days, 180 days.

Results

Comparison of deflection results (mm) and average load (N) of fracture for each material and test conditions with ISO specification limits are summarized in Table 1. Although acetal resin specimens showed permanent deformation, they did not break at the maximum applied force in the three-point bending test. Since these specimens showed no fracture in the three-point bending test, no transverse strength value could be calculated in this study, and maximum deflection values could not be measured. Maximum applied force in the three-point bending test caused the acetal resin specimens to jump out of the test machine. Coincidentally, permanent deformation occurred on the acetal resin specimens. Maximum applied force values are shown in Table 1.

Comparison among the mean values of deflection for each material at three water-storage time periods is shown in Table 2 with three-way ANOVA. There were significant differences between material, load, and water storage times; between material and load; and between load and time ($p < 0.05$).

Results of Duncan's multiple range tests for comparison of mean transverse strength of PMMA for each time period are shown in Table 3. The ultimate transverse strength of PMMA is significantly different only between two water storage time periods (50 hours and 60 days) ($p < 0.05$). Results of Duncan's

Table 3 Multiple range tests for comparison of transverse strength values for PMMA specimens in all treatment conditions

Time	Mean (SD)
50 hours	85.84 (3.22)*
30 days	82.32 (4.15)
60 days	79.96 (3.22)*
180 days	83.49 (2.62)

*Significant difference ($p < 0.05$) was found only between 50 hours and 60 days storage time.

multiple range tests for comparison of mean deflection values for each time period by resin group (for 50 N) are shown in Table 1. As the water storage time increased, the deflection values of PMMA showed no significant difference ($p > 0.05$). Deflection values of white and pink acetal resins began to increase as the water storage time was increased from 50 hours to 30 days, and same trend was observed consistently as the water storage duration was increased. Duncan's multiple range test revealed that significant increase in deflection occurred between 50 hours and 60 days, and between 50 hours and 180 days of water storage for white and pink acetal resins. The increase in deflection for pink acetal resin between 60 and 180 days of water storage was significantly different than the increase in deflection for white acetal resin between 60 and 180 days of water storage ($p < 0.05$).

Comparisons of mean deflections for each resin group by time with one-way ANOVA are shown in Table 1. PMMA resin specimens showed significantly less deformation than acetal resin specimens in all treatment cycles ($p < 0.05$). The differences, however, were small, with the control specimens demonstrating the least value. Overall deflection values of PMMA and pink and white acetal resins were within the ISO specification limits (Table 1).

Discussion

This study demonstrated the time-dependent effect of water storage on the flexural properties of acetal resin denture base polymer test specimens. Information on this parameter is important for clinical application of acetal resin as an alternative denture base material. Comparison of mean deflection for each group by time demonstrated an overall significant difference between PMMA and white acetal resin and between PMMA and pink acetal resin, the PMMA being more resistant to flexure. However, the differences in actual values were small between largest and smallest mean deflection values (3.67 to 3.34), and all results satisfied the criteria in ISO 1567[21] for denture base resins, specifying transverse deformation limits from 1 to 2.5 mm for a force of 15 to 35 N and 2 to 5 mm for a force of 15 to 50 N; this was satisfied by all tested groups' results. Deflection values of white and pink acetal resins began to increase as the water storage duration increased from 50 hours to 30 days, and the same trend continued in the following stages. No significant increase in deflection values from 50-hour to 30-day storage was shown, but a significant increase in deflection was observed between 50 hours and 60 days and between 50 hours and 180 days. The increase in deflection for pink acetal resin between 60 and 180 days of water storage was significantly different than the increase in deflection for white acetal resin between 60 and 180 days of water storage.

As a result of the applied maximum force, acetal resin specimens did not break, but they could not return to their original shape due to applied maximum force. Examinations of acetal resin's failure characteristics showed that acetal resin, while having a relatively low flexural strength, was in fact an extremely tough material. This implies that clinically a denture component made from acetal resin would be very resistant to fracture from a high level of force, disregarding the effects of any fatigue. Since polyacetal denture base resins did not break at the maximum applied force in the three-point bending test, no transverse strength values for acetal resins could be calculated in this study. In all stages, acetal resin specimens showed significantly greater deflection than PMMA specimens did.

Previous studies have shown a decrease in transverse strength for PMMA denture base resins after water sorption has occurred.[1-3] Stafford and Smith[2] believed that 30-day storage in water was necessary to maximize the effect. Dixon et al[1] stated that variations in the transverse strength values for resins during water storage were not statistically significant, but all resins began to increase in transverse strength values from 60 to 90 days of water storage, and longer storage was needed to confirm this trend. This study tested the specimens for longer water storage up to 180 days. Although transverse strength of PMMA showed a small decrease after 30 days of water storage, it increased after 60 and 180 days, and variations were not statistically significant. Our study confirms previous studies.[1-3] According to ISO 1567, the mean breaking force of acrylic resin should not be less than 55 N.[21] In this study, a mean breaking force of 71.2 N was found for PMMA resin. In this respect, the control material (PMMA) satisfied the standard.

Variations in the deflection values for PMMA during 50 hours, 30, 60, and 180 days of water storage, were not statistically significant. Deflection values of white and pink acetal resins began to increase as the water storage duration increased from 50 hours to 30 days, and the same trend continued in the following stages. There was no significant increase in deflection values from 50-hour to 30-day storage, but a significant increase in deflection was seen between 50 hours and 60 days and between 50 hours and 180 days. The increase in deflection for pink acetal resin between 60 and 180 days of water storage was significantly different than the increase in deflection for white acetal resin between 60 and 180 days of water storage.

Stafford and Smith[2] pointed out that plastic test specimens have uniform shape, whereas one of the important features of the strength of a denture is its form. Thus a final assessment of any material must lie in an extended clinical trial. This was confirmed by Stafford et al,[3] who noted that the parameters for a denture base are not so easy to define or to reproduce because of the complex and variable nature of the oral environment. When a new material is introduced, the present specifications are not always of real value, because they lack clinical parameters that can be used for assessments.[4] Clinical trials and experience are also required to assess the evaluation of physical properties of acetal resins in a clinical setting.

Conclusions

Within the limitations of the study design, the following conclusions were drawn:

(1) Water storage time (50 hours, 30, 60, and 180 days) had no significant effect on the transverse strength of PMMA specimens ($p > 0.05$).
(2) All materials tested, demonstrated deflection values in compliance with ISO 1567.
(3) Acetal resin can be used as a denture base material from the standpoint of transverse strength.

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