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Comparative study on the flexural properties and hardness of a PMMA-based dental composite

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ABSTRACT: This work investigates the flexural properties and surface hardness (SH) of poly(methyl methacrylate) (PMMA)-based dental materials developed with urethane dimethacrylate (UDMA) as a crosslinker and zirconia (ZrO_2) as a filler. The composites were prepared using heat-polymerizing PMMA with varying zirconia loadings (0.25–2.50 wt%). Flexural strength (FS) and flexural modulus (FM) were determined through a three-point bending test, while SH was measured using microhardness testing. The addition of zirconia improved FM and SH significantly ($p < 0.05$) and slightly enhanced FS. Incorporating UDMA into unfilled PMMA increased the FS and SH but had no effect on the FM. However, when both ZrO_2 and UDMA were added to PMMA, their impact on the mechanical properties varied. In terms of FS, PMMA exhibited a lower value compared to the control group. Conversely, for SH, their combination resulted in better properties. As for the FM, the addition of ZrO_2 continued to raise it, with no influence from the presence of UDMA. These results emphasize that while ZrO_2 and UDMA can independently enhance the mechanical properties of PMMA, their combination may diminish FS. Consequently, the study recommends cautious use of this combination in dental materials where FS is critical.

KEYWORDS: methyl methacrylate, urethane dimethacrylate, zirconia (ZrO_2), flexural properties, surface hardness

INTRODUCTION

Artificial teeth in Thailand are constructed using two main types of imported materials: acrylic resins and porcelain. While acrylic-based artificial teeth have notable wear-related downsides in clinical settings [1, 2], they offer the benefit of easy grinding without fracturing. They can be adjusted and polished easily, adhere well to the denture base, and cause less wear on natural occlusion teeth [3, 4]. Additionally, acrylic-based teeth are relatively affordable. Due to these advantages, they are still widely utilized despite their drawbacks in terms of surface hardness (SH) [5] and wear resistance compared to porcelain teeth [2].

In dental applications, the primary components used to produce acrylic materials consist of two main parts: a liquid methacrylate monomer and poly(methyl methacrylate) (PMMA) powder. These two components are combined and polymerized with the help of an initiator [4, 6]. Additionally, it is common to include tertiary amine catalysts in the liquid fraction to expedite the decomposition of the initiator [7, 8].

In response to the issue of mechanical properties in acrylic artificial teeth, several approaches have been explored. These include incorporating crosslinking agents and reinforcing the polymer matrix with inorganic fillers to create composite teeth [9]. The inclusion of crosslinking agents has been successful in enhancing tooth strength, hardness, and resistance to crazing [9], but has led to a decrease in denture adhesion strength [10]. On the other hand, the addition of inorganic fillers has proven beneficial in improving the properties of composite materials. The effectiveness of this improvement depends on the type and quantity of fillers used [11, 12]. Composite teeth with inorganic inserts usually offer superior strength and hardness compared to traditional acrylic teeth.

Zirconium dioxide (ZrO_2), also known as zirconia, a metal oxide with various advantages such as high mechanical strength, toughness, stiffness, wear resistance, chemical tolerance, and good thermal stability, is valuable for reinforcing dental materials like denture bases, and research has shown that incorporating ZrO_2 nanoparticles into PMMA polymer can enhance its physical and mechanical properties [13–15]. Yttrium-oxide-partially stabilized zirconia, a fully tetragonal fine-grained ceramic material achieved by incorporating around 2 to 3 mol% yttrium oxide (Y_2O_3) as a stabilizing agent, is widely utilized in dentistry for its remarkable qualities, such as higher flexural strength (FS), excellent mechanical properties, biocompatibility, and aesthetic potential [16]. This material finds extensive applications in various dental prosthetics, including root canal posts, frameworks for all-ceramic posterior crowns, as well as fixed partial dentures (FDPs), dental implants, and implant abutments [17–21].

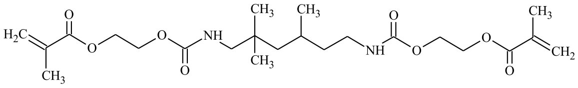
A crosslinking agent is a type of monomer with a minimum of two polymerizable groups per molecule, where each active group can engage in polymer chain growth during the polymerization process, leading to the formation of a crosslink between two chains [22]. Dimethacrylates are monomers that undergo polymerization to form a highly crosslinked network, making them suitable for dental applications [23, 24]. Among the various crosslinking agents used for methyl methacrylate (MMA), ethylene glycol dimethacrylate (EGDMA) is the most frequently employed, creating a brief cross-linkage between the linear monomer chains [25]. The other dimethacrylate monomers commonly used in commercial dental materials include dimethacrylate 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy) phenyl] propane (Bis-GMA), Urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA) [24, 26, 27]. The overall network elasticity of the dimethacrylate monomer depends on its molecular elasticity. Aliphatic hydrocarbon and oligoether chains contribute to increased polymer elasticity, while cycloaliphatic and aromatic units lead to enhanced polymer stiffness [24].

Although numerous studies have focused on enhancing PMMA, limited data are available on characterizing the combination of fillers and crosslinkers. This research aims to investigate the flexural properties and SH of a dental material based on PMMA crosslinked with UDMA and combined with ZrO_2 filler. The findings of this study could contribute to a better understanding of how both the dimethacrylate crosslinker and filler affect the enhancement of composite resin.

MATERIALS AND METHODS

Materials

PMMA powders with a molecular weight of 350,000 g/mol purchased from Sigma-Aldrich (St. Louis, MO, USA) were used in this research. The MMA monomer and UDMA were also supplied by Sigma-Aldrich and stored at 4 °C. The chemical structure of UDMA is shown in Scheme 1. Materials were made heat-curable by adding benzoyl peroxide (BPO), *N*, *N*-dimethyl-*p*-toluidine (DMPT) (Sigma-Aldrich), and hydroquinone (HQ) (Ajax-Finechem, New South Wales, Australia). The schematic representation of the polymerization process in the study is provided in Scheme 2. ZrO_2 nanopowders (40 nm) (grade TZ-3Y-E, Tosoh USA, Inc., Grove City, OH, USA), partially stabilized ZrO_2 powder with uniform dispersion of 3 mol% yttria, were used as inorganic fillers for preparing the nanocomposite. Although the size of ZrO_2 is 40 nm reported from manufacturing, it is presented in agglomeration form in Fig. 1. The SEM image shows that the tetragonal zirconia's secondary particles have a uniform, spherical morphology.



Scheme 1 Urethane dimethacrylate (UDMA) structures.

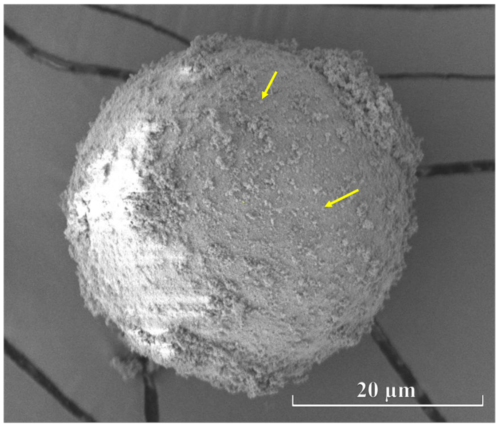


Fig. 1 SEM image of TZ-3Y-E ZrO_2. ZrO_2 powders revealing a hierarchical structure: individual particles are nanosized, but these then aggregate into larger secondary particles. The yellow arrow points to the ZrO_2 primary nanoparticle.

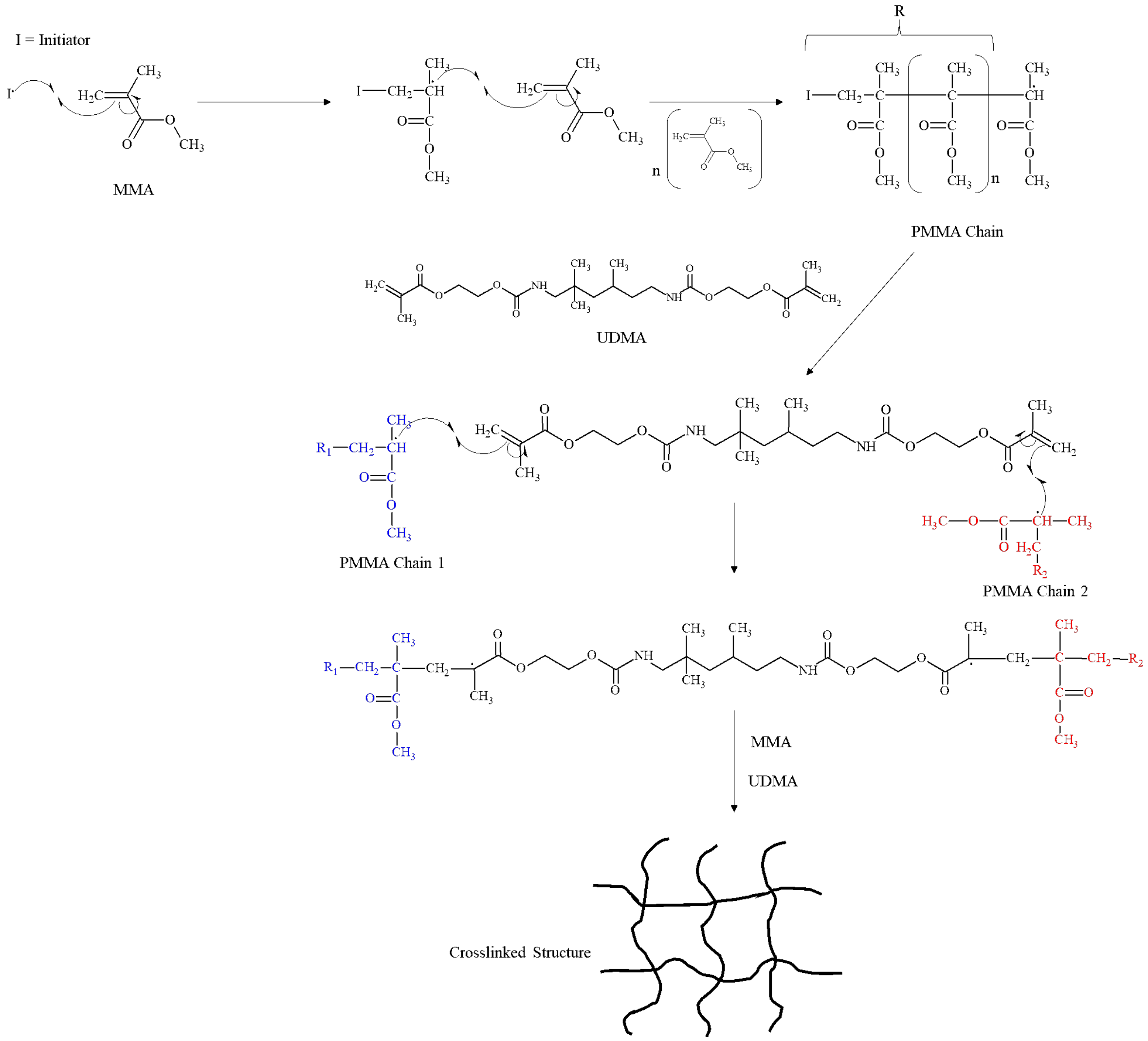
Sample preparation

Heat-curing PMMA-based materials were prepared via a dough-forming method. The powder and liquid parts were mixed at the ratio of 3:1 Powder/Liquid ratio (by weight). The powder component mainly consisted of PMMA powder and ZrO_2 filler added with 0.90 wt% BPO, whereas the liquid component was a mixture of MMA monomer added with 0.30 wt% DMPT and 60 ppm HQ. The pre-weighed ZrO_2 was added to the powder part and thoroughly blended to achieve an equal distribution before being mixed with the liquid part. The powder and liquid parts were mixed manually in a silicone cup at room temperature and stirred until homogeneous within 30 s. Upon reaching the dough state, the mixture was poured into the mold and then shaped using a compression molding machine at 90 °C for 30 min and cooled for 5 min. The composition details of the PMMA powder and filler used in the powder component are described in Table 1.

To prepare the crosslinked materials, UDMA was included in the liquid phase prior to blending it with the powder phase. Subsequently, the mixture was

Table 1 Composition of PMMA powder and filler used in the powder component.

ZrO_2 Content (wt%)	Powder (g)			Liquid (g)
	PMMA Powder	ZrO_2	BPO	
Control (0.00)	7.410	0.000	0.090	2.500
0.25	7.385	0.025	0.090	2.500
0.50	7.360	0.050	0.090	2.500
1.00	7.310	0.100	0.090	2.500
1.50	7.260	0.150	0.090	2.500
2.00	7.210	0.200	0.090	2.500
2.50	7.160	0.250	0.090	2.500



Scheme 2 Polymerization and crosslinking processes.

molded using the aforementioned method.

Characterization

FS and FM were established according to ISO 10477:2020. Five measurements were performed for each study group, based on rectangular samples (dimensions: 2 × 2 × 25 mm). The samples were stored in a glass bottle with the addition of RO water and incubated at 37 °C for 24 h. The sample was wiped dry prior to testing. The three-point flexural properties were measured by the Instron 5566 Universal Testing Machine (UTM) (Instron Co., Norwood, MA, USA) with the crosshead speed of 1 mm/min and a length support span of 20 mm used in this test. The equations for calculating FS and FM are as follows:

Formula for FS (σ):

$$\sigma = \frac{3FL}{2bd^2} \tag{1}$$

Formula for FM (E):

$$E = \frac{FL^3}{\delta 4bd^3} \tag{2}$$

where

F is the maximum force applied,

δ is the deflection,

L is the length of the support span,

b is the width of the sample,

d is the thickness of the sample.

The fracture surfaces of samples after the flexural test were observed by SEM using a TESCAN Mira 4

(TESCAN, Brno, Czech Republic). The fracture surfaces were sputter-coated with gold prior to examination.

The SH test was performed with a Falcon 608FA (INNOVATEST Europe BV, Maastricht, The Netherlands). The force was applied to the midpoint of the base material by a diamond tip. The diagonals of the resulting indentation were then measured by a microscope and calculated to provide hardness values. The values reported were the average of five spots for each condition under a load of 2942 mN.

Statistical analysis

All statistical analyses were performed using IBM SPSS Statistics (version 26) analysis software, with all data expressed as the mean±standard deviation. Data on the FS, FM, and SH of PMMA mixed with ZrO_2 and mixed with ZrO_2 and UDMA were of normal distribution and analyzed using one-way analysis of variance (ANOVA). The multiple comparisons of means were deployed in the case of significant differences ($p<0.05$) using Tukey's HSD.

RESULTS

The impact of inorganic fillers on dental materials made from PMMA-based resin was initially observed. The results revealed that ZrO_2 led to an improvement in FS, FM, and SH, as shown in Fig. 2. The FS slightly increased when ZrO_2 content increased, reaching the maximum value at 1.00 wt% loading and becoming significantly higher in comparison to the control ($p <$ 0.05). The same trend could also be observed on FM, but the maximum value shifted to 1.50 wt% loading and was also significantly higher than the control ($p <$ 0.05). Moreover, with increasing ZrO_2 content, higher SH was found. The highest SH value was detected at 2.00 and 2.50 wt%, significantly higher than the control ($p < 0.05$) but with no sign of decline. It might have increased further than this point if the loading of ZrO_2 had continued to increase. The maximum mean values in each mechanical property between the composite and control samples were significant ($p <$ 0.05).

The distribution of ZrO_2 fillers within PMMA is depicted in Fig. 3. It shows that the particles adhered well to the PMMA matrix. Moreover, the fracture surface of low ZrO_2-loaded PMMA showed a more uniform fracture when compared to the unloaded PMMA samples, leading to the development of mechanical properties.

The effects of ZrO_2 on FS, FM, and SH on cured PMMA composite were compared with the control, cured PMMA, and uncured PMMA composite in Fig. 4. PMMA-based with UDMA showed the highest FS, while PMMA with UDMA and ZrO_2 exhibited the lowest FS (Fig. 4a) ($p < 0.05$). For FM, the highest value was PMMA with ZrO_2, but did not show any statistical difference when PMMA was added with UDMA and ZrO_2 (Fig. 4b) ($p > 0.05$). The SH of PMMA with 10 wt% UDMA and 2.5 wt% ZrO_2 provided the highest values, and all modifications significantly improved SH when compared to the control (Fig. 4c) ($p < 0.05$).

The fracture surfaces of the control, cured PMMA, PMMA composite, and cured PMMA composite are presented in Fig. 5. The incorporation of UDMA in PMMA presented a smooth and homogeneous fracture. Samples with the addition of ZrO_2 displayed more uniform fractures when compared with the control.

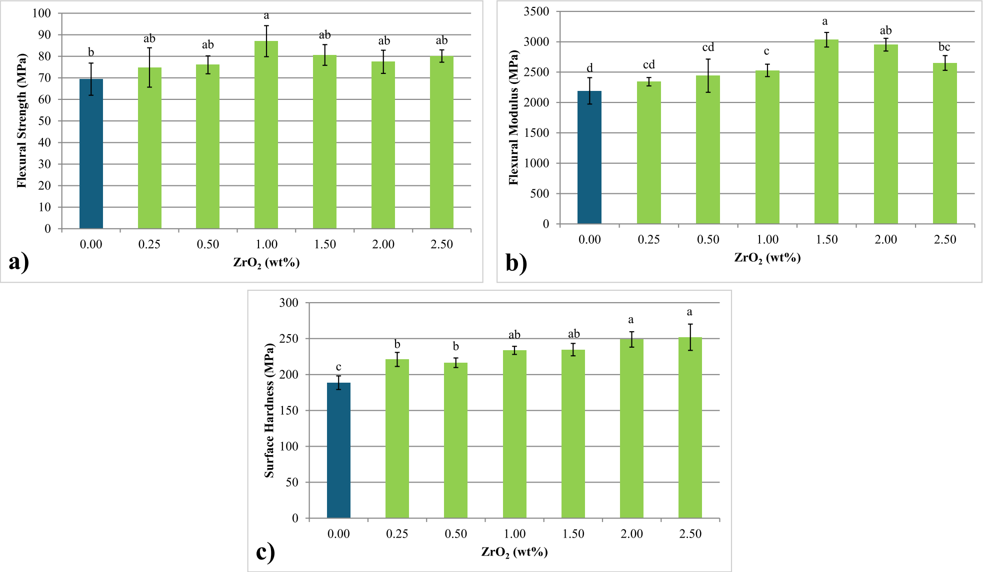


Fig. 2 Flexural strength (a), flexural modulus (b), and surface hardness (c) of PMMA mixed with ZrO_2. Bars with different letters are significantly different at $p < 0.05$.

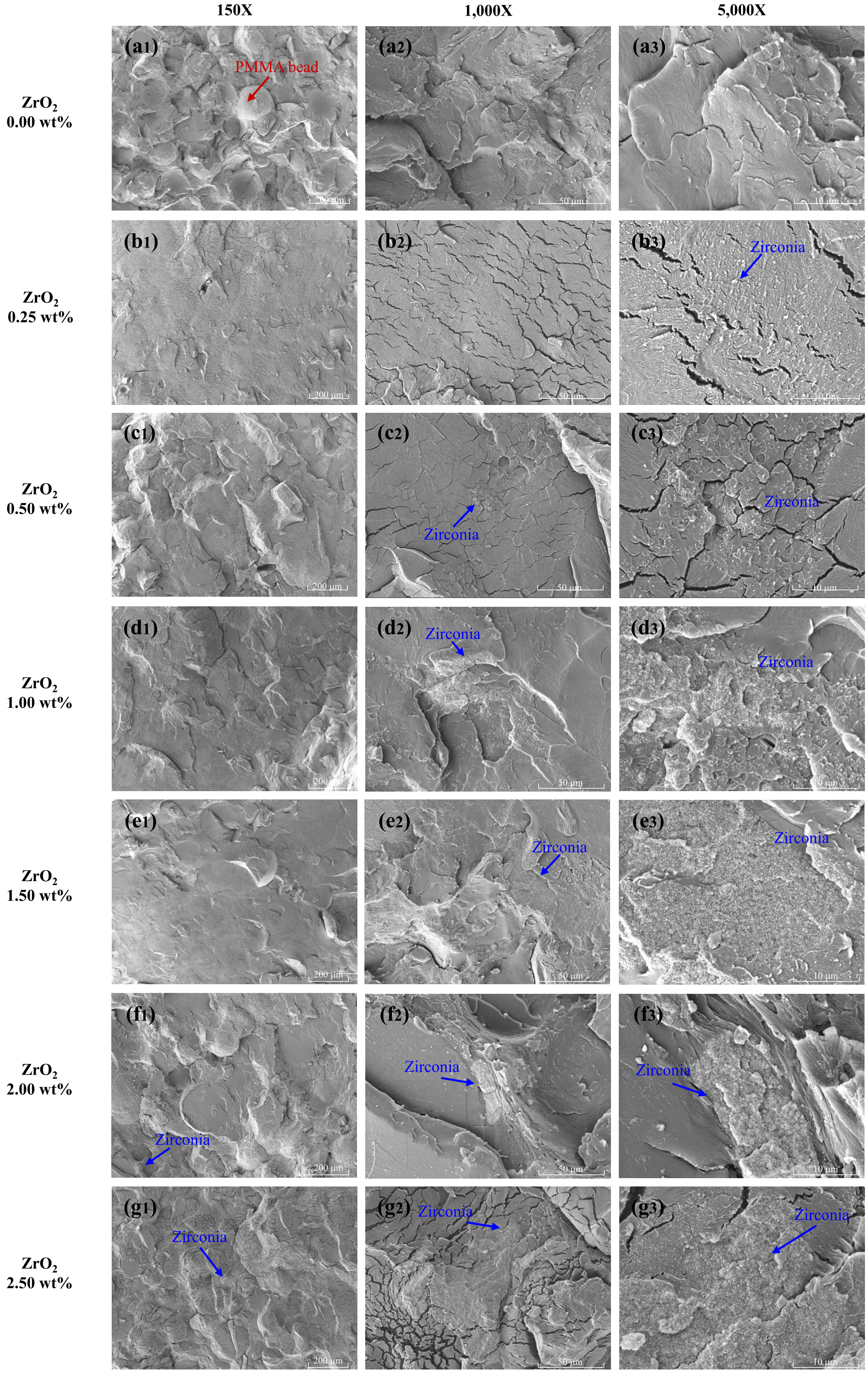


Fig. 3 SEM images showing the surface fracture of TZ-3Y-E ZrO$_2$/PMMA composite at different ZrO$_2$ loadings (a) 0.00 wt%, (b) 0.25 wt%, (c) 0.50 wt%, (d) 1.00 wt%, (e) 1.50 wt%, (f) 2.00 wt%, and (g) 2.50 wt%. The numbers (1–3) after the alphabet indicate different magnifications, where 1 = 150X, 2 = 1,000X, and 3 = 5,000X. The red arrow signals the PMMA bead, while the blue arrow points to ZrO$_2$ reinforcements.

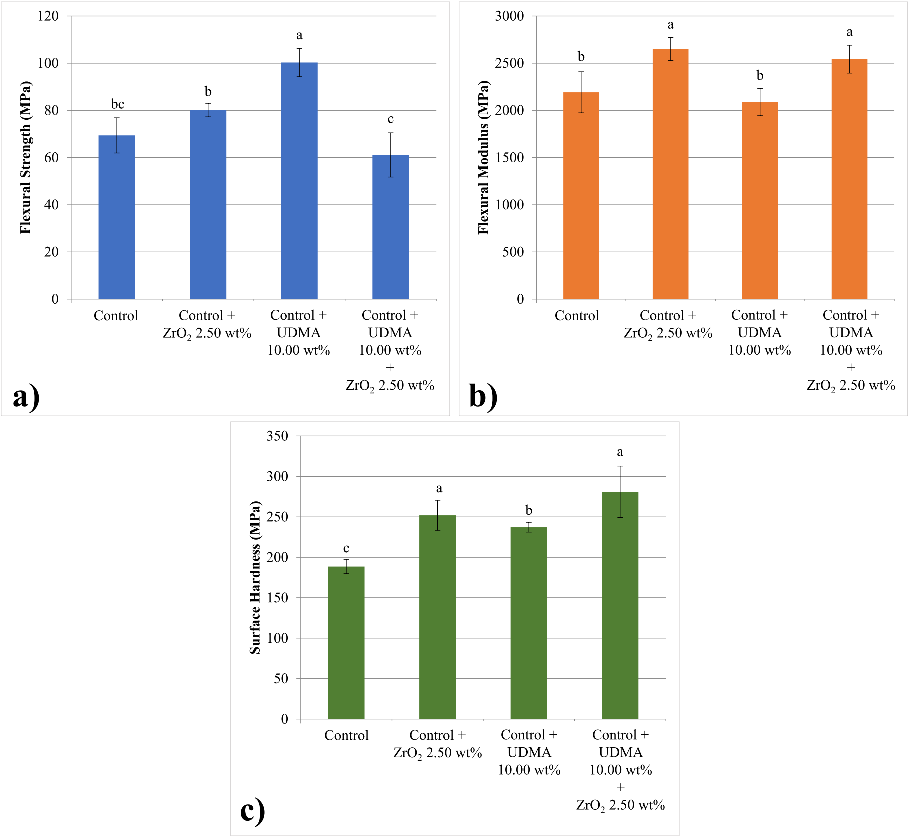


Fig. 4 Flexural strength (a), flexural modulus (b), and surface hardness (c) of PMMA mixed with ZrO_2 and UDMA. Bars with different letters are significantly different at $p < 0.05$.

However, the fracture of cured or crosslinked PMMA with ZrO_2 showed the PMMA beads clearly and indicated a poor interface between the PMMA beads and the matrix component.

DISCUSSION

For the mechanical properties of dental materials made from PMMA without UDMA, the FS and FM values slightly increased when the ZrO_2 content increased and reached the maximum value at a specific loading, but with no sign of decline in SH, and might have increased further. The decline in strength with increased filler percentage can be attributed to a combination of factors, including a higher number of defects influencing the material's strength, clustering of particles within the resin, and the presence of extra filler particles causing interruptions in the resin matrix continuity once it reaches saturation [14, 15].

Earlier research has demonstrated that the addition of fillers to the polymer matrix leads to an improvement in mechanical properties. This enhancement is significantly affected by factors such as the type of filler, arrangement of filler particles, and their ability to bond with the matrix [1, 28–31]. Gad et al [28] found that the addition of 7.5% nano-ZrO_2 to acrylic resins resulted in enhanced FS. Similarly, Reyes-Acosta et al [13] observed an increase in the thermal properties, hardness, and elastic modulus of PMMA by introducing a small amount of ZrO_2 nanoparticles. Salih et al [32] improved FS from 80.792 MPa to 143.498 MPa and FM from 2.05 GPa to 8.63 GPa by adding 3%v/v ZrO_2 to Vertex™ Castavaria PMMA matrix, produced via the autoclave method.

SEM images revealed the fracture of unfilled PMMA (Fig. 3(a1)) and clearly presented that they were PMMA beads dispersed in the newly formed PMMA matrix network. The presence of PMMA beads in a perfect sphere shape, as well as the hole in loosened PMMA beads, confirms the poor adhesion between PMMA beads and the PMMA matrix. At low ZrO_2 loading, the PMMA beads were hardly visible on the fracture surface compared to the unloaded sample (Fig. 3(a1)). The uniform fracture observed in low-ZrO_2 PMMA was due to the energy absorption ability of tetragonal zirconia polycrystal; it transitioned to the monoclinic phase with the application of stress [33]. The SEM images also revealed that although the nano-ZrO_2 was presented in agglomeration form (Fig. 1) at the beginning, they were crushed and distributed through the matrix phase during fabrication and pre-

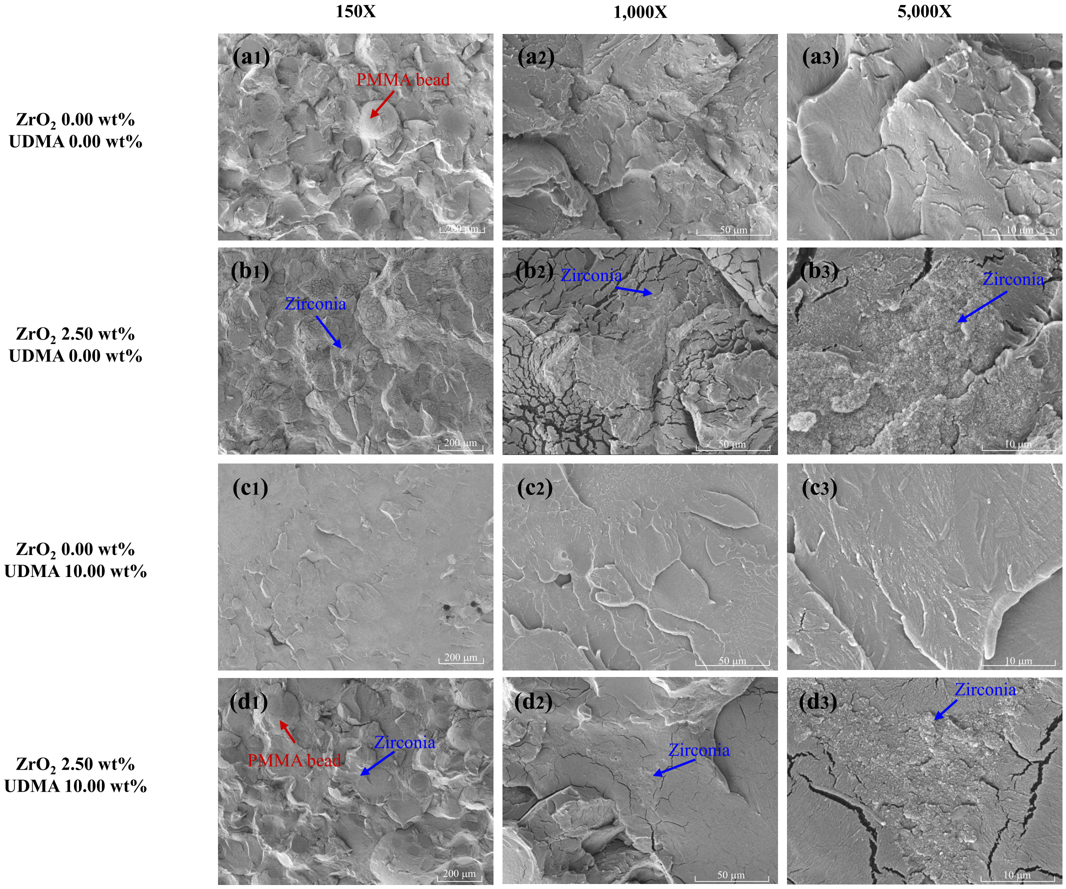


Fig. 5 SEM images showing the surface fracture of (a) uncured and unfilled PMMA, (b) PMMA composite with 2.50 wt% ZrO_2, (c) cured PMMA with 10.00 wt% UDMA, and (d) cured PMMA with 10.00 wt% UDMA loaded with 2.50 wt% ZrO_2. The numbers (1–3) after the alphabet indicate different magnifications, where 1 = 150X, 2 = 1,000X, and 3 = 5,000X. The red arrow signals the PMMA bead, while the blue arrow points to ZrO_2 reinforcements.

sented at the nano-size level. At a higher percentage, the investigation revealed that ZrO_2 exhibited agglomeration, resulting in an uneven dispersion throughout the workpiece. The agglomeration became noticeable around 1.00–1.50 wt% samples, then after this concentration, the fracture surface agglomerates of ZrO_2 could be clearly observed. This agglomeration effect on FS and FM led to a decline in both values. The breaking of the ZrO_2 cluster provides evidence to support that the force was not transferred to the matrix but went straight to the defect in the cluster, allowing it to break with lower force. For SH, the agglomeration did not have any effect on this property and still increased with higher ZrO_2 content.

Denture material polymers are typically composed of a mixture of PMMA powder and MMA liquid, with the addition of small quantities of crosslinker in the monomer composition. The polymerization process results in the creation of a semi-IPN (interpenetrating polymer network) situated between the PMMA beads and the crosslinked polymer matrix [34]. The main purpose of incorporating crosslinking agents into PMMA is to improve the material's ability to withstand cracking [35]. These techniques have been extensively employed to address significant shortcomings in linear PMMA, including low strength, rigidity, modulus, and stiffness [36]. Crosslinking induces alterations in various characteristics such as glass transition, thermal degradation, particle size, pore volume, pore size, surface area, craze resistance, and creep [37]. Consequently, the resulting crosslinked polymers exhibit heightened thermal stability, attributable to the three-dimensional network's capacity to constrain the mobility of polymer chains, leading to improved abrasion and heat resistance in the resulting materials [36, 38].

According to ISO 10477:2020, the minimum FS required is 50 MPa, and all samples in this study meet the criteria. Although FM and SH requirements are not mentioned in the standard, further study on the crosslinker addition focuses on the improvement of SH, resulting in the choice of 2.50 wt% ZrO_2. Moreover, our previous study (data not shown) suggests that FS can be developed by adding UDMA up to 10 wt%.

The utilization of both the crosslinker and filler does not appear to yield any superior outcomes compared to the use of each of them individually. The incorporation of only UDMA or ZrO_2 significantly improves the FS of PMMA. When UDMA monomer is combined with ZrO_2, the resulting composite exhibits a decrease in FS, and the value is even inferior to the control. The strength of UDMA is not solely reliant on chemical crosslinking but also on physical crosslinking resulting from hydrogen bonding. The decrease in FS can be attributed to a reduction in both chemical and physical crosslink density. According to a review by Barszczewska–Rybarek [24], the addition of UDMA to copolymers can lead to an increase in both tensile and FS. They confirm that in terms of FS, the presence of physical crosslinks is more critical to their strength. Regarding this present study, when UDMA is added, the samples show improved FS, but the addition of ZrO_2 causes a significant drop. This suggests that the insertion of filler may disrupt these physical crosslinking and initiate defects in the material. In terms of FM, materials with ZrO_2 exhibit a higher FM compared to those without. However, no significant difference in FM is revealed between the cured and uncured states for either the PMMA materials or PMMA/ZrO_2 composites. The FM of both cured and uncured PMMA materials falls within the same range, and similarly, the FM of cured and uncured PMMA/ZrO_2 composites also remains consistent. The increase in FM is an inherent property of ZrO_2. Fortunately, the combination of UDMA and ZrO_2 demonstrates an improved SH value.

When adding ZrO_2 to PMMA without UDMA, the fillers become crushed and adhere well to the PMMA matrix. However, the fracture of cured PMMA/ZrO_2 composite at 150X also presents PMMA beads and even shows poor adhesion between the beads and matrix. At higher magnification (Fig. 5(d2) and Fig. 5(d3)), poor filler distribution at the boundary between the matrix and filler can be sharply distinguished. The main reason for these findings is the chemical inertness of unmodified nano-zirconia, which hinders its ability to form robust bonds with the resin matrix [39]. This behavior is also responsible for the drop in mechanical properties.

ZrO_2 and UDMA act through different pathways. ZrO_2 performs as a rigid inclusion, increasing FM and SH, while UDMA increases FS and SH by creating a denser network through chemical and physical crosslinking. When combined, FS decreases below the control sample. This is because: (i) the rigid fillers interfere with the physical crosslinks created by UDMA, and (ii) unmodified ZrO_2 causes defects at the interface, showing up as clear boundaries between the PMMA beads and matrix and nonideal filler dispersion. For applications prioritizing stiffness and hardness, ZrO_2-filled PMMA without UDMA is recommended. Where FS is critical, UDMA-modified PMMA without ZrO_2 is preferable. Combining the two should be accompanied by interfacial engineering to avoid the FS penalty. This study uses unmodified nano-zirconia prone to agglomeration and manual blending/compression molding. Future work will aim to functionalize ZrO_2 and improve dispersion to control agglomeration and recover strength.

CONCLUSION

The influence of ZrO_2 on FS showed an improvement, with an obvious increase in FM values, peaking at 1.50% by weight. As anticipated from the filler, SH presented a notable increase, with no apparent limit within the scope of this study. On the other hand, the presence of UDMA in traditional PMMA evidently results in the promotion of FS and SH values, with no enhancement of FM. Both the crosslinker and filler demonstrate a modest development in FS and FM, while significantly enhancing the SH of the PMMA material. Nonetheless, subsequent examinations reveal that the combined influence of these components on mechanical properties may be different. The FS of PMMA appears to be lower compared to the control group, probably due to the disruption in the crosslinking network caused by the filler. In contrast, when it comes to SH, their combination gives an improved hardness level. In terms of FM, the addition of ZrO_2 continues to increase it, unaffected by the presence of the crosslinker.

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