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Original Article

Effects of Fritted Dental Porcelain Particles on Tensile and Flexural Properties of Poly Methylmethacrylate Matrix

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ABSTRACT

Purpose: The aim of this study was to assess influence of varying concentrations of fritted dental porcelain (FDP) particles, at levels of 5, 10, and 15 wt.%, as reinforcement material on the mechanical properties of poly methylmethacrylate (PMMA) matrix.

Methods: An array of methodologies was employed to analyze the raw materials associated with the preparation of FDP. Specimens of tests were created and classified into four groups according to FDP particles concentration into PMMA matrix to assess both tensile and flexural properties. The control group was composed of PMMA matrix containing 0 wt.% of FDP particles. The flexural testing was conducted in compliance with ASTM D790-86 using a three-point bending test machine, whereas the tensile testing was conducted in accordance with the specifications of ASTM D638.

Results: The results demonstrate that as FDP particles concentrations increased into PMMA matrix as tensile modulus and flexural modulus values increased, while simultaneously, the tensile strength and flexural strength values decreased as FDP particles concentrations increased into PMMA matrix.

Conclusions: Among various concentrations of FDP examined, the addition of a low concentration (5 wt.%) of FDP particles to the PMMA matrix is found to be the most effective in augmenting the mechanical properties of PMMA matrix.

Keywords: PMMA, Filler, Reinforcement, Tensile, Flexural, Properties.

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INTRODUCTION

In prosthetic dentistry, PMMA is one of the most frequently employed materials. Thanks to its beneficial features, which include straightforward processing and pigmentation, adequate mechanical properties, and cost-effectiveness, the predominant application of this biomaterial is denture production.[1] The optimal material for denture bases should demonstrate a range of essential physical properties vital for effectively creating polymeric denture bases. The cured polymer must maintain a level of stiffness adequate to secure the teeth in proper occlusion during mastication while minimizing the risk of uneven loading on the mucosa. Important properties to consider include biocompatibility, aesthetic quality, effective bonding strength with the artificial teeth, simplicity of repair, and must possess appropriate mechanical attributes, ease of repair, and sufficient mechanical integrity. In addition, the denture base must display an appropriate level of resilience to cope with the functional and parafunctional forces that occur during chewing. The material used for denture bases exhibits outstanding optical characteristics, minimal toxicity, and favorable biocompatibility with human tissues. From a mechanical perspective, the material needs to possess high mechanical properties, as the current levels do not adequately meet the necessary mechanical strength requirements.[2]

The mechanical characteristics of PMMA are insufficient to endure significant occlusal forces, as it exhibits low flexural and impact strength.[3] Consequently, the application of pure PMMA in dentistry is not favored. The effort to improve one property can inadvertently result in the degradation of another, making it a considerable challenge to develop a denture base material that incorporates all the desired characteristics.

Recent research has explored a variety of filler particles incorporated into PMMA, yielding promising results.[2,4,5] Notably, the investigation performed by[6] indicated that augmenting the filler ratio into PMMA nano-composites leads to an improvement in their mechanical properties. Furthermore,[7] documented that the mechanical properties exhibit enhancements as the concentration of nanoparticles increases. Additionally, they noted enhancements in stiffness when comparing pure PMMA with reinforced PMMA.[8] Research efforts focused on reinforcement strategies are directed toward enhancing the mechanical properties of materials. However, significant obstacles persist in developing a substance that can adequately fulfill the requirements of dental applications. Consequently, integration of filler material in pure PMMA is essential. The application of FDP particles as a reinforcing filler within a PMMA matrix is not extensively documented in the existing literature. The incorporation of filler particles in PMMA matrix offers several benefits, primarily enhancing the mechanical properties. The advancements in these composites render them increasingly appropriate for a range of dental applications; as they are capable of enduring the challenges presented by the mouth cavity while also offering a lifelike aesthetic. Improvement denture base characteristics are significantly determined by several key factors, such as filler particle types, the ratio of filler to the matrix, and the interactions that occur at the interface between the filler particles and the polymer matrix. The choice of filler material, its concentration, and the adhesion of filler particles to the polymer matrix are critical factors that significantly affect the performance of PMMA composites. The present study adopts a research methodology that seeks to develop properties of the denture base for dental applications by incorporating FDP particles, which act as reinforcement material. FDP particles are inert materials if used as part of PMMA composite denture materials. It is very stable in the mouth environment. Therefore, this study aimed to assess the influence of varying concentrations of FDP particles on the tensile strength, tensile modulus, flexural strength, and flexural modulus of the PMMA matrix..

MATERIALS AND METHODS

TPMMA (996,000 GPC) was sourced from Aldrich (U.S.A.), along with Benzoyl peroxide (BPO) as the initiator obtained from Merck Chemical (Germany), while Methyl methacrylate (MMA) was procured from Fluka (UK) and hydroquinone Additionally, a

Field Emission Scanning Electron Microscope (FESEM) ZEISS-SUPRA 35 VP was utilized to assess average particle size and morphology. The phases present in the materials were confirmed using X-ray diffraction (XRD) with a Bruker X-ray Powder Diffractometer (D8 Advance), and the elemental composition was determined through X-ray Fluorescence (XRF) using a Rigaku Spectrometer (model RIX 3000).

Preparation of FDP particles

The raw materials used to prepare FDP particles are detailed in Table 1. The process begins with drying the materials to eliminate moisture, followed by mixing them in a plastic container with zirconia balls for blending. A Multi-Drive mixer is used for a 2-hour mixing period, after which the powder is moved into an alumina crucible. This crucible is then exposed to a melting temperature of 1350 °C for 2 hours in a Lenton glass melting furnace. The melted product is rapidly cooled through quenching in cold water, creating internal stresses and causing cracking. These resulting fragments, known as frit, are then dried and crushed into fine powder using a fast mill machine. This entire process is referred to as fritting, a type of pyrochemical reaction.

Table 1. Materials used to prepare FDP particles.

Name of Compound	Raw Materials	Weight (%)
Na-feldspar	$Na_2O_3.Al_2O_3.6SiO_2$	27.91
K-feldspar	$K_2O.\ Al_2O_3.6SiO_2$	51.46
Kaolin	$Al_2O_3.2SiO_2.2H_2O$	0.70
Silica	SiO_2	11.00
Alumina	Al_2O_3	1.06
Sodium carbonate	Na_2CO_3	1.38
Potassium carbonate	KC_2O_3	2.95
Magnesium carbonate	$MgCO_3$	0.28
Calcium carbonate	$CaCO_3$	1.29
Strontium carbonate	$SrCO_3$	0.08
Barium carbonate	$BaCO_3$	0.28
Titanium oxide	TiO_2	0.11
Lithium carbonate	$Li2CO_3$	1.50

Composite specimen's preparation

The preparation of specimens involves both solid and liquid phases. The solid phase comprises three different weight percentages of FDP particles, specifically 5%, 10%, and 15%, which are integrated into PMMA and BPO. A planetary ball milling technique was utilized to mix the solid components (PMMA, BPO, and FDP particles) for a total of one hour. To avoid overheating and the risk of premature polymerization, the mixing was paused every 15 minutes and then resumed after the same interval. In the liquid phase, a cross-linking agent known as EGDMA was employed, with MMA acting as an activator. Additionally, hydroquinone was incorporated as an inhibitor. The method employed to blend the powder and liquid adhered to the guidelines prescribed for dental laboratory operations. Upon achieving a dough-like consistency, the mixture was placed into a mold and subjected to a pressure of 14 MPa at room temperature for 30 minutes. The final curing phase occurred in a water bath maintained at 78±1°C for one and a half hours. Following this, the mold was allowed to cool gradually to 25±1ºC. After the completion of finishing and polishing, the specimens were immersed in distilled water at a controlled temperature of 37±1ºC for 48 hours. This procedure was necessary to eliminate any residual monomer, relieve any residual stress, and ensure that the denture base materials were kept in a semi-oral environment.

Mechanical properties

The tensile testing was carried out following the guidelines set forth by ASTM standard D-638. The apparatus used for this evaluation was a universal testing instrument (Instron 5582). At least five specimens from each formulation were analyzed, with precise documentation of the tensile strength and tensile modulus. Tensile tests provide essential data on PMMA's mechanical properties. These properties are vital for understanding how PMMA will behave under various loading conditions. The flexural test was conducted according to ASTM D790-86. The test was performed at a speed of 0.2 cm/min. The support span was set at 5 cm. The loading nose diameter was 2 cm, while the supports had a diameter of 1 cm. flexural strength (MPa) indicates the maximum stress experienced by the material before failure occurs.

Flexural modulus (GPa) reflects the material's stiffness during bending. This procedure was executed in a laboratory environment.

Statistical analyses

Data analysis was executed using SPSS version 19. ANOVA was implemented, and subsequently, Tukey's post-hoc analysis was performed. The criterion for determining statistical significance was defined as $P<0.05$.

RESULTS

Particle size analysis of raw materials

The assessment of particle size distribution of raw materials was fundamentally conducted using the laser diffraction technique. Table 2 illustrates the average sizes of particles expressed in micrometers. Most of the powders have sizes less than 8 µm. Except for lithium carbonate, sodium carbonate, potassium carbonate, and alumina have sizes bigger than 10 µm. The average particle size for barium carbonate and strontium carbonate is almost the same (2 µm) and that for Na-feldspar, silica, and calcium carbonate is about 8 µm. This similarity in particle size would lessen the demixing problem of components.

Table *2*. Average size of raw materials particles.

Raw Materials	Particle Size(µm)
Calcium carbonate	7.93
Lithium carbonate	11.57
Barium carbonate	1.65
Magnesium carbonate	2.31
Sodium carbonate	15.44
Strontium carbonate	1.69
Potassium carbonate	17.76
Titanium oxide	0.54
Kaolin	3.05
Silica	7.60
Alumina	13.09
K-feldspar	5.50
Na-feldspar	7.35

FESEM of raw materials

Several micrographs of some raw materials were taken at magnification powers of 500X, 1.00 KX, and 1.50 KX. Figures 1 to 5 show the FESEM for alumina, kaolin, K-feldspar, Na-feldspar and silica, respectively. It is clear that the range of particle size of alumina is from 7.3 µm to 24 µm, with an irregular shape and sharp edges. The kaolin particle size ranged from 3.1 µm to 30.7 µm with an irregular shape. The K-feldspar particle size ranged from 3.3 µm to 4.3 µm with noticeable similarity in shape of the type of alumina. For Na-feldspar, the size of particles varies between 6.3 µm and 25.3 µm, whereas silica demonstrates a particle size range extending from 2.7 µm to 168.0 µm.

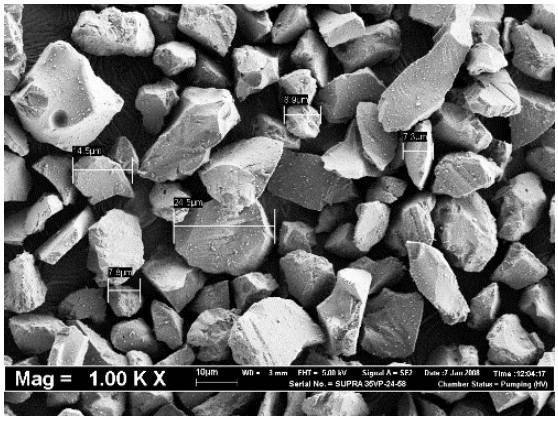

Fig 1. Micrograph for alumina.

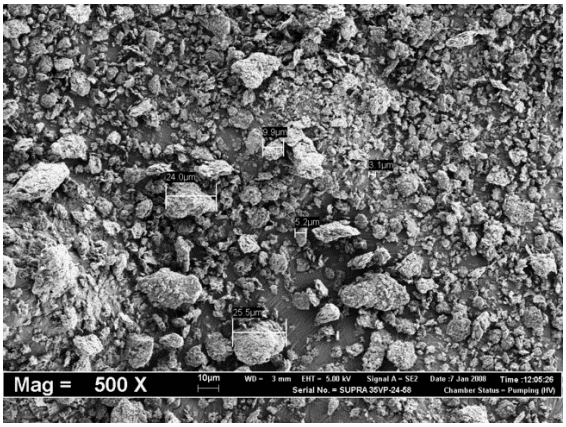

Fig 2. Micrograph for Kaolin.

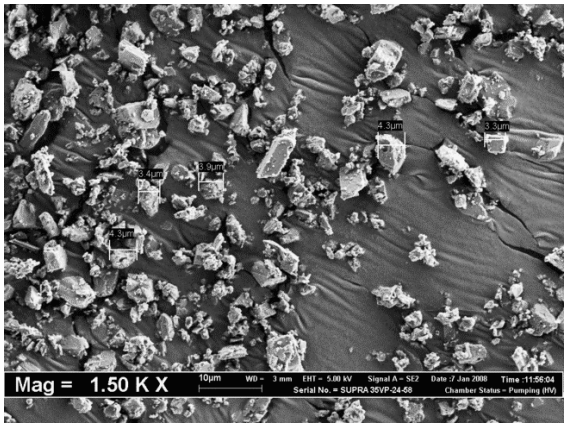

Fig 3. Micrograph for K-feldspar.

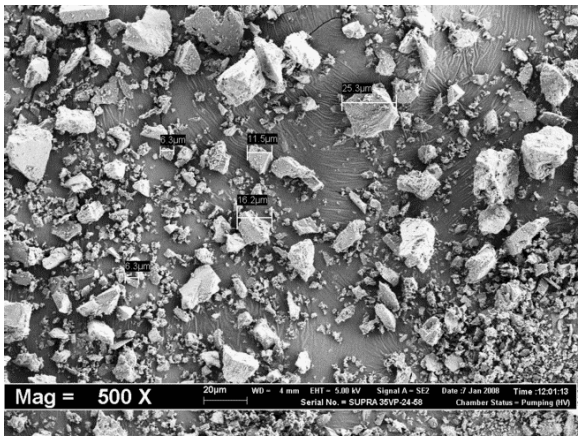

Fig 4. Micrograph for Na-feldspar.

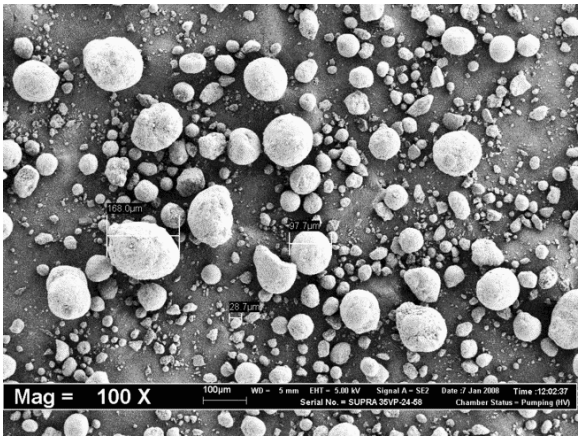

Fig 5. Micrograph for Silica.

XRF of raw materials

The findings from the XRF analysis are presented in Table 3. This examination concentrated on the five primary raw materials, which include (Na-feldspar, K-feldspar, alumina, limestone, and silica). XRF analysis indicated that the above major raw materials are relatively pure. Alumina, calcium carbonate, and silica are considered to have the highest purity (exceeding 99 wt %); while Na-feldspar contains 5.4 wt.% Na2O and 2.2 wt.% K2O and for K-feldspar it contains 11 wt.% K2O and 2.3 wt.% Na2O. The rest of the trace elements are considered of low amount (less than 1 wt.%) for each material.

XRD of raw materials

An analysis of the raw material samples was conducted through X-ray diffraction (D8 Advance).

The XRD testing specifically targeted the raw materials used in the preparation of FDP particles. A phase comparison across all samples was executed, adhering to the standards established by the International Center for Diffraction Data (ICDD). Table 4 shows the outcomes of XRD analysis. The XRD analysis corroborated that all raw materials involved in the preparation of FDP particles included the essential compounds. It was evident that the key raw materials, namely (K-feldspar, Na-feldspar, alumina, calcium carbonate, and silica), possessed the appropriate minerals. Additionally, similar patterns were observed in the minor components, including (kaolin, alumina, sodium carbonate, potassium carbonate, and magnesium carbonate).

Table 3. Micrograph for Silica.

Wt.% of elements (Metal oxide)	K-feldspar	Na-feldspar	Alumina	Calcium Carbonate	Silica
Na_2O	2.30	5.40	-	-	-
Al_2O_3	19.00	19.25	99.766	0.017	0.2
SiO2	67.00	71.00	0.088	0.23	99
P_2O_5	0.17	0.26	-	0.023	-
SO_3	0.027	0.028	0.034	0.048	-
K_2O	11.00	2.2	-	-	0.026
CaO	0.11	1.6	0.039	99.651	0.015
Fe_2O_3	0.12	0.18	0.055	0.013	0.054
NiO	0.025	0.014	0.011	-	0.016
Rb_2O	0.27	0.04	-	-	-
ZrO_2	-	0.028	-	-	Trace
CuO	-	-	-	-	Trace

Table 4. Micrograph for Silica.

Name of Compound	Materials	ICDD File No.
Na-feldspar	$Na_2O_3.Al_2O_3.6SiO_2$	09-466
K-feldspar	$K_2O.\ Al_2O_3.6SiO_2$	2-675
Kaolin	$Al_2O_3.2SiO_2.2H_2O$	6-221
Silica	SiO_2	3-444
Alumina	Al_2O_3	10-173
Sodium carbonate	Na_2CO_3	19-1130
Potassium carbonate	KC_2O_3	16-820
Magnesium carbonate	$MgCO_3$	8-479
Calcium carbonate	$CaCO_3$	5-586
Strontium carbonate	$SrCO_3$	5-418
Barium carbonate	$BaCO_3$	5-378
Titanium oxide	TiO_2	21-1276
Lithium carbonate	Li_2CO_3	2-1141

Particle size analysis of FDP particles

The investigation into the particle size distribution of FDP particles utilized in this study employed the laser diffraction technique with the assistance of Sympatec. The ceramic material was subjected to crushing and subsequent sieving to achieve a fine powder consistency. The median size of FDP particles was determined to be 2.36 µm. Table 5 presents the statistical data concerning the size of FDP particles analyzed in this study. Additionally, Figure 6 depicts the distribution of FDP particle size, indicating its appropriateness to be used as reinforcement filler in composite materials.

Table 5. Particle size analysis of FDP particles.

Sample	Particle size (µm)		
	X0.1	X0.5	X0.99
FDP particles	0.53	2.36	22.43

X0.5: Median particle size 50%.
X0.1: The particle size below which 10 %.
X0.99: The particle size below which 99 %.

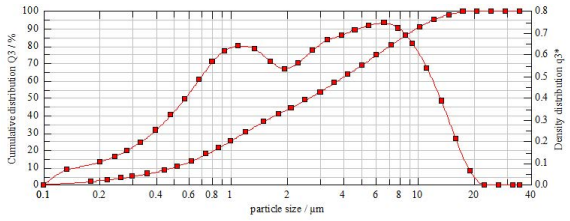

Fig 6. Particle size distribution of FDP particles.

Tensile properties

Figure 7 illustrates the tensile modulus of both the PMMA matrix and the PMMA/FDP composites, highlighting the significant influence of filler content on the tensile modulus value. The PMMA matrix exhibited a tensile modulus of 2.2 GPa, while the tensile modulus of a PMMA/FDP composite containing 5, 10, and 15 wt.% of filler particles reached a peak of 2.23 GPa, 2.28 GPa and 2.36 GPa respectively. As the concentration of FDP particles increases in the PMMA matrix, the tensile modulus values increase.

In contrast, the findings depicted in Figure 8 reveal that the addition of FDP particles into the PMMA matrix caused a decline in the tensile strength values. Notably, as the concentration of FDP particles increased, there was a corresponding decline in tensile strength of the PMMA matrix. The PMMA matrix exhibited a tensile strength of 58.7 MPa, while the tensile strength of a PMMA/FDP composite containing 5, 10, and 15 wt.% of filler particles reached a peak of 50.1 MPa, 48.4 MPa, and 44.2 MPa respectively. The statistical evaluation revealed notable differences ($P<0.05$) in the tensile strength measurements among all composite groups that included FDP particles when contrasted with the control group.

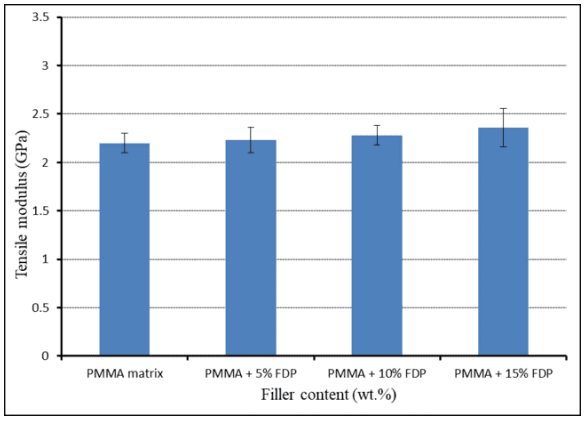

Fig 7. Tensile modulus of PMMA and PMMA/FDP.

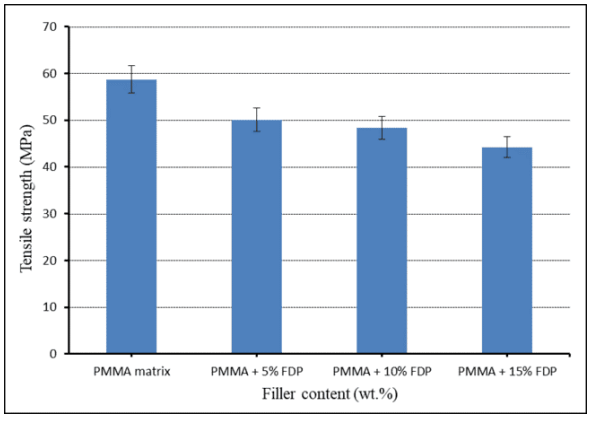

Fig 8. Tensile strength of PMMA and PMMA/FDP.

Flexural properties

Figure 9 illustrates the flexural modulus results of the PMMA matrix and PMMA matrix incorporated with FDP particles at different concentrations of 5, 10, and 15 wt.%.The graph illustrates a positive relationship between the concentrations of filler particles and the PMMA matrix regarding flexural

modulus values. The PMMA matrix exhibited a flexural modulus of 2.1 GPa, while the flexural modulus of a PMMA/FDP composite containing 5, 10, and 15 wt.% of filler particles reached a peak of 2.5 GPa, 2.6 GPa and 2.9 GPa respectively. Statistical analysis demonstrates that as the filler increased, there were significant increases in the flexural modulus values ($P < 0.05$).

The results illustrated in Figure 10 demonstrate flexural strength values of PMMA matrix and PMMA matrix incorporated with FDP particles at different concentrations of 5, 10, and 15 wt.%. The results reveal a decline in flexural strength as the concentration of the filler particles increases. The PMMA matrix exhibited a flexural strength of 85.9 MPa, while the flexural strength of a PMMA/FDP composite containing 5, 10 and 15 wt.% of filler particles reached a peak of 75.1 MPa, 69.8 MPa and 66.3 MPa respectively.

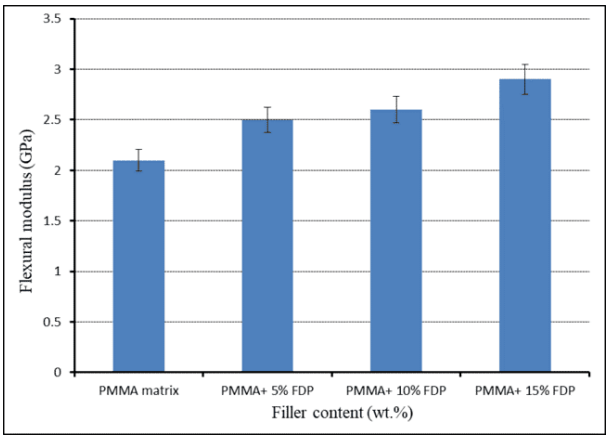

Fig 9. Flexural modulus of PMMA and PMMA/FDP.

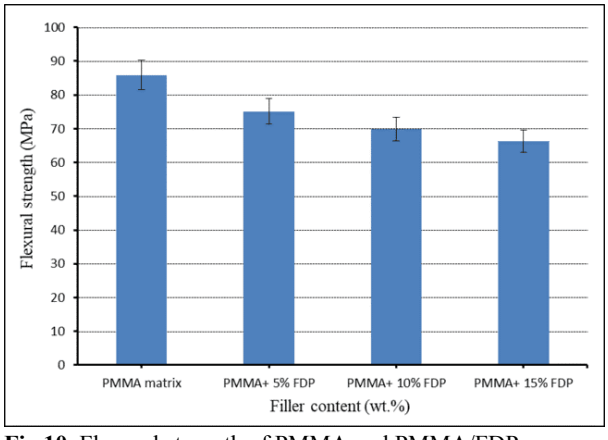

Fig 10. Flexural strength of PMMA and PMMA/FDP.

DISCUSSION

The tensile modulus of heat-cured PMMA denture base materials is generally reported to be lower than that of other modern materials used in dental applications. The tensile modulus, which indicates a material's stiffness, is a critical property for denture bases as it affects their performance and longevity. According to,[10] the tensile modulus of heat-cured PMMA typically ranges from 2.2 to 3.8 GPa depending on the formulation and any reinforcing agents used. Therefore, maintaining a tensile modulus of at least this rate is critical to ensure that heat-treated PMMA denture base materials perform effectively in clinical settings. The effect of incorporating two different types of reinforcing particles on the tensile properties of the heat-cured polymer was assessed by,[11] who noted that the maximum modulus of elasticity reached 1.85 GPa. In contrast,[12] indicated that heat-cured polymers exhibit a tensile modulus of approximately 2.4 GPa. The increase in tensile modulus observed in PMMA/FDP composites can be elucidated by the filler's role in resisting stress and limiting the mobility of molecular chains within the PMMA matrix when exposed to external forces. The substantial increase in tensile modulus with elevated filler content can be ascribed to the filler's considerable rigidity. According to,[13] the interphase between the filler and the polymer matrix acts as a crucial bridge that facilitates effective stress transfer. This interaction helps to enhance the modulus of the composite material, as a denser interphase can lead to better reinforcement of the composite. Conversely, a weak interfacial adhesion can diminish this effect under load.

The tensile strength of PMMA as a denture base can vary significantly, typically ranging from 1.126 MPa to 66 MPa. This variation is influenced by several factors, including the specific formulation of the PMMA, crosslinking, crystallinity, the presence of additives or fillers, and the processing methods used during fabrication. The tensile strength of PMMA denture bases is critical for their performance and durability. The tensile strength is reported at approximately 1.126 MPa for heat-cured acrylic resin, which is considered the lower end of the

spectrum.[14] Research indicates that when PMMA is blended with natural nanoparticles or other fillers the tensile strength can significantly increase. For instance, a composite material with clove powder reached a tensile strength of 65 MPa.[15] The baseline tensile strength for unmodified PMMA is often reported as around 48.41 MPa, with variations based on specific formulations and processing conditions.[16] In this study the tensile strength decline can likely be explained by inadequate compaction between the fillers and the polymer matrix, coupled with a non-uniform distribution of the particles. Additionally, the agglomeration of filler particles further contributes to the reduction in tensile strength. Consequently, the imposition of stress can result in a non-uniform distribution throughout the composite material, leading to localized stress concentrations. These concentrations may induce cracking near the regions where the filler particles interact with the polymer matrix and with one another. A study carried out by[17] reveals the significant role that the agglomeration of filler particle dispersion plays in determining the properties of composite materials. It indicates that the inevitable agglomeration leads to a reduction in strength due to the inherently lower strength characteristics of the agglomerates compared to individual filler particles. This finding aligns with earlier research by,[18] which noted that excessive filler loading can have the opposite effect and reduce the strength of the composite due to poor adhesion. The mechanical performance of composite materials is significantly influenced by the distribution of filler particles, micromechanical processes, and interfacial adhesion, which create weak points for crack initiation and propagation.

According to ISO specification 1567-2000, a denture base polymer must achieve a flexural modulus of at least 2 GPa. The findings of this study regarding the increase in flexural modulus values align with the conclusions drawn by,[18] which indicated that the addition of filler particles to the resin matrix results in an improvement in flexural modulus. This advancement is connected to the augmented brittleness and stiffness of the composite material, arising from the rigid distribution of the filler particles. A uniform distribution of filler particles contributes to greater resistance against flexural forces. Furthermore, the efficient wetting of the filler particles by the PMMA matrix facilitates a homogeneous distribution, enabling the composite to endure higher stress levels. Additionally, the incorporation of filler particles restricts the mobility of the matrix phases in their surrounding area, resulting in a notable increase in the flexural modulus. The strategic incorporation of high-modulus fillers into polymer composites significantly enhances their mechanical strength. This makes them highly valuable in various industrial applications where performance under stress is critical. The evaluation of the flexural strength of dentures is vital for determining the efficacy of resin materials when they are exposed to the mechanical forces generated by the mastication process. Clinical failures in upper and lower acrylic dentures are mainly ascribed to three types of failures: fatigue failure, midline failure, and impact failure. These failures are predominantly a result of the insufficient flexural and impact strength inherent in the materials utilized.

According to the ISO specification 1567:2000, a denture base polymer must achieve a flexural strength of at least 65 MPa for heat-curing polymers. This value is determined when the materials are tested in water at a temperature of 37±1 °C. This requirement ensures that the materials used in the construction of dentures are sufficiently strong to withstand the forces encountered during normal use, thereby enhancing their durability and functionality. This trend aligns with the research conducted by,[19] which established that higher filler concentrations are associated with decreased flexural strength. The observed reduction was consistent across all formulations of PMMA/FDP composites, primarily due to the agglomeration of the dispersed filler particles. Incorporating 5 wt.% of filler particles into the PMMA matrix is effective for enhancing its mechanical properties. However, exceeding this threshold can result in a notable decrease in flexural strength. This decline may be attributed to issues such as agglomeration or uneven distribution of the filler within the resin matrix.

When fillers are added beyond 5 wt.%, they may cluster together, creating regions of high particle concentration that lead to stress concentrations and weaken the overall material. When external loads are applied to these composites, the stress is concentrated on the neighboring particles adjacent to any propagating cracks. This concentration of stress accelerates crack growth, ultimately resulting in brittle failure. A systematic review examining the influence of filler on the properties of the PMMA matrix was conducted.[20] Their research findings demonstrated that the integration of fillers, at levels reaching 5 wt.%, substantially enhances the flexural strength along with other properties of the resin. However, at higher filler loadings, the flexural strength tends to be reduced. The observed reduction is ascribed to the possibility that fillers may interfere with the polymer matrix, resulting in a decline in mechanical integrity. This occurs even though enhancements in other characteristics, such as fracture toughness, can be achieved when fillers are effectively dispersed and chemically bonded. The study indicates that while lower filler concentrations can reinforce denture bases, exceeding this threshold may negatively impact the overall performance of the material, suggesting a careful balance must be maintained in filler loading to optimize mechanical properties without compromising strength.

CONCLUSIONS

The incorporation of inert FDP particles (5-15 wt.%) into the PMMA matrix demonstrates that increased filler concentration correlates with an increase in both the tensile and flexural modulus of the PMMA matrix, while simultaneously, the tensile and flexural strengths decrease as the filler concentrations increase. Among the different filler concentrations used, incorporating a low concentration (5 wt.%) of FDP particles into the PMMA matrix shows the optimal filler concentration and is effective for enhancing the mechanical properties of the PMMA matrix. Therefore, FDP has the very potential to be applied as a PMMA denture base component subjected to further clinical investigation.

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