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Original article

Effects of Treated Fritted Dental Porcelain Particles on the Fracture Toughness and Surface Hardness of Poly Methylmethacrylate as Denture Base

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ABSTRACT

This research aimed to assess the fracture toughness and surface hardness properties of polymethyl methacrylate (PMMA) reinforced with fritted dental porcelain (FDP) particles at various weight percentage levels. Eighty specimens were prepared and divided into four groups. The PMMA matrix was the control group, with others having 5, 10, and 15 wt.% FDP particles. The three-point bending test was carried out to determine fracture toughness (KIC). The surfaces of the fractured specimens were subsequently examined using a Field Emission Scanning Electron Microscope (FESEM). Additionally, the surface hardness of the specimens was assessed through the Vickers hardness (VH) test. The results indicate that the KIC value of FDP/PMMA composite slightly decreased as FDP particles containing increased. The surfaces of the PMMA matrix specimens exhibit a smooth appearance, whereas the FDP/PMMA composite containing 5 wt.% FDP particles displays a rougher surface and FDP particles appearing partially embedded and adhered to the PMMA matrix. With an increase in amount of FDP particles increases from 10 to 15 wt.%, a tendency for particle agglomeration is observed, suggesting that the particles are not fully wetted by the PMMA matrix. The FDP/PMMA composite with different FDP particle concentrations had greater hardness than the PMMA matrix. The optimal results were observed at 5 wt.% FDP particles.

Keywords: PMMA; FDP; Filler Reinforcement; Fracture Toughness; Surface Hardness.

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يهدف هذا البحث إلى تقييم تحمل الكسر. وخصائص صلابة سطح البولي ميثيل ميثاكريلات (بى إم إم أى) المعززة بجزيئات البورسلين السني الدقيقة (إف دى بى) بمستويات نسب وزنية مختلفة. تم تحضير ثمانين عينة وتقسيمها إلى أربع مجموعات. كان راتنج بى إم إم أى هو المجموعة الضابطة، بينما كانت المجموعات الأخرى تحتوي على 5 و10 و15% بالوزن من جزيئات إف دى بى. تم إجراء إختبار الإنحناء ثلاثي النقاط لتحديد صلابة الكسر. (كى أى سي). تم بعد ذلك فحص أسطح العينات المكسورة باستخدام مجهر المسح الإلكتروني (إف إي إس إي إم). بالإضافة إلى ذلك ، تم تقييم صلابة سطح العينات من خلال اختبار صلابة فيكرز (فى إتش). تشير النتائج إلى أن قيمة كى أى سى لمركب إف دى بى/ بى إم إم أى إنخفضت قليلا كلما زادت كمية جزيئاتت إف دى بى. أظهرت أسطح عينات راتنج بى إم إم أى مظهر أملس، في حين أن مركب إف دى بى/ بى إم إم أى الذي يحتوي على 5 بالوزن.٪ جزيئات إف دى بى أظهر سطح أكثر خشونة وظهرت جزيئات إف دى بى جزئيا منغرسة وملتصقة في راتنج بى إم إم أى. مع زيادة كمية جزيئات إف دى بى من 10 إلى 15 بالوزن.٪، لوحظ ميل لتكتل للجزيئات، مما يشير إلى أن الجزيئات لا يتم ترطيبها بالكامل بواسطة راتنج بى إم إم أى. كان لمركب إف دى بى/ بى إم إم أى بتركيزات جزيئات إف دى بى المختلفة صلابة أكبر من راتنج بى إم إم أى. وقد لوحظت النتائج المثلى عند نسبة 5٪ بالوزن من جزيئات إف دى بى.

INTRODUCTION

Currently, PMMA is the predominant material employed in the production of denture bases. While various other materials are available for dental prosthetics, none exhibit the unique characteristics of PMMA. Approximately ninety percent of dentures are constructed from this acrylic polymer, attributed to its superior aesthetic qualities, affordability, ease of handling, and compatibility with straightforward processing equipment. The optimal PMMA formulation must incorporate several critical properties for the effective creation of polymeric denture bases, as the cured polymer needs to be sufficiently rigid to maintain the alignment of teeth during chewing and to reduce uneven stress on the mucosal surfaces. An investigation by [1] examined the characteristics of PMMA and its various modifications, emphasizing the importance of rigidity and durability in preventing breakage and promoting even distribution of forces during chewing.

Despite the fact that traditional partial and complete dentures remain the preferred treatment option in numerous instances due to economic and health-related considerations in comparison to dental implant therapy, it is important to note that no existing denture base material possesses all the desired characteristics, biocompatibility, and aesthetic qualities. Each type of denture material is accompanied by its own set of limitations.

Although PMMA's widespread use in denture fabrication, it has notable limitations due to its vulnerability to fractures and insufficient hardness. Dentures fabricated from PMMA are especially prone to fractures, particularly when exposed to mechanical stresses, such as impacts from dropping or substantial occlusal forces during functional use. The full upper dentures are especially susceptible to midline fractures, largely because of the breakability of the PMMA material [2]. While PMMA demonstrates satisfactory surface hardness, it remains comparatively lower than that of certain modified variants. The surface hardness of PMMA is particularly problematic. The hardness of PMMA is generally lesser than that of modified variants. For instance, a study by [3] revealed that conventional PMMA exhibits a Vickers hardness value close to 18.11. Pure PMMA is constrained by its surface hardness and overall durability; nevertheless, advancements in material science, especially with the integration of different types of reinforcement, have uncovered promising possibilities for improving its mechanical properties.

The quest for optimal materials for denture bases has been a longstanding endeavor among researchers and manufacturers. Despite considerable progress, challenges regarding the properties of these materials continue to exist. The improvement of PMMA's mechanical characteristics has been realized by integrating various materials. Creating a strong interfacial bond between the organic and inorganic materials is crucial for the overall enhancement of the material's characteristics [4]. Numerous efforts have been directed towards augmenting the properties of acrylic materials, particularly PMMA, in the context of denture production. The strategic addition of diverse fillers has proven to be instrumental in enhancing these properties, resulting in significant advancements in mechanical performance and biocompatibility.

The incorporation of glass fibers has led to notable advancements in the characteristics of polymer. Research findings indicate that the integration of glass fibers within the denture base plays a crucial role in determining its mechanical properties; for instance, incorporating fibers on the tensile stress side can enhance both flexural modulus and toughness, while positioning them in areas of neutral stress can bolster overall flexural toughness [5]. Furthermore, the addition of ZrO_2 nanoparticles has been associated with improved flexural strength and impact resistance, indicating their potential as an effective enhancement for PMMA-based denture materials [5, 6].

The addition of metal oxides to PMMA enhanced its properties significantly. This enhancement includes better thermal conductivity and reduced curing shrinkage and water sorption, which are critical for the longevity and comfort of dentures [7]. Moreover, the flexural strength and modulus of PMMA matrix were increased with the incorporation of 5 wt% of filler [8]. The efficacy of microfillers is frequently influenced by their size and distribution into resin matrix. Ongoing investigation into the most effective types and concentrations of fillers is crucial for enhancing the performance of PMMA in high-demand applications such as dental prosthetics. However, specifically the fracture toughness and surface hardness of PMMA dental incorporated with treated FDP particles is never been reported. Therefore, this study is reported on both properties when FDP incorporated into PMMA to serve as reinforcement filler particles.

METHODS

Polymer matrix are consisting of PMMA Aldrich U.S.A, benzoyl peroxide (BPO) Merck Chemical, Germany, and liquid component comprised of MMA (fluka UK), with hydroquinone plus ethylene glycol dimethacrylate (EGDMA) Aldrich U.S.A. FDP (particle size less than 75 μm) were prepared according to our study [9] which used as reinforcement materials. Coupling agents are utilized to create a durable connection between surfaces that would otherwise be nonbonding and incompatible. Silane coupling agent, 3-methacryloxypropyltrimethoxysilane (γ-MPS) was supplied by Sigma-Aldrich. It is used for enhancing the interaction between FDP particles and the organic matrix PMMA because it has two different reactive groups in their molecules of which one can be hydrolyzed.

Treatment of FDP particles

The FDP particles are treated using a mixture of 70% acetone and 30% water, mixed with 10 wt% of the γ-MPS relative to the filler particles. The mixture is stirred for four hours (see Figure 1). This duration ensures thorough dispersion of the silane on the filler particles, facilitating effective surface modification, and then sonicated for about 20 minutes to break down agglomerates and promote uniform coating of the filler particles with the silane. Following the mixing process, the treated blend is subjected to drying at 110°C for twenty-four hours. This step is crucial as it removes any residual solvent and allows the silane to chemically bond to the external side of the fillers, enhancing their hydrophobic properties. This method has been referenced in various studies focusing on dental materials, where γ-MPS is known to improve the compatibility between reinforcement particles and resin matrix, thereby enhancing mechanical properties in composite materials [10, 11]. The treatment results in a hydrophobic surface on the fillers, which is beneficial for applications in dental restorations and other composite materials [12].

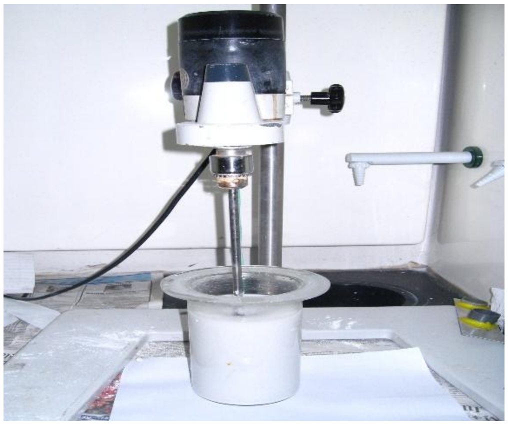

Figure 1: Filler Stirrer Machine

FTIR of FDP particles

Fourier Transform Infrared Spectroscopy (FTIR) is an essential analytical technique that provides detailed insights into the chemical bonding and molecular structure of a sample. The vibrational modes of chemical bonds occur at specific frequencies, and when these bonds are exposed to infrared radiation, they absorb energy at frequencies that correspond to their vibrational characteristics. The measurement of this energy absorption across various frequencies produces a spectrum that can be employed to identify the compounds present in the sample. In this investigation, FTIR analyses were conducted on both untreated and silane-treated FDP particles to assess the chemical structure following treatment. A sample consisting of approximately 5 mg of FDP particles mixed with 95 mg of potassium bromide (KBr) was prepared and compacted into thin pellets using a hydraulic press, applying a force of 8 tonnes for duration of 3 minutes. The resulting pellets were subsequently analyzed with a Nicolet Avatar 360 FTIR spectrophotometer.

Specimen preparation

The specimen preparation process encompasses both solid and liquid phases. The solid phase is composed of PMMA and 0.5 wt.% BPO as the initiator, along with treated FDP particles. To achieve a homogeneous mixture of the solid components, the planetary ball milling technique was utilized for duration of one hour, with intermittent pauses every fifteen minutes to mitigate the risks of overheating and premature polymerization. The liquid phase is primarily made up of methyl methacrylate, which acts as the principal reactive agent for polymer synthesis, supplemented by 10% by volume of ethylene glycol dimethacrylate, which functions as a cross-linking agent. Additionally, a small quantity of hydroquinone (0.0025 wt.% of the liquid monomer) is incorporated to prevent undesired reactions during storage and handling. This formulation is commonly employed in applications that necessitate durable acrylic materials. The ratio of powder to liquid in the mixing process is maintained at 3:1. Once the mixture attains a dough-like consistency, it is placed into a mold and subjected to a pressure of 14 MPa at room temperature for thirty minutes. The specimen polymerization is conducted in a water bath at 78°C for one and a half hours, after which the mold is allowed to cool gradually at room temperature.

Fracture toughness test

The fracture toughness of a material is a critical property that indicates its capacity to withstand fracture in the presence of a crack, making it essential for nearly all design applications. A total of 40 rectangular specimens were produced from molded plate with dimensions of 4 mm in thickness, 20 mm in width, 64 mm in span length, and an overall length of 80 mm, featuring a notch length of 4 mm. These specimens were categorized into four distinct groups: the control group consisted of a pure PMMA matrix, while the other three groups incorporated varying concentrations of FDP particles (5, 10, and 15wt.%) into the PMMA matrix. Each group contained ten specimens (n = 10). The fracture toughness was assessed using a single edge notch bending test (SEN-B) in accordance with ISO 13586:2000 standards. The SEN-B specimens underwent testing at a crosshead speed of 1.00 mm/min. The fracture toughness values (K_{IC}) were computed using the equation: $K_{IC} = 3PSa^{1/2}y/2tw^2$. The geometry correction factor (y) was derived from the following formula: $y = 1.93 - 3.07\,(a/w) + 14.53\,(a/w)^2 - 25.11\,(a/w)^3 + 25.8\,(a/w)^4$, where P represents the peak load (N), S denotes the span length (mm), a indicates the notch length (mm), t is the specimen thickness (mm), and w is the specimen width (mm).

Field emission scanning electron microscope (FESEM)

The surface structure and the morphology of the fracture surface of the composite samples were analyzed by the angles and energies of electrons scattered by the surface atoms using an FESEM, specifically the Leica Cambridge S-360 model, capable of producing images with micron-level resolution.

Vickers Hardness (VHN) Test

The evaluation of hardness was performed following the ASTM 384-2008 standard. A calibrated Vickers Hardness Tester FV (Future-Tech) was utilized to exert a force of 0.3 Kgf (kilogram-force) through a diamond indenter for a period of 10 seconds on the surface of the specimen, with the diagonal length measured optically in micrometers (μm). The dimensions of the specimen used for the Vickers hardness test were defined as: length of 10 mm, width of 10 mm, and thickness of 3 mm. In total, 40 specimens were fabricated according to the concentration of FDP particles incorporated into the

PMMA matrix (n = 10 each group) were tested and the average of readings was calculated. Three indentations were performed at various locations on one side of each specimen, and a mean value was subsequently determined. The Vickers hardness number was computed according to the equation specified in the ASTM standard: $HV = P/A_S = 2P \sin(\alpha/2)/d^2 = 1.8544\ P/d^2$. In this equation, P represents the load in kilogram-force (Kgf), A_S denotes the surface area of the indentation in square millimeters (mm^2), d refers to the average of the two diagonals of the indentation, d_1 and d_2, measured in millimeters, and α indicates the face angle of the indentation, which is 136º. Given that grams-force and micrometers are more commonly utilized than kilogram-force and millimeters, the Vickers hardness number can be conventionally expressed as $HV = 1854.4\ P_1/d_1^2$, where P_1 is the load in grams-force (gf) and d1 is the mean diagonal of the indentation in micrometers (μm). The improvements in hardness are calculated using the following formula: HV improvements (%) = (HVm-HVm) / HVm × 100, where c and m denote the composite and matrix, respectively.

Statistical analysis

The analysis conducted with SPSS version 19 utilized an ANOVA, which was subsequently followed by Tukey's post-hoc analysis, establishing a significance threshold of 0.05.

RESULTS AND DISCUSSION

FTIR of FDP particles

Coupling agents are typically utilized with fillers to improve the mechanical characterizes of filler/resin matrix. This study focused on improving interfacial adhesion by creating a chemical bond between the FDP particles and the Polymer matrix. To validate the silanation treatment, Fourier Transform Infrared (FTIR) spectra of FDP particles were examined both prior to and following treatment with the silane coupling agent (refer to Figure 2). The detection of bands corresponding to the carboxyl group C=O (at a wave number of 1720 cm^{-1}) and the C-H stretching band (at a wave number of 2946 cm^{-1}) confirmed the presence of the coupling agent on the surface of the filler particles. The spectra suggest that the coupling agent γ-MPS has been successfully grafted onto the FDP particles.

A study by [8] focused on silane-modified fillers and demonstrated that the band at 1720 cm^{-1} serves as an indicator of successful silanization, which significantly improves the interfacial compatibility between the filler and the polymer matrix. The detection of this peak implies that the silane coupling agent has effectively enveloped the filler particles, thereby enhancing their dispersion and adhesion within the polymer matrix. The C=O stretching vibration is typically observed in the range of 1710-1740 cm^{-1} for saturated aldehydes and ketones, with aliphatic aldehydes specifically showing a C=O stretch around 1720 cm^{-1}, while esters exhibit similar absorption patterns. FTIR spectra of silanated fumed silica samples reveal the characteristic adsorption bands associated with MPS. The band at 1718 cm^{-1} corresponds to the free C=O stretching vibration, whereas the band at 1700 cm^{-1} is attributed to the C=O group that is hydrogen-bonded with silanol groups [13]. Additionally, the C-H stretching band observed in the spectrum of γ-MPS at approximately 2946 cm^{-1} is crucial for identifying the presence of methylene ($-CH_2-$) and methyl ($-CH_3$) groups in the molecular structure. This band may result from a combination of asymmetric C-H stretching modes, typically found around 2926 cm^{-1}, and symmetrical modes near 2853 cm^{-1}. The intricate structure of γ-MPS, along with the interactions among various functional groups, likely contributes to this spectral characteristic. C-H stretching vibrations for alkanes are generally noted between 2850 and 2960 cm^{-1} [14]. Also [15] reported that the aliphatic C–H stretching vibration band appeared at 2946 cm^{-1}.

Silane coupling agents are indeed recognized as the most prevalent products used to chemically bond inorganic filler particles to organic resin matrices. Their primary role is to enhance the adhesion between fillers and polymers, while also improving hydrolytic stability, which is crucial for the longevity and durability of composite materials. The organofunctional silane γ-MPS has been widely utilized in dental applications, particularly in the treatment of filler particles for dental resin composites. Its main purpose is to strengthen the bond between particles of reinforcement and the polymer matrix, thereby augmenting the mechanical properties and overall efficacy of dental materials [16, 17]. Silane coupling agents possess two distinct reactive groups within their molecular structure, one of which is capable of hydrolysis. The interaction between γ-MPS and polymer matrices, especially in composite materials, is essential for enhancing mechanical properties and overall performance. The primary facilitation of these interactions occurs through strong bonding mechanisms present at the interface between the reinforcement particles and the polymer. γ-MPS serves as a coupling agent that establishes chemical bonds between inorganic fillers and the polymer matrix. This bonding is achieved through hydrophilic groups on γ-MPS that engage with polymer chains, effectively forming a molecular bridge that enhances adhesion and stress transfer between the two phases [18]. The strong connections formed by γ-MPS with both the polymer matrix and filler particles lead to several advantageous outcomes, including improved mechanical properties, reduced polymerization shrinkage, and enhanced thermal stability.

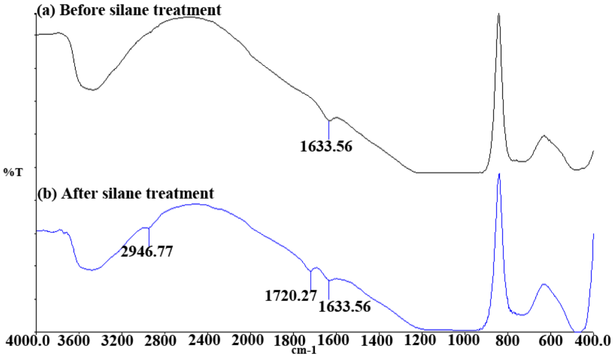

Figure 2: FTIR spectra of FDP particles

Fracture toughness

Figure 3 illustrates K_{IC} value of the PMMA matrix and FDP/PMMA composite at different concentrations. It is noted that PMMA matrix exhibits slightly higher K_{IC} value than do the FDP/PMMA composite. The K_{IC} value of FDP/PMMA composite specimen containing 5, 10 and 15 wt % filler particles formulations are found slightly decreased comparing to PMMA matrix specimen. This finding aligns with the research conducted by [19], who noted that pure PMMA demonstrates enhanced fracture toughness compared to its composite variant. According to [20] who conducted a study on the assessment of fracture toughness and impact strength of PMMA reinforced with nanoparticles and fiber as advanced denture base materials, it was found that fracture toughness values increased with increasing filler content up to 3 wt%. However, beyond this threshold, specifically at 5 wt% and above, a decrease in fracture toughness values was observed. Diverse trends have been previously observed in the variation of K_{IC} with filler addition where some researchers reported an increase in the K_{IC} while others reported a decrease. As per [19] neat PMMA showed better K_{IC} properties than PMMA composite. At low HA levels (up to 5 wt.%) [21] found that the K_{IC} of PMMA increased. The reasons for this heterogeneity have been ascribed to several elements including the volume and the type of filler.

The underlying reason for this study lies in the relatively weak bonding between the PMMA matrix and the filler particles, which results from the partial embedding of these particles within the matrix. Additionally, the anisotropic orientation of the particles within the composite contributes to an increased resistance to local plastic deformation, thereby rendering the composite more brittle. Consequently, crack propagation occurs rapidly due to the composite's limited capacity to absorb energy through plastic deformation. As the concentration of filler particles rises, the availability of PMMA as a matrix diminishes, leading to a reduction in the composite's toughness. Fracture toughness quantitatively measures a material's resistance to brittle fracture in the presence of a crack; materials with high fracture toughness values are more likely to experience ductile fracture, whereas those with low values are prone to brittle fracture. Denture fractures typically occur due to stress concentration and flexural fatigue, with stress concentration manifesting at specific points where the material endures heightened stress, often resulting from design imperfections such as sharp notches or insufficient thickness. In dentures, common areas for stress concentration include the labial frenum notch and regions of reduced thickness [22, 23].

The number of fillers significantly influences the mechanical characteristics of composite materials. An increase in the polymer content typically results in enhanced flexibility, toughness, and improved binding with the filler. Conversely, a reduction in polymer constituents can lead to structural integrity issues. When a load is applied, stress concentrations develop within the material, while the distribution of stress is hindered, resulting in cracking at vulnerable locations. Further application of stress may render the already compromised matrix incapable of withstanding additional forces. However, if the filler is uniformly distributed and exhibits strong interfacial adhesion with the polymer, an optimal balance of stiffness, strength, and toughness can be achieved. For example, the addition of fillers to polypropylene composites has been demonstrated to improve toughness, especially when these fillers are well-integrated within the matrix. The ability of fillers to improve toughness is dependent on their effective dispersion throughout the polymer matrix. The effective dispersion of fillers is crucial for achieving a more consistent distribution of stress during mechanical loading, which in turn alleviates stress concentrations that may result in material failure. In contrast, inadequate dispersion of fillers can introduce vulnerabilities within the composite, thereby compromising its overall mechanical strength [24, 25].

To enhance the strength characteristics of inorganic filler-resin composites, the coupling agent γ-MPS is typically utilized. Silane coupling agents are characterized by their reactive silanol groups, which can undergo condensation to form siloxane (Si-O-Si) bonds. This reaction takes place at the interface of dissimilar materials, such as a polymer and a silica surface. The establishment of these siloxane connections significantly improves the composite properties by fostering a strong interfacial bond [26, 27].

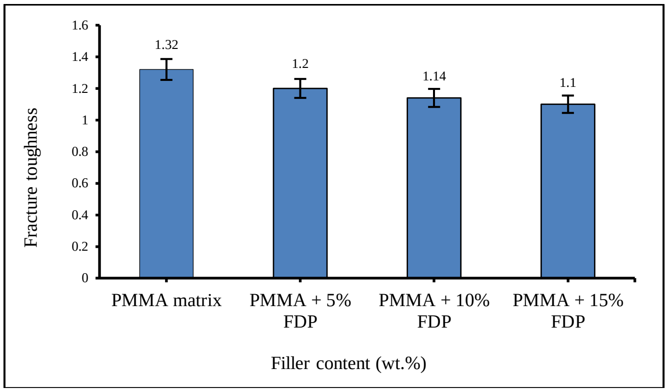

Figure 3: K_{IC} test values

FESEM

Figures 4 to 7 depict the FS resulting from a fracture test conducted on both the PMMA matrix and the FDP/PMMA composite at varying concentrations of FDP particles, as observed in FESEM micrographs. Figure 4 presents the FS of the PMMA matrix, characterized by a stripped pattern that arises from crack propagation originating from the initiation point. This pattern signifies stable crack propagation, while the smooth morphology of the fracture surface suggests uniform crack propagation indicative of brittle behavior within the PMMA matrix. In Figure 5, the FS of 5 wt.% FDP/PMMA composite is displayed. Here, the surface appears rough, with filler particles appearing partially embedded and bonded to the matrix. The increased surface roughness suggests a longer crack path and a release of greater fracture energy, thereby enhancing the fracture toughness of the PMMA matrix filled with FDP particles.

Figures 6 and 7 illustrates the FS of 10 and 15 wt.% FDP/PMMA composite, respectively. The interaction between the FDP particles and the polymer in these formulations exhibits similar characteristics. A brittle fracture is evident, as indicated by the irregular, flake-like rough surface, a trait commonly associated with brittle failure. As the filler loading increases from 10 to 15 wt.%, a tendency for filler particle agglomeration is observed, suggesting incomplete wetting of the particles by the matrix. Consequently, some filler particles appear detached from the polymer matrix, indicating a weak filler-matrix interaction that facilitates de-bonding before the full development of plastic deformation.

The relationship between filler particles and the PMMA matrix is essential for determining the mechanical characteristics of composite materials. Increased amount of filler particles may result in agglomeration, which diminishes the effective surface area for bonding with the PMMA matrix. Such agglomeration can considerably impair interfacial adhesion, leading to the detachment of filler particles from the matrix [28, 29]. As the concentration of filler increases, the detachment phenomenon becomes more evident. At lower filler levels, detachment occurs under relatively low stress, whereas at higher levels, detachment and shear yielding occur concurrently [28]. This suggests that an overabundance of filler can compromise the mechanical stability of the composite material.

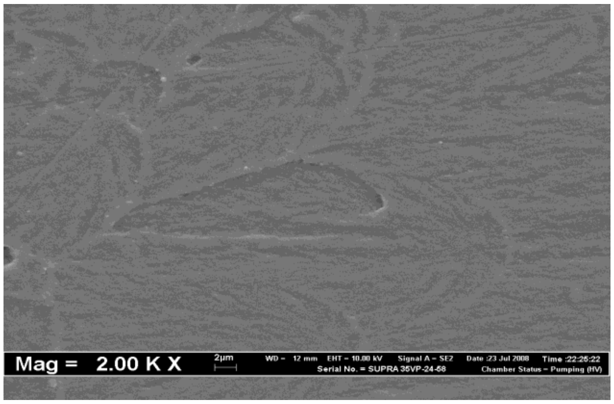

Figure 4: FS of PMMA matrix

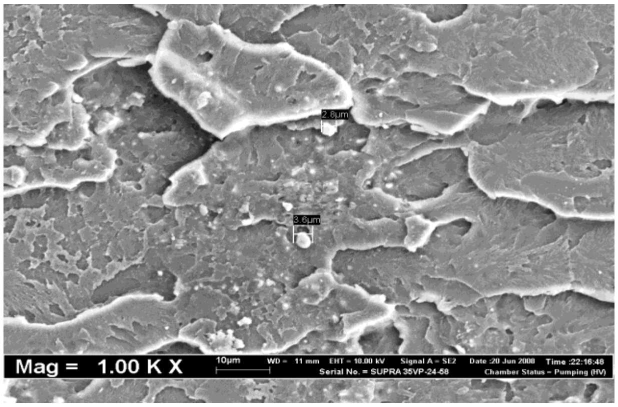

Figure 5: FS of 5 wt.% FDP/PMMA composite

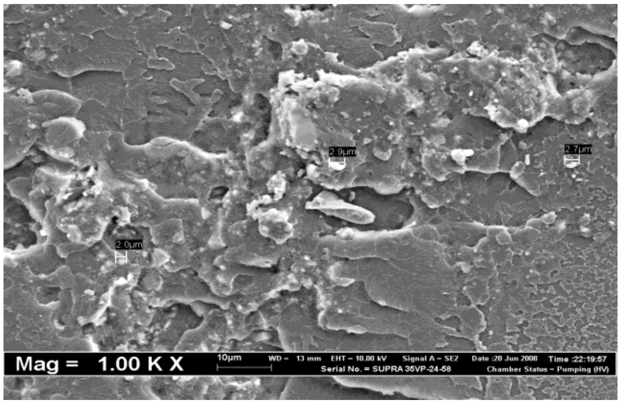

Figure 6: FS of 10 wt.% FDP/PMMA composite

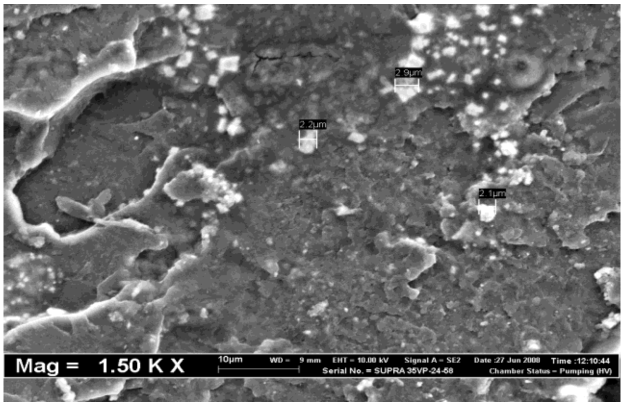

Figure 7: FS of 15 wt.% FDP/PMMA composite

Surface hardness

Table 1 presents the Vickers hardness values (Kg/mm²) and the percentage improvements in hardness for both the PMMA matrix and the FDP/PMMA composite with comprising various concentrations of FDP particles. The data indicate a notable increase in surface hardness for all formulations of the

FDP/PMMA composite when compared to the PMMA matrix ($p < 0.05$). Nevertheless, the results from the Tukey test reveal that there are no statistically significant differences in surface hardness among the formulations that incorporated FDP particles at different concentrations. The results are in agreement with previous research, which has revealed a significant augmentation in surface hardness of PMMA denture bases through the addition of filler particles at varying levels [2, 30]. The increase in hardness values observed in the FDP/PMMA composite is primarily influenced by the content of the filler, which contributes to the elevated hardness of the dispersed phase. Furthermore, the even distribution of the dispersed phase restricts the deformation of the matrix. This enhancement in properties is realized through the addition of particles to polymer. An increase in the concentration of FDP particles is associated with a heightened hardness of the polymer composite. This relationship is corroborated by the research conducted by [31, 32], who documented a notable increase in surface hardness with the augmentation of filler particle concentration, while the acrylic resin with the least filler content demonstrated the lowest hardness.

The hardness of a material is a critical characteristic when evaluating restorative materials, as it allows for an assessment of the material's ability to withstand permanent surface indentation or penetration. The Vickers hardness test serves as a method to determine the resistance of a material's surface to penetration by a point under a specified load. In an ideal plastic material, the Vickers hardness value correlates directly with the yield stress. The resulting indentation, or depression from penetration, is a consequence of plastic deformation occurring beneath the indenter. The interpretation of hardness measurements for PMMA is affected by various experimental factors, especially at shallow penetration depths. Significant variables include the rate of loading, duration of holding, surface preparation, and the roughness of the surface. Notably, PMMA hardness exhibits sensitivity to both loading rate and holding time, with an increase in loading rate resulting in a higher elastic modulus and hardness, whereas an extended holding time tends to reduce these properties [33]. Surface preparation is critical in ensuring consistent hardness measurements. The method of preparing the PMMA surface-whether through polishing, grinding, or other techniques-can alter the surface characteristics, leading to variability in hardness results. For instance, a smoother surface generally yields higher hardness values due to reduced microstructural defects that can absorb energy during indentation [2, 34]. The roughness of a surface significantly influences the outcomes of hardness testing. An increase in surface roughness may cause irregular contact between the indenter and the material, which can subsequently yield lower recorded hardness values. Studies have shown that rougher surfaces tend to scatter the load applied by the indenter, which may not accurately reflect the material's intrinsic hardness. The scattering effect occurs because the uneven surface causes variations in the contact area, leading to inconsistent measurements [35]. Therefore, maintaining a controlled surface roughness is essential for reliable hardness measurements. PMMA surfaces at low penetration depths show a peculiarly harder response, influenced by penetration depth, applied normal load, and deformation rate [36]. An increase in surface roughness can result in discrepancies between the hardness values measured at depths similar to the roughness scale and the true hardness of the material. This effect arises from the fact that surface irregularities alter the contact area during hardness testing, which in turn leads to measurement inaccuracies. Consequently, as surface roughness intensifies, these discrepancies may become more significant, thereby undermining the reliability of the recorded hardness values.

Table 1: Vickers Hardness test values

Formulations	Vickers Hardness (Kg/mm^2)	Hardness Improvement (%)
PMMA matrix	12.56 ± 0.5	-
FDP/PMMA composite (5wt %)	16.06 ± 1.4	27.86
FDP/PMMA composite (10wt %)	16.67 ± 1.3	32.64
FDP/PMMA composite (15wt %)	18.18 ± 0.4	44.74

CONCLUSION

In light of the limitations inherent in this study, it can be concluded that FDP/PMMA composite with different FDP particles wt.% have been effectively produced. The experimental results indicate that the mechanical properties can be adjusted by varying the proportion of FDP particles. Notably, the PMMA matrix demonstrates a slightly superior fracture toughness compared to FDP/PMMA composite containing various concentrations of FDP particles. Furthermore, FESEM analysis of the FS of FDP/PMMA composite specimens with differing FDP concentrations reveals a rough surface, with an observed tendency for FDP particle agglomeration as the content increases from 10 to 15 wt.%, suggesting incomplete wetting of the FDP particles by the matrix. The surface hardness of the FDP/PMMA composite at varying wt.% of FDP particles exhibits a significant increase in comparison to the PMMA matrix. This enhancement in hardness values for the FDP/PMMA composite can be attributed to the filler content, which contributes to the overall hardness due to the hardness of the filler particles. Therefore, FDP/PMMA composite with 5 wt.% FDP exhibited the most favorable characteristics and should be well-suited for use in denture applications.

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