New Materials, Compounds and Applications
Vol.8, No.3, 2024, pp.363-372
https://doi.org/10.62476/nmca83363

# COMPARISON OF SOME MECHANICAL PROPERTIES OF ADDING $ZrO_2$ NANOPARTICLES TO MICROWAVE-CURED ACRYLIC AND HOT-CURED DENTURE BASE ACRYLIC MATERIALS

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**Abstract.** *Background:* The mechanical properties of denture-base acrylic resin have declined over the past decade, it is imperative to take immediate action to investigate alternative possibilities, such as employing metal oxide nanoparticles, which have shown considerable promise in diverse applications in the fields of nanobiotechnology, specifically in biological and health care fields. *Aims:* Exploring the effect of incorporating zirconium oxide ($ZrO_2$) nanoparticles into microwave-cured acrylic and hot-cured acrylic denture base materials, on the tensile strength, impact strength and structural chemical changes. *Methods:* A total of 66 samples were utilized. The manufacturer's instructions have been followed for the production of specimens for each product. The impact of addition 0%, 3% and 5% zirconium oxide ($ZrO_2$) nanoparticles were examined. *Results:* Addition of 3% and 5% $ZrO_2$ nanoparticles improved the tensile and impact strengths with no chemical alterations changes with the addition of $ZrO_2$ NPs, with 3% $ZrO_2$ showing a greater enhancement than 5%. *Conclusions:* Incorporating 3% $ZrO_2$ nanoparticles into both microwave-cured and hot-cured acrylic denture base materials enhances tensile and impact strengths more effectively than 5%, with no chemical changes observed.

***Keywords:*** *Impact strength, nanoparticle, resin, tensile strength, zirconium oxide.*

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***Received:*** *2 June 2024;* ***Accepted:*** *1 October 2024;* ***Published:*** *10 December 2024.*

## 1. Introduction

Zirconia nanoparticles (NPs) have drawn interest in recent years as a prospective curative for enhancing the mechanical characteristics of polymethylmethacrylate acrylic resin (PMMA) (Elzahar *et al*., 2022). PMMA is commonly utilized for the production of dentures since it is easy to work within a laboratory setting, inexpensive, stable in the mouth, capable of matching colours, aesthetically pleasing and non-toxic. Nevertheless, there are some disadvantages associated with it, such as limited ability to withstand impact and susceptibility to fatigue failure, particularly when using the traditional curing procedure (water bath) (Alqutaibi *et al*., 2023; Chhabra *et al*., 2022; Zafar, 2020).

**How to cite (APA):**
Aziz, R.R., Shehab, M.M., Hasan, R.H., Abdulwahhab, G.H. & Abduo, J. (2024). Comparison of some mechanical properties of adding $ZrO_2$ nanoparticles to microwave-cured acrylic and hot-cured denture base acrylic materials. *New Materials, Compounds and Applications, 8*(3), 363-372 https://doi.org/10.62476/nmca83363

To address these problems, many altered forms of PMMA have been introduced, such as the utilization of microwave energy to stimulate polymerization reactions in PMMA. This process enhances characteristics such as porosity, surface roughness and dimensional stability. However, microwave polymerization is not widely accepted in therapeutic settings, despite its advantages (Ajay *et al*., 2019; Salih, 2015; Wang *et al*., 2016; Zhang *et al*., 2014).

Tensile stresses, the primary factor contributing to fractures of dentures within the mouth, are multifaceted stress that results in intra-oral fractures (Asopa *et al*., 2015; Gad *et al*., 2018a; 2018b).

The inclusion of NPs into PMMA has attracted interest, particularly $ZrO_2$ NP, which enhance the physical and mechanical characteristics of the PMMA/$ZrO_2$ nanocomposite. $ZrO_2$ NP possesses elevated levels of hardness, compressive strength and fracture toughness. Its exceptional biocompatibility greatly enhances the mechanical characteristics of heat-cured acrylic resin, such as flexural strength, surface hardness and impact strength (Asopa *et al*., 2015; Elhazar *et al*., 2022; Gad *et al*., 2018b; Salih, 2015; Wang *et al*., 2016; Zhang *et al*., 2014).

The objectives of the current study propose to determine and contrast the tensile strength and impact strength of hot acrylic denture base materials and microwave-cured acrylic (MCA) with different concentrations (control 0%, 3%, 5%) of zirconium oxide $ZrO_2$ NPs.

The null hypothesis posited that the inclusion of zirconium oxide ($ZrO_2$) NPs at concentrations of 3% and 5% by weight in hot and MCA resin would have no impact on the tensile strength and impact strength nor the chemical structure of the modified acrylic resin.

## 2. Materials and Methods

**Sample Preparation:** A total set of 66 samples was generated and two groups underwent testing. The specimens in the first group were produced by heat-polymerizing acrylic (HPA) resin (Major), while the specimens in the second group were cured using a microwave (Acron MC). There were three subgroups for each of the 33 samples. A set of five samples of $ZrO_2$ NPs was utilized for each concentration within each group. Particles of nanoscale Zirconium dioxide ($ZrO_2$) measuring 50 nm in diameter were introduced into the specimens at weight percentages of 0%, 3% and 5% (US Research Nanomaterials Inc., Houston, USA). Before testing, all samples were preserved in distilled water at 37°C for 48 hours.

The ideal concentrations of $ZrO_2$ NP for this inquiry were determined by referencing results from prior research that employed weight percent combinations of 10% and 15%, as well as 3%, 5%, 7% and 5%. The impact strength exhibited a decline as the weight percent of $ZrO_2$ NP increased beyond 5%. The highest impact strength was observed with $ZrO_2$ NP concentrations of 3% and 5%. Based on their outcomes, we have decided to utilize concentrations of $ZrO_2$ NPs of 0%, 3% and 5%.

The electronic balance (Shimadzu, Germany) was utilized to weigh the $ZrO_2$ NPs and acrylic particles with a four-decimal-point accuracy, as per the specified percentages. To ensure precise and uniform dispersion of the $ZrO_2$ NP granules within the acrylic monomer, it was incorporated into the monomer via hand agitation followed by approximately ten minutes of ultrasonic vibration, combining the monomer and granules subsequently. Ten minutes were spent incorporating the ingredients until the mixture

assumed a dough-like consistency. At that moment, the mixture had progressed to the working stage, which is the stage of homogenous dough. Every specimen composed of HPA resin was manufactured following established acrylization protocols. The MCA specimens were subjected to microwave polymerization. This was accomplished by combining 100 mg/43 ml of powder and liquid, placing the mixture in the microwavable flask mould (FRP Flask. GC, Tokyo174-8585, Japan), inserting the corresponding bolts and ensuring consistent pressure was maintained with the included wrench. Following this, the specimens underwent a three-minute microwave polymerization process at 500 W in a Sanyo Microwave oven (EM M 553 T). For every specimen made of heat- and microwave-polymerized acrylic resin, NP was mixed with the monomer as suggested by prior investigations. Packing, curing and finishing are conducted based on BS EN ISO 20795-1:2008 guidelines and ISO 20795-1:2013 standards.

**Tensile Testing:** Specimens in the shape of conventional dumbbells with the following dimensions (75mm×12.75mm×2.5±0.03mm) have been fabricated to undergo tensile testing. These specimens adhere to the guidelines outlined in ADA Specification No. 12, 2002. The specimens were affixed to a Universal Testing Machine (GESTER, Fujian, China) using a vice and were aligned at the centre of the attachment of the testing unit. The objective of this alignment was to direct the load along the long axis of each specimen using a 20 mm/min chart and a 5 kN load cell. Using this apparatus, the tensile test was performed. Following this, tension loading was applied to the specimens until they fractured at a crosshead velocity of 0.5 mm/min. The measured feasibility load is in Newtons (N). The act of recording the load of failure in Newtons (N). The tensile strength (in MPa) was determined by dividing the load at failure (N) by the surface area ($mm^2$). The tensile strength value is subsequently calculated using the following formula:

$TS = F/A$

TS = tensile strength ($N/mm^2$)

F = force at failure (N)

A = area of a cross-section at failure ($mm^2$).

**Impact strength test:** Following the ISO 20795-1:2013 standards, the experiment was conducted using rectangular samples (60mm×7mm×4mm). A machine was used to conduct the test. The "Charpy Impact Tester (Tokyo Koki Seizosho, Japan)" contains 40-mm hole between two fixed supports. A v-shaped notch was created, with a depth of 0.8 mm and an effective depth of 3.2 mm below the notch. The notch is intended to induce a regional stress concentration, hence favouring a fragile fracture over a bendable fracture. Impact strength calculated based on the formula

Impact strength ($KJ/M^2$) = E/tw.

E = amount of energy absorbed in kilojoules (KJ),

t = thickness of the specimen

w = residual width at the notch base in square meters ($m^2$).

"**Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR)":** It is a widely utilised spectroscopic technique employed in the analysis of polymer structures. By utilising FTIR spectroscopy, changes in the chemical composition of biomaterials are applied with extreme sensitivity. A total of six samples were produced, one specimen for each group with a dimension of 10×4×4 ±0.02mm, consisting of heat polymerizing resin and MCA resin.

## 3. Results

The Shapiro-Wilk test, which was used to evaluate the distribution of the data, indicated that all variables had a normal distribution. The statistical analysis for all the data was conducted using the SPSS system (version 28).

The tensile strength for HPA resin and MCA denture base materials are presented in Figure 1. These materials were tested with the addition of $ZrO_2$ NPs at three different concentrations: 0%, 3% and 5%. The addition of 3% $ZrO_2$ NPs resulted in the HAP resin samples exhibiting the highest value of 71±1.50 N/mm$^2$, while the samples from the Acron MC-MCA control group had the lowest value of 63±2.073 N/mm$^2$.

The analysis of variance (ANOVA) carried out regarding the tensile strength of different groups of acrylic denture base materials (Table 1) demonstrated statistically significant variations. The HPA resin exhibited an F ratio of 11.146 and a P value of 0.002, while the Acron MC-MCA denture base materials showed an F ratio of 22.217 and a P value less than 0.001.

The Duncan's multiple range test (DMRT) was conducted on the tensile strength of Ivoclar and Major Acrylics. The results showed substantial changes between the group treated with 3% $ZrO_2$ NPs and the control groups treated with 0% and 5% $ZrO_2$ NPs, with a significance level of P≤0.05. The addition of 5% $ZrO_2$ increased tensile strength; however, it did not reach a statistically significant level compared to the control group. However, the DMRT analysis of Acron MC-MCA denture base materials revealed a notable difference in tensile strength between the control group and the other tested groups at 3%, 5% and $ZrO_2$ NPs (P value≤0.05) (Figure 1).

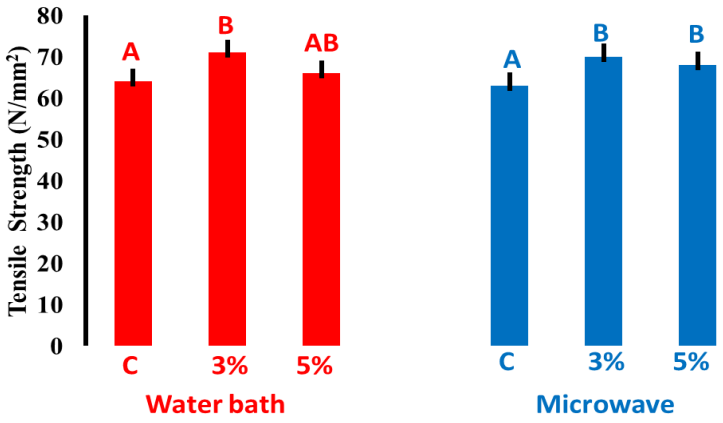


**Figure 1.** Tensile strength MPa (N/mm$^2$) for (Ivoclar, Major) HPA resin and Acron MC-MCA denture base materials after adding $ZrO_2$ NPs (c (control), 3%, 5%). Data expressed as mean±SD, the different letters mean significant at (P value ≤0.05) according to DMRT.
DF: Degree of Freedom, *significant difference at P≤ 0.05

**Table 1.** ANOVA of the tensile strength (N/mm$^2$) for (Ivoclar, Major) and Acron MC acrylic denture base materials after adding $ZrO_2$ NPs (0%, 3%, 5%)

| | | Sum of Squares | df | Mean Square | F | P- value |
|---|---|---|---|---|---|---|
| W.B | Between Groups | 142.300 | 2 | 71.150 | 11.146 | 0.002* |
| | Within Groups | 76.600 | 12 | 6.383 | | |
| | Total | 218.900 | 14 | | | |
| M.W | Between Groups | 119.233 | 2 | 59.617 | 22.217 | .000* |
| | Within Groups | 32.200 | 12 | 2.683 | | |
| | Total | 151.433 | 14 | | | |

Figure 2 displays the impact strength for the fundamental materials of HPA resin and MCA denture base materials after including $ZrO_2$ NP (0%, 3%, 5%). The HPA resin samples comprising $ZrO_2$ Np at a concentration of 3% had a higher value of 29.4±3.781. In contrast, the samples in the control group, which consisted of Acron MCA, had the lowest value of 22±1.224.

The impact strength of HPA resin indicated a substantial variation among all the tested groups, as evidenced by the F ratio of 3.335 and a corresponding P value of 0.040. There was a notable difference seen in the tested groups of Acron MCA denture base materials following the addition of $ZrO_2$ NPs (0%, 3% and 5%). The F ratio of 9.905 and a P value of 0.003*, which is less than the threshold of 0.05, indicate that this difference was statistically significant (Table 2).

DMRT analysis revealed substantial differences in the impact strength of heat-cured acrylic when $ZrO_2$ NPs were introduced at concentrations of 0%, 3% and 5%. Specifically, the impact strength at 3% $ZrO_2$ NPs was significantly different from the impact strength seen in the other two groups (0% and 5%) at a significance level of P≤0.05. However, the addition of 5% $ZrO_2$ NPs to the Acron denture base material resulted in a rise in impact strength to 23.6 KJ/m$^2$. Nevertheless, this increase did not demonstrate any statistically significant distinctions compared to the other groups.

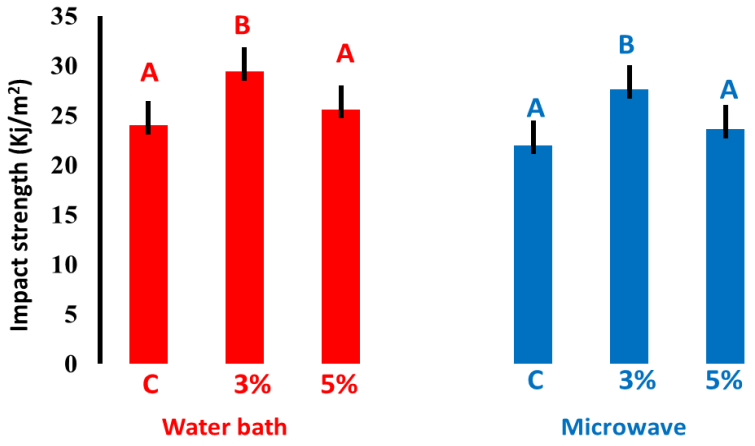


**Figure 2.** Impact strength MPa (N/mm$^2$) for HPA resin (Ivoclar, Major) and MCA denture base (Acron MC) materials after adding $ZrO_2$ NPs (c (control), 3%, 5%). Data expressed as mean±SD, the different letters mean significant at (P value ≤0.05) according to DMRT

**Table 2.** Impact strength (KJ/m$^2$) for (Ivoclar, Major) and Acron MC acrylic denture base materials after adding $ZrO_2$ NPs (0%, 3%, 5%)

| | | Sum of Squares | Df | Mean Square | F | P value |
|---|---|---|---|---|---|---|
| W.B | Between Groups | 76.933 | 2 | 38.467 | 3.335 | 0.070 |
| | Within Groups | 138.400 | 12 | 11.533 | | |
| | Total | 215.333 | 14 | | | |
| M.W | Between Groups | 83.200 | 2 | 41.600 | 9.905 | 0.003* |
| | Within Groups | 50.400 | 12 | 4.200 | | |
| | Total | 133.600 | 14 | | | |
| DF: Degree of Freedom, *significant difference at P≤ 0.05 using One-Way ANOVA | | | | | | |

Table 3 displays the results of the independent T-test conducted to compare the tensile strength and impact strength between two groups: HPA resin and MCA denture base materials, after the inclusion of $ZrO_2$ NPs at different percentages (0%, 3% and 5%). The evaluation revealed that there were no statistically significant differences observed among all groups at a threshold for significance of $p \leq 0.05$ after the addition of varying percentages of $ZrO_2$ NPs.

**Table 3.** Tensile strength ($N/mm^2$) and impact strength ($KJ/m^2$) between (Ivoclar, Major) and Acron MC acrylic denture base materials after adding $ZrO_2$ NPs (0%, 3%, 5%)

| | | | N | Mean | Std. Deviation | F | Sig |
|---|---|---|---|---|---|---|---|
| Tensile strength (MPa) | Control | Heat cured | 5 | 64.200 | 3.114 | 0.373 | 0.558 |
| | | Microwave cured | 5 | 63.400 | 2.073 | | |
| | 3% | Heat cured | 5 | 71.400 | 1.516 | 0.022 | 0.885 |
| | | Microwave cured | 5 | 70.000 | 1.581 | | |
| | 5% | Heat cured | 5 | 66.200 | 2.683 | 5.550 | 0.064 |
| | | Microwave cured | 5 | 68.200 | 1.303 | | |
| | | | N | Mean | Std. Deviation | F | Sig |
| Impact strength (MPa) | Control | Heat cured | 5 | 24.000 | 2.449 | 0.914 | 0.367 |
| | | Microwave cured | 5 | 22.000 | 1.224 | | |
| | 3% | Heat cured | 5 | 29.400 | 3.781 | 0.038 | 0.851 |
| | | Microwave cured | 5 | 27.600 | 3.209 | | |
| | 5% | Heat cured | 5 | 25.600 | 3.781 | 3.843 | 0.894 |
| | | Microwave cure | 5 | 23.600 | 0.894 | | |

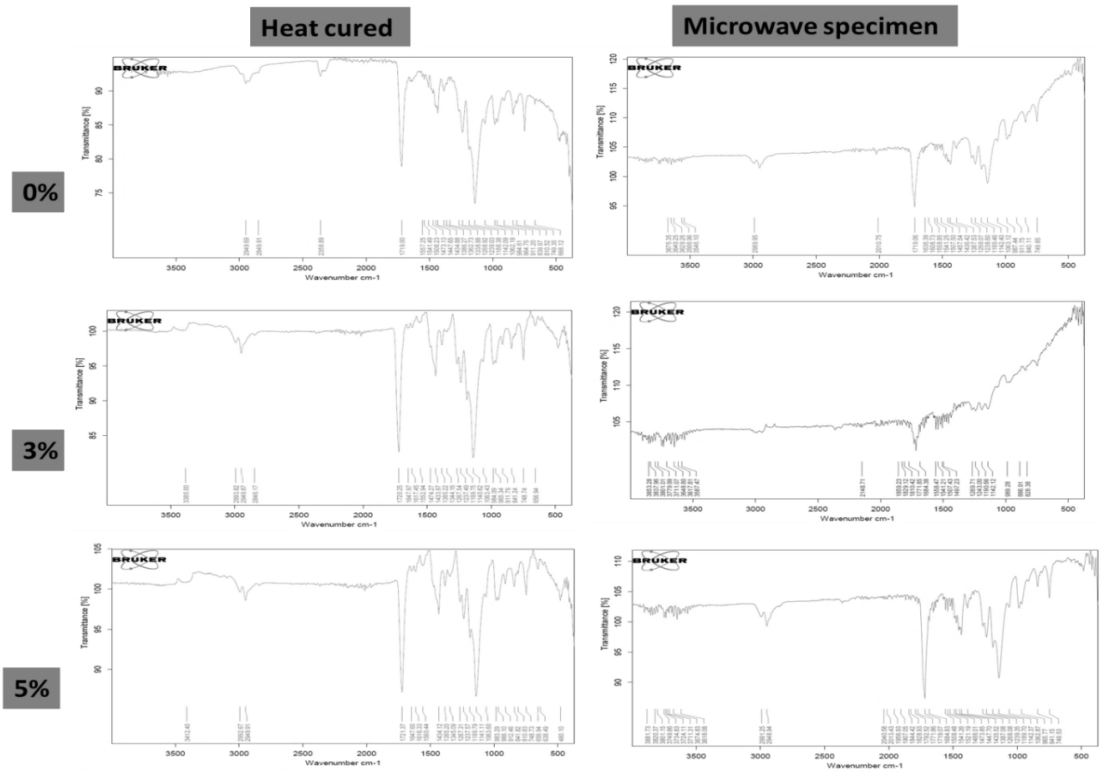


**Figure 3.** ATR-FTIR ATR-FTIR spectra: 0% control, 3% $ZrO_2$ and 5% $ZrO_2$ in heat-cured and MCA specimens

Figure 3 depicts the ATR-FTIR charts of the heat and MC MCA, showing that the addition of 3% or 5% $ZrO_2$ NPs did not cause significant structural chemistry alterations. However, no chemical reaction with $ZrO_2$ NPs since no new peaks were produced, indicating that no new materials had been generated.

## 4. Discussion

PMMA/composite polymers were synthesised by including fibres, fillers and nano-fillers. One of the most extensively researched additions for reinforcing PMMA is $ZrO_2$ NPS (Gad *et al*., 2016; 2017). Fan et al. (2013) investigated the dispersion of $ZrO_2$ NPs inside a PMMA matrix using two mixing strategies: If $ZrO_2$ NPs were introduced to the monomer, it would be more beneficial than adding them to the powder using physical and chemical means. Melt mixing, ultrasonic vibration or high-energy ball milling are examples of physical procedure used (Fan *et al*., 2013). The combination of NPs with an MMA monomer was used, in this process, the inorganic $ZrO_2$ NPs represents the core, while the shell was the monomer leading to grafting (polymerization). $ZrO_2$ NPs endures surface chemical alteration by the monomer providing dispersion of $ZrO_2$ NPs in the polymer lattice uniformly perhaps preventing. Particle aggregation and reducing the chance for phase separation (Aboshama *et al*., 2022; Zidan *et al*., 2019).

**Tensile Testing:** The findings of the present study confirmed that the addition of $ZrO_2$ NPs have increased the tensile strength of PMMA for the types of acrylic material used, these results were harmonized with previous studies suggesting that these NPs is directly interrelated with enhancement in flexural, impact and tensile strength (Gad *et al*., 2016; 2018a) perhaps this could be potentially linked to proper dispersion of $ZrO_2$ NPs fillers inside the texture body of the matrix body probably due to filling the gaps inside the latex body by the nano-sized dispersed particles providing additional beneficial effects on the strength of the latex body, thereby enhancing tensile strength and minimizing porosity, minimizing water resorption (Ajay *et al*., 2019). Nonetheless, the capacity of zirconia to assemble as monoclinic phase which provide the latex with a shape helping to dissipate energy and thereby invoke energy absorption in cracks tackling their stress on the latex of the system (Gad *et al*., 2018a; 2016; Ilyas *et al*., 2022).

**Impact strength test:** These aforementioned impacts enhance mechanical characteristics promoting fracture deflection and crack suppression. Moreover, proper dispersion has been acknowledged by their provided increased surface area improving mechanical interlocking and improving flexibility of the nanocomposites.

Previous studies has tested different concentration of zirconia, Hameed and Rahman (2015) used 5% nano-$ZrO_2$ in the PMMA resin enhancing strength and improving the physicochemical properties (Asar *et al*., 2013; Gad *et al*., 2018a; 2016). Moreover, a greater amount incorporation of zirconia provide reciprocal increase in strength (Al-Flayeh & Al-Noori, 2023) with no further improvement beyond 7% (Gad *et al*., 2016). The impact strength seen in our current investigation aligns with the findings of prior studies, except the results obtained for the 5 wt% $ZrO_2$/PMMA sample (Asar *et al*., 2013; Gad *et al*., 2018a; Hameed & Rahman, 2015), where a concentration of 5 wt% is believed to be the optimal amount for enhancing particle dispersion evenly and minimising amalgamation, agglomerations and cluster formations, which ultimately weaken the material instead of strengthening it (Asopa *et al*., 2015; Gad *et al*., 2017).

The findings of our study confirmed that there were non-significant differences for the studied materials at each concentration of $ZrO_2$ NPs (0%, 3%, 5%), except the MCA

tensile strength greater at 5% $ZrO_2$ NPs than the hot-cured acrylic This finding is consistent with the research conducted by Al-Flayeh and Al-Noori, (2023) and Spartalis et al. (2015), with an alternate viewpoint to Rasan and Farhan (2023), who demonstrated that a mechanical features of MCA materials probably linked proportionally to residual monomer when compared with HPA.

$ZrO_2$ NPs have been shown to strengthen denture bases; yet, $ZrO_2$ concentrations are not proportional to advancements in tensile or impact strength. 3% of groups yield the best outcomes. Since high filler fractions might potentially result in more flaws and make the material more vulnerable to damage, the greater surface area of the fillers and stress concentration are two of the many hypothesised causes of the crack percentage. The act of aggregation that causes the deterioration of a substance (Alshaikh *et al*., 2022). Moreover, the interface region is affected by the higher concentration of nano-powder, resulting in a decrease in impact strength due to a reduction in energy dissipation per unit volume (Hamdy, 2024; Srivastava *et al*., 2023). The positive impact of increasing filler content would thereafter be reduced, possibly due to the occurrence of a break in the resin matrix's continuity once saturation is reached. This break leads to a drop in the strength of the reinforced specimens. The results are consistent with the findings of previous studies (Al-Flayeh & Al-Noori, 2023; Al-Hiloh & Ismail, 2016; Asar *et al*., 2013; Spartalis *et al*., 2015).

**ATR-FTIR Analysis:** The absence of any structural chemical changes in the resin is confirmed by the ATR-FTIR when 3% or 5% $ZrO_2$ NPs were employed, in contrast to the control groups (0% $ZrO_2$ NPs) of the acrylic resin cured by heat or microwave. The FTIR charts illustrate that the spectral peaks of the control and tested groups are comparable. The lack of newly generated peaks serves as confirmation that no novel substances are being formed and that there was no chemical reaction involving $ZrO_2$ NPs (Figure 4). The absence of chemical changes can be attributed to no chemical reaction between saturated acrylic resins and saturated $ZrO_2$ NPs, as well as the absence of functional groups in both compounds, as determined by the molecular interaction at the level of ATR-FTIR analysis (Kumar *et al*., 2019).

## 5. Conclusions

Improving the efficiency of acrylic resin can be accomplished by fortifying it with $ZrO_2$ NPs. This study convincingly shows that the addition of $ZrO_2$ NPs, at concentrations of 3% and 5%, improves the tensile strength and impact strength of PMMA, with no changes in the chemical structures detected in both MCA and hot-cure acrylic denture base materials. Significantly, the most remarkable enhancement was noticed at a concentration of 3% $ZrO_2$ NPs, where the utmost values were recorded for both tensile and impact strength.

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