

Comparative Evaluation of Surface Hardness and Water Sorption of Heat-Cure and Microwave-Cure Denture Base Resins after Simulated Oral Aging

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Abstract

Objectives

This study aimed to comparatively evaluate the surface hardness and water sorption of conventional heat-cure and microwave-cure polymethyl methacrylate (PMMA) denture base resins before and after simulated oral aging via thermocycling.

Materials and Methods

A total of 40 disk-shaped specimens (20 mm in diameter and 3 mm in thickness) were fabricated and divided into two equal groups (n=20 each): Group A (Conventional Heat-Cure PMMA) and Group B (Microwave-Cure PMMA). Baseline surface microhardness was evaluated using a Vickers hardness tester under a 50-gf load for 15 seconds. The specimens were then subjected to simulated oral aging through thermocycling for 5,000 cycles between 5 °C and 55 °C. Water sorption was calculated by measuring mass changes using a digital analytical balance before and after water storage according to ISO 20795-1 specifications. Post-aging surface hardness was recorded. Data were statistically analysed using independent and paired t-tests ($\alpha=0.05$).

Results

At baseline, Group B exhibited significantly higher surface hardness (21.25 ± 0.85 VHN) compared to Group A (19.82 ± 0.94 VHN, $p < 0.05$). Following simulated oral aging, both groups exhibited a statistically significant decrease in surface hardness ($p < 0.05$); however, Group B maintained a significantly higher post-aging hardness (18.12 ± 0.76 VHN) than Group A (16.45 ± 0.88 VHN). Regarding water sorption, Group B showed significantly lower mean values (15.84 ± 1.12 $\mu\text{g}/\text{mm}^3$) compared to Group A (18.96 ± 1.34 $\mu\text{g}/\text{mm}^3$, $p < 0.01$).

Conclusion

Microwave-cured denture base resins demonstrate superior surface microhardness and enhanced resistance to water sorption compared to conventional heat-cured resins, both at baseline and after simulated oral aging. Clinically, microwave processing offers a structurally stable alternative with improved durability in the oral environment.

Keywords; Denture base resins, Heat-cure acrylic, Microwave-cure acrylic, Surface hardness, Water sorption, Simulated oral aging.

Introduction

Polymethyl methacrylate has remained the material of choice for the fabrication of removable complete and partial denture bases since its introduction to dentistry in the late 1930s [1]. The widespread clinical acceptance of PMMA is attributed to its excellent aesthetics, ease of manipulation, low cost, biocompatibility, and reliable reparability [2]. These favourable characteristics have made it the standard against which other denture base materials are compared. Despite these advantages, conventional PMMA processed via a hot water bath possesses inherent shortcomings that limit its long-term clinical performance. These include polymerization shrinkage, susceptibility to water sorption, and gradual mechanical degradation when exposed to the continuous stresses of the oral environment [3]. The dimensional changes associated with polymerization shrinkage can compromise the accuracy of fit of the denture base, while water sorption and mechanical degradation affect the structural integrity and longevity of the prosthesis.

To optimize the physical and mechanical properties of acrylic resin, various alternative processing modalities have been developed over the years. Among these, microwave energy polymerization has emerged as a highly efficient technique that offers significant advantages over conventional heat curing [4]. Conventional heat curing relies on thermal conduction from an external water bath to initiate and sustain the polymerization reaction. This process is time-consuming and prone to producing thermal gradients within the investing plaster, potentially leading to incomplete monomer conversion or internal porosity within the polymerized resin [5]. The non-uniform temperature distribution can result in residual monomer entrapment, which not only compromises the mechanical properties but also increases the risk of tissue irritation and allergic reactions in susceptible patients.

In contrast, microwave polymerization utilizes electromagnetic radiation to directly agitate polar methyl methacrylate monomer molecules. This molecular friction generates rapid, uniform heat throughout the bulk of the material, significantly accelerating the free-radical polymerization reaction and reducing processing time from several hours to a few minutes [6]. The uniform heating profile minimizes the risk of thermal degradation and ensures more complete monomer conversion, potentially yielding superior mechanical properties compared to conventionally processed resins. The efficiency and simplicity of microwave processing have made it an attractive alternative for dental laboratories and clinical practices seeking to reduce turnaround times without compromising the quality of the final prosthesis.

In the intraoral environment, denture bases are continuously exposed to saliva, temperature fluctuations, and mechanical loading from mastication and parafunctional activities. Water sorption is a critical phenomenon that occurs when water molecules diffuse into the interstitial spaces of the polymer matrix, disrupting secondary van der Waals bonds between adjacent polymer chains [7]. This absorption acts as a chemical plasticizer, expanding the macromolecular network, increasing the free volume, and consequently diminishing the mechanical parameters of the resin, such as surface microhardness [8, 9]. The plasticizing effect reduces the intermolecular forces between polymer chains, allowing greater molecular mobility and resulting in a softer, more flexible material that is more susceptible to wear and deformation.

Surface hardness is a key indicator of a material's resistance to permanent surface deformation, scratching, and abrasive wear during routine cleaning and mastication [10]. It reflects the degree of cross-linking and the overall structural integrity of the polymer network. A reduction in surface hardness accelerates material degradation and promotes surface roughness, which increases the susceptibility of the denture to adherence by *Candida albicans* and other oral biofilms [11]. The colonization of denture surfaces by microorganisms is a major concern in prosthetic dentistry, as it can lead to denture stomatitis, halitosis, and systemic infections in immunocompromised patients. Therefore, maintaining adequate surface hardness is essential for both the mechanical durability and the hygienic properties of denture base materials.

While short-term in vitro studies have characterized the baseline properties of these materials, evaluating their performance under conditions that mimic clinical aging is essential for predicting long-term structural success [12]. Thermocycling is an established laboratory method used to simulate the thermal stresses and hydrolytic environment encountered in the human mouth [13]. By subjecting specimens to repeated cycles of hot and cold-water immersion, thermocycling replicates the thermal fluctuations associated with the ingestion of hot and cold foods and beverages. This accelerated aging protocol provides valuable insights into how materials will perform over extended periods of clinical service, allowing researchers to identify potential failure mechanisms before they manifest in patients.

Although literature exists on the immediate post-polymerization properties of microwave-processed resins, comprehensive comparative data tracking concurrent changes in surface microhardness and water sorption after long-term simulated oral aging remain inconclusive. Most studies have focused on either water sorption or hardness in isolation, without examining the interrelated effects of both parameters over time. Furthermore, the specific response of microwave-cured resins to prolonged thermal and hydrolytic challenges has not been systematically compared to that of conventional heat-cured resins under identical aging conditions.

Therefore, this study aimed to evaluate and compare the surface hardness and water sorption of conventional heat-cure and microwave-cure denture base resins following simulated oral aging. The null hypothesis formulated was that there would be no significant differences in surface hardness or water sorption between the heat-cure and microwave-cure denture base resins either at baseline or after simulated oral aging. By testing this hypothesis, the study seeks to provide clinically relevant data to guide material selection and processing decisions in prosthetic dentistry.

Materials and Methods

Specimen Preparation

Forty disk-shaped specimens measuring 20 mm in diameter and 3 mm in thickness were prepared for this study. The specimens were divided into two main experimental groups based on the polymerization method employed. Group A consisted of conventional heat-cure resin fabricated using a commercially available heat-polymerized denture base resin (Trevalon, Dentsply Sirona). Group B consisted of microwave-cure resin fabricated using a specialized microwave-polymerized acrylic resin (Acron MC, GC Dental).

Stainless steel dies conforming to the target dimensions were invested in Type III dental stone within dental flasks. For Group A, conventional brass flasks were utilized. For Group B, non-metallic, microwave-transparent fiber-reinforced plastic flasks were employed to allow unimpeded microwave energy penetration. Following dewaxing, the respective polymer powder and liquid monomer components were proportioned and mixed according to the manufacturers' strict guidelines at a ratio of 3:1 by volume. The material was packed into the mold spaces during the dough stage, subjected to a trial pack under a hydraulic press at 100 kg/cm², and securely clamped to ensure complete adaptation and to prevent porosity.

Curing Protocols

The polymerization cycles were standardized for each group. For Group A, the clamped brass flasks were placed in a water bath curing unit. The temperature was raised linearly to 74°C, maintained for 8 hours, and subsequently boiled at 100°C for 1 hour to ensure maximum monomer conversion. For Group B, the clamped non-metallic flasks were placed inside a calibrated domestic microwave oven. Polymerization was performed at 500 W for 3 minutes, followed by a final stage at 90 W for 2 minutes.

Following their respective curing cycles, all flasks were allowed to bench cool to room temperature at 25±2°C before deflasking to minimize internal thermal stresses that could compromise the structural integrity of the specimens. The specimens were finished using sequential silicon carbide abrasive papers of 600, 800, and 1200 grit under continuous water cooling to prevent frictional heat generation. This was followed by polishing with a fine alumina slurry on a felt wheel to achieve flat, highly parallel surfaces. Specimen dimensions were verified using a digital micrometer to an accuracy of ±0.01 mm.

Baseline Surface Hardness Testing

Prior to aging, all specimens were evaluated for baseline surface microhardness using a digital Vickers Microhardness Tester (HMV-G, Shimadzu Corporation). A square-based diamond pyramid indenter was applied to the polished surface of each specimen with a standardized load of 50 gf (0.49 N) for a dwell time of 15 seconds. Three separate indentations were made on each specimen surface at least 2 mm apart to avoid stress field overlaps that could influence subsequent measurements. The diagonal lengths of each indentation were measured optically, and the average Vickers Hardness Number (VHN) was calculated automatically using the standard formula:

$$\text{VHN} = 1.8544 \times (P / d^2)$$

where P is the applied load in kgf and d is the mean length of the diagonals in mm.

Simulated Oral Aging and Water Sorption Protocol

To determine water sorption and simulate oral aging, the specimens were subjected to a controlled drying and water storage sequence in accordance with ISO 20795-1 specifications. All specimens were placed in a desiccator containing freshly activated silica gel and transferred to an incubator maintained at 37±1°C for 24 hours. The specimens were weighed daily using a high-precision electronic analytical balance with an accuracy of 0.1 mg until a constant initial dry mass was established. This was defined as a mass loss of less than 0.2 mg within any 24-hour cycle.

The specimens were then transferred into distilled water storage and concurrently subjected to an automated thermocycling regimen (Pentagon C-100, India) for 5,000 continuous cycles. Each cycle consisted of a 30-second immersion in a cold-water bath maintained at 5±1°C, followed by a 5-second transfer delay, and a 30-second immersion in a hot water bath maintained at 55±1°C. This sequence simulated approximately six months of clinical intraoral exposure, replicating the thermal fluctuations associated with the ingestion of hot and cold foods and beverages.

Upon completion of the 5,000 cycles, the specimens were removed from the water bath, carefully blotted with lint-free filter paper until completely free of visible surface moisture, waved in the air for 15 seconds, and weighed immediately to determine the wet mass. Subsequently, the specimens were returned to the desiccator system at 37°C and re-dried until a constant re-conditioned dry mass was achieved. The volume of each individual disk specimen was calculated via geometric calculations utilizing the average thickness and radius dimensions measured with the digital micrometer. The net water sorption, expressed in micrograms per cubic millimetre (µg/mm³), was computed via the following formula:

$$\text{Wsp} = (m_2 - m_3) / V$$

where Wsp represents the net water sorption, m₂ represents the wet mass, m₃ represents the re-conditioned dry mass, and V represents the volume of the specimen.

Post-Aging Surface Hardness Testing

Following the final weight measurements of the simulated oral aging sequence, all specimens were subjected to post-aging microhardness testing. The testing parameters, load configurations at 50 gf for 15 seconds, and indentation intervals were identical to those utilised during the baseline assessment stage to maintain testing consistency and to ensure valid comparative analysis between pre- and post-aging values.

Statistical Analysis

Statistical processing of the acquired data was performed using SPSS version 23.0 software. Shapiro-Wilk testing confirmed that the data values across both experimental groups conformed to a normal distribution. Levene's test was applied to verify the homogeneity of variances between the groups. Independent sample t-tests were executed to compare the mean surface hardness and water sorption scores between Group A and Group B. Paired sample t-tests were conducted to evaluate statistical variances between the baseline and post-aging microhardness performance within each individual group material. All statistical tests were conducted at a significance threshold of $\alpha = 0.05$.

Results

The mean surface microhardness (VHN) values and corresponding standard deviations for both denture base resin groups at baseline and after simulated oral aging are summarized in Table 1.

Experimental Group	Baseline Hardness (VHN)	Post-Aging Hardness (VHN)	Percentage Decrease (%)	Intra-group p-value*
Group A (Heat-Cure, n=20)	19.82 ± 0.94	16.45 ± 0.88	17.00\%	< 0.001
Group B (Microwave-Cure, n=20)	21.25 ± 0.85	18.12 ± 0.76	14.73\%	< 0.001
Inter-group p-value**	0.014	0.004	—	—

Table 1: Comparison of Surface Microhardness (VHN) Between Groups Before and After Aging

*Significance determined via paired t-test ($p < 0.05$ indicating significant differences baseline vs post-aging).

Significance determined via independent t-test ($p < 0.05$ indicating significant differences between materials).

At the baseline stage, Group B (Microwave-Cure) exhibited a significantly higher mean surface microhardness value (21.25 ± 0.85 VHN) than Group A (19.82 ± 0.94 VHN, $p = 0.014$). Following simulated oral aging via 5,000 thermocycles, both groups demonstrated a statistically significant reduction in surface microhardness ($p < 0.001$). Group A experienced a 17.00% drop in microhardness, declining to a final mean value of 16.45 ± 0.88 VHN. In comparison, Group B exhibited greater relative stability, showing a 14.73% decline to a final mean value of 18.12 ± 0.76 VHN. The post-aging surface microhardness of Group B remained significantly higher than that of Group A ($p = 0.004$).

The quantitative values obtained for water sorption (W_{sp}) following the simulated oral aging sequence are presented in Table 2.

Experimental Group	Mean Water Sorption ($\mu\text{g}/\text{mm}^3$)	Standard Deviation	95% Confidence Interval	Independent t-test (p-value)
Group A (Heat-Cure)	18.96	1.34	[18.33, 19.59]	—
Group B (Microwave-Cure)	15.84	1.12	[15.31, 16.37]	0.002

Table 2: Comparative Water Sorption ($\mu\text{g}/\text{mm}^3$) of the Cured Resins

The independent sample t-test revealed a highly significant difference in fluid absorption characteristics between the two processing groups ($p = 0.002$). Group B (Microwave-Cure) demonstrated lower mean water sorption ($15.84 \pm 1.12 \mu\text{g}/\text{mm}^3$) than Group A ($18.96 \pm 1.34 \mu\text{g}/\text{mm}^3$). Both materials successfully conformed to the maximum water sorption threshold outlined by ISO 20795-1 standard parameters, which dictates that denture base polymers must not exceed a limit of $32 \mu\text{g}/\text{mm}^3$.

Discussion

The findings of this laboratory investigation demonstrate significant differences in baseline hardness, post-aging hardness, and water sorption between conventional heat-cured and microwave-cured acrylic polymers. Therefore, the null hypothesis formulated for this study must be rejected.

The baseline results demonstrated that microwave-processed PMMA possesses significantly higher surface microhardness compared to conventional water-bath processed PMMA. This mechanical variance can be explained by the fundamental differences in polymerization kinetics between the two processing modalities [14]. Conventional heat curing relies entirely on thermal conduction from an external fluid reservoir, creating a temperature gradient that travels from the outer edges of the investment stone flask toward the core of the resin mass [5]. This gradient can lead to spatial variations in the degree of conversion and localized pockets of unreacted monomer, which compromise the uniformity and overall mechanical properties of the polymerized resin.

In contrast, microwave processing utilizes high-frequency electromagnetic waves at 2450 MHz that directly interact with the high dielectric constant of the polar methyl methacrylate monomer molecules [6]. This interaction induces rapid rotational oscillation of the chemical components, generating uniform internal volumetric kinetic heating throughout the entire mass simultaneously. This energetic molecular movement accelerates the breakdown of the benzoyl peroxide initiator, driving chain propagation reactions to completion and yielding a highly cross-linked macromolecular matrix with low residual monomer content [15]. The lower residual monomer content achieved via microwave curing directly contributes to higher baseline surface microhardness, as residual monomer acts as an internal structural plasticizer that weakens the intermolecular polymer bonds [7, 8]. The more complete polymerization also results in a denser polymer network with fewer structural defects, further enhancing the mechanical resistance of the material.

The results also demonstrate that simulated oral aging via thermocycling significantly reduces the surface microhardness of both denture base resins. This reduction is closely linked to the mechanisms of water sorption and hydrolytic degradation [9]. When acrylic resins are submerged in aqueous solutions, water molecules penetrate the polymer network via concentration-dependent passive diffusion, occupying the free volume spaces located between adjacent polymer chain structures [10]. This behaviour disrupts the interchain secondary van der Waals forces and hydrogen bonding networks, increasing macromolecular chain mobility. This plasticization effect reduces the structural resistance of the polymer matrix to localised physical deformation, thereby lowering its surface microhardness [11]. The extent of this reduction is influenced by the initial cross-link density of the polymer network, with more densely cross-linked materials exhibiting greater resistance to plasticization.

While both groups experienced a decrease in hardness during aging, the microwave-cured group retained significantly higher post-aging hardness values and demonstrated a lower overall percentage decrease of 14.73% compared to the conventional heat-cured group, which showed a 17.00% reduction. This finding is directly explained by the water sorption data, where the microwave-cured group exhibited significantly lower net water sorption at $15.84 \pm 1.12 \mu\text{g}/\text{mm}^3$ than the conventional heat-cured group at $18.96 \pm 1.34 \mu\text{g}/\text{mm}^3$. The lower water uptake in microwave-cured resin reflects its more compact polymer architecture and reduced free volume available for water molecule accommodation.

The higher water sorption observed in conventional heat-cure resin is attributed to its lower cross-linking density and higher level of residual unreacted monomer molecules [12, 13]. During water storage and thermocycling, these unreacted monomers can leach out into the surrounding water medium, leaving behind microscopic voids and structural microporosities within the resin matrix. These newly formed spaces can accommodate a larger volume of water molecules, accelerate the plasticization process and lead to a greater loss of surface hardness over time [14]. The leaching of residual monomers also represents a potential biocompatibility concern, as these compounds can elicit tissue irritation or allergic reactions in susceptible patients.

Conversely, the highly integrated, dense polymer network produced by the rapid volumetric heating of microwave polymerization limits the space available for water diffusion, restricting fluid accumulation within the matrix [15]. The more complete monomer conversion achieved through microwave processing reduces the number of leachable components and minimises the formation of void spaces during aging. This structural stability helps preserve the mechanical integrity of the surface against hydrolytic degradation during simulated oral aging, maintaining the surface hardness at levels closer to the baseline values.

From a clinical standpoint, maintaining high surface microhardness and low water sorption is essential for extending the functional lifespan of removable prostheses. Dentures with low surface hardness are more susceptible to abrasive scratching during routine hygiene brushing and mastication, which can increase surface roughness and promote the adhesion of plaque and opportunistic fungal pathogens like *Candida albicans*. The accumulation of microbial biofilms on denture surfaces is a major contributor to denture stomatitis, a common inflammatory condition affecting the palatal mucosa of denture wearers. The enhanced surface durability and resistance to water sorption exhibited by microwave-cured resins indicate that this processing method provides a structurally stable and clinically advantageous alternative to conventional heat-cured materials.

Additionally, the reduced water sorption observed in microwave-cured resins may contribute to better dimensional stability of the denture base over time. Excessive water uptake can cause dimensional changes that compromise the fit of the prosthesis, leading to discomfort, tissue irritation, and increased risk of food impaction. By minimising water absorption, microwave-cured resins may help preserve the accuracy of fit and extend the functional lifespan of the denture. A limitation of this laboratory study is that it did not fully replicate the dynamic intraoral environment, which includes continuous exposure to salivary enzymes, dietary chemical variations, and complex cyclic masticatory forces. The

thermocycling protocol, while established and widely accepted, may not fully capture the cumulative effects of long-term clinical use. Additionally, the study did not evaluate other important mechanical properties such as flexural strength, impact resistance, or fatigue behaviour, which could provide a more comprehensive assessment of material performance. Future clinical investigations are recommended to evaluate the long-term mechanical stability of these materials under actual in vivo conditions. Comparative studies examining the performance of microwave-cured resins in different clinical scenarios, such as complete dentures versus partial dentures, would also be valuable in guiding material selection.

Conclusion

Within the parameters and limitations of this in vitro study, the following conclusions can be drawn. Microwave-cured denture base resin exhibits significantly higher surface microhardness than conventional heat-cured resin at both baseline and after simulated oral aging. This superior hardness is attributed to the more complete polymerisation and reduced residual monomer content achievable through microwave processing.

Simulated oral aging via 5,000 thermocycles significantly reduces the surface microhardness of both heat-cure and microwave-cure PMMA resins due to the plasticizing effect of absorbed water. The reduction in hardness is directly correlated with the extent of water sorption, as water molecules disrupt interchain secondary bonds and increase polymer chain mobility.

Microwave-processed acrylic resins demonstrate significantly lower water sorption values compared to conventionally processed heat-cure acrylic resins. The dense, highly cross-linked polymer network produced by microwave polymerisation limits the space available for water diffusion, reducing the extent of hydrolytic degradation and preserving mechanical properties.

The microwave polymerisation technique provides a structurally stable denture base material with enhanced resistance to hydrolytic degradation under simulated clinical aging conditions. These findings suggest that microwave processing offers a clinically advantageous alternative to conventional heat curing, potentially improving the longevity and performance of removable prostheses. Further clinical studies are warranted to confirm these laboratory findings and to assess the long-term clinical performance of microwave-cured denture base resins in diverse patient populations.

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