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EFFECT OF SILANIZED ZIRCONIA ON SOME MECHANICAL PROPERTIES OF HEAT-POLYMERIZED POLY METHYL-METHACRYLATE DENTURE BASE MATERIAL

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ABSTRACT

Objective: The purpose of this work was to study the effect of addition of silanized zirconia powder on flexural strength, deflection, modulus, toughness and micro-hardness of heat-polymerized poly methyl methacrylate (PMMA) denture base resin.

Materials and Methods: One hundred specimens of heat-polymerized PMMA were prepared for flexural and hardness tests, fifty for each. Zirconia (ZrO_2) powder was treated with silane coupling agent then added to acrylic powder in percentage of 0%(control), 0.5%, 1%, 2% and 5% by weight. Specimens for flexural test were prepared in the shape of flat strips with dimensions of (65×10×2.5mm) using compression molding technique. Flexural test was performed by three-point bending test using universal testing machine. Flexural strength, flexural deflection, flexural modulus and toughness were obtained from the software of the testing machine. Specimens for micro-hardness test were prepared in dimensions of (10×10×3mm). Micro-hardness was determined using Vickers Micro-hardness Tester. Digital microscope was used to capture photomicrographs at x500 magnification of the surface of randomly selected specimen for each group to show the distribution of different percentages of zirconia. One-way Anova followed by Scheffe's post hoc multiple comparison test was performed with 5% level of significance for testing the significance between the means of tested groups.

Results: There is a significant difference among the mean values of flexural deflection, flexural modulus, toughness and micro-hardness (P<0.05%). The mean values of flexural strength were not significantly different (P>0.05%). The digital microscope has shown nearly even distribution of zirconia particles within the acrylic matrix at 0.5% and 1%, while clusters started to form at 2%, and 5% zirconia.

Conclusion: Silanized zirconia powder could be used to improve the properties of heat-polymerized acrylic denture base resin. The most appropriate percentages were 1% and 2%.

KEY WORDS: Zirconia powder, Silane coupling agent, Modified denture acrylic resin**,** Flexural test, Micro-hardness.

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INTRODUCTION

Poly methyl methacrylate, broadly termed 'acrylic resin', is the material of choice for full denture base fabrication and for adding 'gum work' to cast metal frameworks. Although many researches have been performed to find alternatives with better properties, it remains the most common polymer system for these applications.[1]

Heat-polymerized acrylic resin has many advantages particularly, good esthetics, low density, accurate fit, stability in the oral environment, inexpensive equipment and ease to process and repair. However, this material is not ideal because of its inferior mechanical performance. Therefore, there is a need to improve its mechanical properties.

Many attempts have been studied to overcome its drawbacks including chemical modification to produce graft copolymer high-impact resins[2] and mechanical reinforcement through the inclusion of glass, aramid, nylon fibers[3], ultra high modulus polyethylene Fibres,[4] metal fillers[5] and metal oxides.[6] The recent approach to improve the properties of acrylic resins is the addition of zirconium dioxide (zirconia, ZrO_2) as a filler. Some studies have shown that zirconia is a biocompatible and esthetics more than other metal fillers.[7,8] Zirconia possess high flexural strength, high hardness and excellent modulus of elasticity. [9] All of these properties enabled it to be an attractive option to enhance the mechanical properties of polymers.

The flexural properties of denture base materials are of importance for predicting their performance upon loading in the oral cavity. Dentures are commonly subjected to flexures during mastication. Repeated flexures can lead to failure in well-fitting denture occluding against natural teeth.[10]

Hardness of the polymers has been found to be sensitive to the residual monomer content in it. Because of easy preparation of specimens, simple test method and available equipment, hardness has been widely used as an indirect method of evaluating depth of cure of resin-based composite materials and for characterization of the mechanical quality of a polymer.[11-13]

Few researches have been dealing with the effect of varying ratios of zirconia on the properties of heat-cured acrylic resin. Therefore, the objective of this study was to investigate the effect of incorporation of 0.5, 1, 2 and 5% zirconia on the flexural strength, flexural deflection, flexural modulus, toughness and micro-hardness of heat-polymerized acrylic resin. The null hypotheses of this study are: (i) there is no effect of different percentages of zirconia on the flexural properties of heat-polymerized acrylic resin. (ii) zirconia has no effect on the micro-hardness.

MATERIALS AND METHODS

Preparation of silanized zirconia powder:

Zirconia (ZrO_2) powder with particle size of 7-18 μm (Zirconium oxide, Promochem GmbH Postfach, Wesel, Germany) was treated with silane coupling agent (Silikon- & Wachs-Entspanner, Dental future systems, Riedenburg, Germany).

A 0.3 gm. solution of silane coupling agent in 100 ml acetone (pure toluene solvent) was used to treat a 30 gm. of ZrO_2 powder into a glass flask then sonicated at 37 ºC for 20min. The ZrO_2 powder were stirred in the coupling agent/acetone solution with a magnetic stirrer (Wisestir® MSH-20A, Wisd laboratory instruments, DAIHAN scientific Co., Korea) for 60 minutes, after which acetone was completely evaporated using a rotary evaporator (BÜCHI Rotavapor R-200 AG, B-490, Germany) at rotation of 150 rpm for 30 min under vacuum. The silanized powder was dried in a vacuum oven (Gallen bamp, England) at 60ºC for 20 hours, then stored at room temperature before use.[14]

Preparation of silanized zirconia/acrylic powder

After silanization, zirconia powder was weighed using an electronic balance (Precisa, PAG OERLIKON AG, Zurich, Switzerland) then added to the acrylic powder in percentage of 0%(control), 0.5%, 1%, 2% and 5% by weight. Acrylic polymer powder was mixed thoroughly with silanized zirconia powder to obtain a consistent and uniform mix.

One hundred specimens of heat-polymerized acrylic resin (Acrostone; Acrostone Dental factory, under exclusive license of whw England, A.R.E.) were prepared for flexural and micro-hardness tests, fifty for each.

Flexural test

Fifty specimens, ten for each percentage, of heat-polymerized acrylic resin were prepared in the shape of rectangular flat strips with dimensions of 65mm length, 10mm width and 2.5mm thickness according to ADA specification no 12 for denture base polymers.[15]

Preparation of specimens

Rectangular metal dies with dimensions of 65mm length, 10mm width and 2.5mm thickness were used for preparation of acrylic specimens of flexural test. Each die was painted with pure petroleum jelly (Magnum, Great World Cosmetics, Area Delhi, India) as a separating medium then invested in a dental flask using dental stone (Elite dental stone, Zhermack, Badia Polesine, Italy). After stone setting, the two halves of the flask were separated and the die was removed leaving mold space. The measured acrylic powder and liquid were mixed, packed at the dough stage in the mold space then cured according to the manufacturer's instructions using compression molding technique. After processing, the specimens were trimmed with an acrylic stone, polished and stored in distilled water at 37^{o}C for 7 days before testing.

Testing procedure

Flexural properties were measured by the three-point bending test using universal testing machine (Instron®, Corporation Road, High Wycombe, Bucks HP12 3SY, UK) and data were recorded using computer software (Bluehill®LE testing Software). The test assembly consists of a loading wedge and a pair of supporting wedges spaced 50 mm apart with 5 kN load cell, at a crosshead speed of 5mm/min. This loading wedge has engaged the center of the upper surface of the specimen until the specimen has broken. The specimen was deflected until fracture occurred. The testing machine was computer-aided to process the data with a specifically designed program. The thickness and width of the specimen was edited in the computer. The software of the testing machine can conduct the following properties:

1. Flexural strength: It represents the maximum stress developed in a rectangular beam loaded in the center of the span which can be calculated according to the following equation: [11]

Flexural strength (S) = $3PL/2bd^2$

Where P is the load, L is the distance between two supports (50.0 mm), b is the specimen width, and d is the specimen thickness.

2. Flexural deflection: It represents the amount of deflection expected, which can be calculated according to the following equation: [11]

Flexural deflection= $3PL^3/4Ebd^3$ where E is flexural modulus.

3. Flexural modulus: It can be determined from a stress-strain curve by calculating the ratio of stress to strain or the slope of the linear region of the curve. [11]
4. Toughness: It is related to the area under the load-deflection curve and represents the energy required to stress the material to the point of fracture and reported in units of Joules. [11]

Micro-hardness test

Fifty specimens of heat-polymerized acrylic resin in the dimensions of (10×10×3 mm) were prepared for micro-hardness test, ten for each percentage. Preparation of specimens was performed as described in the flexural test. Specimens were stored in distilled water at 37°C for 7 days before testing. Surface micro-hardness was determined using Digital Display Vickers Micro-hardness Tester (Model ZHVμ-S, Zwick Roell Indentec Hardness testing machine, West Midlands, UK). In each specimen, ten indentations were made using the diamond indenter with load of 25 gm. applied for 10 sec., and the mean VHN was obtained digitally. The indentations were observed using ×10 magnification lenses.

Microscopical examination

Digital microscope (Digital Microscope Model: A005, Supereyes, China) was used to capture photomicrographs of the surface of randomly selected specimen for each group to show the distribution of different ratios of zirconia. Photomicrographs were taken at x500 magnification.

Statistical analysis

Mean values and standard deviation of tested properties were calculated for each group. Statistical analysis was carried out using QI Macros software (KnowWare International, Inc., Denver, USA). One-way analysis of variance (ANOVA) followed by Scheffe's post hoc multiple comparison test was performed with 5% level of significance for testing the significance between the mean values of all groups.

RESULTS

Flexural test

Mean values and standard deviation of flexural strength, flexural deflection, flexural modulus and toughness of heat-polymerized acrylic resin (control) and zirconia-modified acrylic resin at different percentages are listed in table (1) and shown in figure (1). One way ANOVA has revealed that there was significant difference among the mean values of flexural deflection, flexural modulus and toughness of all groups ($P<0.05\%$) while the difference among the mean values of flexural strength was not significant ($P>0.05\%$).

Micro-hardness test

Mean values and standard deviation of micro-hardness of heat-polymerized acrylic resin (control) and zirconia-modified acrylic resin at different percentages are reported in table (1) and shown in figure (1). There was a significant difference among the mean values of micro-hardness of all groups as illustrated by one way ANOVA ($P<0.05\%$).

TABLE (1) Mean values and standard deviation of tested properties of control group and zirconia-modified acrylic resin groups

	0% (control)	0.5%	1%	2%	5%	One way Anova (P-value)
Flexural strength (MPa)	89.91±4.55	91.46±8.49	94.08±4.26	92.26±5.69	90.82±8.50	0.762
Flexural deflection (mm)	1.338^{a} ±0.05	1.291^{ab}±0.12	1.095±0.08	1.268^{abc}±0.04	1.328^{ac}±0.12	0.037^{*}
Flexural modulus (GPa)	2.034^{a}±0.27	2.132^{ab}±0.22	2.301^{abc}±0.39	2.313^{bce}±0.29	2.547^{ce}±0.12	0.016^{*}
Toughness (Joule)	0.200±0.03	0.124^{a}±0.03	0.149^{ab}±0.02	0.148^{abc}±0.02	0.153^{abc}±0.03	0.016^{*}
Micro-hardness(kg/mm^2)	12.07^{a}±1.30	13.97^{a}±1.88	17.57^{b}±2.64	21.5±2.69	17.71^{b}±3.57	0.000^{*}

P< 0.05 significant difference
Values with identical letters in the same row indicate no significant difference after Scheffe's post hoc multiple comparison test

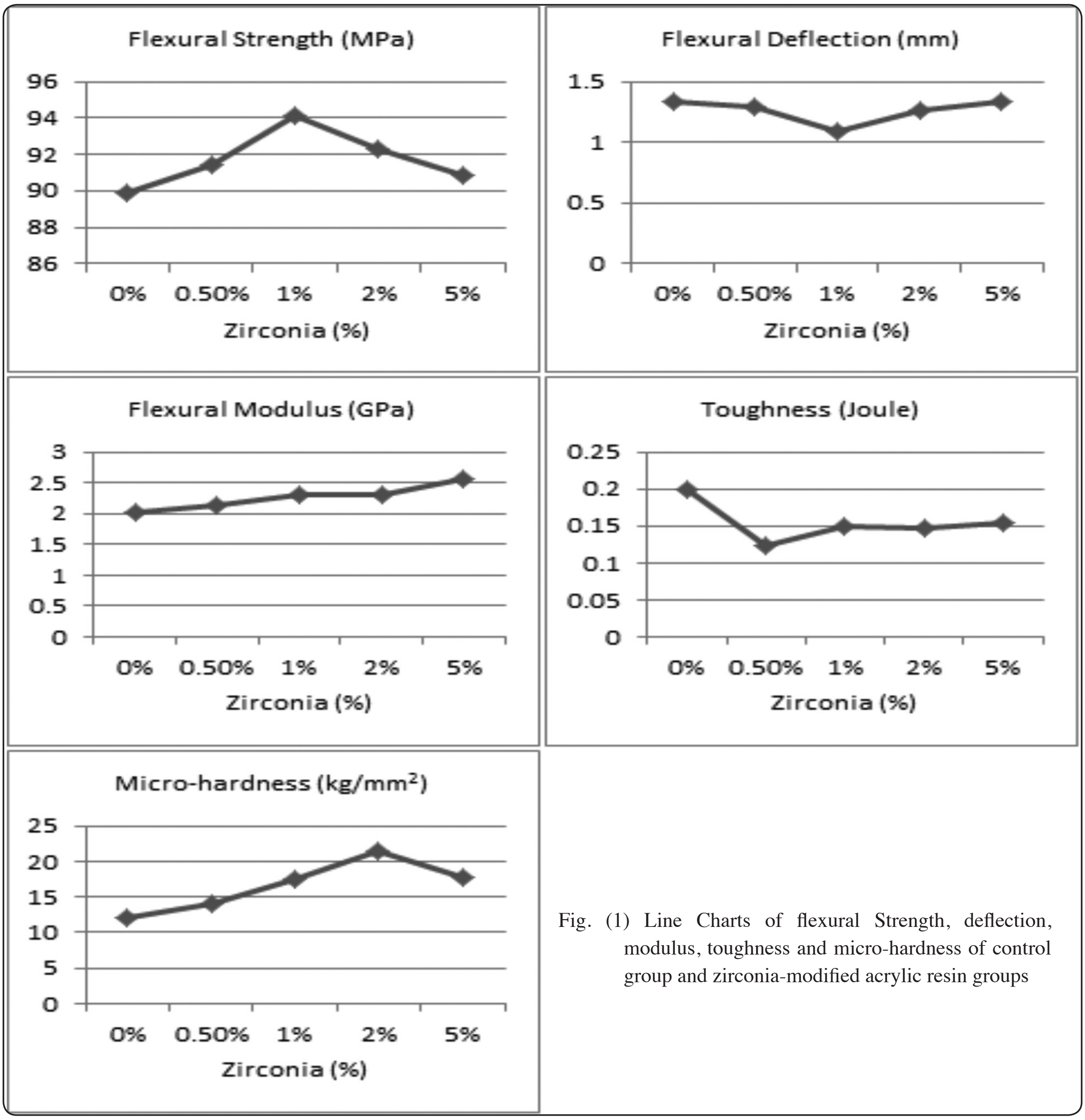

Fig. (1) Line Charts of flexural Strength, deflection, modulus, toughness and micro-hardness of control group and zirconia-modified acrylic resin groups

Microscopical Findings

The digital microscope has shown homogeneous matrix of the PMMA of the control specimen (Fig. 2A). At 0.5% and 1% zirconia (Fig. 2B and 2C), zirconia particles are widely and evenly distributed within the matrix keeping its homogeneity. At 2%, zirconia particles start to cluster but still keep the matrix continuity (Fig. 2D) while 5% zirconia has produced more clustering of the particles within the resin which leads to interruption in the resin matrix continuity as shown in Fig (2E).

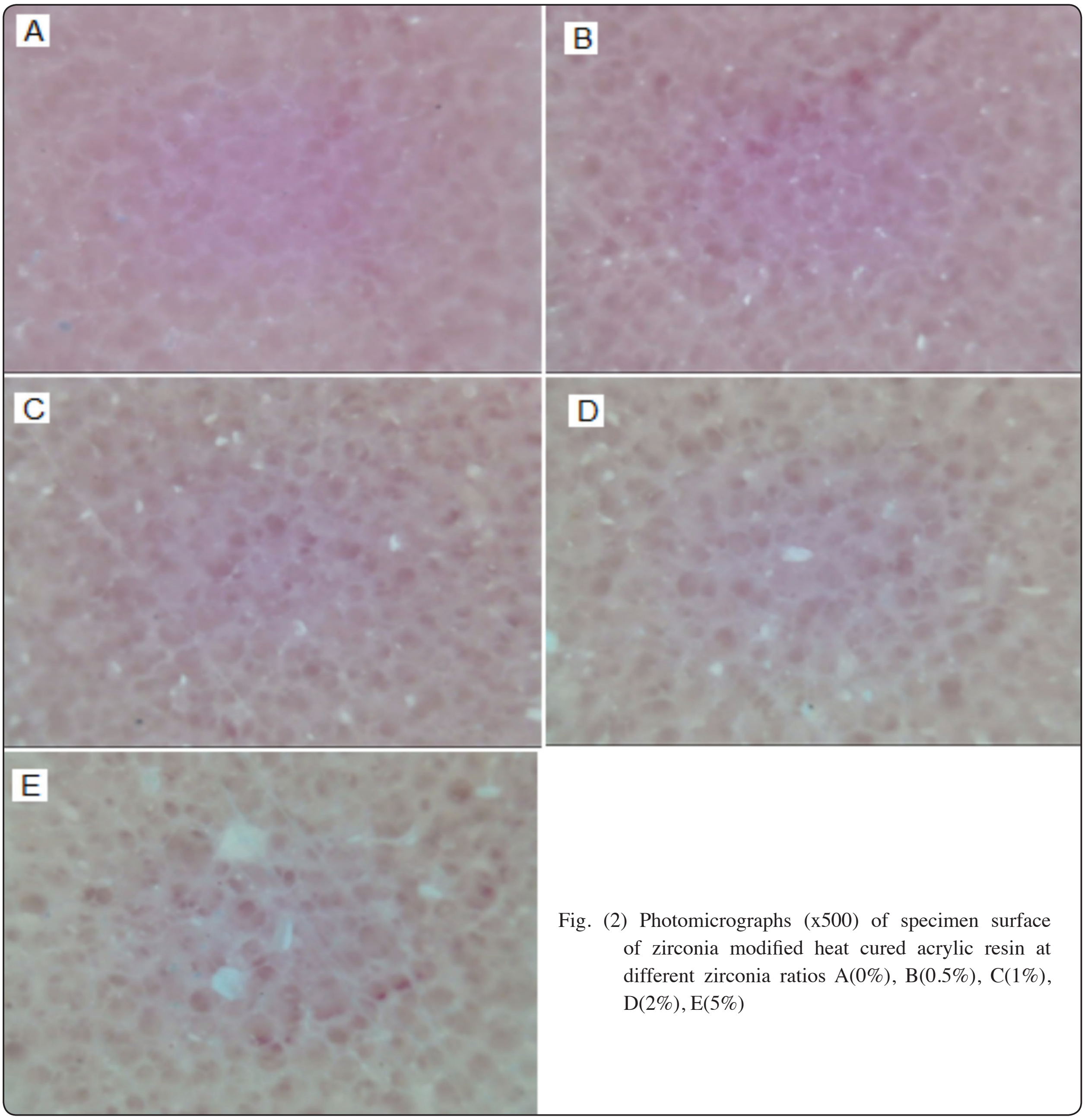

Fig. (2) Photomicrographs (x500) of specimen surface of zirconia modified heat cured acrylic resin at different zirconia ratios A(0%), B(0.5%), C(1%), D(2%), E(5%)

DISCUSSION

Although polymethylmethacrylate (PMMA) has been widely used as a denture base since 1930`s, it sometimes has failed or cracked clinically.[16] To overcome these problems, many attempts were made to improve the mechanical properties including flexural strength, deflection, modulus, toughness and micro-hardness of the PMMA.[17]

The present study has investigated the effects of ZrO_2 filler on the flexural strength, deflection, modulus, toughness and micro-hardness of heat-polymerized acrylic resin. ZrO_2 was used because it has white color which less likely to change the appearance of the denture base. Moreover, it has excellent biocompatibility.[18]

The size of filler particles in the polymer matrix and strong adhesion at the interface play major role on the mechanical properties of filled polymer composites.(19,20)

The size of filler particles should be small for proper processing.(21) The particle size of ZrO_2 (7-18 μm) used in this study are much smaller than that of powder resin particles (up to 100 μm).(22) Therefore, zirconia particles will fill the interstitial of polymer particles and interfere with the displacement of the segments of polymer chain.(23)

Percentage of filler used to reinforce acrylic resin is another important factor affecting mechanical properties. Filler percentage should be such that the filler particles disperse homogeneously into the resin matrix without interrupting its continuity.(24) Therefore, the percentage of zirconia chosen in this study was 0.5%, 1%, 2% and 5%.

The inorganic filler usually display high surface energy because of the hydrophilic ionic nature of it. However, the hydrophobic polymer does not bond with the filler due to the difference in surface energies. Therefore, the filler surface should be modified to improve surface adhesion between the filler and matrix.(25)

Silane coupling agents play a central role in improving bonding between filler and the resin matrix, and they subsequently improved the resin's properties as concluded by Gad et al.(26) In the current study, prior to admixing with the polymer beads, the filler was treated with silane coupling agent to create strong interphase between filler and the resin matrix.(23)

Silane coupling agent is a bi-functional monomer, with the hydroxymethyl groups substituted by hydroxyl groups for attachment to the fillers. This decreased the surface tension of the particles and influenced the spatial distribution of fillers. On the other hand, silane coupling agent contained C=C bonds which reacted with PMMA matrix during the curing process, thereby acting as a "molecular bridge" to establish chemical bonding between PMMA matrix and the particles.(27)

The flexural strength test is useful in comparing denture base materials because it simulates the type of stress applied to the denture during function in the oral cavity.(11) The greater the flexural deflection potential of a denture base material is, the greater will be the possibility of the denture separating from the alveolar ridge during masticatory function, causing loss of retention and stability of the prosthesis in the mouth which might contribute to greater bone loss in residual ridge.(28)

The elastic modulus describes the stiffness or rigidity of a material within the elastic range. The interatomic forces of the material are responsible for the property of elasticity. The stronger the basic attraction forces, the greater are the values of the elastic modulus and the more rigid is the material. This property is quite dependent on the composition of the material.(11)

The results of the present study have showed that the addition of different percentages of zirconia has an influence on the flexural properties and micro-hardness of heat cured acrylic resin. Therefore, the first and second hypotheses are rejected.

Flexural strength values of zirconia modified acrylic resin groups at all percentages were higher than that of control group. The strength has increased at 0.5% and the highest value was obtained at 1% zirconia then decreased at 2% and 5%. However, the difference was not statistically significant (P=0.762).

The increase in transverse strength was due to good distribution of the very fine zirconia particles (Fig. 2A, 2B) that enabled them to enter between linear macromolecular chains of the polymer and fill spaces between chains, segmental motions of the macromolecules were restricted leading to increased strength and rigidity of the resin and decrease in the ability to deformation.(21)

As zirconia percentage increased, there was a decrease in transverse strength because at higher percentage, zirconia particles tend to clump together when mixed with monomer forming clusters of the particles within the resin which act as inclusive bodies that broke the homogeneous matrix of the PMMA [29] as shown in Fig.(2E). These clusters could result in mechanical weak points (structural defects) which would adversely affect the mechanical properties of the zirconia-modified acrylic resin.[30,31] In addition to the difference in density between PMMA (1.18 g/cm^3) and zirconia (6.15 g/cm^3) [9] that has favored zirconia to be settled down during mixing and has participated in inhomogeneity.

The lowest deflection value was recorded at 1% zirconia modified acrylic resin. Decreased deflection of acrylic resin with addition of zirconia was consistent with Hameed and Abdul Rahman. [17]

The mechanical properties of the polymer are important in the fabrication of denture bases as the cured polymer should be stiff enough to hold the teeth in occlusion during mastication and to decrease the uneven loading of the mucosa.[32]

The high stiffness of filler particles can lead to increase of a composite's stiffness.[33]

The modulus of elasticity of zirconia (205 GPa) [9] exceeds too much that of PMMA (2.03 GPa). So, addition of zirconia to PMMA has increased the flexural modulus significantly. This result was in agreement with Zuccari et al.[25] The highest value was recorded at 5% zirconia modified acrylic resin. Silanized zirconia cross-linked with methyl methacrylate monomer. Cross-linking provides a sufficient number of bridges between linear polymer chains to form a three-dimensional network that increases rigidity of the resin and resistance to solvents. On the other hand, cross-linking has only modest influence on strength.[34]

According to Wang et al.[35] the smaller particle size yielded higher mechanical properties due to larger specific surface area or more contact points between the matrix and filler that provided good mechanical interlocking in between and therefore increased its stiffness properties.

The results of toughness showed that the modified acrylic resin at all ratios of zirconia have required significantly less energy absorption to break compared with the control group ($p < 0.05$). Therefore, the flexural modulus of zirconia modified acrylic was improved at the expense of the toughness, producing a denture base with a different brittle behavior.

Micro-hardness of heat-cured acrylic resin has increased after addition of zirconia up to 2% ratio then decreased at 5% ratio. This improvement in hardness was in accordance with Zuccari et al[25]

Improvement in micro-hardness with the increase in percentage of zirconia filler up to 2% is due to inherent characteristics of the zirconia particles. Zirconia possesses strong ionic interatomic bonding giving rise to its desirable material characteristics, that is, hardness and strength. The small particle size provides sufficient surface area to make good adhesion with polymers that increase the hardness of the filled PMMA composite.[18,36]

In addition, inclusion of zirconia which has very high hardness (11.7 GPa i.e. 117000 MPa)[9] into low hardness acrylic resin (12.07 kg/mm^2 i.e. 118.37 MPa) with the use of silane coupling agent that bonds between them producing homogenous surface and enhancing the measured micro-hardness. However, at 5% zirconia, the micro-hardness of zirconia-modified acrylic resin has decreased because the clusters formed at this percentage (Fig. 2E) led to depletion of many areas on the specimen from hard zirconia particles with the net result of decreased micro-hardness of the specimen.

CONCLUSION

Within the limitation of this study, it may be concluded that silanized zirconia powder could be used to improve the properties of heat-polymerized acrylic denture base resin. The most appropriate percentages were 1% and 2%.

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