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The efficacy of reinforcement of glass fibers and ZrO₂ nanoparticles on the mechanical properties of autopolymerizing provisional restorations (PMMA)



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KEYWORDS

Glass fibers; Provisional restoration; ZrO₂ nanoparticles **Abstract** *Objective:* to investigate and compare the reinforcing effects of glass fibers (GFs) and ZrO₂ nanoparticles at different ratios on the Flexural Strength (FS), Microhardness (MH), and Surface Roughness (SR) of autopolymerizing provisional PMMA.

Methods: A total of one hundred and twenty specimens of autopolymerizing PMMA were prepared for FS, MH, and SR tests and grouped as follows: no additives (control group), for the tested groups, different ratios of GFs and ZrO₂ at 5% of autopolymerizing PMMA were incorporated. The ratios of GFs/ZrO₂ nanoparticles were 0%-5%, 1%-4%, 2%-3%, 2.5%-2.5%, 3%-2%, 4%-1% and 5%-0% (n = 5). The FS was evaluated using the three-point bending test, MH was evaluated using the Vickers microhardness tester and SR was evaluated using a contact-type profilometer. Data were analyzed using ANOVA, Tukey's test, and Person correlation at 0.05 level of significance.

Results: The unreinforced group had the lowest FS, MH, and SR mean values followed by (0% GFs + 5% ZrO₂), (1% GFs + 4% ZrO₂), (2% GFs + 3% ZrO₂), (2.5% GFs + 2.5% ZrO₂), (3% GFs + 2% ZrO₂), (4% GFs + 1% ZrO₂) and (5% GFs + 0% ZrO₂) which had the highest values. *Conclusion:* Hybrid reinforcement with GFs, ZrO₂ nanoparticles, or a combination of them effectively improved flexural strength and microhardness of autopolymerizing provisional PMMA that would create provisional restorations with extended clinical service. GFs demonstrated superior reinforcing effects compared to ZrO₂ nanoparticles. However, reinforcement with 2.5–5% GFs increased the surface roughness for provisional restoration.

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1. Introduction

Provisional or temporary restorations are essential treatments in fixed prosthodontics. They facilitate biomechanical and biological refinement before the manufacture of the final restoration, protect the prepared abutment teeth, and maintain esthetics, function, and periodontal health. They also aid in preserving occlusal parameters, especially in complex restorative rehabilitation (Burns et al., 2003).

Provisional restorations are exposed to masticatory forces inside the mouth, hence studying their mechanical characteristics is necessary to evaluate the long-term performance, particularly in some clinical situations including, long-term fixed restorations in full mouth rehabilitation, treatments for patients with parafunctional habits as well as oral implantation treatments (Astudillo-Rubio et al., 2018; Naik and Mathur, 2017).

Over the period of many years, numerous dental materials were developed for provisional restorations such as polymethyl methacrylate, polyvinyl methacrylate, and bis-acryl composites. Traditional autopolymerizing PMMA is widely used to fabricate provisional restorations owing to their acceptable esthetics, low cost, lightweight, and biocompatible (Akay and Avukaty, 2019; Jamel and Yahya, 2022). However, their clinical use is limited by unsatisfactory properties that may cause an unwanted effect on their clinical performance (Astudillo-Rubio et al., 2018). PMMA-based materials are relatively weak and brittle. They have a tendency to mechanical failure resulting in a high risk of cracks and fractures. Several clinical studies revealed that autopolymerizing PMMA has high polymerization shrinkage, stainability, residual monomer release, and poor wear resistance (Alhotan, et al., 2021 a, Cascione et al., 2021; Jamel and Yahya, 2022).

Composite resins have been commonly used because they have many beneficial properties over traditional autopolymerizing PMMA, for instance, good mechanical properties, excellent aesthetics, and less polymerization shrinkage (Mehrpour et al., 2016). Nowadays, Rubberized urethane resins, CAD/ CAM and three-dimensional additive printers PMMA, and composite resins have been introduced. These materials give more attention to anatomic details with extremely lower polymerization shrinkage, perfect marginal adaptation, and better mechanical properties than traditional PMMA (Revilla-León et al., 2021; Vellingiri et al., 2020).

With the advancement of technology, numerous techniques and materials have been used to promote the characteristics of PMMA (Hamouda and Beyari, 2014; Zhang et al., 2014). These techniques included the addition of various fibers, for instance, polyethylene, carbon, and glass fibers in addition to different nanoparticles (Gopichander et al., 2015; Chang et al., 2019) and the modification of these particles by preimpregnation with silane coupling agents or resin monomers (Abdulrazzaq et al., 2018; Al-Thobity, 2020).

Glass fibers (GFs) are silica-based glass inorganic materials added to different resins to produce GFs-reinforced composite materials. These materials offer advantages such as high toughness, optimum flexural strength, and biocompatibility. Impregnating GFs using a silane coupling agent can successfully facilitate adherence of the GFs to the resin matrix and promote fracture resistance (Gad et al., 2017; Safwat et al., 2021). Other authors tried to reinforce the PMMA acrylic resins with metal oxide nanoparticles. Nanotechnology recently introduces a new era for reinforcing materials; because of nanomaterials' physical, chemical, mechanical, and biological properties (Elhatery, 2019; Gad et al., 2019a; Vikram and Chander, 2020). Nowadays, zirconium oxide (ZrO₂) nanoparticles have been applied as a reinforcing agent owing to their biocompatibility, advantageous mechanical properties as well as antifungal properties of ZrO₂ (Alhavaz et al., 2017; Akay and Avukat, 2019).

Adding GFs or ZrO_2 in a certain amount is needed to reinforce autopolymerizing PMMA and enhance its properties such as fracture toughness, flexural strength, elastic modulus, hardness, wear resistance, water sorption, and solubility (Jamel, 2020; Cascione et al., 2021; Chęcińska et al., 2022; Hata et al., 2022; Kaga et al. 2023).

Several previous studies have been performed to assess the effects of incorporating GFs or ZrO₂ nanoparticles alone into different types of PMMA acrylic resins (Azmy et al., 2022; Chowdhury et al., 2021; Sabri et al., 2021) However, there were insufficient data about the impact of a combination of GFs with ZrO₂ nanoparticles on the mechanical properties of provisional auto polymerizing PMMA. Hence, this invitro study aimed to investigate and compare the reinforcing effects of GFs and ZrO₂ nanoparticles at different ratios on the Flexural strength (FS), Microhardness (MH), and Surface roughness (SR) of provisional autopolymerizing PMMA. The null hypothesis was that the incorporation of different ratios of GFs and ZrO₂ nanoparticles would not affect the mechanical properties of provisional PMMA.

2. Materials and methods

The materials used in this experimental study were: autopolymerized poly methyl methacrylate for provisional restoration (Temporary. Cold. V, Major Prodotti Dentari S.P.A, Italy). Glass fibers (Gulf glass fiber, Tech. Ind. Saudi Arabia) of 10 μ m in diameter. Zirconium oxide (ZrO₂) nanoparticles (size 10–30 nm and 99.5% purity Houston, USA). Monobond Plus silane coupling agent (Ivoclar/Vivadent Schaan, Liechtenstein).

2.1. Specimen fabrication

The GFs used in the study were rolled to form bundles of fibers in an aluminum foil, then they were chopped into 0.5 \pm 0.1 mm in length using a scalpel blade. Chopped GFs and ZrO₂ nanoparticles were weighed using an electronic digital balance (KERN & Sohn GmbH, Version 1.3, Germany) with an accuracy of 0.0001 g to prepare different ratios of GFs/ZrO₂, and PMMA acrylic powder mixtures. Pre-weighed GFs were impregnated in a silane coupling agent in an average of 1.5 ml of silane for each 1gm of GFs at room temperature for 1 min so as to wet the fibers and improve the bonding of GFs with the resin matrix, then were dried completely for 5-10 min at 110-120 °C (Hamouda and Beyari, 2014; Gad et al., 2019b). The addition of salinized GFs and ZrO₂ nanoparticles was determined at 5 wt% of autopolymerizing PMMA acrylic powder. The ratios of GFs/ZrO2 nanoparticles added to acrylic powder were as follows: (Gad et al., 2019b).

Group I: 0% (0% glass fibers + 0% ZrO₂ nanoparticles) 100% PMMA acrylic powder.

Group II: 5% (0% glass fibers + 5% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group III: 5% (1% glass fibers + 4% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group IV: 5% (2% glass fibers + 3% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group V: 5% (2.5% glass fibers + 2.5% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group VI: 5% (3% glass fibers + 2% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group VII: 5% (4% glass fibers + 1% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

Group VIII: 5% (5% glass fibers + 0% ZrO₂ nanoparticles) 95% PMMA acrylic powder.

The mixture of GFs/ ZrO_2 and PMMA was stirred thoroughly for 30 min using a mixer machine at a speed of 300 rpm to obtain a fine particle distribution.

2.2. Mixing procedure

Based on the manufacturer's instructions, acrylic resin (with and without additives) was dispersed in methyl methacrylate monomer with a powder/liquid ratio of 2:1 by volume. After the mixture reached a dough state, it was filled in the molds with a spatula. A weight of 1.5 kg was applied to the glass slab located on the mold to allow the material to completely flow into the mold and remove any excess. (Mehrpour et al., 2016). After complete polymerization, specimens were removed from the molds and precisely examined to detect any porosities or defects, deformed specimens were excluded. Finally, whole specimens were finished and wet-polished using 600, 800, and 1200-grit abrasive polishing paper, rinsed with distal water, dried, and stored in distilled water for 72 h in an incubator at 37°C prior to testing to simulate the oral environment.

A total of one hundred twenty prepared specimens of autopolymerizing PMMA acrylic were divided into eight groups per test (FS, MH, and SR). Each group contains five specimens (n = 5).

2.3. Flexural strength test

Forty bar-shaped specimens of PMMA with different ratios of additives were prepared for the FS test using a stainless-steel split mold with dimensions of $(2 \text{ mm} \times 2 \text{ mm} \times 25 \text{ mm})$. The preparation of the specimens was done in the same procedure mentioned earlier. FS was measured by a three-point bending test using a universal testing machine (Sans testing machine Co., Ltd. Shenzhen; China) at a crosshead speed of 0.5 mm/min. Each specimen of PMMA acrylic resin was placed on two support pins (20 mm apart). A load was applied by a centrally positioned rod until a fracture occurred and the maximum value was recorded. The FS was calculated in Mega-Pascal from the following equation: (Mehrpour et al., 2016).

 $FS = 3FL/2WH^2.$

Where FS = Flexural strength (MPa).

F =load at fracture (N).

W = width of the sample (mm).

H = thickness of the sample (mm).

2.4. Vickers microhardness test

Forty disc-shaped specimens with different ratios of additives were prepared for the Vickers microhardness test using silicone molds with dimensions (9 mm diameter \times 3 mm thickness) in the same mixing procedure mentioned previously (Karawatthanaworrakul and Aksornmuang, 2020). Vickers microhardness test was performed using a Vickers tester (OTTO WOLPER-WERKE, Germany). A diamond indenter with a 0.5 kg load and a dwell time of the 30 s was used, three indentions were achieved on each surface of specimens and the mean value of the three indentions was considered. Vickers Microhardness Number (VHN) for each specimen was calculated in Kg/mm² from the following formula:

 $VHN = 1.8544L / D^2$.

Where L = applied load (Kg).

D = mean diagonal length (mm).

2.5. Surface roughness test

Forty specimens with different ratios of additives were prepared for the SR test by silicone molds with dimensions of (10 mm diameter \times 2 mm thickness) (Chowdhury et al., 2021). The specimens were tested using a contact type of profilometer surface roughness device (TAYLOR HOBSON, Talysurf 10, Leicester, England, U.K.). This device contains a diamond stylus tip with a radius of 0.5 µm that moves at a constant speed of 0.5 mm/sec. The distance of the profilometer's needle is 2.5 mm. The tip of the stylus made direct contact with the specimen surface. The tip of the detector is supplied with a stylus and it records all the irregularities that characterize the test specimen's polished surface and measures SR in micrometers. (Abdullah et al., 2021). Three readings per specimen were recorded and the mean of them was considered. A flowchart of the specimen's preparation and testing procedure is presented in Fig. 1.

2.6. Statistical analysis

Data were analyzed and processed using SPSS (Statistical Package for social science) version 24. The Analysis of variance (ANOVA), Person correlation, and Tukey's test of FS, MH, and SR were performed to reveal the relation between each. *P*-value ≤ 0.05 was considered significant.

3. Results

The mean values, standard deviations, ANOVA, Tukey's test, and Pearson correlation of FS, MH, and SR of the tested groups are presented in Table 1. One-way ANOVA exhibited a significant change among the tested groups (*P*value ≤ 0.001). The investigational groups reinforced with the GFs and ZrO₂ nanoparticles exhibited a significant increase in FS compared with the control group (75.639 \pm 0.9 MPa). Besides, there were significant changes in the FS of the groups reinforced with GFs and ZrO₂ nanoparticles 79.89 \pm 0.087, 82.05 \pm 1.4, 86.58 \pm 0.9, 87.31 \pm 1.07, 89.99 \pm 0.7, 93.77 \pm 0.64, and 96.8 \pm 0.96 MPa, respectively as shown in Table 1. Samples reinforced with 5% GFs + 0%

L = distance between supports (mm).



Fig. 1 Flowchart of specimens preparation and testing.

 ZrO_2 exhibited the highest FS, while specimens free of GFs and 5% ZrO_2 had the lowest values.

The investigational groups reinforced with the GFs and ZrO₂ nanoparticles exhibited a significant increase in MH compared to the control group (15.85 \pm 0.93 kg/mm²). Furthermore, there were significant changes in the MH of the groups reinforced with GFs and ZrO₂ nanoparticles 20.2 \pm 0.64, 23.8 \pm 1.09, 26.80 \pm 0.59, 27.72 \pm 0.72, 29.61 \pm 0.54, 31.20 \pm 0.5, and 34.91 \pm 0.66 kg/mm² respectively in Table 1. Samples reinforced with 5% GFs + 0%ZrO₂ revealed the highest MH, while specimens free of GFs and 5% ZrO₂ exhibited the lowest values. Different ratios of GFs and ZrO₂ nanoparticles yielded different effects on the FS and MH, the values gradually increased from group I (0% glass fibers + 5% ZrO₂) to group IV (2% glass fibers + 3% ZrO₂) with no significant differences between group IV (2% glass fibers + 3% nanoZrO₂) and group V (2.5% glass fibers + 2.5% nanoZrO₂).

The groups that reinforced with (2.5% glass fibers + 2.5% ZrO₂, 3% glass fibers + 2% ZrO₂, 4% glass fibers + 1% ZrO₂, and 5% glass fibers + 0% ZrO₂) revealed a significant increase in SR compared to the (0% glass fibers + 0% ZrO₂, 0% glass fibers + 5% ZrO₂, 1% glass fibers + 4% ZrO₂, and 2% glass fibers + 3% ZrO₂) as shown in Table 1. Specimens reinforced with 5% glass fibers + 0% ZrO₂ showed the highest SR. In addition, there was a positive correlation between FS, MH, and SR.

4. Discussion

The present study showed that group I (0% glass fibers + 0% ZrO₂) had the lowest mean FS and MH values. Altering the mixture ratio (increasing or decreasing) resulted in different

mean FS and MH values. With increasing the amount of GFs and decreasing ZrO_2 nanoparticles away from group II (0% glass fibers + 5% ZrO₂), a continuous increase in FS and MH is observed in group III (1% glass fibers + 4% ZrO_2) followed by group IV (2% glass fibers + 3% ZrO_2), group V (2.5% glass fibers + 2.5% ZrO_2), group VI (3% glass fibers + 2% ZrO_2), group VII (4% glass fibers + 1% ZrO_2) and finally, group VIII (5% glass fibers + 0% ZrO_2) which showed the highest mean FS and MH values. However, even with the lower FS and MH values of group II (0% glass fibers + 5% ZrO_2), the results were still significantly higher than the unreinforced group. These results denoted that the incorporation of GFs and ZrO_2 enhanced the mechanical properties of provisional PMMA compared to the unreinforced PMMA. Based on these results the null hypothesis was rejected.

FS and MH improved with increasing GFs and decreased ZrO₂ ratios. These results may be related to two reasons. First, the increase in FS and MH is mostly related to the rate of GFs which revealed a more pronounced effect than that of ZrO_2 , this is noticeable when the concentration of GFs at 5% and ZrO₂ at 0% where the FS revealed (96.8 MPa) and MH (34.91 kg/mm^2) , while the group reinforced with 5% ZrO₂ and 0% GFs revealed FS (79.89 MPa) and MH (20.2 kg/ mm²). The strong adhesion between salinized GFs and the matrix interferes with crack propagation and prevents fracture (Gad et al., 2019b). Second, the incorporation of the ZrO_2 nanoparticles did not play a significant role in reinforcing the autopolymerizing PMMA prominently overall. In addition, higher concentrations of ZrO₂ led to a decrease in the mean values of FS and MH. Recent studies have shown that the incorporation of high proportions of ZrO₂ into PMMA often has antagonistic effects on mechanical properties. Excess nanofillers diminish the mechanical properties of the PMMA

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because they	y form v	oids and	aggregate	es in the	resin matrix.
Nanoparticl	e aggrega	ation and	cluster f	ormation	act as stress
concentratio	n areas	that wea	aken the	PMMA	(Alshahrani,
et al., 2021;	Azmy et	al, 2022;	Kaga et	al. 2023).	

The concept of reinforcing effects of GFs is based on the prevention of the initiation and propagation of microcracks by transmitting the stress from the weak resin to the salinized GFs that have good tensile strength (Gad et al., 2019b; Alhotan et al., 2021a). In this study, GFs were treated with the silane coupling agent containing 3-methacryloxypropyl trimethoxysilane (3-MPS) which has hydroxyl groups that bond to the GFs and carbon bonds that react with PMMA during chemical polymerization. This provided good fiber distribution in the matrix and prevented agglomeration. The results coincided with previous studies (Hamouda and Beyari, 2014; Al-Thobity, 2020).

ZrO₂ nanoparticles enhanced the properties of the provisional PMMA, this can be explained by the transformation toughening mechanism of ZrO₂ nanoparticles. During crack propagation, a transformation of ZrO₂ nanofillers from a tetragonal crystalline phase to the stable monoclinic phase occurs, which consumes the energy of crack propagation and thus stops it. Besides, The ZrO₂ nanoparticles used in this study were very small in size (10-30 nm) which greatly increased the surface area that was very successful in dissipating energy and reducing the chance of crack propagation (Gad et al., 2018). The size of nanoparticles being < 100 nm allowed them to penetrate between linear macromolecule chains limiting their movement. (Nejatian et al., 2020; Alshahrani et al., 2021). Recently, it has been concluded that the inclusion of nanoparticles into PMMA significantly enhances physical and mechanical properties (Alshahrani et al., 2021, Chowdhury et al., 2021). Alhotan et al. (2021b) studied the effect of adding GFs and nanoparticles to PMMA denture bases and found that adding GFs attained superior improvement in mechanical characteristics, followed by ZrO₂.

The results revealed that the SR values increased with increasing the GFs ratio from 2.5% to 5% compared to the other tested groups and the highest values were recorded at 5% GFs. This result can be explained by the possibility of GFs protruding from the specimen's surface and the random orientation of GFs on the surface. This was in agreement with Gad et al. (2018). The increase in the proportion of GFs may cause poor adhesion of GFs to resins, plaque accumulation, and tissue irritation which may be the main cause of patient discomfort and associated with problems by promoting biofilm aggregation and microbial growth (Pradhan et al., 2022). Thus, choosing an appropriate proportion of GFs may be one possible way to reduce their potential effects in addition to using any type of polishing technique (chemical or mechanical) which is important to reduce the SR and produce a sufficiently smooth and glossy surface thus preventing bacterial plaque deposition (Vishwanath et al., 2022).

The reinforced groups $(2\%GFs + 3\%ZrO_2, 1\%GFs + 4\%ZrO_2, and 0\%GFs + 5\%ZrO_2)$ did not show any significant change compared to the unreinforced group, this result may be due to ZrO_2 nanofillers are very small in size and can be well distributed in the resin substance (Abdulrazzaq et al., 2022). Moreover, the nanoparticles fill the pores of the polymer matrix and improve the SR. These results agreed with the other studies (Al-Harbi et al., 2018; Fouda et al., 2021; Abdulrazzaq et al., 2022; Mârt et al., 2022).

Table	I The mean, sta	undard deviation,	ANOVA, Tukey'	s test, and Pearso	n correlation of Fle	xural Strength, M	licrohardness, and	Surface Roughn	less of the tested	l groups.	
	Mean ± SD								ANOVA	Pearson Correlation	
Group	Group I 0% GFs+0% ZrO2	Group II 0% GFs+5% ZrO2	Group III 1% GFs+4% ZrO2	Group IV 2% GFs+3% ZrO2	Group V 2.5% GFs+2.5% ZrO2	Group VI 3% GFs+2% ZrO2	Group VII 4% GFs+1% ZrO2	Group VIII 5% GFs+0% ZrO2	F-value P- value	FS MH S	SR
FS	$75.639 \pm 0.9^{\rm A}$	$79.89 \pm .087^{B}$	$82.05 \pm 1.4^{\rm C}$	$86.58 \pm 0.9^{\rm D}$	87.31 ± 1.07^{D}	$89.99~\pm~0.7^{\rm E}$	$93.77~\pm~0.64^{\rm F}$	$96.8 \pm 0.96^{\rm G}$	272.378 0.000	1 O.906 0	.972
(MPa) MH (kg/	$15.85 \pm 0.93^{\rm A}$	$20.2 \pm 0.64^{\rm B}$	$23.8 \pm 1.09^{\rm C}$	26.80 ± 0.59^{D}	27.72 ± 0.72^{D}	$29.61 \pm 0.54^{\rm E}$	$31.20~\pm~0.5^{\rm F}$	34.91 ± 0.66^{G}	344.438 0.000	0.974 0.866 1	_
mm ²) SR (μm)	$0.5 \pm 0.02^{\rm A}$	$0.71~\pm~0.02^{\rm A}$	$0.75 \pm 0.02^{\rm A}$	$0.84~\pm~0.03^{\rm A}$	$1.4~\pm~0.19^{\rm B}$	$1.7 \pm 0.04^{\mathrm{BC}}$	$1.8~\pm~0.13^{\mathrm{BC}}$	$1.87 \pm 0.059^{\rm C}$	572.754 0.000	0.974 1 0	.866
Group	s with dissimilar le	tters are significan	tly different, Numb	oer of samples $= 5$	i, Abbreviations: SD:	Standard Deviatio	n, FS: Flexural Sti	ength, MH: Micro	ohardness, SR: Su	urface Roughr	less.

The limitations of this experimental study can be summarized as follows: the test conditions did not fully mimic the oral environment, such as the presence of saliva, occlusal function, and temperature variation. One type of provisional material was tested, and only one type of nanoparticles and GFs was used. Thus, it is recommended to carry out further studies in vitro and in vivo with different types and ratios of provisional materials, nanoparticles, and GFs.

5. Conclusion

The following conclusions were drawn from the results of this in vitro experiment: hybrid reinforcement with GFs, ZrO_2 nanoparticles, or a combination of them effectively improved flexural strength and microhardness of autopolymerizing provisional PMMA that would create provisional restorations with extended clinical service. GFs demonstrated superior reinforcing effects compared to ZrO_2 nanoparticles. However, reinforcement with 2.5–5% GFs increased the surface roughness for provisional restoration.

Ethical statement

This study was approved by the Research Scientific Committee board at the University of Mosul, College of Dentistry in Iraq (approval No.: 6398 on 30/11/2021).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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