

The effect of adding ZrO₂ nanoparticles on the transverse strength and hardness of microwave-cured acrylic and heat-cured acrylic denture base materials

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ABSTRACT

Background: One drawback of acrylic denture base materials is their liability to fracture, requiring methods to increase fracture resistance. Adding nanoparticles (NPs) represented one of these methods. **Purpose:** The objectives of this study are to evaluate and compare transverse strength and hardness when adding zirconium oxide nanoparticles (ZrO₂ NPs) at concentrations of 0%, 3%, and 5% to heat-cured acrylic denture base materials (Ivoclar, Major) and to microwave-cured acrylic (Acron MC). **Methods:** Transverse strength was tested with a Universal Testing Machine (GESTER, Fujian, China), while hardness tests were conducted by using a Shore-D hardness durometer (Shou, China). The 90 samples were prepared and then divided into three groups for each material. Attenuated total reflectance–Fourier transform infrared (ATR-FTIR) spectroscopy was used to analyze the microstructure. The samples were prepared following the manufacturer's instructions for each material. **Results:** The results revealed that the addition of ZrO₂ NPs (3%, 5%) improves the transverse strength and hardness of polymethyl methacrylate acrylic resin for both types (microwave-cured and heat-cured acrylic resins). The addition of ZrO₂ NPs at 3% concentration shows the highest values for both transverse strength and hardness. The ATR-FTIR confirms no structural chemical changes with the addition of ZrO₂ NPs. **Conclusion:** The study concludes that the incorporation of ZrO₂ NPs (3%, 5%) into microwave-cured and heat-cured acrylic resins improves transverse strength and hardness.

Keywords: denture base; hardness; PMMA; transverse strength; zirconium oxide nanoparticle

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INTRODUCTION

A typical denture base material should have proper mechanical and physical properties, besides biocompatibility and aesthetics. Transverse strength includes tensile and compressive strengths. It has some elements of the proportional limit and elastic modulus. It is also considered as the modulus of rupture or flexural strength, stiffness, and resistance to fracture.¹

The addition of nanoparticles (NPs) into polymethyl methacrylate acrylic resin (PMMA) has recently gained attention. Zirconium oxide nanoparticles (ZrO₂ NPs), which are a bioceramic for many dental uses,^{2,3} such as crowns, bridges, screws, implant fixtures, abutments, orthodontic brackets⁴, and maxillofacial materials,⁵ is used to enhance the mechanical and physical properties of

PMMA. Zirconia is of a high flexural strength (900 to 1200 MPa), high fracture toughness (9–10 MPa m^{1/2}), and high hardness (1200 HV).⁶ It also has better biocompatibility than other ceramics, such as alumina. Many studies that modify conventional heat-cured resins with ZrO₂ NPs show that this remarkably improves the resins' mechanical properties, including surface hardness, flexural, and impact strength.^{7,8}

ZrO₂ NPs greatly enhance transverse strength and hardness, and studies have found that transverse strength has linear correlation with hardness number.^{9,10} ZrO₂ NPs have also recently been proven to enhance the transverse strength of repaired denture bases.^{11,12} However, this effect is shown to decrease when the concentration of ZrO₂ NPs is increased due to the agglomeration phenomena, resulting in a worsening effect on material.¹³

Using a water bath is the conventional method of acrylic resin curing; it has a low cost, but it requires a longer amount of time than other methods. Problems associated with water bath curing include hypersensitivity reactions, easy breakage, and increased surface porosity. Several types of modified PMMA have been introduced to overcome the mentioned problems. Recently, microwave energy has been used to provide thermal energy that induces polymerization reactions in the acrylic resin; a non-metallic fiber-reinforced plastic FRP flask must be used specifically with formulated microwave processing resin. PMMA cured by microwave radiation is better than PMMA cured in a water bath because the interior and exterior of the substance are equally and promptly heated, which eradicates the time required to maintain the heat gained in the water bath until the resin is inside the flask. Polymerization via microwave energy improves multiple physical and mechanical properties, such as surface roughness, porosity, and dimensional stability.^{14–16} The aims of this study are to assess and contrast the transverse strength and hardness of heat-cured acrylic (Ivoclar, Major) and microwave-cured acrylic (Acron MC) denture base materials when ZrO₂ NPs are incorporated at different concentrations (control 0%, 3%, 5%).

MATERIALS AND METHODS

A total of 90 samples (Figure 1) were collected; 45 samples were divided into three groups for each material of each test (five for each ZrO₂ NP concentration). Tests were carried out on two groups (heat-polymerized acrylic resin specimens (Major) and microwave-polymerized specimens (Acron-MC)). Nano-ZrO₂ at 50 nm¹⁷ (US Research Nanomaterials, Inc., Houston, USA) was added to the specimens in concentrations of 0%, 3%, and 5% by weight. All samples were reserved in distilled water at 37°C for 48 h before testing.

Determining proper percentages of ZrO₂ NPs: Weight percentages of ZrO₂ NPs in the present study were specified based on previous research (1.5 wt.%, 3.0 wt.%, 5.0 wt.%, 7.0 wt.%, 10.0 wt.%, and 15 wt.% mixtures), where the most appropriate percentages giving the best results were at 3wt.% and 5 wt.% ZrO₂ NPs. Based on Zidan et al.'s study¹⁷ that concluded ZrO₂ NPs added to PMMA at 3 wt.% and 5 wt.% resulted in optimal mechanical properties for denture base uses, a decision was made to adopt those concentrations (0 wt.%, 3.0 wt.%, 5.0 wt.% of ZrO₂ NPs) in the present study.

Mixing of ZrO₂ NPs with PMMA: The ZrO₂ NPs and acrylic powders were weighed according to the specific percentages by an electronic scale (accuracy of three decimal points). The ZrO₂ NP powder was incorporated into the acrylic monomer and mixed properly via shaking by hand and then via ultrasonic vibrations for approximately 10 min to ensure the proper and equal distribution of all the powder within the monomer. Then, the powder and the monomer were mixed together. The mixing continued for approximately 20 min until the mixture reached a dough-like state. When the mixture reached a homogeneous dough stage (working stage), it was handled into a mold by hand. The packing and curing processes were done in accordance with British Standards Institution: Dentistry Base Polymers (BS EN ISO 20795-1:2008) and British Standards Institution: Specification for Denture Base Polymers (BS 2487:1989).^{18,19}

All the heat-cured acrylic resin samples were cured by using standard protocols of polymerization,²⁰ while the microwave-polymerized specimens (Acron MC) were cured by mixing powder and liquid at a ratio of 100mg/43ml. Then, it was packed into a microwavable flask mold (FRP FlaskH.K. TYPE) where the associated bolts were inserted. Constant pressure was applied by the supplied wrench and submitted to 500 W microwave irradiation for 3 min in a microwave oven (EM-M 553 T,

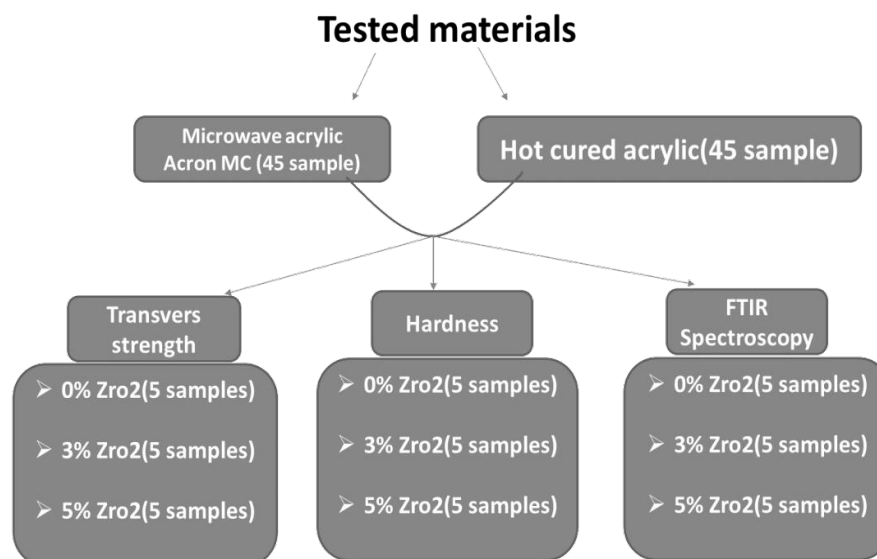


Figure 1. Study design reflecting the steps to follow to test materials for transverse strength, hardness, and FTIR spectroscopy.

Sanyo) for the microwave curing of Acron MC specimens.²¹ All samples were kept in distilled water at 37°C for 48 h before testing. Adding ZrO₂ NPs (0%, 3%, 5%) by weight to the microwave-cured acrylic (Acron MC) and heat-cured acrylic denture base materials (Ivoclar, Major) and then to the monomer proved to be advantageous over adding it to the powder. This finding is confirmed by many other researchers who also mixed ZrO₂ NPs with the monomer and saw similar benefits.²²

Transverse strength: Specimens with dimensions of 65 mm in length, 10 mm in width, and 2.5 mm in thickness were prepared according to International Standards Organization (ISO) Specification No. 1567. Transverse strength was tested using a Universal Testing Machine (GESTER, Fujian, China). The transverse strength of acrylic resin samples was measured at three bending points. The values of transverse strength were calculated with the following equation: transverse strength (S) = $3PI/2bd^2$ (N/mm²), where: P = Maximum force (N), I = Distance between supports (mm), b = specimen width (mm), and d = a specimen depth (mm).²³

Hardness: To measure surface hardness, specimens of 30 mm in length, 10 mm in width, and 2.5 mm in thickness were prepared according to International Standards Organization (ISO) Specification No. 1567. Hardness tests were conducted by using a Shore-D hardness durometer (Show, China); five readings were taken for each specimen and then the mean was calculated.

Attenuated total reflectance–Fourier transform infrared (ATR-FTIR) spectroscopy: ATR-FTIR spectroscopy is one of the most prevalent spectroscopic methods for analyzing the structure of polymers. FTIR spectroscopy presents a sensitive analysis tool to detect chemical composition changes in biomaterials. Thirty specimens of heat-cured resin and microwave-cured acrylic resin were prepared.²⁴

Statistical analysis for all data was done in SPSS (version 24). Data was expressed as mean±SD. The differences were considered statistically significant at $p \leq 0.05$. Analysis of variance (ANOVA) with Duncan tests were conducted for different concentrations within the group and t-tests were conducted between both groups to compare heat-treated versus microwave-treated specimens.

RESULTS

Figure 2 demonstrates the mean values and standard deviation of transverse strength for heat-cured acrylic (Ivoclar, Major) and microwave-cured acrylic (Acron MC) denture base materials after adding ZrO₂ NPs (0%, 3%, 5%). Heat-polymerized acrylic resin samples from the control group (0% ZrO₂ NP) show the lowest value, 89.717 ± 1.486 , while 3% ZrO₂ NP concentration samples from the microwave-cured acrylic group show the highest value, 109.3 ± 11.322 .

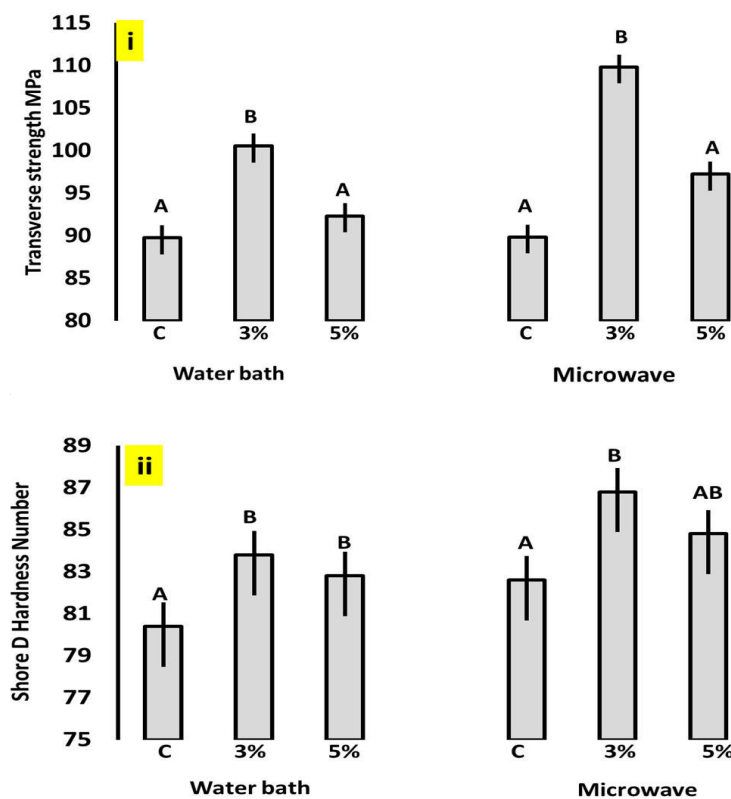


Figure 2. Transverse strength MPa (N/mm²): (i) Shore D hardness (ii) for heat-polymerized acrylic (Ivoclar, Major) and microwave-cured acrylic (Acron MC) denture base materials after adding ZrO₂ NPs (control (C), 3%, 5%). Data is expressed as mean±SD. The different letters indicate the significance of the p value (≤ 0.05) according to DMRT.

The ANOVA of transverse strength shows that there are statically significant differences among all tested groups (heat-polymerized and microwave-cured acrylic denture base materials) after adding ZrO₂ NPs (0%, 3%, 5%). The F ratio for heat-cured acrylic resins was 7.333 and the p value was 0.008, while the F ratio for microwave-cured acrylic resins was 8.967 and the p value was 0.004, showing a

significant difference in terms of $p \leq 0.05$. Duncan multiple range tests (DMRT) illustrated a significant increase in transverse strength for both heat-polymerized acrylic (Ivoclar, Major) and microwave-cured acrylic (Acron MC) resins at 3% ZrO₂ NPs with a p value of ≤ 0.05 . However, the addition of 5% ZrO₂ NPs to both types of denture base materials demonstrated increased transverse strength, but,

Table 1. Transverse strength MPa and Shore D hardness number for heat-polymerized acrylic resin (Ivoclar, Major) and microwave-cured acrylic resin (Acron MC) denture base materials after adding ZrO₂ NPs

Groups		Transverse strength MPa	F	Sig
		mean±SD		
Control	Heat-cured (n=5)	89.7±1.5	0.322	0.586
	Microwave-cured (n=5)	89.9±1.9		
3%	Heat-cured	100.5±6.7	2.953	0.124
	Microwave-cured (n=5)	109.3±11.3		
5%	Heat-cured	92.4±4.2	0.013	0.911
	Microwave-cured (n=5)	97.2±5.5		
		Shore D hardness number		
Control	Heat-cured	80±1.1	0.913	0.367
	Microwave-cured (n=5)	82±2.2		
3%	Heat-cured	83.8±0.8	4.500	0.067
	Microwave-cured (n=5)	86.8±1.6		
5%	Heat-cured (n=5)	82.8±0.8	1.645	0.236
	Microwave-cured (n=5)	84.8±1.9		

Data expressed as mean±SD, Significant difference at $p \leq 0.05$.

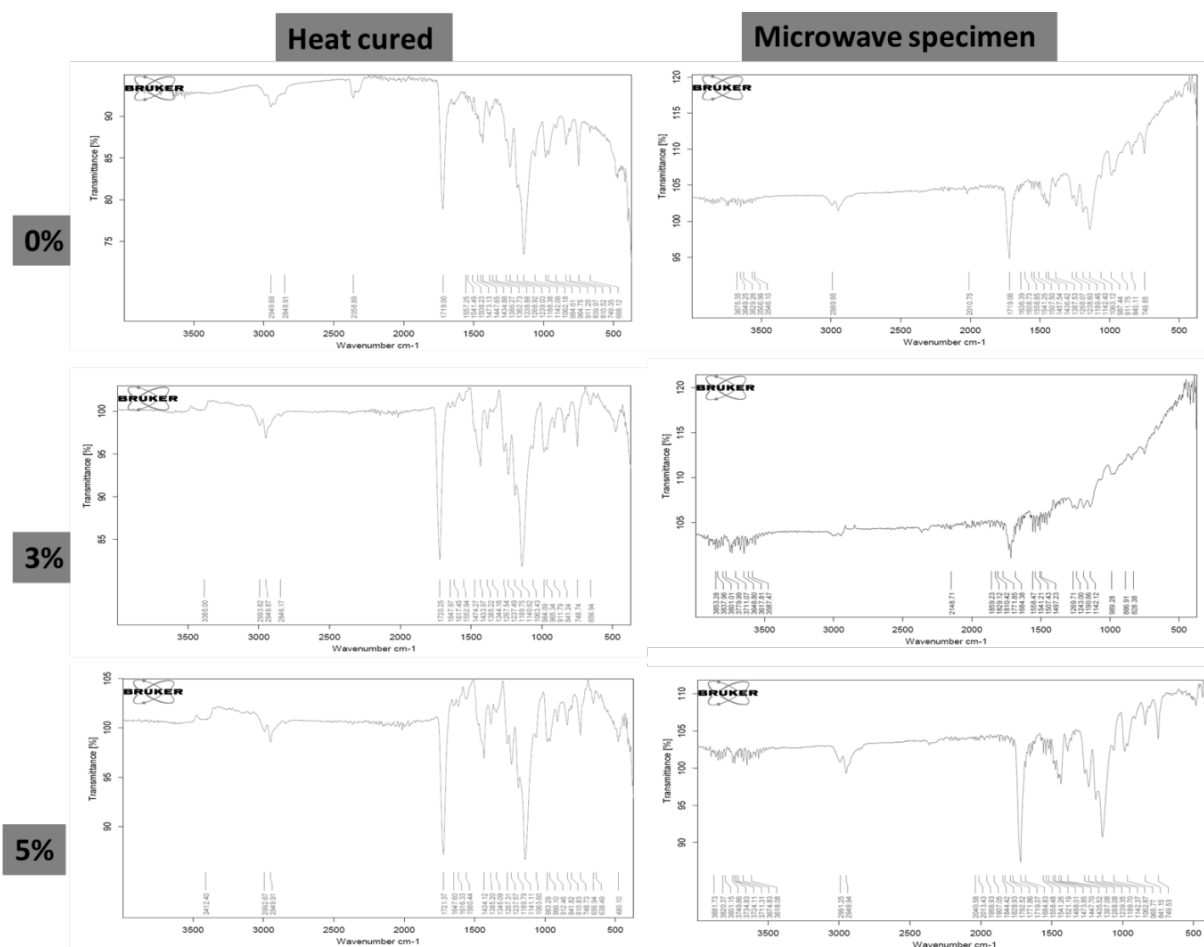


Figure 3. ATR-FTIR spectrum for heat-cured acrylic and microwave-cured acrylic (0% control, 3% ZrO₂, 5% ZrO₂).

statically, this did not reach a level of significant difference. As shown in Figure 2 (i), the different letters mean the level of significance in terms of $p \leq 0.05$.

Figure 2 (ii) illustrates the mean values and standard deviation of Shore D hardness for heat-polymerized acrylic (Ivoclar, Major) and microwave-cured acrylic (Acron MC) denture base materials after adding ZrO₂ NPs at 0%, 3%, and 5% concentrations. The control heat-polymerized acrylic resin samples showed the lowest value of 80.4 ± 1.140 , while samples of microwave-cured acrylic resins had a higher value of 86.8 ± 1.643 after adding ZrO₂ NPs at 3%. The ANOVA of Shore D hardness numbers for heat-polymerized and microwave-cured acrylic resin materials indicate that there are significant differences among all tested groups after adding ZrO₂ NPs (0%, 3%, 5%), with a p value of ≤ 0.05 .

Duncan's multiple range tests of the Shore D hardness numbers for heat-polymerized acrylic resins shows that there is significant increase in the hardness numbers with 3% and 5% ZrO₂ NP concentrations, with the control group having a p value of ≤ 0.05 . After the addition of ZrO₂ NPs, microwave-cured acrylic denture base materials showed a statically significant change at 3% ZrO₂ NPs, with the control group having a p value of ≤ 0.05 . They also exhibited an increase in hardness at 5% ZrO₂ NPs, but no statically significant change was seen for other groups.

Table 1 explains the independent t-tests conducted for transverse strength and the Shore D hardness numbers for the two groups, heat-cured acrylic resins and microwave-cured acrylic resins, after adding ZrO₂ NP concentrations (0%, 3%, 5%). No significant differences were found between the two types of acrylic denture base materials after adding ZrO₂ NPs (5%) in terms of p value.

The ATR-FTIR charts (Figure 3) confirm the absence of any structural chemical changes after adding 3% or 5% ZrO₂ NPs as compared to the control groups (0% ZrO₂ NPs). However, the FTIR charts show similarity in their spectral peaks compared to those of the control group. The absence of newly formed peaks confirms that no new materials were formed; thus, no chemical reaction occurred with ZrO₂ NPs.

DISCUSSION

The results of this study show that the addition of ZrO₂ NPs (3% and 5%) increases transverse strength when compared to the control groups of both heat-cured and microwave-cured acrylic resins. This confirms previous research findings that proved the beneficial effect of adding ZrO₂ NPs to PMMA denture base materials on transverse strength.^{2,8,11,25,26} The increase in transverse strength may be a result of a good dispersion of the ZrO₂ NPs fillers within the matrix. Their nano size allows them to spread through the basis of the interstitial filling of the acrylic resin matrix in a uniform manner, which interrupts crack propagation, thus improving the strength of the resin.^{25,27}

The improvement in transverse strength of the PMMA may be related to the higher conversion of the monomer into the polymer. Moreover, the size of the particles and the average polymer chain length could undoubtedly affect the transverse strength of the acrylic resin.²⁸

It has also been found that the best results were obtained with the 3% ZrO₂ NP concentration, which is in agreement with other studies that argued that the addition of high concentrations of ZrO₂ NPs results in a decrease in transverse strength. This undervalue can be attributed to the presence of such a high filler content, meaning it cannot be incorporated by the resin because it reaches a saturation point. Any attempt to add filler particles to the already saturated matrix would lead to continuity interruption in the resin's matrix, thus causing an undervalue in the strength of the reinforced samples.^{17,29,30}

This study shows that hardness increases with the addition of 3% and 5% ZrO₂ NP concentrations when compared to the control groups for both heat-cured acrylic and microwave-cured acrylic resins. This finding is in agreement with other research.^{3,11,31} This increase in hardness could be the result of an improvement in the mechanical properties due to the addition of ZrO₂ NPs, including mechanisms of crack refraction and restriction.^{2,8,12,25}

Enhancement in the mechanical and physical properties may be a result of better dispersion of ZrO₂ NPs. This enhancement would lessen the cohesion tendency in the composites, while the large interface surface area of the NPs would result in extra contact points between the ZrO₂ NPs and PMMA. Moreover, this enhancement would accelerate mechanical interlocking, thus offering additional flexibility and indentation resistance of the nanocomposites.³²

The highest hardness values were obtained with the 3% ZrO₂ NPs concentration, which is in agreement with various research.^{29,31} A number of studies have proved that adding more than a 5% concentration of ZrO₂ NPs to the resin would seriously affect its mechanical properties.^{8,33,34} For example, Zhang et al.⁴ conducted a similar study with NP concentrations (1 wt.%, 2 wt.%, 3 wt.%, and 4 wt.%), and Gad et al.⁸ used concentrations of 2.5 wt.%, 5 wt.%, and 7 wt.%. The aforementioned scholars attributed the lowering in surface hardness to the high filler loading due to weak adherence of the particles to the resin matrix. They also attributed it to filler clumping within the resin.

The results show there are no remarkable differences in transverse strength or hardness tests between microwave-cured and the heat-cured acrylic resins at the chosen concentrations of ZrO₂ NPs (0%, 3%, 5%) ($p \leq 0.05$). However, microwave-cured acrylic resin showed greater values for both transverse strength and hardness, which is in agreement with Spatalis et al.¹⁶ yet contradictory to Ozkir et al.¹⁴, who noted a reduction in the mechanical properties of microwave-cured acrylic when compared with conventionally heat-cured materials, especially in terms of transverse strength. This decrease, according to Ozkir et al.,¹⁴ may be related to the high vapor of the monomer during microwave polymerization.

The ATR-FTIR confirms the absence of any structural chemical changes in the resin after adding 3% or 5% ZrO₂ NP concentrations when compared to the control groups (0% ZrO₂ NPs) for both heat-cured and microwave-cured acrylic resin. The FTIR charts show similarity in the spectral peaks of both the tested and the control groups. The absence of newly formed peaks confirms that no new materials are formed and no chemical reaction occurred due to ZrO₂ NP concentrations. The results shown in Figure 3 demonstrate that no chemical changes occurred because no chemical reaction between saturated acrylic resins and saturated ZrO₂ NPs had taken place and also because of the absence of functional or active groups in both materials. These results were obtained through ATR-FTIR qualitative and quantitative structural analysis, which is based on interactions at the molecular level with no need to cure acrylic in thin film specimens.^{35,36}

The results of the study show that adding ZrO₂ NP concentrations (3%, 5%) improves the transverse strength and hardness of PMMA for both heat-cured and microwave-cured acrylic resin and that the 3% concentration results in the highest values for both transverse strength and hardness. No changes in the chemical structures were detected for both heat-cured acrylic resin and microwave-cured acrylic resin after adding 3% or 5% ZrO₂ NP concentrations, so the additives evaluated throughout this study possess the capability to serve as a very efficient reinforcing aid for denture base resins polymerized using either the water bath technique or using microwave energy.

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