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# Evaluation of Color Change, Porosity, and FTIR on Modified Heat Cured Acrylic Resin by Zinc Oxide Nanoparticles and CAD/CAM Denture Base Materials

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**Abstract**

**Aims**: the present study aimed to analyze the effects of the incorporation of Zinc Oxide nano-fillers (ZnO NPs) with size (10-30 nm) at one concentration (1%) on color change and porosity and FTIR compared to milled CAD/CAM and conventional denture base materials. **Materials and methods:** the number of samples were 53 which divided into twenty samples for color change test (control, 1 % ZnO NPs), thirty samples for porosity test and three specimens for FTIR test for (control, 1 % ZnO NPs,CAD/CAM ) . **Results**: the results demonstrated that there was a significant change in color, with comparable FTIR spectra for the PMMA-ZnO nanocomposite at ZnO nanoparticles (1%), when compared to control and non-significant reduction to porosity test. CAD/CAM denture base showed significant low mean value as compared between control and (1%) zinc oxide group and also showed similar with non-significant difference spectra on FTIR test as compared to the control group. **Conclusions**: there is unacceptable color change in addition that use of ZnO nanoparticles as dental fillers at 1% by weight of PMMA denture base material and no chemical interaction between zno nanoparticles and CAD/CAM PMMA denture base which has superiority in porosity test.

**الخلاصة**

**الأهداف**: تهدف الدراسة الى دراسة تأثير دمج جزيئات الزنك أوكسيد النانوية بحجم (10-30 نانومتر) بتركيز( ١٪) على خاصية تغيير اللون وأختبار خاصيه المسامية وأيضا دراسة المكونات الكيميائيه عن طريق (FTIR) لراتنج الأكريليك المعالج بالحرارة ومقارنته بمادة اطقم الاسنان المصنع عن طريق انظمة الكمبيوتر (CAD/CAM) . **المواد و طرائق العمل**: كان العدد الإجمالي للعينات ثلاث وخمسون مقسمة إلى عشرون عينة ل فحص خاصيه تغيير اللون لكل مجموعة عشرة (مجموعة السيطرة ، ١٪ الزنك أوكسيد النانوي ) وثلاثون عينة للمجموعات الثلاث لفحص خاصية المسامية و ثلاث عينات ل فحص (FTIR) مقسمة عينة لكل مجموعة (مجموعة السيطرة ، ١٪ الزنك أوكسيد النانوي ،CAD/CAM) . **النتائج**: أظهرت النتائج أن مجموعة (1% ZnO) تغيير لون ملحوظ عند مقارنته بمجموعة السيطرة و ايضاً ليس هنالك اي تغيير كيميائي لمركب النانوي المتولد في جزيئات الزنك أوكسيد النانوي (١٪) و ايضا لا تغيير كيميائي في مجموعة (CAD/CAM) ، عند مقارنتها بمجموعة السيطرة , مجموعة (CAD/CAM) اقل معدل قيم لفحص خاصية المسامية عند مقارنتها بمجموعتا (مجموعة السيطرة ، ١٪ الزنك أوكسيد النانوي ). **الاستنتاجات** :عند إضافة الزنك أوكسيد بتركيز (١٪) إلى البولي ميثيل ميثاكريليت المعالج بالحرارة له تأثير سلبي على خاصيه اللون وتاثير غير ملحوظ في خاصيه المسامية وايضالايوجد اي تعارض كيميائي عند اضافه المادة النانوية ولا يوجد اي تغيير كيميائي في مادة اطقم الأسنان المصنعة عن طريق أنظمة الكمبيوتر (CAD/CAM) .

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## INTRODUCTION

Most people are really concerned about tooth loss. The continuance of daily life depends on their replacement with artificial substitutes, such as dentures made of acrylic resin. Multiple treatment procedures are available to restore lost teeth, regardless of the underlying cause [(1)]. Nowadays, removable partial dentures and complete denture bases are made of poly methyl methacrylate (PMMA), a polymer that was initially introduced in 1937. It is still utilized because it is simple to manipulate, fits properly, dimensionally stability, and aesthetic [(2)].

However, a few significant drawbacks are found in this resin, such as poor strength, especially under fatigue failure inside the mouth, allergy to residual monomer , abrasion resistance was low , and brittleness on impact. These problems frequently lead to denture fractures [(3)].

Efforts to strengthen denture bases to decrease the risk of denture fracture can be characterized as; reinforcement of denture -base by adding filling materials, changing the chemistry of the denture base polymer by co-polymerization or cross-linking of resin materials, or fabricating new materials that are more fracture resistant [(4)].

Incorporating various nanoparticles into PMMA to act as the reinforcing material is one of the most important approaches recently used to overcome these problems [(5)]. To deal with these drawbacks and improve the resin's mechanical properties, the composition includes a variety of fillers, metal oxides, and carbon graphite fibers [(5)].

Nano-fillers zinc oxide (ZnO) is highly effective against both bacteria and fungi. ZnO may considerably enhance the biological properties of acrylic resins and the properties of denture base resins. [(6)]

Since the 1980s, computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies have been used in around prosthodontics to overcome some of these drawbacks. [(7)] CAD/CAM technology was presented to provide a denture base acrylic resin manufactured by machining. As a result, polymerization shrinkage of the resin is minimized, and the fit of the denture base is improved over that produced conventionally. [(8)]

This research aimed to study the influence of zinc oxide nanoparticles addition at concentration (1%) and compare to conventional heat cured acrylic resin on color change and compare to pre-polymerized milled CAD/CAM and conventional heat cured denture base materials on FTIR test and porosity test.

## MATERIALS AND METHODS

**Sampling:** the number of specimens were 53 which divided into twenty specimens for color change test and sub grouped into 10 for (control, 1 % ZnO NPs), and one specimen for each group (control, 1 % ZnO NPs,CAD/CAM) for FTIR test, also 30 specimens for porosity test, 10 specimens for each group (control, 1 % ZnO NPs,CAD/CAM). The study was done at the college of dentistry and technical institute at the University of Mosul. Approval of the study was from the Scientific Research Committee / Department of Prosthodontics / College of Dentistry (UoM. Dent/DM.L.39/22).

**Preparation of the samples**

During the mold preparation, a conventional flasking procedure was operated for complete dentures. Separating medium (Alginic isolator, Zhermack®) was employed and let to dry for the layer of plastic before putting the lower part of metal flasks filled with die stone (Elite® stone, Zhermack®) and combined in vibration according to the directions of the manufacturer to remove the trapped air, then left to set. Acrylic sheets were used to make the plastic model, assembled using computer software (AutoCAD), and then cut with a computer-controlled laser cutting machine. The length, width, and thickness of the plastic models utilized in mold fabrication were precisely designated according to the specifications needed for each test. Samples of all groups were then stored in distilled water at 37ºC for two days using an incubator. [9]

The specimens were first prepared by mixing the weight of zinc oxide nano powder with heat-cured PMMA" monomer, which was sonicated and dispersed in the monomer for 3 minutes using an ultrasonic of 20W and 60 kHz, and then the Heat-cured PMMA polymer powder was added and manually mixed to avoid particle agglomeration [10].

The CAD/CAM specimens; a 3D specimens design with dimensions were virtually designed using SketchUp Pro (2020) were saved to as standard tessellation language (STL) file form. Then, STL files were imported to CAD software (Exocad Dental DB) which eventually connected to the milling machine to be used for fabricating the milled samples from (Ivotion CAD Base) pre-polymerized PMMA blocks (SPEC 98.5x30 shade Pink Monolayer) with CAD/CAM milling machine (MAXX DS 200-5Z, Korea) with milling PMMA burs (2.5, 1, 0.5 mm) respectively; the number of the axis was five-axis with dry milling subtractive technique. [11]

**Color Change test**

The specimens with a constant size of 10 × 2 ±0.02 mm (Diameter, thickness), as presented in the figure (1), were formed according to. [12]

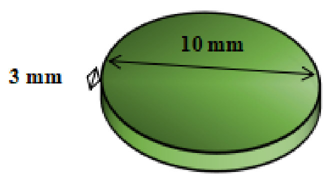


Figure (1): Dimensions of color change testing specimen

Specimens were tested using a colorimeter testing device. The mechanism of the colorimeter is to flash the device's light into the specimen to be examined through a small rounded aperture, which was secured to the clear white, shining surface board to achieve correct data, as shown in figure (2).

This approach is based on three color-definition parameters: L*, a*, and b *. Where L* denotes lightness-darkness; so, the higher the L*, the lighter the specimen. The a* denotes the chroma along the red-green axis; positive a* reflects the amount of redness, while negative a* shows the amount of green in the specimen. While b* on the chromatic scale measures chroma along the yellow-blue axis, where positive b* refers to the degree of yellowness and negative b* refers to the amount of blue of the specimen. The color changes (ΔE) of the specimens were assessed using the equation: [13]

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

All the data required, including L, a, b, ΔL, Δa, Δb and ΔE was displayed on the device screen; nevertheless, if no color change is noticed after testing, the ΔE ratio will be zero. In vitro clinically acceptable research had an ΔE ratio was (3.7) or less, while in vivo clinically acceptable research had an ΔE ratio of (6.8) or less [14]


Figure (2): Colorimeter portable device for color change measurement

**Fourier Transform Infrared Spectroscopy Test (FTIR)**

The FTIR test was performed to determine if a chemical reaction or change occurred following the addition of the additives employed in this study (Zinc oxide) [15]

Three specimens, one for each group, with dimensions of 10*4*4 ±0.03mm (length, breadth, and thickness, respectively) using a CL Alpha-P FTIR spectrophotometer with a resolution of 2 $cm^{-1}$ and the wavenumber region 400–4000 $cm^{-1}$ (figure 3), specimens were made and conditioned for 48 hours in distilled water at 37°C ± 1°C. [16]

After conditioning, the samples were removed from the water, dried in the fresh air, and stored in freshly dried silica gel until they were completely dehydrated. The investigation was achieved by combining the ground powder of the specimens with potassium bromide salt and compressing it under pressure to form a pellet that was then examined by FTIR spectroscopy. [17]

**Porosity test**

Thirty specimens were prepared, ten for each group, with the following dimension 10×12×4 ±0.2mm in length, width, and height, respectively as shown in figure (3).[18]

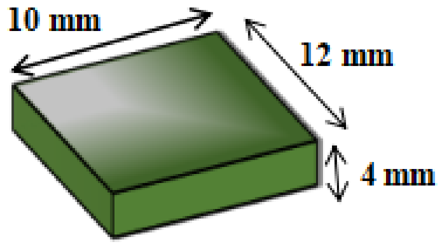


**Figure (3)**: Dimensions of porosity test specimen

After specimens' preparation, the specimens were dried in a desiccator that contains freshly dried silica gel at 37°C. An electronic balance device was used to weight the specimens (accuracy of 0.0001 gm) daily until a constant weight was achieved ; two weights were taken; one with samples in the air and one with samples immediately immersed in distilled water; The samples were then stored in distilled water at 37°C. Weighed at regular intervals until a constant mass was reached, indicating a state of water saturation for a period of 30 days. The specimens pulled from the water and excess water removed by blotting with filter paper, and again, the samples were weighed, one in air and the other with the samples immediately immersed in distilled water. The porosity calculations made using the following equations [19]

Vd=(md-md" )/ρw ….. (1)
Vs=(ms-ms")/ ρw ….. (2)
% porosity =[(Vs-Vd)×100]/Vd ….. (3)
Where:

Vd= Volume of dried specimen
md: Mass of dried specimen in air.
md": Mass of dried specimen in water.
ρw: Density of water (1000Kg/m3 ).
Vs: Volume of the specimen with water.
ms: Mass of saturated specimen in air.
ms": Mass of saturated specimen in water.

## RESULTS

**Color Change Test (ΔE)**

Color change (ΔE) is considered accepted in vitro if ΔE is equal to (3.7) or less. As ΔE equals zero, it means there was no color change at all. From the data collected (L*, a*, and b*) was analyzed using the colorimeter device for measuring the color change of the (1% ZnO) group, the mean values of ΔE were (4.53) and the result in N.B.S unit were (4.16), in vitro research which was not acceptable, according to the standard in vitro color change value ($\Delta E \leq 3.7$)

Table (1): Color change according to (CIE L* a* b*) color system.

| Experimental group | ΔE | N.B.S unit | Acceptance in vitro |
|---|---|---|---|
| **1% (ZnO)** | 4.53 | 4.16 | Not Accepted |

**Fourier Transform Infrared Spectroscopy Test (FTIR )**

FTIR spectrum for control heat cured PMMA, figure (4) showed absorption bands at 2992 cm-1 related to aliphatic (ν –CH3) group and at 2949 cm-1 related to aliphatic (ν –CH2) group; strong absorption bands at 1719 cm-1 for (ν C=O) group and at 1140 cm-1 for (ν C-O) in addition to absorption band at 1434 cm-1 related to (δ -CH2) group.

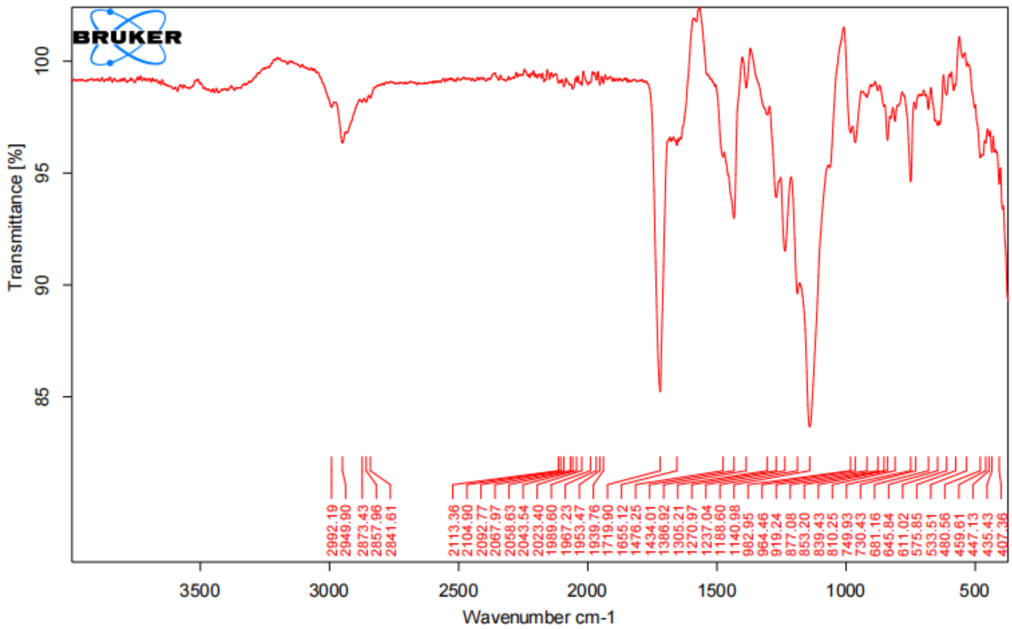


**Figure (4)**: FTIR spectrum of control group

Compared to the spectrum of the control group ,the FTIR spectrum for 1% ZnO nanoparticles Figure (5) reveals comparable FTIR spectral peaks Figure (4). The lack of new peaks suggests that adding zinc oxide nanoparticles at 1% did not stimulate the creation of new products in the PMMA matrix since there were no chemical interactions between the saturated heat cured PMMA and the saturated ZnO nanoparticles. However, a composite was created using nano-sized additives as fillers in a polymer matrix, and this composite may result in a change in physical and mechanical qualities.

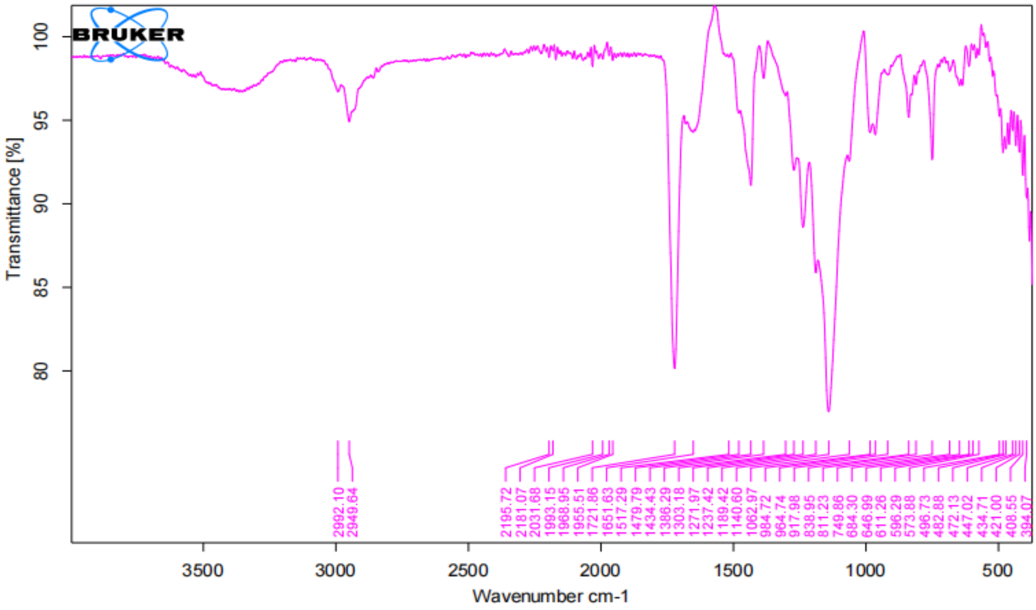


**Figure (5):** FTIR spectrum of experimental group (1%) ZnO.

The FTIR spectrum for CAD/CAM group figure (6) reveal comparable FTIR spectral peaks of control at Figure (4). reveals absorption bands at 2989 cm-1 related to aliphatic (ν –CH3) group and at 2948 cm-1 related to aliphatic (ν –CH2) group; strong absorption bands at 1719 cm-1 for (ν C=O) group and at 1140 cm-1 for (ν C-O) in addition to absorption band at 1447 cm-1 related to (δ -CH2) group.

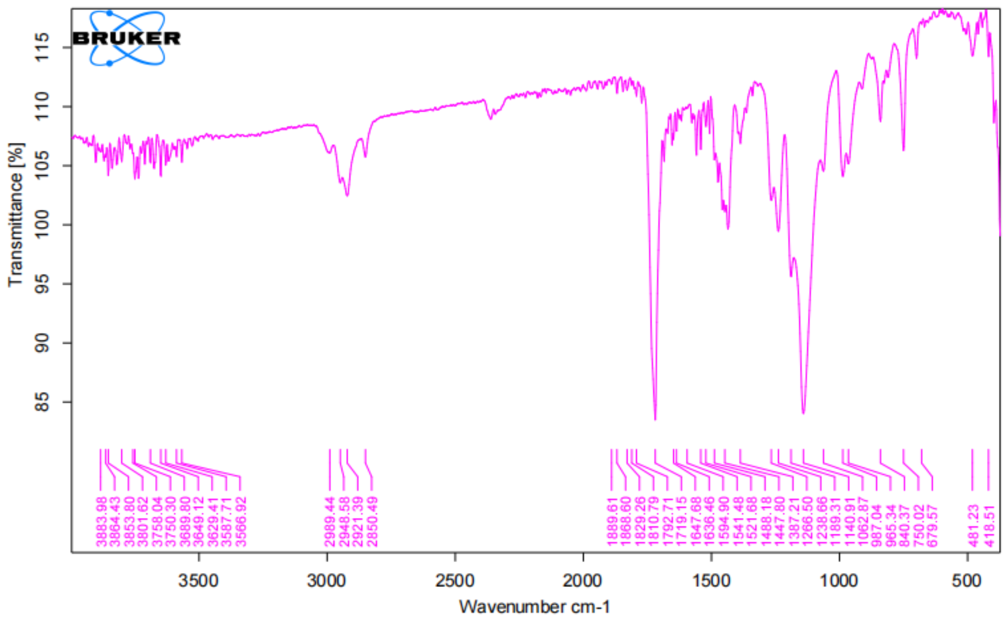


**Figure (6):** FTIR spectrum of experimental group CAD/CAM.

**Porosity Test**

Using the SPSS software version 26, the statistical analysis was performed using inference statistics analysis of variance (ANOVA) and Duncan multiple range tests.

The values of mean and standard deviation for porosity Test of the (control) and experimental groups (1% ZnO) and (CAD/CAM) are shown in figure (8).

The results represented decrease in the Porosity of the experimental group as compared to the control group. The lower percentage of Porosity (0.762 %) was recorded with the (CAD/CAM) .

One-way analysis of variance (ANOVA) was employed to assess the porosity Test data of the control and (1%) Zinc Oxide nanoparticles and CAD/CAM at ($P \leq 0.05$) in table (2), showed significant difference between groups.

**Table (2):** One-way analysis of variance for porosity test of control and experimental groups of (1%)ZnO nanoparticles and CAD/CAM group.

| SOV | SS | DF | MS | F-value | P-value |
|---|---|---|---|---|---|
| **Between Groups** | 0.292 | 2 | 0.146 | 3.762 | 0.036 |
| **Within Groups** | 1.048 | 27 | 0.039 | | |
| **Total** | 1.34 | 29 | | | |

Duncan's multiple range test was illustrated in figure (7) and exhibited that the Porosity was statistically non-significant reduction at (1%) ZnO nanoparticle and significantly reduction in The CAD/CAM group, as compared to the control group, which also had a significant difference in porosity found between experimental groups

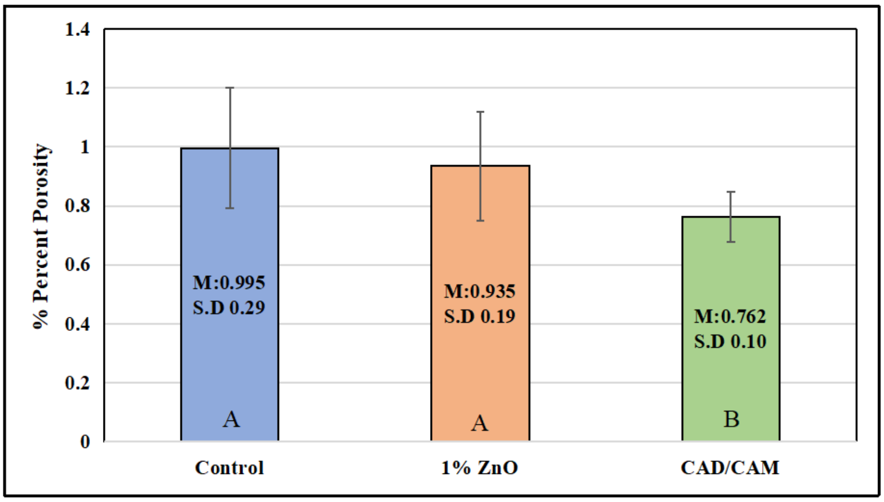


**Figure (7):** Duncan multiple comparison test, Mean, standard deviation for porosity of control and experimental groups of (1%) ZnO nanoparticles and CAD/CAM group

## DISCUSSION

### Color Change Test

The color of denture base materials may change over time for various reasons, such as water absorption, stain accumulation, deterioration of intrinsic pigments, ingredients, food, and beverage dissolution, as well as surface roughness [20].

The type, size, shape, and concentration of the additional fillers affect the optical characteristics of PMMA [21].

The results of the color test which illustrated that the change in the L*a*b* color system of the experimental group of (1%) zinc oxide NPs when compared to that of the control group according to (CIE L*a*b*) that color change (ΔE) values are not acceptable clinically since the values ΔE of the experimental group were less than 3.7 [14].

It was agreed with Kamonkhantikul et al. (2017) [22], who have also shown that incorporating ZnO NPs in acrylic resin PMMA had a negative whitening effect on the optical characteristic of acrylic resin PMMA. Associated with variations in color and form of nanoparticles, denture base resin materials, filler types, and polymerization methods. A composite resin's color may also be affected by its nano-filler content and size as well as additional chemical additives. [21]

Moreover, the result in agreed with the study which showed that the increase in light absorption is significant as a result of light absorption with the increasing of modified nanoparticles concentration [23]. This is obviously because of the presence of nano-ZnO powder in the matrix which is opaque and absorbs more light energy than polymer matrix and appears opaquer. This finding are attributed to the high atomic number of nano-filler (ZnO) compared to the low atomic number of the chemical constituent of acrylic which is dependent on the cube of its atomic number [24].

Additionally, a study That was compatible with the study result by Rudolf et al. (2020) [25] clearly noted that the combination of ZnO NPs and PMMA generated a brighter color when the percentage of nanoparticles was increased. An appropriate pigment might be applied to the resin used to make denture bases and teeth to achieve a color similar to that of the oral mucosa or teeth.

### Fourier Transform Infrared Spectroscopy Test (FTIR)

It is a widely used and well-established spectroscopic method for investigating and characterizing the structure of polymers. FTIR spectroscopy offers a sensitive analysis tool to detect composition changes in biomaterials [26].

FTIR chart of control and 1% zinc oxide nanoparticles groups, figures (4-6 ) show that no chemical interaction happened between them and that just a composite was established for zinc oxide addition. Although the experimental group's FTIR spectra showed the same FTIR absorption bands as the control group and revealed no change in the PMMA spectral range after adding 1% zinc oxide nanoparticles,and

CAD/CAM FTIR sprctra showed the same FTIR absorption bands as the control group with non-significant difference, the data show that only physical and mechanical alterations occurred .

The absorbance band of nanoparticles in heat-cured acrylic resin did not emerge in the FTIR spectroscopy chart because its wavelength is between (200-400 cm-1) which is beyond the range of FTIR spectroscopy (400-4000 cm-1) [(27)].

The result of the zinc oxide group was consistent with Jawad et al. (2016) [(28)] concluded that the incorporation of ordinary ZnO nano-fillers or thermally activated ZnO nano-powders appears to be just incorporating filler without chemical interaction with the resin matrix.

**Porosity Test**

A porous denture is vulnerable to staining, calculus deposition, and adherent substances. So for a denture to be hygienically satisfactory, it should be nonporous. The porous denture is an ideal incubator for species such as Candida albicans. [(29)]

In this study, the results of the milled CAD/CAM samples demonstrated the statistically significant low mean value of porosity percentage followed by Modified heat-polymerized with zinc oxide nanoparticles samples, and conventional heat-polymerized denture base samples showed the higher mean value of porosity, as shown in figure (8). The superiority by low porosity percentage, with the significant difference due to the difference in the processing technique to CAD/CAM group as compared to the control group. The reinforced heat-cured PMMA with (1%) zinc oxide NPs group showed a non-significant decrease in porosity as compared to control groups. However, when comparing the results between experimental groups, Milling CAD/CAM group has a low mean value of porosity percentage than (1%) Zinc oxide NPs.

The results of the study's porosity test of the CAD/CAM group agree with Al-fouzan *et al*. (2017)[(30)], who reported that the milling of prepolymerized acrylic resin decreased the porosity compared to a conventionally processed denture. Consequently, there was a decrease in the retention of Candida albicans on the denture base.

The result of the CAD/CAM group may be attributed to a reduction of internal voids and defects during the polymerization of acrylic resin. During the polymerization of acrylic resin, porosity, and voids can develop between polymeric chains. The use of high-pressure during polymerization improves the conversion of monomer to polymer and increases crosslinks between polymer chains. Furthermore, reducing porosity and voids in PMMA may decrease water sorption [( 31)].

The result of this (1%) Zinc oxide NPs showed that non-significant reduction of porosity, and the mean values of the examined porosity were less than that of the control group. There was a slight reduction in acrylic resin's porosity after adding the

ZnO nano-filler. This study agrees with Nafea *et al*. (2016) [32] found that adding ZnO powder to heat-cured acrylic resins slightly affect their physical properties.

The causes could be due to the existence of such fillers within acrylic resin, increasing its density. It was found that there was a negative relationship between the density and porosity, where an increase in the density results in a decrease in porosity and vice versa. [33]

## CONCLUSION

CAD/CAM showed superiority in porosity test and no chemical difference as compared to conventional heat cured PMMA, in addition, that use of ZnO nanoparticles as dental fillers at 1% by weight significant change in color with a slight non-significant reduction in porosity and no chemical interactions with resin matrix of PMMA denture base material.

**Declaration of interest**

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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