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Hardness, Flexural Strength, and Flexural Modulus Comparisons of Three Differently Cured Denture Base Systems

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Abstract

Purpose: This study compared the surface hardness, flexural strength, and flexural modulus of a light- and heat-cured urethane dimethacrylate (UDMA) to two conventional polymethyl methacrylate (PMMA) denture base resins. The effect of less-than-optimal processing condition on the hardness of internal and external surfaces of UDMA specimens was also investigated.

Materials and Methods: The materials tested were Eclipse (light- and heat-cured UDMA), Meliodent (heat-cured PMMA), and Probase Cold (auto-cured PMMA). Eclipse specimens were prepared by adapting the material onto the master cast and light curing in the processing unit for 10 minutes. Meliodent and Probase Cold specimens were prepared according to the manufacturers' instructions. Twenty rectangular specimens measuring $65 \times 10 \times 2.5$ mm^3 were prepared for each material. They were stored in water at 37°C for 30 days before testing. The surface hardness was measured using Vickers Hardness (VHN) test, and flexural strength and flexural modulus were measured using a 3-point bending test. Twenty-five additional Eclipse specimens were similarly prepared and were processed at various times of less than 20 minutes of curing. Vickers Hardness was determined on both the external and internal surfaces of specimens. Data were analyzed using a one-way ANOVA for comparisons of hardness, flexural strength, and flexural modulus between the three denture base materials and for hardness values of both the internal and external surface of Eclipse specimens with curing times. Post hoc analyses (Scheffé test) determined the difference between the groups. Student *t*-test was used for comparison of hardness between the external and internal surfaces of Eclipse specimens.

Results: The hardness (VHN) values were 19.4 ± 0.7, 17.0 ± 0.4, and 16.0 ± 0.4; the flexural strengths (MPa) were 103 ± 4, 78 ± 3, and 63 ± 4; and the flexural moduli (MPa) were 2498 ± 143, 1969 ± 55, and 1832 ± 89 for Eclipse, Meliodent, and Probase Cold materials, respectively. A comparison among the three polymers showed there were significant differences in surface hardness, flexural strength, and flexural modulus ($p < 0.05$). No significant difference in surface hardness (VHN) between the internal (19.1 ± 0.6 to 19.4 ± 0.7) and external surfaces (18.9 ± 0.4 to 19.2 ± 0.6) of irradiated Eclipse specimens was observed at 10-, 12-, and 14-minute polymerization times.

Conclusion: The surface hardness, flexural strength, and flexural modulus of light- and heat-cured UDMA (Eclipse) were significantly higher than the values obtained for heat-only cured (Meliodent) and auto-cured (Probase Cold) PMMA denture base systems.

Polymethyl methacrylate (PMMA) is the most commonly used material for denture fabrication; however, its mechanical properties are far from ideal, as illustrated by the ongoing efforts to improve its properties. Rubber[1,2] and fiber reinforcements[3,4] have been used to overcome the mechanical limitations of PMMA denture base polymer. Polycarbonate[5] and nylon[6] have also been investigated as alternative materials for patients who are allergic to methyl methacrylate denture material and its

byproducts.[7] Visible light-cured denture base resin was introduced to the market in the early 1980s and was promoted on the basis that the material does not contain methyl methacrylate monomer and hence could be considered an alternative to heat- and auto-cured PMMA polymers.

The first system of light-activated UDMA denture base polymer, known as Triad, was advocated because of its biocompatibility, low bacterial adherence, ease of fabrication and manipulation, patient acceptance, and ability to bond to other denture base resins, and a lack of requirement for proportioning and mixing.[8] However, its application has been limited because of the material's brittleness and low impact resistance.[9] This has led to the development of a new light- and heat-activated denture base material marketed as Eclipse resin system (Dentsply Trubyte, York, PA). The system is comprised of three resins to form the denture. They are supplied in different packages: Baseplate, Set-up, and Contour resins. The resins were developed to handle like wax and designed to eliminate the need for flasking, boiling out, packing, and water-bath curing as required in conventional compression molding denture construction. According to the manufacturer, a different initiator system from that in earlier Triad (Dentsply Trubyte) material is used in the formulation. The manufacturer advocated a curing time of 10 minutes for baseplate thicknesses of 8 mm or less. Additional curing on the tissue side of the denture is not necessary unless it is for relining and repair. High-intensity visible light of 400 to 500 nanometer wavelength results in deep polymerization of the material up to the recommended maximum thickness.

A clinical report[10] has described the use of Eclipse in aiding clinical and laboratory procedures for prosthesis fabrication. Laboratory investigations by the manufacturer[11,12] showed that the flexural and impact strength of Eclipse was higher than acrylic denture base materials.

Another property of importance to be evaluated for any new denture base material is its surface hardness. Hardness is defined as the resistance of a material to permanent surface indentation or penetration. The value provides a possible indication of the abrasiveness of the denture material, and it can easily be measured on both the polished and intaglio surfaces of the specimen. Material with a higher surface hardness could withstand excessive wear by denture cleanser, toothbrush, and food better than a softer material.[13] The Vickers hardness (VHN) test uses a square-based diamond pyramid as the indenter and the indentation has pyramidal geometry. Previously, Triad (UDMA-based) was shown to exhibit higher surface hardness than PMMA-based polymers, results that were related to the different compositions, filler content, and modes of polymerization between the two materials.

Therefore, it was the objective of this study to compare the hardness and 3-point flexural strength and flexural modulus of a light- and heat-curing UDMA (Eclipse) to heat-only curing (Meliodent) and auto-curing (Probase Cold) PMMA denture base systems. It was also the objective to investigate the effect of less-than-optimal processing conditions by measuring differences in degree of cure of Eclipse material at the external and internal surfaces of irradiated specimens using hardness values collected during the first 20 minutes of curing.

Materials and methods

The denture base materials used in this study are shown in Table 1.

Eclipse specimens were prepared by investing a Perspex block ($70 \times 50 \times 3$ mm^3) to make a stone mold in a conventional metal flask. The mold was preheated in a special oven (Conditioning Oven, Dentsply Trubyte, No: 904968) at 55°C for 2 minutes before the material was adapted using finger pressure. Separating agent (Al-Cote, Dentsply Trubyte, Lot No: 050414) was applied beforehand onto the mold. A glass slab was then pressed on top of the material to extrude the excess and to allow a uniform thickness of the specimens.

Table 1 Denture base materials used

Material	Type	Powder/liquid processing method	Manufacturer	Lot & Batch No.
Eclipse	Single paste component Matrix: UDMA* Filler: silica and PMMA beads*	Prepacked Light-polymerized	Dentsply, York, PA	030204
Meliodent	Powder PMMA, Benzoyl peroxide† Liquid MMA, EGDMA†	23.4 g/10 ml Heat-polymerized Water bath 70°C for 7 hours	Bayer Dental, Newbury, UK	A1397B-2
Probase Cold	Powder PMMA, Benzoyl peroxide, catalyst‡ Liquid MMA, EGDMA, catalyst‡	20.5 g/10 ml	Ivoclar Vivadent Ltd., Schaan, Liechtenstein	D53289

UDMA = urethane dimethyl methylacrylate; PMMA = polymethyl methyl acrylate; EDGMA = ethylene glycol dimethacrylate; MMA = methyl methylacrylate.

*Personal communication with BJ Sun, Dentsply International, York, PA, January 2006.

†Meliodent Heat Cure, user instruction sheet.

‡Probase Cold, user instruction sheet.

Air barrier coating (Eclipse ABC, Dentsply Trubyte, Lot No: 050209) was applied on the resin surface to prevent inhibition of polymerization by oxygen. The polymerization process was carried out by placing the mold in the center of the rotating table of the light-curing unit (Eclipse Processing Unit, Dentsply Trubyte, p/No: 95-0348-01) and exposing it to 400-500 nm visible light for 10 minutes. Six halogen lamps (Eclipse Replacement Lamp, Dentsply Trubyte, Lot No: J218) of 41 V each within the unit were required for polymerization. The curing unit is programmed with various menus to cover the different processing times for different denture procedures. The manual instruction and the menu screen of the unit allowed the selection of the correct program. The manufacturer stated that by pressing menu button #1 on the screen, the maximum temperature of 129°C could be achieved within the unit. No attempt was made in this study to measure the maximum temperature achieved or the curing unit output.

Meliodent and Probase Cold specimens were prepared with the same method as described for Eclipse resin. The powder:liquid ratio of the resin mixture for these materials was mixed and packed according to the manufacturers' instructions (Table 1). Meliodent was heat polymerized in a thermostatically controlled water bath (Acrydig10, Menfredi, Torino, Italy, No: 4680), and Probase Cold was autopolymerized under constant pressure in a pressure pot (Leone s.p.a., Firenze, Italy, Model No. T133S-00).

The processed block was cut manually into four specimen strips using a band saw. The specimens were then trimmed and wet ground on a polishing machine (Metaserv® 2000t, Buehler Ltd, Lake Bluff, IL, Model No 45-28229) with silicon carbide paper discs of grade 600 and 1000 to the size of 65 × 10 × 2.5 mm^3. Each specimen was individually measured to a final dimension by use of a micrometer (Digimatic Micrometre, Mitutoyo Ltd., Tokyo, Japan, Model No. CD-6-CS). For Eclipse specimens, trimming of excess was only performed on the exposed surface of the irradiated specimens. All specimens were immersed in water at 37°C for 30 days prior to testing.

Vickers hardness test was employed to measure the surface hardness, using an indenter point in the shape of a square-based pyramid. The test was made using a microhardness tester (Shimadzu Microhardness Tester, Shimadzu Corp., Tokyo, Japan, Model No. HMV -2000) with an applied load of 300 g at a 15-second dwell time at 23°C. The testing procedure was conducted on five specimens of each material at room temperature immediately after removal from the water. For each specimen, three indentations were made at different points along the specimen.

For the flexural strength and modulus, a three-point bending test was conducted on the Instron universal testing machine (Instron, Inc., High Wycomb, UK, Model No:4302) according to the ISO 1567:1999 specification[14] for denture base polymers. The crosshead speed during the test was 5 mm/min, and the test was carried out in a water bath at 37°C. The deflection was measured from the testing apparatus as the crosshead traveled and not by using strain gauges on the actual specimen. For each material, ten specimens were prepared. The flexural strength (S) was computed from the equation $S = 3NI/2bd^2$ and flexural modulus (E) from the equation $E = Fl^3/4ybd^3$ where N equaled maximum force exerted on the specimen, I was the distance between supports (50 mm), b was the breadth of specimen, d was the depth of specimen, and y was the deflection at point proportional limit.

Surface hardness was again employed to investigate the effect of less-than-optimal processing conditions on the difference in hardness between the external and internal surfaces of irradiated specimens. Twenty-five more specimens were prepared and processed as before and divided into five groups. Each group was polymerized with one of the following curing times: 4, 6, 8, 12, or 14 minutes. The hardness value obtained previously with 10 minutes of curing was also included in the analysis. The polymerized specimens were removed from the mold, and the external and internal surfaces were identified. The external surface was the surface directly exposed to the light irradiation, and the internal surface was where it touched the bottom of the mold.

Data were analyzed using a one-way ANOVA for comparisons of hardness, flexural strength, and flexural modulus between the three denture base materials and for comparison of hardness values of Eclipse specimens when irradiated at different curing times on the internal and external surfaces of specimens. Post hoc analyses (Scheffé test) were carried out to determine the difference between the groups. Student *t*-test was used for comparison of hardness between the external and internal surfaces of Eclipse specimens.

Results

The mean surface hardness, flexural strength, and flexural modulus values of Eclipse, Meliodent, and Probase Cold denture base polymers are presented in Table 2. One-way ANOVA indicated that there were significant differences in the surface hardness, flexural strength, and flexural modulus ($p < 0.05$) among the three denture base polymers. Post hoc Scheffé test indicated that for all mechanical properties investigated, the values were significantly different from one another ($p < 0.05$). Eclipse yielded the highest mean surface hardness, flexural strength, and flexural modulus values ($p < 0.05$).

The mean values for the external and internal surface hardness of Eclipse resin at different curing times are shown in Table 3. One-way ANOVA of external or internal surface hardness indicated there was a significant difference in the hardness values with different curing times ($p < 0.05$). Post hoc analysis indicated that, for both surfaces at 4-, 6-, and 8-minute polymerization times, no significant difference in the hardness values was observed ($p > 0.05$); however, there was a significant increase in the hardness value with the 10-minute polymerization curing time ($p < 0.05$). No significant difference in hardness value was observed with 10, 12, and 14 minutes ($p > 0.05$).

For each curing time, Student *t*-test comparing the hardness between the external and internal surfaces indicated that at 4, 6, and 8 minutes of polymerization time, there was a significant difference in the hardness between them (Table 3); however, when polymerized for longer times (10, 12, and 14 minutes) no significant difference in the hardness values between the two surfaces were observed ($p > 0.05$).

Table 2 Surface hardness, flexural strength, and flexural modulus of three denture base polymers

Material	Vickers hardness (VHN), mean ± SD (n = 15)	Flexural strength (MPa), mean ± SD (n = 10)	Flexural modulus (MPa), mean ± SD (n = 10)
Eclipse	19.4 ± 0.7[a]	103 ± 4[a]	2498 ± 143[a]
Meliodent	17.0 ± 0.4[b]	78 ± 3[b]	1969 ± 55[b]
Probase Cold	16.0 ± 0.4[c]	63 ± 4[c]	1832 ± 89[c]

One-way ANOVA and Scheffé post hoc tests ($p < 0.05$) indicated differences among the materials as indicated by difference in the superscripts.

Discussion

Denture base materials may undergo changes due to continued water uptake after curing. For this reason, in this study, to allow water saturation,[15] all specimens were immersed in water for 30 days, which is longer than that specified in ISO specification 1567:1999.[14]

In this study, three differently cured denture base systems' hardness, flexural strength, and flexural modulus were compared. A hardness test was used in this study, not so much to measure the property of the denture base material, but to provide a possible indication of the abrasive resistance of the material.[16] Denture base materials should have sufficient abrasion resistance to prevent excessive wear of the material by abrasive denture cleansers or food. The results of this study showed that the hardness of the relatively new light- and heat-curing UDMA was higher than the conventional heat-curing and self-curing PMMA denture base polymers. The findings in this study were in agreement with the study by Khan et al[17] who observed that Triad (UDMA-based) exhibited higher surface hardness (18.02 ± 0.99) than a PMMA denture resin (17.08 ± 0.49). Between the two PMMA-based materials (Meliodent, Probase Cold), the difference in hardness values was small, but statistically significantly different. In comparison, the hardness values obtained for heat-cured PMMA (Meliodent) in this study were in agreement with those observed in the previous study.[17]

This study also showed that with the shorter than recommended curing time, the inner surface of UDMA specimens showed a small but significantly lower hardness value than the external surface. Therefore, this finding affirmed the manufacturer's recommendation of 10 minutes of curing for Eclipse baseplate resins to achieve uniform polymerization across the denture base.

Table 3 Vickers hardness (VHN) of irradiated and non-irradiated surfaces of UDMA Eclipse polymer at various polymerization times

Polymerization time (min)	Vickers Hardness (VHN), mean ± SD	
	Irradiated surface n = 15	Non-irradiated surface, n = 15
4	18.1 ± 0.8[aA]	16.6 ± 0.5[aB]
6	18.3 ± 0.6[aA]	16.5 ± 0.7[aB]
8	18.2 ± 0.4[aA]	17.8 ± 0.5[aB]
10	19.4 ± 0.7[bA]	19.2 ± 0.6[bA]
12	19.2 ± 0.4[bA]	19.2 ± 0.5[bA]
14	19.1 ± 0.6[bA]	18.9 ± 0.4[bA]

One-way ANOVA and Scheffé post hoc tests ($p < 0.05$) indicated differences among irradiation times as shown by differences in lowercase superscripts and differences between surfaces as indicated by differences in uppercase superscripts.

Results obtained for flexural properties showed Eclipse exhibiting significantly higher flexural strength and flexural modulus than the two PMMA denture polymers. This was in agreement with that reported by the manufacturer,[18] where the flexural strength and flexural modulus of Eclipse (113.1 to 125.5 MPa and 2895.8 to 3343.9 MPa, respectively) were higher than quoted for acrylic resins (80.0 to 96.5 MPa and 2482.1 to 3033.6 MPa, respectively). The manufacturer claimed that the improved strength and rigidity were attributed to the initiator in the formulation and the mode of polymerization where both heat and light are required for the conversion. The light triggered the polymerization process and the maximum temperature of 129°C attained by the processing unit ensured complete polymerization. The manufacturer described the resin as shape-stable semi-crystalline with high glass transition temperature. It was also thought that the crystallizable nature of the formulation, and favorable increased rate of polymerization at higher temperature, had an impact on the mechanical properties of the denture base material.[12]

This study showed that Eclipse material fulfilled the ISO 1567:1999[14] requirement for type IV (light-curing) denture base material for flexural strength and modulus. A denture with adequate strength should be able to resist denture base fracture. Even though fatigue fracture is more clinically relevant, as it simulates the clinical failure mechanism more closely, the assessment of flexural strength is easier and has been used by other researchers.[19-21]

The present study also showed that the self-cured PMMA had lower flexural strength and modulus than heat-cured PMMA, which was in agreement with the findings in another study.[9] The flexural strength of heat-cured PMMA obtained in this study was in agreement with the values obtained by Pfeiffer et al[22] for Paladon 65 PMMA denture resin (78.6 ± 5.5 MPa); however, the flexural modulus values were lower that those reported by Vallittu[23] for heat-cured (2550 ± 71 MPa) and self-cured (2418 ± 128 MPa) PMMA resins. According to ISO 1567:1999[14] the minimum acceptable flexural modulus of type 1 (heat-cured) denture base materials should not be less than 2000 MPa. The apparent decrease in flexural moduli values for the materials in this study could have been related to the inaccuracy in the deflection measurement, as they were not directly measured on the specimens.

The mechanical properties investigated and compared in the present study were only a limited view of the materials, and the in vitro study was limited in simulating the clinical condition. The size of the specimens, which conformed to the actual denture configuration, could be recommended for future study when investigating the properties of denture base materials.

Conclusion

Within the limitations of the current study, the following conclusions can be made:

- Light- and heat-cured UDMA denture base polymer showed statistically higher values of surface hardness, flexural strength, and flexural modulus than both heat-only curing and self-curing denture base polymers ($p < 0.05$).
- A comparison between the two PMMA denture base polymers showed that heat-polymerized denture base polymer showed statistically higher values of surface hardness, flexural strength, and modulus than chemically polymerized denture base polymer.
- There was a significant difference in surface hardness between the external and internal surfaces of Eclipse materials when polymerized for less than 10 minutes ($p < 0.05$), but no significant difference was observed when polymerized for 10, 12, and 14 minutes ($p > 0.05$).

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