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Effect of Addition Of Zirconium Oxide Nanoparticles on Flexural Strength and Porosity of Heat Cure Acrylic Resin

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Abstract

Heat cure denture base is the most commonly used material for fabrication of removable prosthesis to the present day. However difficulties persist in fabrication of satisfactory prosthesis due to poor mechanical properties which have resulted in frequent repairs in dental practice. The present study is aimed to investigate the effect of Zirconium oxide nanoparticles (ZrO_2NPs) on flexural strength and porosity of denture base and its correlation. X-ray diffraction (XRD) was used to check the purity of NPs. NPs was dispersed at 1%, 3% and 5% by weight to the monomer of methyl methacrylate with aid of probe sonicator. In addition, Scanning Electron Microscope (SEM) was used to observe agglomeration of particles within the acrylic. The results revealed significant flexural strength difference ($p<0.05$) between each concentration of ZrO_2NPs. The analysis showed 17% and 11% reduction for 1% and 3% ZrO_2NPs respectively while 5% caused a drastic reduction by 32% in reference to control. In regards to porosity, the results present no statistically significant difference among the concentrations in contrast to control. Pearson correlation showed strong and a negative relation (-0.83) between flexural strength and porosity. However, the results was not statistically significant ($p=0.369$). Within the limitations of this study, it can be concluded that the addition of ZrO_2 caused reduction in flexural strength for all

concentrations added. While it caused non-significant effect on porosity of acrylic. Considering the negative effect it had on mechanical strength it won't be considered a suitable additive to enhance the properties of PMMA.

Keywords: Zirconium oxide nanoparticle, flexural strength, porosity, planimeter.

تأثير إضافة جزيئات أكسيد الزركونيوم النانوية على قوة الانضغاط ومسامية المعالجة الحرارية للأكريليك

الخلاصة

تعتبر مادة الأكريليك هي المادة الأكثر استخدامًا لتصنيع الأسنان حتى يومنا هذا٠ تهدف هذه الدراسة إلى تأثير جزيئات أكسيد الزركونيوم على قوة الانثناء ومسامية٠ تم استخدام XRD للتحقق من نقاء جسيمات أكسيد الزركونيوم النانوية قبل استخدامها في الدراسة٠ تم اضافه الجسيمات النانوية ب ١٪ ، ٣٪ و ٥٪ بالوزن إلى مونومر الميثيل ميثاكريلات مع مساعدة Sonicator ٠ اتم إعداد أربعين عينة٠ بالإضافة إلى ذلك ، تم إجرا فحص SEM لمراقبة تكتل الجزيئات داخل الأكريليك٠أظهرت النتائج اختلاف كبير في قوة الانثناء بين كل تركيز من جسيمات أكسيد الزركونيوم النانوية٠ بحيث اضهرت انخفاضاً بنسبة ١٧٪ و ١١٪ لـ ١٪ و ٣٪ من أكسيد الزركونيوم على التوالي في حين أن ٥٪ تسبب في انخفاض حاد بنسبة ٣٢٪٠ في فيما يتعلق بالمسامية ، يضهر النتائج فروق ذات دلالة إحصائية٠ أظهرت Person correlation علاقة سلبية بين قوة الانثناء والمسامية ومع ذلك ، لم تكن النتائج ذات دلالة إحصائية٠ ضمن حدود هذه الدراسة ، يمكن الاستنتاج أن إضافة أكسيد الزركونيوم تسبب في انخفاض في قوة الانثناء لجميع التركيزات المضافة في حين أنه يؤدي إلى تأثير غير معنوي على مسامية الأكريليك وبالنظر إلى التأثير السلبي الذي لديه على القوة الميكانيكية ، فإنه لن يعتبر مادة مضافة مناسبة لتعزيز خصائصPMMA ٠

1. Introduction

Throughout the years, researchers are trying to improve the quality of biomaterials for fabrication of denture base because of increase in life expectancy of human beings and increasing demand of patients for better aesthetics, function and comfort. [1] Complete and partial dentures that are made from acrylic considered the most popular method because it provides a much affordable option for reconstruction than other available prosthesis. [2] Numerous materials with their modifications have been put to the market with enhanced mechanical and biological properties; despite this, there is still no single material that can fulfil the ideal requirements for denture base material. [3]

Polymethyl methacrylate (PMMA) is a derivative of acrylic acid, which related to as acrylic resin [4] that was introduced in 1937, by Walter Bauer, which gradually took the place of the traditional metal base and became most widely used material in clinical practice [5,6] because of its good biocompatibility, dimensional stability, the absence of taste and odour, good tissue response and outstanding toxicity profile.[7] Despite those excellent properties, PMMA demonstrates high porosity [8] and frequent fracture under load due to fatigue and chemical degradation.[9,10] Fracture resistance of PMMA does not show satisfactory results.[11] According to a survey, 68% of dentures had broken within 3 years of their construction. [12]

Due to their unsatisfactory properties, PMMA remain as an active material for research.[7] Generally speaking, there are three methods manufacture uses to enhance mechanical properties of denture base: discovery or development of new material to PMMA; chemical alteration of PMMA to fabricate high impact resin; and reinforcement of PMMA by other materials (e.g. nanoparticles). [13–15] Addition of nanoparticles (NPs) to the polymer matrix will result in alteration of mechanical properties by providing resistance against stress causing cracking. [14, 16, 17] Depending on the way the material will be enhanced it would be based on the size, shape, type and concentration of the added material. [17]

Alveolar ridge resorption is a continuous and irregular operation that causes uneven prosthesis support [18] with perpetual chewing cycles will result in generation of micro-

cracks in the material and continual application of force will cause propagation of cracks and flexural fatigue that commonly manifest itself as midline fracture. [15, 19] Studies had tried to improve the flexural properties of the material to overcome the consequence of fracturing and enhancing patient's satisfaction. [20] Study by Xin-jing et al. [21] that added nano Zirconium oxide (ZrO_2) NPs to acrylic at seven different concentrations (0.5%, 1%, 1.5%, 2%, 2.5%, 3% and 3.5%). Out off all the proportions 1.5% showed the highest mean value. On the same year, Al_2O_3 and ZrO_2 particles were incorporated to PMMA at 5% that improved flexural property. [22] Also, elevation of flexural value was demonstrated in another study that used 1.5%, 3%, 5% and 7% ZrO_2 in PMMA; the results revealed a direct relation between concentrations and strength value.[23] Furthermore, 10% and 20% ZrO_2 showed a statistically significant effect on increase of flexural mean of high impact acrylic by 32% and 23% respectively when compared to control.[24] Gad and his colleagues in 2016 [17] showed a raise in flexural strength at 2% only, while opposite effect was seen for 5%. Recently, Ergun et al.[25] added ZrO_2 NPs to heat cure PMMA at 5%, 10% and 20%. Results showed inverse relation with statistically significant effect for all concentrations.

Porosity considered as a quality of solid that is related to their structure and is expressed in the presence of voids between separate grains, layers, crystals, and other elements of a coarse structure of solid. [26] Porosity of PMMA is one of the most unfavourable characteristics because it causes high internal stress and increase susceptibility to distortion and warpage. [27] It has been attributed to variety of factors that include: monomer contraction during polymerization; air entrapment during mixing; monomer vaporization associated with exothermic reaction; presence of residual monomer; inadequate pressure on the flask and processing temperature higher than 74 °C. [28,29] It has been declared that 11% porosity in acrylic resin is linked to poor esthetic and reduction in mechanical properties. [30] The main concerns with the spongy areas is the harbour microorganisms and retain fluids which cause calculus deposition and staining that does not follow the definition of hygienically acceptable prosthesis.[9, 14]

Several methods have been utilized to calculate porosity of acrylic samples which are the classical, [31] photographical method that uses microscope [27] and mercury

porosimetry.[32] In this study, photographical method with aid of planimeter was used. To this date the influence of NPs on porosity of acrylic resin is very much restrained by the limitation of literatures available. A research by Acosta- Torres and his colleagues[33] that used classical method to measure the effect of TiO_2 and iron oxide (Fe_2O_3) NPs at 0.0150g and 0.009g respectively on porosity of PMMA. The result showed reduction in porosity of acrylic by 5.9% in contrast to unmodified. The findings suggested that metal oxide NPs can be a suitable additive for improvement of PMMA since high porosity considered as a critical drawback in Prosthodontic applications. [34] Furthermore, impregnated ZrO_2 NPs at 3%, 5% and 7% to heat cure PMMA revealed decrease in porosity by 18%, 38% and 55% for 3%, 5% and 7% ZrO_2 respectively. [35]

This study is attempted to investigate the effect of ZrO_2 NPs on flexural strength and porosity as well as it is correlation on conventional heat cure acrylic.

2. Materials and Methods

The ZrO_2 NPs were characterized using X-ray diffraction (PANalytical X'pert powder) with Cu-Kα X-ray source at a wavelength of λ= 1.54060$\dot{A}$. Diffraction data was recorded at 2θ range from 10° to 79.9950° with step size 0.0100° per 0.5s. Low scan speed was elected to provide higher sensitivity for detection of impurities. [36] The PANalytical software was then used to compare X-ray patterns to identify the nanoparticle.

In addition, SEM was used to check for agglomeration of ZrO_2 NPs within acrylic. The samples had dimensions of 30x10x2.5 mm (Length x width x thickness) and were cut in the middle, width wise. The specimens were cleaned for 10 minutes using ultra-sound cleaning device and then dried.[37] Looking at nonconductive material such as acrylic causes negative charge accumulation. [38] To overcome this, gold was sputter coated at a thickness of 300 $\dot{A}$ for 300s. Planetary rotation was used during coating process to provide a more uniform thickness. [39] Scanning was done using Quanta 450 SEM. Photographs were taken at 15,000x at accelerated voltage 15.00kV [40] using high vacuum mode.

The NPs was bought from Richest Group Company in China with 50nm in size and surface treated with saline coupling agent. The saline agent will provide a better bond between particles and acrylic [41] and minimizes agglomeration. [17,30] In order to reduce agglomeration of NPs, they were added to acrylic monomer first and then mixed with powder [23,25,29] with aid of probe sonicator (Biosafer 900-02) using the settings provided below:

a. Power Rate: 80%
b. Power: 900w
c. Probe size: 12 Φ
d. Processing time: 1-minute
e. Pulse on: 8s
f. Pulse off: 2s
g. Temperature warning: 37 °C.

Sonication was started with 8s on and 3s off to keep the temperature within the set range. After the sonication time was over, polymer powder was added immediately to the suspension (within 10s) to prevent re-agglomeration of NPs. [16] The mix was polymerized using conventional flasking and pressure packing technique. After polymerization was over, de-flasking was done and samples with gross porosity were divorced according to revised ADA specification number 12 for denture base polymer.[42]

3. Flexural strength test

For flexure strength test, 5 samples were prepared for each concentration of ZrO_2 making a total of 20 samples including the unmodified group. Each sample had a dimension of 65 mm length x 10mm width x 3mm thickness and were stored in distilled water at 37 ± 1 °C for 50 ± 2 hours, in compliance with International Standard Organization (ISO) 1567:1999 Denture Base Polymers. [43] The flexural value was obtained by doing a three-point bending test using universal testing machine. The samples were placed on circular supports (3.2mm in diameter and 10.5mm in length) that are 50 mm apart (Figure 1). Then force was applied perpendicular to the centre of the specimen using the loading nose at a crosshead speed of

5.00 ± 1 mm/ min until it breaks.[43] The maximum load required to fracture the specimen was recorded.

Then flexural strength was calculated using the formula: $\delta = \frac{3Fl}{2bh^2}$

Where: F: is the maximum load, in Newton, exerted on the specimen; l: is the distance, in millimetre, between the supports, accurate to ±0.01; b: is the width, in millimetre, of the specimen measured immediately prior to water storage and h: is the height, in millimetre, of the specimen measured immediately prior to water storage.

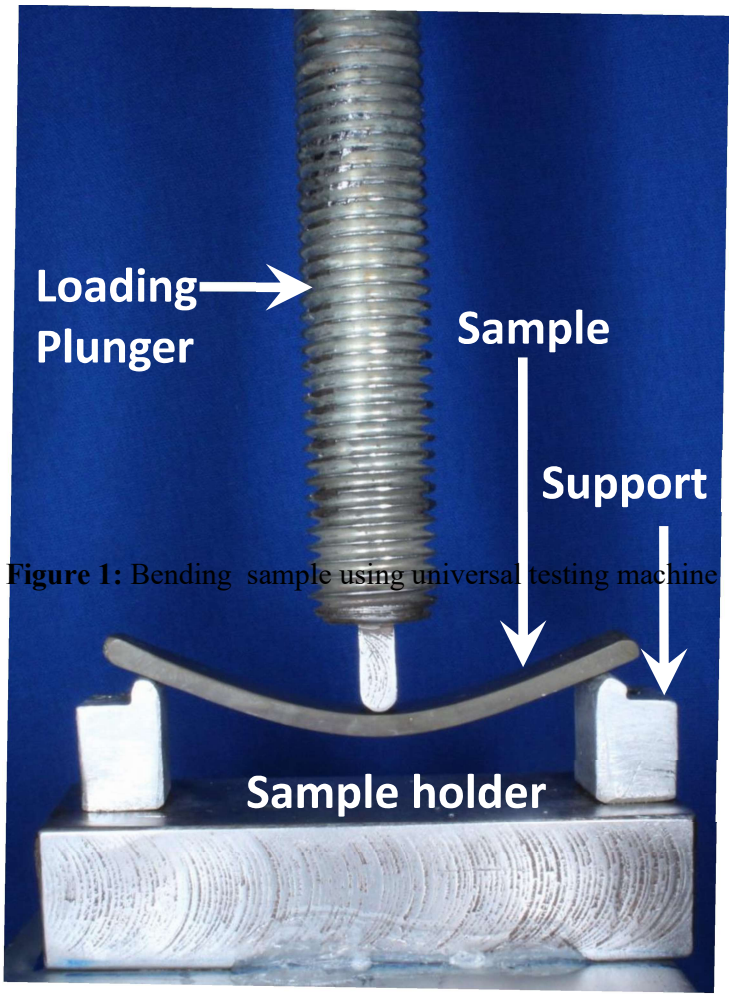

Figure 1: Bending sample using universal testing machine

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4. Porosity test

For this test, five samples were prepared for each concentration in addition to five samples for control group. Each sample had dimensions of 40 x 10 x 3mm [44] (Figure 2a). Each specimen was cut in three parts to produce three cross-sectional areas (Figure 2b). First cut was 1mm from the start of the sample; second and third cuts were 13mm apart. The surfaces were polished and observed under Maozua Digital Microscope under 8x magnifications and an image of the sample was captured and printed. Area of each pore evident on the photographs was outlined using a fine pen then perimeter of each pore was measured using a planimeter (Figure 3). Then total areas of pores on each surface were calculated using the formula:

$$\frac{Total\ porous\ area\ of\ specimen\ (mm^2)}{Cross\ sectional\ area\ of\ specimen\ (mm^2)}\ X\ 100 = \text{Area (\%)}$$

After that, the average of the three cross sectional areas was taken to be considered as a total porous area for a single sample.

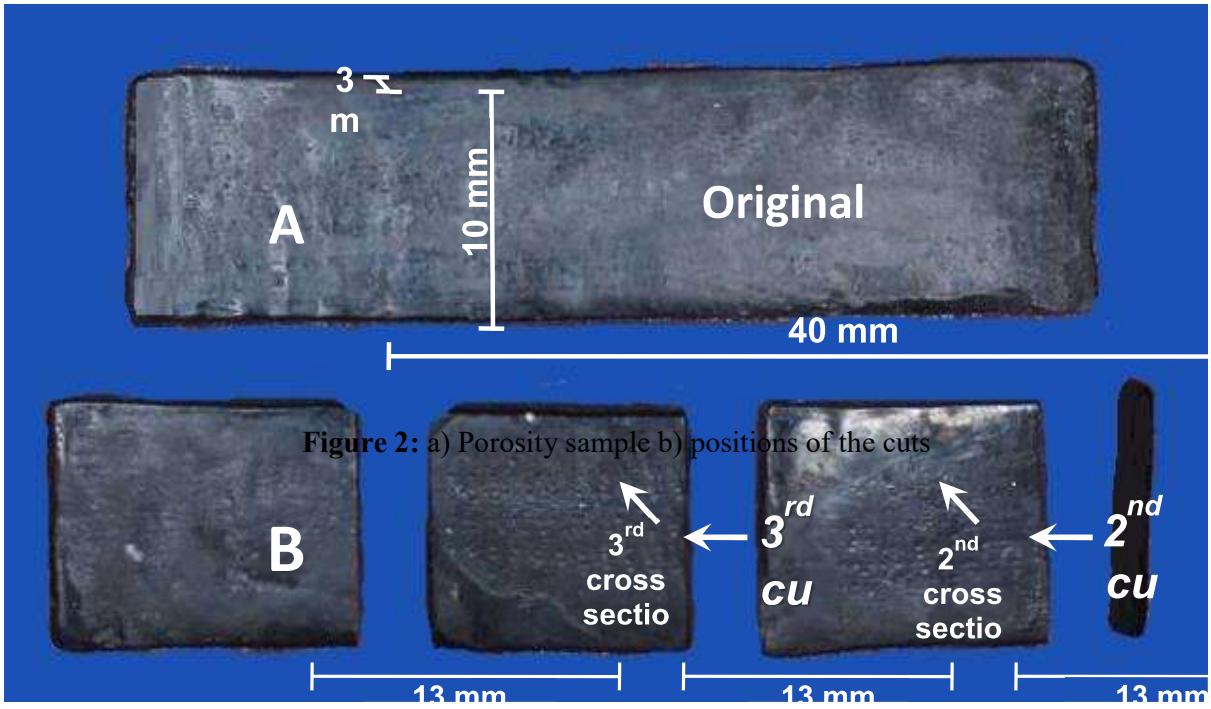

Figure 2: a) Porosity sample b) positions of the cuts

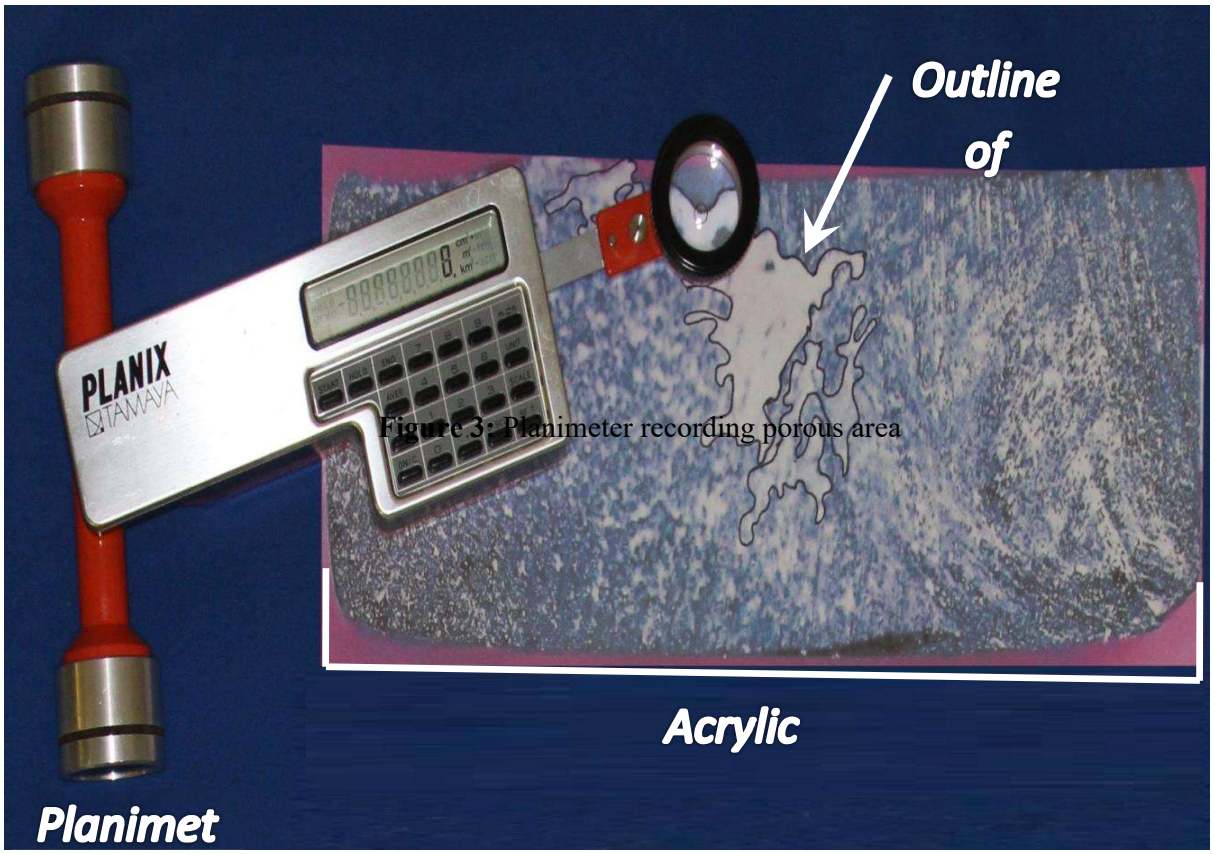

Figure 3: Planimeter recording porous area

The results were analyzed using Statistical Package for Social Sciences (SPSS, version 23). One-way ANOVA with Tukey's Honest Significant Difference (HSD) was performed to determine if there is a statistically significant difference between each concentration of ZrO_2 when compared to control. In addition, Pearson correlation coefficient (PCC) was used to measure and describe the relation between flexural strength and porosity. For statistical analysis, significant level $\alpha = 0.05$ was considered.

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5. Results

ZrO_2 NPs was analyzed using XRD. The PANalytical software was used to compare the XRD pattern. The Sample was identified as ZrO_2 NPs and it is diffraction pattern is shown in Figure 4.

The data that was obtained from flexural strength and porosity test were analysed using one way ANOVA with HSD to find out the effect of addition of NPs at the three concentrations (1, 3, and 5%). The flexural strength results, revealed statistically significant differences ($p< 0.05$) between each concentration of ZrO_2 in reference to control. The results for 1% and 3%, revealed 17% and 11% reduction in flexural mean respectively, while 5% showed a drastic reduction by 32%. Is worth mentioning that the flexural mean for 1% and 3% ZrO_2 NPs did not jeopardize the strength beyond the standard requirement (65 MPa) that has been set for Type 1 denture base polymer [43] except for 5% (59.43 MPa) that did not exceed the minimal limit. The highest value is registered for control group (87.205 MPa) that presented in Table 1.

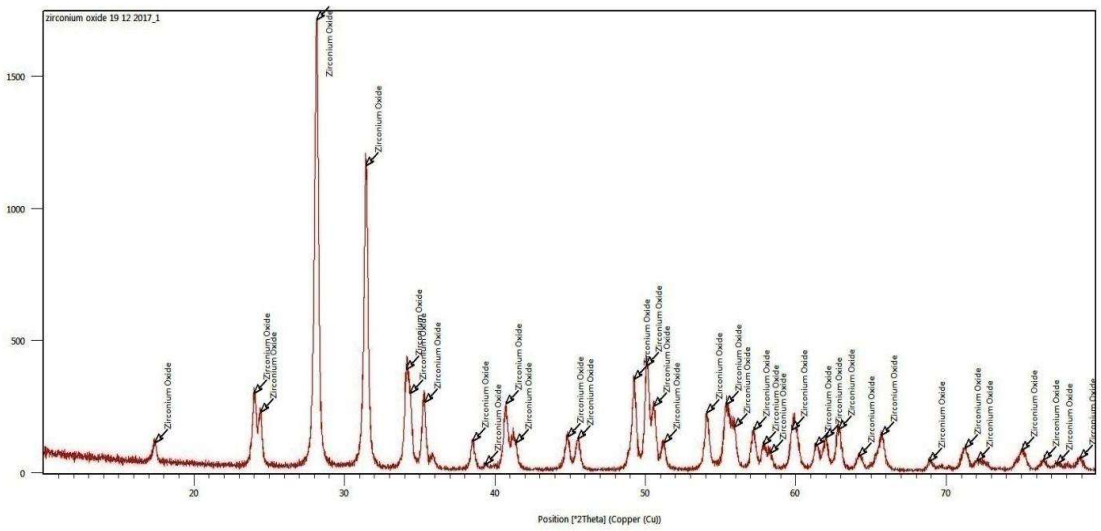

Figure 4: XRD pattern for Zirconium oxide nanoparticle

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Table 1: Descriptive statistics and comparisons of each concentration of ZrO_2 NPs with control group for flexural strength

Nanoparticle	Concentration %	Mean (MPa)	SD	ANOVA P value of concentration with control
Zirconium oxide	1	72.278	1.492	0.00
	3	77.281	0.874	0.00
	5	59.425	1.943	0.00
Control		87.205	2.499	

In regards to porosity test, all the concentrations did not show statistically significant difference when compared to control because their p values were greater than 0.05. 1% and 3% addition of ZrO_2 showed 11% and 6% reduction respectively however 5% caused a 4% increase in porosity in regard to control. The summary for porosity values is illustrated in Table 2. As the table shows the highest porosity mean was for 5% (13.30%) while 1% (11.34%) showed the lowest value among the group.

Table 2: Descriptive statistics and comparisons of each concentration of $ZrO_2$with control group for porosity test.

Nanoparticle	Concentration (%)	Porosity mean (%)	SD	ANOVA P value of concentration with control
Zirconium oxide	1	11.338	2.688	0.450
	3	11.942	0.598	0.817
	5	13.300	0.982	0.940
Control		12.766	0.550	

With regards to correlation, PCC test showed strong and a negative relation (-0.83) between flexural and porosity for ZrO_2. However, the results did not show statistically significant because their p values were greater than 0.05 (p=0.369).

Based upon the data obtained from flexural strength test, ZrO_2 3% and 5% was sent for scanning to check for dispersion and agglomeration of NPs within the matrix of heat cure PMMA. SEM images were taken at 15,000x magnification for all samples. SEM image of 3% (Figure 5) and 5% (Figure 6) of ZrO_2 showed drastically different agglomeration state. The 5% revealed a very high agglomeration within the acrylic in contrast to 3%.

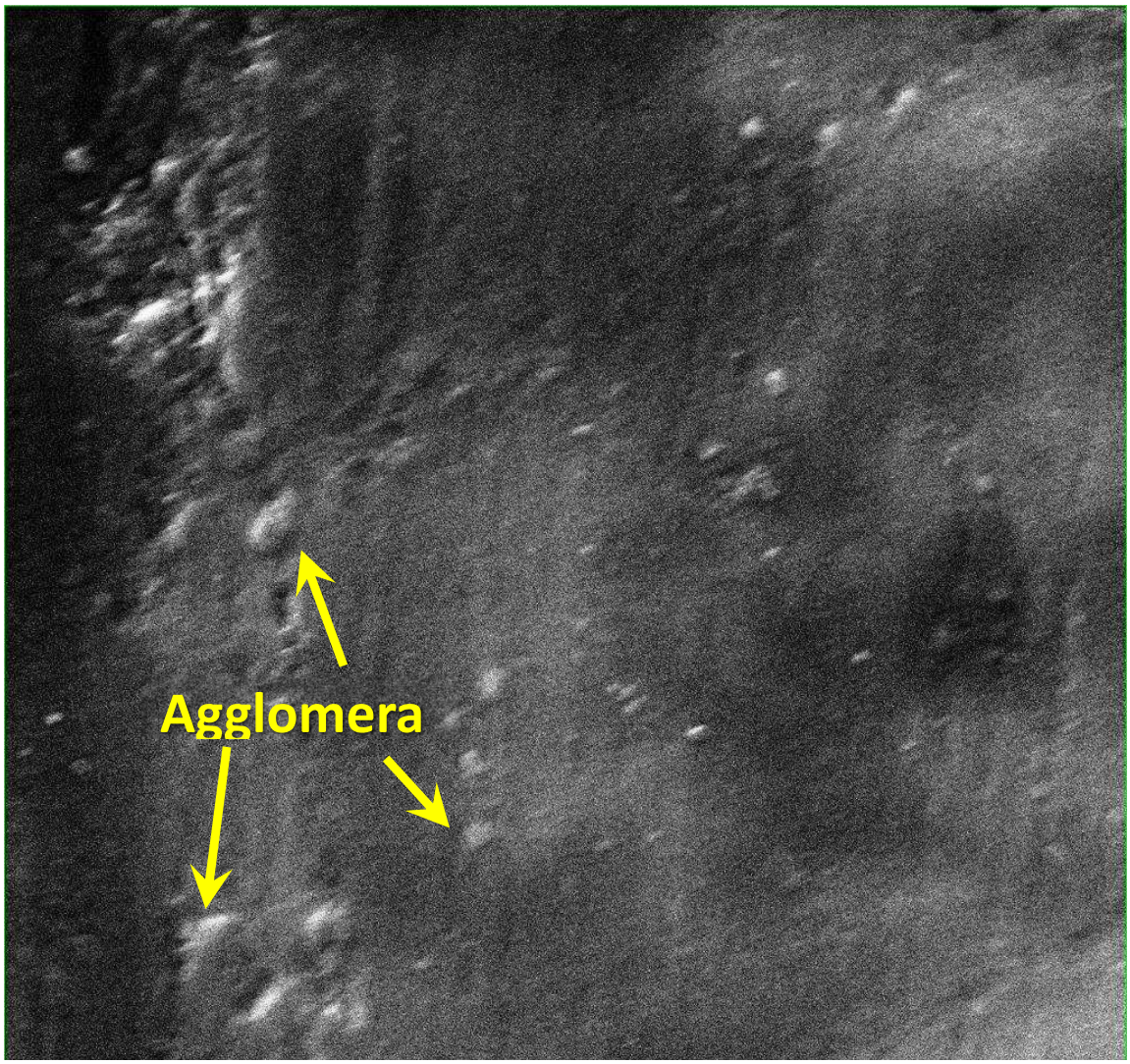

Figure 5: SEM image represents agglomeration of ZrO_2 NPs at 3%.

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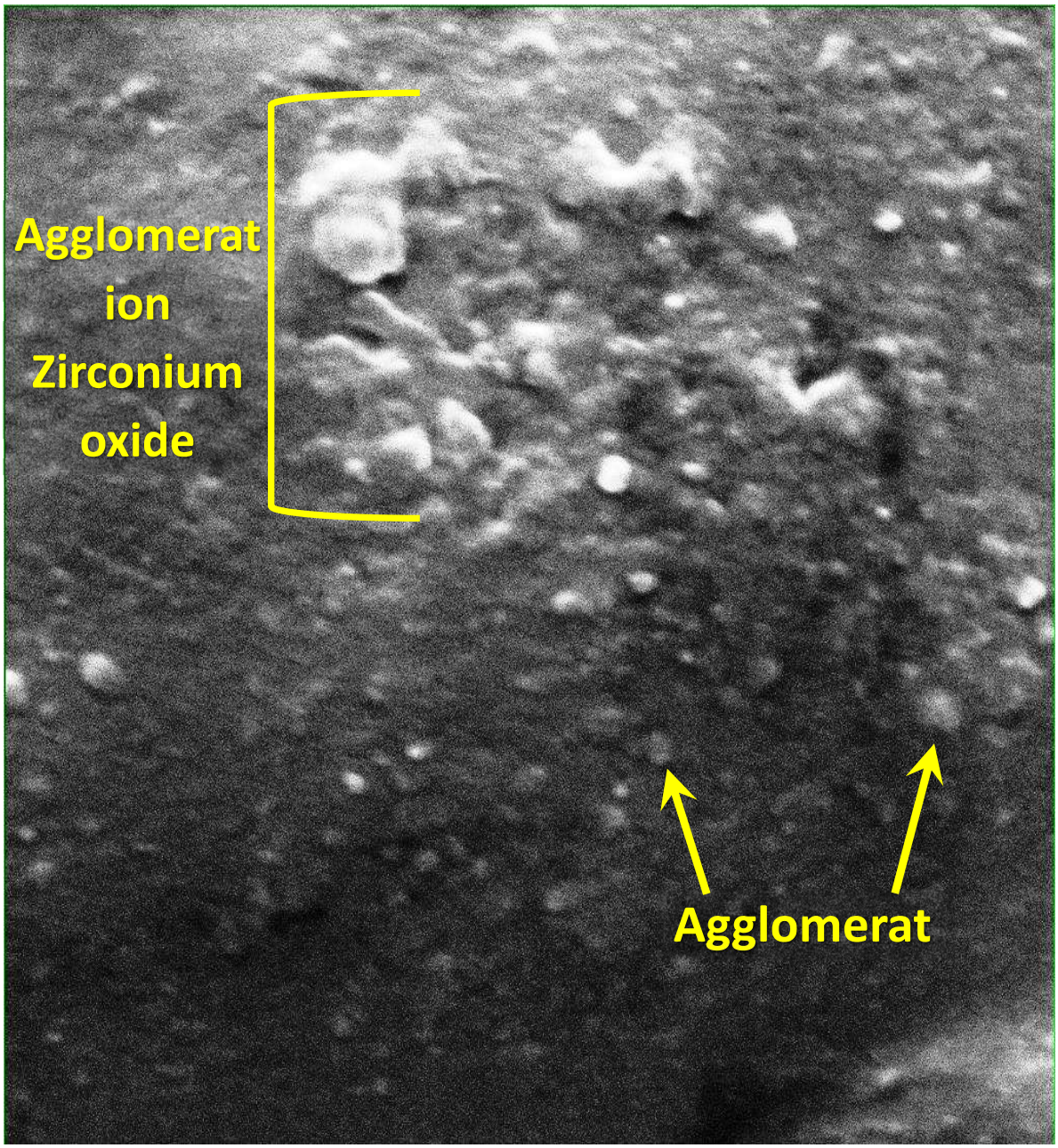

Figure 6: SEM image represents agglomeration of ZrO_2 NPs at 5%.

6. Discussion

Acrylic resin is the most commonly used material in fabrication of dental prosthesis [45] due to its aesthetics, good tissue response and ease of manipulation [9] but its mechanical properties is not satisfactory. [46] The occurrence of denture fracture has been reported in the literature with high rate which compromises the longevity of the prosthesis. [23] Movement and distortion of the denture during mastication with gradual bone resorption will result in unsupported prosthesis which in turn cause stress formation. [47] This commonly termed as flexural fatigue that manifest itself as midline fracture. [48] To mimic the clinical condition the material faces during their use, flexural strength was conducted.

The results of this study showed when ZrO_2 NPs were dispersed in acrylic at 1%, 3% and 5% caused a drastic reduction in flexural strength by 17%, 13%, and 32% respectively. The findings are in disagreement with Xin-jing et al. [21], Alhareb [22] and Ahmed et al.'s[23] research that revealed enhancement in flexural value. As it can be seen from the bending test, the values decreases progressively with increase in NPs concentration which is in agreement with Gad et al.'s [17] and Ergun et al.'s [25] findings. This effect can be linked to many factors. One of the causes is dispersion of NPs which had shown to play an important role in reinforcing material; increasing the filler content in addition to poor dispersion will results in suspending resin matrix continuity and creating defect in the material which weakens it in the outcome. [24] It will also interfere with the reaction of methylmethacrylate which causes an increase in uncreative monomer that behaves as a plasticizer.[34,49] The plasticizer capable in getting between polymer chains which induces the separate chains to become more tenuous and reduce the attraction force between molecules as a consequence the acrylic will be more flexible but brittle. [50]

Another consequence of improper dispersion is agglomeration which causes reduction in strength [51] since too many fillers will act as a stress concentrating point [12,51–53] that will alter the modulus of elasticity of the resin and mode of crack propagation through the polymerized specimen as well as hindering integrity of polymer matrix. [17,54] This concurs with our flexural strength data that showed 5% ZrO_2 had the lowest value (59.43 MPa) in

contrast to 3% (77.28 MPa) which revealed a superior effect that can be linked to a better dispersion with less agglomeration. This can clearly be seen in SEM findings (Figure 5 and 6). Furthermore, agglomeration will brings about micro-cracks and micro-pores as a structural defect which endangers mechanical properties of the polymer.[34] Another reason, is the polymer has reached to a saturation point and the resin cannot incorporate further more filler particles any effort after reaching this point will cause suspending in matrix continuity and compromising the flexural strength of reinforced material in the outcome. [24] In addition to this, NPs might have acted as impurities within the PMMA which led to unfavourable decrease in mechanical strength [55, 56] due to lack of chemical bond will result in poor adhesion between particles and acrylic resin. [51, 57]

Porosity is complex phenomena of multifactorial origin.[32] Several factors have contributed to porosity that includes; polymerization method, thickness and the material of chose, air entrapment during mixing; monomer vaporization associated with exothermic reaction and presence of residual monomer. [28,29] Porosity in a material considered unfavourable condition that can weaken the denture and cause high internal stress which result in a greater vulnerability to distortion.[31]

Based on the results of this study, ZrO_2 NPs at 1% and 3% caused no significant reduction in porosity by 11% and 6% respectively. The obtained results are in consistence with Acosta-Torres et al.'s [33] and Hameed and Rahman's [35] that showed reduction in porosity when NPs were added to acrylic. This is due to well dispersion nano-sized particles that is capable of entering between linear macromolecular chains of the polymer and occupy the spaces between them in the result will decrease the porosity by increasing the density of acrylic without changing the basic structure of PMMA chains. [35,53] Another reason is the fillers will occupy the space inside material leading to a decrease in the number of pores that open up to the external surface. [35] While, 5% caused a 4% increase with the same statistical effect as the other concentrations in reference to control. It has been stated that the concentration of benzoyl peroxide is linked to porosity in an inverse manner.[30,58] Since, 5% ZrO_2 were added to acrylic without reduction in monomer this might increased in the amount of unreacted monomer that will raise the chance for porosity. In addition, NPs in

high concentration will also act as impurity that will interfere with the polymerization reaction thus acting as a plasticizer leading to increase in amount of residual unreacted monomer. [52, 54] This explains the direct proportional relation of NPs concentrations with porosity.

In regards to the relation between flexural strength and porosity, 5% ZrO_2 showed highest porosity as well as lowest flexural value because excessive addition of NPs will affect the polymerization of PMMA resulting in an increase in porosity as well as making the polymer more brittle as a result it will be more prone to breakage during bending test. [50, 59] It is noticeable that all concentrations of NPs had caused porosity of heat cure acrylic to be more than 11%; this would cause unsatisfactory aesthetic and inferior mechanical properties according to the literature. [30]

7. Conclusions

Within the limitations of this study, it can be concluded that the addition of Zirconium oxide caused a reduction in flexural strength for all concentrations added. While it result in a non-significant effect on porosity of acrylic. Considering the negative effect it had on mechanical strength it won't be considered a suitable additive to enhance the properties of PMMA.

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