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ORIGINAL ARTICLE

Effect of The Addition of Zirconium Dioxide Filler on The Diametral Tensile Strength of Light-Activated Polymethyl-Methacrylate

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ABSTRACT

Introduction: Acrylic resin serves as a foundational material for fabricating aesthetically pleasing denture bases, with one of its choice is light-activated acrylic resin. However, one notable drawback, in comparison to heat-polymerized variants, is their relatively lower fracture resistance. Incorporating zirconia ceramic filler (ZrO_2) could enhance the diametral tensile strength (DTS) of the material that shall contribute to fracture resistance. **Materials and Method:** An experimental study was conducted to evaluate the effects of ZrO_2 on the DTS of light-activated acrylic resin. Cylindrical acrylic samples were prepared by incorporating ZrO_2 at concentrations of 3%, 5%, 7%, and 9% by weight. Cylinder samples with 6 mm diameter and 3 mm height size were subjected to DTS test using a Universal Testing Machine, operated at a constant crosshead speed of 1 mm/min, until the appearance of cracks indicated sample failure. **Result:** The incorporation of ZrO_2 filler resulted in a decrease in DTS compared to the control samples, with the positive control (28.799 MPa) and the negative control (30.409 MPa). In the treatment samples, the DTS values ranged from 8.573 to 13.560 MPa, with the highest strength with the 3% addition filler. These findings suggest that the ZrO_2 concentration must be carefully optimized to ensure a uniform dispersion within the resin matrix, avoiding any disruption to its structural continuity. **Conclusion:** An increase in the concentration of zirconium dioxide in light-activated acrylic resin is associated with a decrease in DTS. Further investigations are needed to explore alternative filler proportions or dispersion techniques to achieve potential reinforcement.

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Keywords: Acrylic resin light-activated, zirconium dioxide (ZrO_2), diametral tensile strength, filler, mechanical properties

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INTRODUCTION

Dental acrylic resin is widely used as the primary material for fabricating dentures due to its affordability and favourable aesthetic qualities (1). Structurally, acrylic resin is derived from ethylene derivatives and contain vinyl groups in its chemical structures (2). This material is preferred for various application in dentistry because of its gingiva-like colour and texture for improved oral aesthetics, relatively low water absorption, and minimal dimensional alterations (3). Chemically, dental acrylic resin is an ester derived from methacrylic acid (CH_2= $C(CH_3)COOH$) and acrylic acid (CH_2= CHCHOOH). It is utilised extensively as a base material in production of dentures, orthodontic bases, pediatric bases and maxillofacial devices. Light polymerized acrylic resins, which are based on urethane dimethacrylate (UDMA), are activated by light. Light-activated acrylic resins have the advantage of easy handling to control prior to curing, allowing sufficient time for manipulation and adaptation before polymerization begins (4). Additionally, light-activated acrylic resins offer reduced polymerisation shrinkage, lower level of residual monomers, and decreased bacterial adhesions when compared to heat- and chemically-polymerised alternatives (4).

However, the fracture resistance of acrylic resin remains inferior to that of metal-based denture materials (5). Diametral tensile strength is a critical property in denture base materials, as most fractures occur due to tensile stress (6). To address this limitation, the incorporation of zirconium dioxide (ZrO_2) ceramic fillers into acrylic resins has been investigated for its potential to enhance the impact and transverse strength of denture bases (6). Ceramic materials, including zirconia, possess a strong and directional ionic bond between metal and oxygen, providing significant strength but limited tolerance to deformation (7).

Ceramics fillers can be categorised into several types: silicon dioxide, alumina, and zirconium dioxide. Among these, zirconia ceramic filler is a zirconium dioxide (ZrO2) crystal that exhibits mechanical properties similar to metals. Zirconia has better fracture resistance than alumina and glass ceramic (7). Zirconium dioxide added to acrylic resins can increase impact and transverse strength more than adding alumina. The reinforcement of fracture resistance in denture bases is largely supported by improvements in transverse strength, impact strength, and tensile strength (7,8).

MATERIALS AND METHODS

Sample Preparation

Zirconium dioxide (ZrO_2; Zirai Guangzhou Hongwu Material Technology Co. Ltd., China), with submicron particle size, was utilised in this study as a reinforcing agent for light-activated acrylic resin. The experimental procedure involved two main stages: The initial blending of zirconium dioxide (ZrO_2) and light-activated acrylic resin and subsequent with compression testing following the American Dental Association (ADA) and American National Standards Institute (ANSI) specification no.27 to identify diametral tensile strength (DTS) in dental resin composites (9,10). For this study, 37 grams of light-activated acrylic resin (Shandong Huge Dental Material Co. Limited) was dissolved in 25 ml of tetrahydrofuran (THF) solvent (EMSURE®, ACS, Reag. Ph Eur) in a 250 ml beaker. The solution was stirred for 60 minutes using a magnetic stirrer (Thermo Scientific™ Cimarec™) (11). Prior to mixing, all materials were precisely weighed using a digital analytical balance (Sartorius). Zirconium dioxide (ZrO_2) was then introduced to the solution in four different weight percentages: 3%, 5%, 7%, and 9%. A 0% ZrO_2 concentration was designated as the positive control group, while a negative control group consisted of untreated acrylic resin without dissolution.

After thorough mixing, the prepared solution was stored in a light-shielded laboratory hood for 24 hours to prevent premature polymerization (11). The mixture was subsequently molded into cylindrical plastic molds, each with a diameter of 6 mm and a height of 3 mm, before being transferred to a desiccator for a curing period of 48 hours (11). Polymerization was performed using the Vertex Eco Light Box according to the manufacturer's instructions, with alternating exposures for 10 minutes each on the top and bottom surfaces.

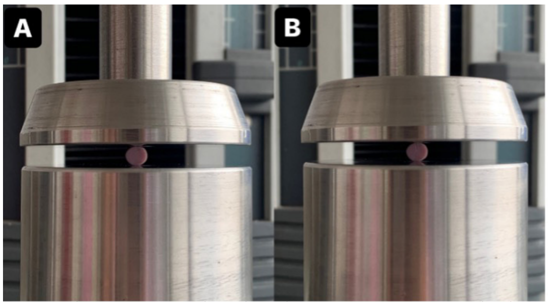

Fig 1: Samples are formed with a diameter of 6 mm and a height of 3 mm. Samples are formed after mixing completed, the mixture stored in a laboratory hood to keep light away for 24 hours. (A) positive control sample, (B) 3% sample, (C) 5% sample, (D) 7% sample, (E) 9% sample

After polymerization, the samples were polished with pumice to ensure a smooth finish, and the final dimensions of each sample were verified to be 6 mm in diameter and 3 mm in height, as illustrated in Fig. 1. A diametral tensile strength test was then conducted on the prepared samples to evaluate the effects of ZrO_2 incorporation on the mechanical properties of the acrylic resin.

Diametral Tensile Strength Test

The samples were subjected to mechanical testing using a Universal Testing Machine (Lloyd LRX-Plus Instruments, Fareham, England) operating at a constant crosshead speed of 1 mm/min until visible cracks appeared in the sample, as shown in Fig. 2B. The diametral tensile strength (σ) was determined based on the maximum load sustained (P) by the sample (D the diameter and T the thickness of the sample) prior to failure. The calculation of diametral tensile strength follows Formula 1:

$$\sigma = \frac{2P}{\pi DT}$$

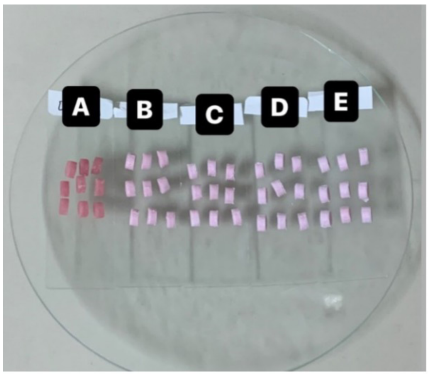

Fig 2: Diametral tensile test of samples on Universal Testing Machine. The samples were tested using a Universal Testing Machine (Lloyd LRX-Plus Instruments, Fareham, England) at a speed of 1 mm/min until cracks occurred in the sample, as depicted in Figure 1B. The diametral tensile strength is obtained from the maximum load acceptable to the sample (A) Before pressurized, (B) After pressurized and cracks appear on the sample

Statistical Analysis

The method of one way ANOVA and Tukey's follow-up test were conducted to determine the significancy statistical analysis of DTS value.

RESULT

The result of diametral tensile strength is obtained from the maximum load acceptable to the sample until cracking occurs in the sample as seen in Table I. The average diametral tensile strength value in group A1 as a control group without treatment was 30.409 MPa; in group A2 as a control with the same treatment procedure, it was 28.799 MPa; in group B with 3% treatment, it was 13.560 MPa; in group C with 5% treatment, namely 9.396 MPa; in group D with 7% treatment, namely 9.002 MPa; and group E with 9% treatment, namely 8.573 MPa.

Table I. Diametral Tensile Strength Test Results

Samples	N	Mean (MPa)	Std. deviation	Variance
Negative control (A1)	9	30.4089	3.26478	10.659
Positive control (A2)	9	28.7989	.81429	.663
3% ZrO_2 filler (B)	9	13.5600	1.78082	3.171
5% ZrO_2 filler (C)	9	9.3956	.85446	.730
7% ZrO_2 filler (D)	9	9.0022	1.51792	2.304
9% ZrO_2 filler (E)	9	8.5733	1.53529	2.357

The mean diametral tensile strength of group A1, the control group without treatment, was 30.409 MPa. Meanwhile, group A2, the control group that used THF as the same treatment procedure, gave 28.799 MPa of DTS result. These findings show that after treatment with tetrahydrofuran (THF), the diametral tensile strength (DTS) value of the treated samples decreased.

A one-way ANOVA test indicates that the diametrical tensile strength data in each group differ, with a p-value < 0.05. When comparing the diametral tensile strength of the negative control and positive control treatment groups to the 5%, 7%, and 9% treatment groups, Tukey's follow-up test findings demonstrate the significance of the p-value of statistical test results between groups. Compared to the other treatment groups, the DTS result for the 3% treatment group was considerably different. In the meantime, there was not a significant difference in the diametral tensile strength between the 5%, 7%, and 9% groups.

DISCUSSION

Diametral tensile strength is an important measure of fracture resistance in acrylic resins. In this study, the addition of zirconium dioxide fillers to acrylic resins directly did not increased the DTS of light-activated acrylic resins. The results indicate that, varying concentration of zirconium dioxide-3%, 5%, 7%, and 9%-led to a decrease in DTS compared to control samples.

Among the treatment groups, the highest DTS was observed with the addition of 3% zirconium dioxide, achieving a value of 13.560 MPa. This was followed by 5% zirconium dioxide with a DTS of 9.395 MPa, 7% zirconium dioxide at 9.002 MPa, and finally, 9% zirconium dioxide, which exhibited the lowest DTS at 8.573 MPa. These findings contrast with earlier research, which showed that lower concentrations of zirconium dioxide (e.g., 2%, 3%, 5%, and 7%) could increase DTS in acrylic resins. In this study, however, a clear trend was observed: as the concentration of zirconium dioxide increased, the DTS of the light-activated acrylic resin decreased.

This indicates that higher concentrations of zirconium dioxide result in a further attenuation of DTS in light-polymerized acrylic resins. None of the zirconium dioxide treatment groups surpassed the DTS values of the control samples. The positive control sample displayed a DTS of 28.799 MPa, and the negative control sample had a DTS of 30.409 MPa, underscoring the limitations of the current approach to enhancing tensile strength with zirconium dioxide fillers.

When added to polymethyl methacrylate (PMMA), zirconia filler particle of varying size aid in the interstitial matrix filling process, potentially enhancing the material mechanical properties. The proportion of filler is a critical factor, it must be carefully calculated to ensure that the zirconia particles are uniformly dispersed within the resin matrix without disrupting its structural integrity. Excessive filler content does not always correlate with improved strength. In fact, beyond a certain threshold, an increase in filler may lead to a decrease in material strength. This phenomenon occurs because, once the resin matrix reaches a saturation point, any additional filler can disrupt matrix continuity, resulting in weakened mechanical properties (12,13).

The statistical analysis of Tukey's follow-up test, showed that the samples of the positive control and negative control groups had significant differences with the treatment group that added with zirconia fillers. However, among the treatment groups with 5%, 7%, and 9% zirconia content, the p-values exceeded 0.05, indicating that while differences in diametral tensile strength (DTS) existed, they were not statistically significant. The results suggest that the method of incorporating zirconia fillers into the acrylic resin plays a crucial role in the mechanical outcomes.

One factor that may have influenced the results is the use of tetrahydrofuran (THF) as a solvent for the light-activated acrylic resin. The THF-dissolved control group exhibited lowest DTS values compared to the untreaded group, indicating a potential alteration in the resin's structure. At room temperature, PMMA chains are still packed randomly. According to the findings, PMMA and THF could be described to combine and generate an amorphous structure that differs from that of bulk PMMA. The PMMA chains are thought to be randomly organised because their interaction with THF weakens their bond, which limits their interaction with the zirconia filler (14). Therefore, there is a concern that it may adversely impact the DTS results in the treatment groups.

The interaction between PMMA and THF could increase chain mobility and reduce inter-chain interactions, potentially affecting the integration of zirconia fillers. THF is hypothesized to enhance chain mobility, thereby reducing the efficiency of filler incorporation. Consequently, achieving a homogeneous dispersion of zirconia within the resin matrix is crucial to improve loading efficiency and reduce localized stress points (14,15). The inability to achieve uniform distribution may explain the observed decrease in DTS values with increasing filler concentrations from 3% to 9%.

This research quantitatively demonstrates a point of diminishing returns when zirconia content exceeds 3%. This indicates that filler concentrations greater than 3% are not just neutral but actively detrimental to the DTS of light-activated acrylic resins. This finding underscores the necessity for precise optimization of filler content, providing a more nuanced understanding of filler-reinforced acrylic systems. It is supporting the theory that an oversaturated matrix fails to integrate fillers effectively. This explanation is not only valuable for dental materials but may also apply broadly to other polymer-filler composite systems in materials science.

A strong bond between zirconia and PMMA, could also be achieved through surface modification, which is known as silanization. This modification could also increase the transverse and impact strength in the resin-based materials (16,17). However, in this research still not used this type of modification as the primary objective was to investigate the direct interaction of zirconia filler and PMMA matrix first. To ensure that the filler diffuses evenly throughout the resin matrix without disrupting its continuity, the treatment and the amount used for reinforcing must be optimised.

CONCLUSION

The addition of zirconium dioxide fillers to light-activated acrylic resins has not been able to increase diametral tensile strength. The higher the concentration of zirconium dioxide added, the lower the value of diametral tensile strength of light-activated acrylic resin. Future research could be conducted to optimized the treatment and the amount of reinforced materials. It should also consider surface treatments to enhance the filler-matrix interface, aiming to improve the mechanical performance of the composite. Optimization of filler content and dispersion techniques is necessary to ensure even distribution throughout the resin matrix, which could prevent disruption of the matrix's continuity and lead to enhanced mechanical properties.

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