

Improvement of Mechanical Properties for PMMA Based Denture Material

Pritosh Tomar¹, Naveen Rana^{2*}, Mohit Sharma³, Mohit Bhargva⁴, Neel Kamal Batra⁵

Abstract

Denture material properties can be improved by various fiber addition. The most often used denture materials are polymethyl methacrylate (PMMA) resins; nevertheless, they do not have a high flexural strength (FS). This investigation compared the mechanical characteristics of an injection-molded polyamide. Deflex, an additional injection-molded PMMA base material, SRIVOCAP, and a typical compression-molded PMMA (Meliodent). The flexural qualities (deflection, bending strength, and bending modulus) of 100 different denture base materials were assessed. Specimens measuring (13 x 4.5 x 4 mm³) and fulfilling Bureau of Indian Standards (BIS) specification number requirements were created. On an Instron testing apparatus with a 5 mm/min crosshead speed, a three-point bending test was performed. To compare microhardness results, the Knoop hardness test was employed. Tensile test and bending test suggested that chopped glass fibre with other natural fiber shows the maximum strength. SEM test shows the good bonding of fibre with PMMA. High amount of GF can also be harmful so optimum wt. percentage composition used in testing procedure. It is observed that, hybrid mixing materials generate an ideal combination in the resulting composite by offering improved balance and stability between the qualities of the combined fillers. Hence, it is recommended to use natural and vegetable fibres for future work.

Keywords: Denture base material, PMMA base denture, Polymer denture, Flexural strength, Composite resin

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INTRODUCTION

Dentism is still a major public health issue globally despite improvements in preventive dentistry (1). Despite the growing popularity of dental implants, the demand for removable dentures, whether they are partial or complete, remains relevant. Due to the oral cavity's exposure to chewing pressures, temperature fluctuations, pH variations, and saliva's abundance in enzymes and bacteria, denture materials must adhere to a variety of specifications. The substance must maintain its inertness throughout usage and not dissolve in the mouth. It also has to have an unobtrusive flavour and odour. The commonly employed materials in prosthetic dentistry are composed of poly (methyl methacrylate) (PMMA). These materials are known for being cost-effective, easily manufacturable, and possessing excellent aesthetic properties (2).

Resin-based materials, categorized as either

dimethacrylates or bis-acryl/composite resins (such as bisphenol A-glycidyl dimethacrylate (Bis-GMA) and urethane dimethacrylate (UDMA)), as well as monomethacrylates (or acrylic resins), encompassing poly-methylmethacrylate (PMMA), can be classified into two distinct groups. (1). These materials exhibit variations in their chemical composition, mechanical properties, and polymerization kinetics [1]– [3].

Bis-acryl/composite resins (BACRs) consist of an organic polymeric matrix along with a coupling agent, pigment, catalyst, and inhibitor. Furthermore, they incorporate fillers such as crystalline quartz, pyrogenic silica, glasses of barium, zinc, and strontium, as well as ceramics [4]. On the other hand, dimethacrylates possess a firm, cross-linked structure due to the presence of highly viscous and plentiful multifunctional monomers, which are capable of forming cross-links with other polymeric chains. This rigid, cross-linked structure grants dimethacrylates favorable mechanical properties when subjected to external forces. These materials are made to be robust, manageable, and polishable thanks to the cross-linking and inorganic loading(3–7). The pressures required to cause a BACR to shatter are within its capacity. They are appropriate for temporary and permanent repairs due to all of these characteristics (8,9). As soon as the stress exceeds the elastic deformation limit, BACRs often shatter without significant plastic deformation. In light of this, they might be referred to as brittle materials(5,6).

In applications such as automotive, aircraft, construction, or medicine, polymer-matrix composites (PMC) have been touted as a great substitute for metals and ceramics. (10) (11) (12)As demonstrated in recent review publications (3,4), the variety of PMC applications is expanding and diversifying with each new advancement. Fiber reinforcements are commonly employed to enhance the strength, stiffness, fracture resistance, impact resistance, and fatigue resistance of Polymer Matrix Composites (PMCs). The strength of the bond between the fibers and the matrix is significantly impacted by the surface conditions of the fibers, thus influencing the overall mechanical performance of the composites. The strength characteristics obtained by fibre reinforcing are also influenced by fibre orientation (5). Advanced computational tools, artificial intelligence models, and more effective manufacturing techniques have all been used to produce goods with ideal composite architectures. Researchers have enhanced the mechanical characteristics of denture bases by focusing on composite materials of fiber-reinforced PMMA [10]– [15]. Among various fiber types such as glass, aramid, carbon, nylon, and ultrahigh-modulus polyethylene, glass fiber is the most commonly employed in denture bases due to its transparent nature and favorable surface properties [15]. The fundamental mechanism underlying fiber adhesion is attributed to the hydroxyl groups present on the surface of glass fibers, which react with resin monomers through the use of silane coupling agents. Prior to silanization, glass substrates are often treated with acid or base to enhance the quantity of surface hydroxyl groups. Notably, woven glass fibers have demonstrated the capability to enhance the impact and fatigue strengths of PMMA denture base materials, in contrast to unidirectional fibers.

Limited data exists regarding the impact of acid or base-treated glass fibers on the mechanical properties of PMMA matrix composite materials employed in denture bases. Therefore, this study aims to explore the influence of HCL and NH₄OH aqueous activation pretreatments on the mechanical properties of PMMA/woven glass fiber composite materials. The vitrification-induced isothermal cure processes' diffusion control and potential interfacial adhesion mechanisms have both been studied. (13)

MATERIAL AND METHOD

Material used was pure PMMA with additives various fiber with different compositions. Samples can be made up of Pure PMMA (10 samples), PMMA+ GF 1.5%+ WF 1.5%(10 samples), PMMA+ GF 1%+ WF 1%(10 samples), PMMA+ WF 1.5% (10 samples), PMMA+ GF 1.5% (10 samples), PMMA+ GF 1% (10 samples), PMMA+ WF 1% (10 samples). Where PMMA is abbreviated as Poly-methyl methacrylate, GF is Glass fiber (mixture of 5mm and 3mm length size) and WF is woolen

fiber (5mm length size). Some machines and tools are used to makes samples according to the standards like notching machine. A notching machine allows you to swiftly prepare bending and welding tasks at precise angles without cutting, such as for support frames or sheet metal trays. Corners must be carefully cut out on workpieces that will be bent into 3-D curves or containers to reduce rework. These hydraulic notching machines offer exceptional repeatability due to their precisely adjustable stops and angles.

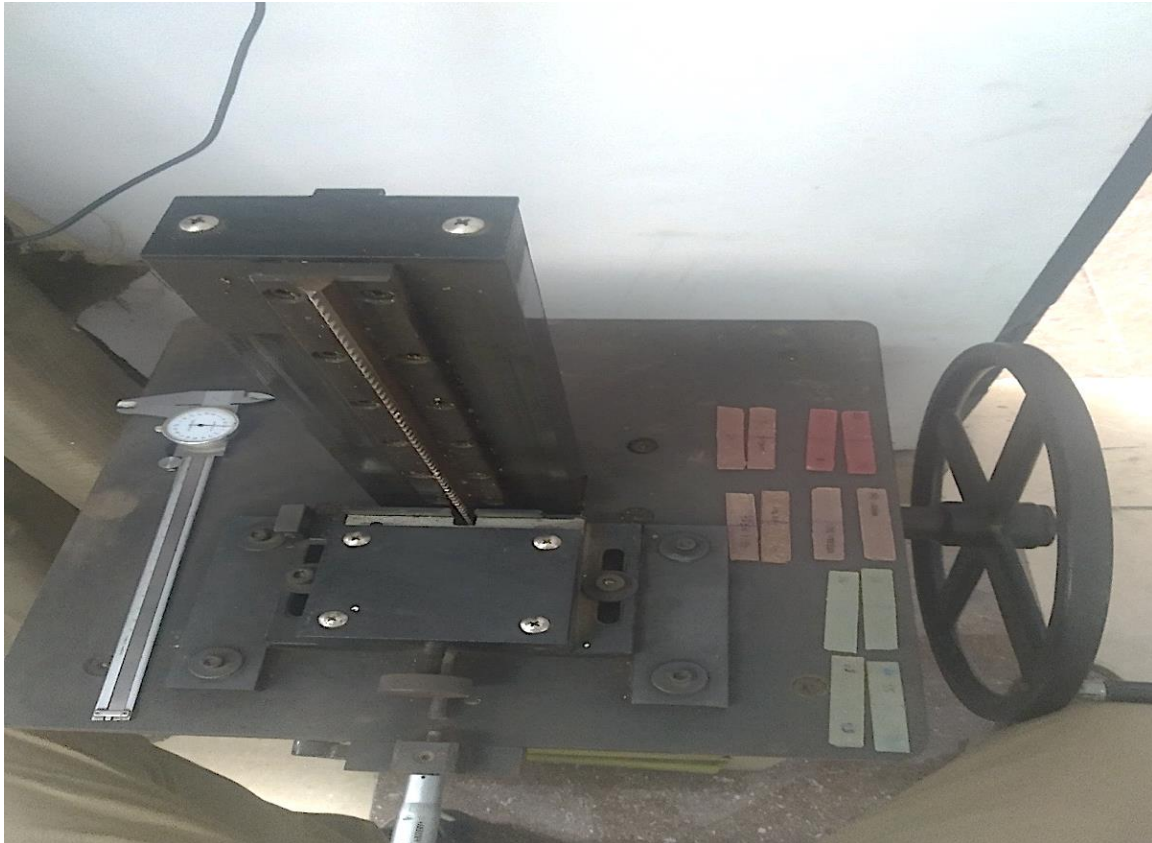


Figure 1. Notch machine used to make notch on samples for impact test according to ISO standard.

Notching machine help to cut notches as per the BIS, ASTM or other specified standard. It is used to cut samples according to requirement of notch angle and depth of cut of notch. So sample preparation work can be accurate and precise. An apparatus depicted in Figure 1, commonly known as a notching machine, is employed. In mechanical engineering and materials science, a deliberate introduction of a V-shaped, U-shaped, or semi-circular discontinuity into a planar material is termed a "notch." Such notches in structural components lead to localized stress concentrations, which can trigger and propagate fatigue fractures. Within materials characterization, notches are utilized to evaluate factors associated with fracture mechanics, including fracture toughness and the rates of fatigue crack propagation. Moreover, notches find frequent application in material impact testing when a precisely controlled origin of a morphological crack is necessary to ensure consistent assessment of the material's resistance to fracture. One of the most widely used tests is the Charpy impact test, wherein a pendulum hammer strikes a specimen featuring a horizontally positioned notch.

The primary mechanisms governing the adherence of glass fibers to denture base resin involve the presence of hydroxyl groups on the fiber surface and the interaction of these groups with resin monomers through silane coupling agents. To enhance surface hydroxyl group content, glass substrates are often subjected to acid or base treatments prior to silanization. These treatments aim to facilitate potential trialkoxysilane surface modification processes. When glass fibers are treated with

3-(trimethoxysilyl) propyl methacrylate (3-TMSPMA), methoxy groups can undergo hydrolysis in an aqueous environment, leading to the formation of silanol groups and the release of methanol. Silanol groups can then undergo self-condensation or condensation with hydroxyl groups present on the glass fiber surface. This results in the creation of covalent links with the substrate, forming polymeric siloxane structures. PMMA Sample after preparation as shown in below Fig. 2. Slightly variation seems in different sample like colour of pure PMMA sample are lighter as WF addition provides colour to the samples. We can test these samples on UTM.

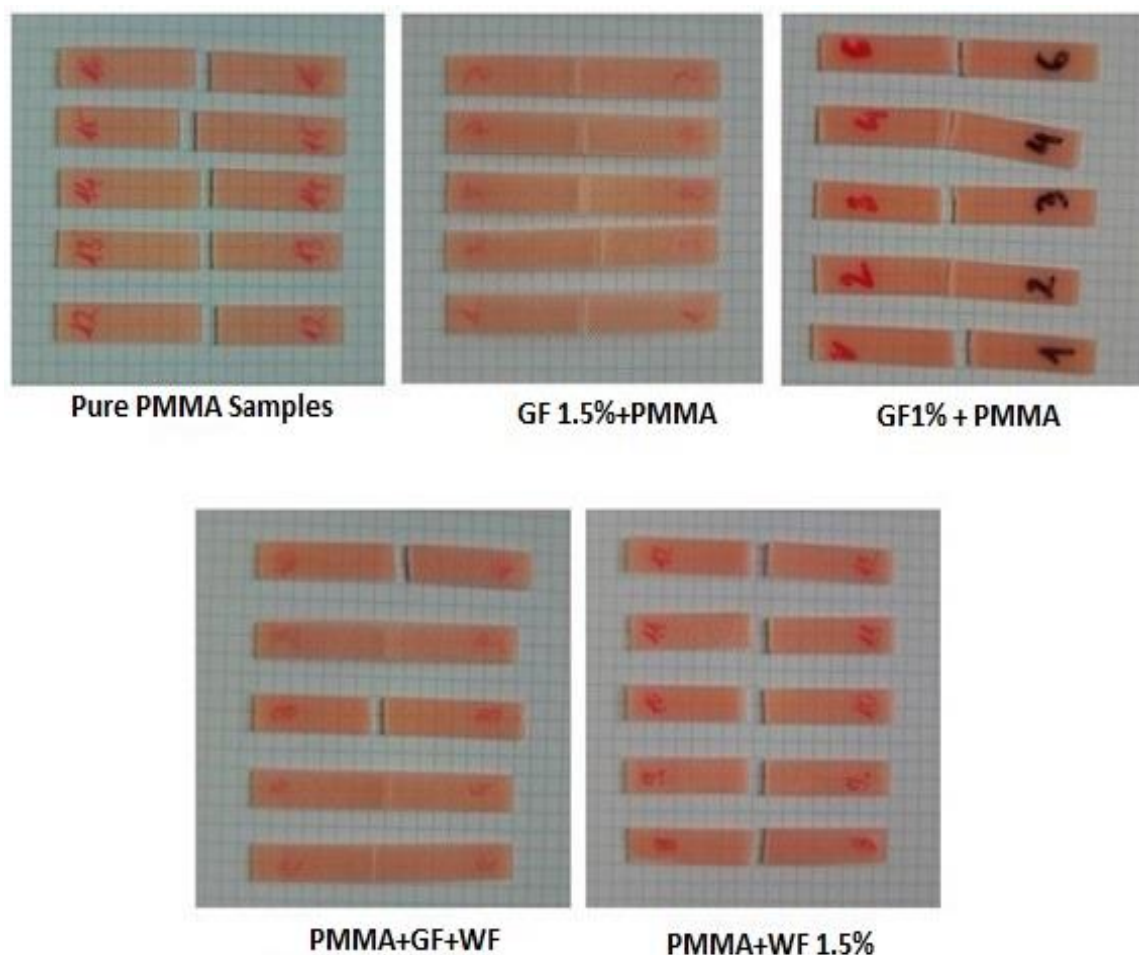


Figure 2. Various composition Samples Made for testing.

MATERIAL AND METHOD

Various tests can be conducted to analyze the correct composition for denture material some are as follows-

Impact Strength

As shown in Figure 3 below it signifies highest impact strength can be shown by the GF 1.5% / WF 1.5% / PMMA type of sample strips and lowest impact strength values can be shown by pure PMMA readings as GF and WF composition increases Impact strength also increases and maximum at 1.5% composition of GF and WF afterwards it starts decreasing lowest compressive strength can be observed in pure PMMA which is taken as reference to all other additives of fibres. Based on a previous study, vegetable fibers and oil palm empty fruit bunch (OPEFB) have been suggested as natural fiber reinforcements for strengthening denture base resins, specifically ramie fiber. The incorporation of OPEFB was observed to enhance the flexural modulus and strength of acrylic resin (14).

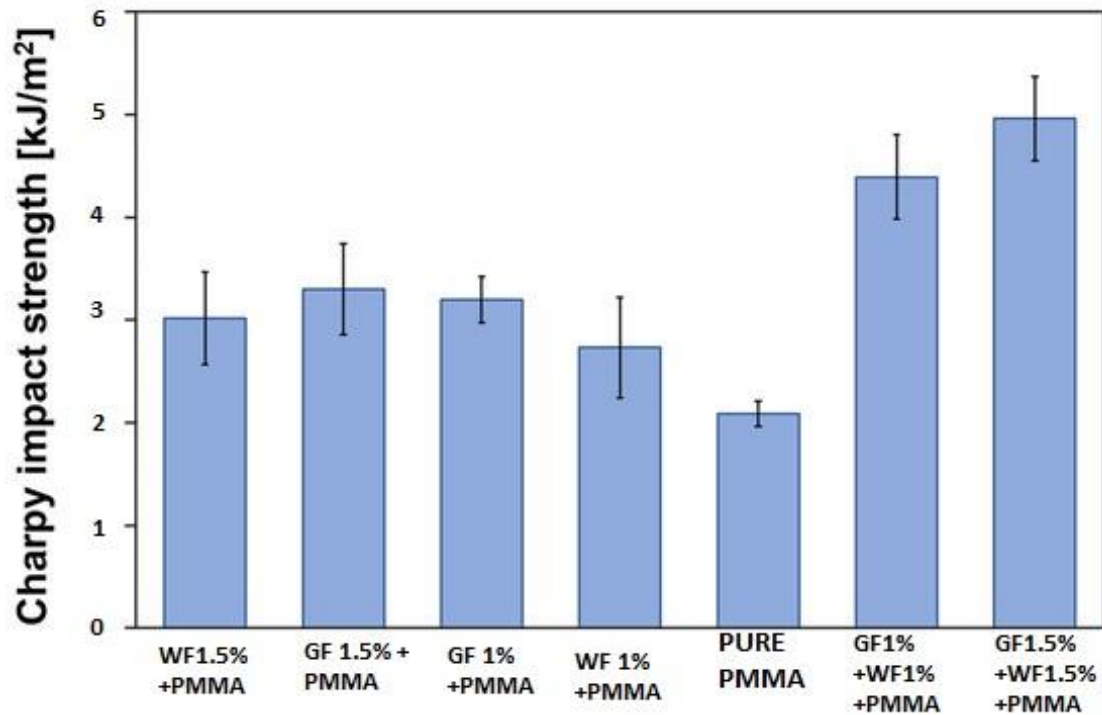


Figure 3. Comparison of Impact Strength data of the sample strips.

The mechanical characteristics of composites are influenced by several elements, none of which can be considered in isolation. As can be observed from the data, pretreating glass fibers with HCl increased their hydroxyl group content. This could potentially influence the chemical bonding facilitated by silane coupling agents and the attachment of the glass fibers to the polymer matrix. It is also reasonable to infer that HCl etching makes the fibers' surfaces rougher, resulting in mechanical interlocking. Nonetheless, due to the leaching of ions from both the fiber surface and the fiber core during H_2SO_4 pretreatment, there is a potential for degradation of the glass fiber structure. In accordance with our results, the bond between the glass fabric and the matrix significantly influences the flexural modulus of composites, surpassing the influence of the matrix itself. Following table 1 values can be converted by suitable conversion factor. (15)

Table 1. UTM and charpy impact test result of material.

Material	Bending Strength (M±SD)	Flexural Modulus (M±SD)	Impact Strength
PMMA	90.9 ± 4.4 a	2.94 ± 0.10 a	9.68
PMMA/GF 1%	119.4 ± 38.5 a	3.11 ± 0.79 a	16.26
PMMA/GF 1.5%	135.1 ± 8.57 a	3.17 ± 0.33 a	25.41
PMMA/WF	118.0 ± 51.5 a	3.57 ± 0.39 b	14.47
PMMA/GF/WF	130.3 ± 23.5 a	3.97 ± 0.34 b	15.95

In linear polymers like PMMA, the entanglement of polymer molecules is evident in the solution's viscosity. These intermolecular interactions play a pivotal role as internal factors influencing the final morphology of electrospun materials. They lead to the formation of structures like cups, rings, and other morphologies even at very low concentrations, rather than just fibers. We analyzed the specific viscosity as a function of PMMA content. At extremely low concentrations of PMMA, viscosity demonstrates a nearly linear relationship with concentration, yielding an intrinsic viscosity of 145.4 cm^3/g . Subsequently, viscosity experiences a nonlinear increase with higher concentrations of PMMA. Comparing the observed intrinsic viscosity of PMMA in DMF with the polymer's anticipated value of 150.3 cm^3/g and its molecular weight of 996,000 g/mol , an excellent agreement is observed. (16)

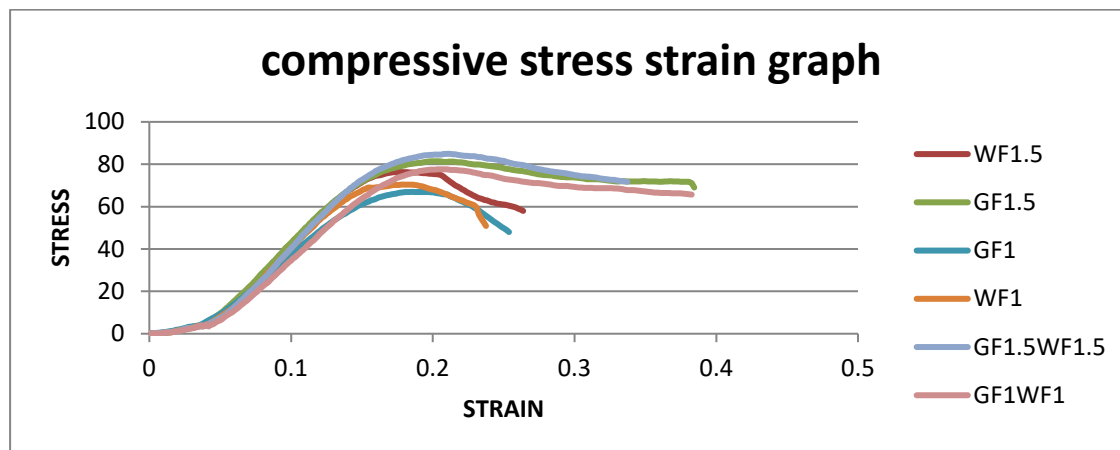


Figure 4. Compressive Stress-Strain Graph.

Compression Test

In the context of full or partial denture applications, poly methyl methacrylate exhibits satisfactory tensile and compressive strength. The compressive strength of a specimen refers to the pressure required for its fracture. Factors impacting the compression failure of composite materials include characteristics of both the matrix and the reinforced material, encompassing volume percentage, interface properties, form, and dimensions of the reinforced elements. The compression test adheres to ASTM standards and is conducted using a universal testing apparatus with a crosshead (strain rate) set at 5 mm/min, applying a load until the specimen fractures. Following the completion of finishing and polishing, the test specimens are submerged in distilled water at $(37\pm 1)^{\circ}\text{C}$ for one week (48 hours). Typically, five specimens are utilized for most tests, which are carried out in an ambient air environment at room temperature $(23\pm 2)^{\circ}\text{C}$. This testing state is vital as mastication forces primarily manifest as compressive forces. The compression test is particularly apt for comparing brittle materials, which exhibit limited resilience under strain and consequently yield relatively lower results. As such, this test finds application in comparing cements, investments, impression materials, and dental amalgam. Materials that are more adaptable to compression forces than tensile forces are classified as malleable. The mechanical qualities of PMMA are considerably improved by glass fibre reinforcement. Vegetable fibre and natural fibres (OPEFB) are both acceptable (17).

To evaluate a material's compressive strength, two axial sets of force are delivered to a sample in the opposite direction to simulate the material's molecular structure. The samples' dimensions should have a 2:1 ratio of length to diameter. When this ratio is surpassed, the specimen may bend in a way that is not desired. As we can see maximum compressive strength shown by the GF1.5WF1.5 as shown in Fig. 4 graph below as GF and WF composition increases compressive strength also increases and maximum at 1.5% composition of GF and WF afterwards it starts decreasing lowest compressive strength can be shown by pure PMMA samples which is taken as reference after that GF 1% shows the compressive strength values and rest PMMA type of denture material. (18)

Fatigue Test

During fatigue testing, a specimen undergoes cyclic stress applications below the yield stress until eventual fracture takes place. The conducted test employed specimens with dimensions $L=100\text{mm}$, $b=10\text{mm}$, and $d=4\text{mm}$, conducted at room temperature in accordance with the machine's manual and ADA Specification No.12, 1999. A fatigue testing apparatus designed for bending under alternating stress (HSM20 fatigue testing machine, HI-TECH Scientific, 1400 rpm, voltage span 230V, frequency 20Hz, nominal power 0.4kw) was used. Any fracture that exacerbates base deformation and subsequently impacts stress distribution can heighten the susceptibility of denture breakage. As flexural fatigue often culminates in midline fractures within dentures, the presence of an enlarged notch played a significant role in this phenomenon. Various factors contribute to the fatigue properties

and fatigue failure of composite materials, including stress concentration, residual stress, material microstructure, interaction between matrix and reinforcement mechanical properties, bond strength between them, constituent volume fractions, loading direction and type, loading frequency, and temperature. Fatigue damage can manifest in a range of ways, including delamination, matrix cracking, fiber failure, matrix crazing, fiber/matrix debonding, and void development. These mechanisms collectively give rise to a distinct yet intricate zone of damage. This depends on factors related to the testing environment, the material's manufacture, and its composition. S-N curves are used to illustrate the results of a fatigue test on two sets of laminated composite specimens and a PMMA matrix. The power formula is used to curve-fit the experimental data from the fatigue test to produce these curves. (19)

The image presents the power equations describing the fatigue behavior of laminated composite materials along with their respective relative correlation coefficients (R^2). The relatively high correlation coefficients indicate a substantial alignment between these equations and the experimental data, underscoring the effectiveness of the power formula in capturing the observed trends. A higher correlation coefficient is indicative of a better fit between the model and the data.

In the context of the long-cycle autoclave polymerization process with a 5 weight percent glass fiber reinforcement, the flexural strength and elastic modulus displayed notably superior values compared to other groups. This suggests that employing glass fiber reinforcement in combination with an extended autoclave polymerization process represents a promising strategy for enhancing the mechanical properties of heat-polymerized acrylic resin.

SEM

Heat-cured acrylic resin's water solubility was directly influenced by the kind and quantity of additives. The mean water solubility is greater in the control group than it is in the experimental group. The introduction of additives led to a significant reduction in the water solubility of the heat-cured denture base resin. The results of the study revealed a clear trend: with higher amounts of the added polymers in the experimental group, the water solubility diminished. This observation was established by evaluating the influence of different levels of polymer concentrations integrated into the denture base acrylic resin. This could be a result of enhanced physical cross-linking within polymeric structures caused by an increase in the concentration of additional polymers, which prevented plasticizer leakage. (20)

Nanopores can be seen on the surface of every manufactured fibre in high-magnification SEM images of the fibre samples (Figure 5&6). The pore sizes varied from 195 ± 63 to 150 ± 142 nm. Each sample of a fibre has a different pore diameter that fluctuates similarly to the fibre diameter. Smaller pore diameters were produced by increased GF loading, however as compared to fibres with lower GF loading, the pore diameter variance was greater. When the GF particle concentration rises, the surface topography varies greatly. The holes are quite round and evenly spaced along the fibre at lower concentrations (0.5, 1, and 1.5 wt%). The pores become increasingly erratic and unpredictable in terms of their distribution, size, and form above 1.5 wt%.

The GF fibres' fibre diameter was 7.06 ± 3.77 mm when there were no WF particles present. Raising the concentration of GF particles from 0.5 wt% to 1.5 wt% prompted the creation of thicker fibers exhibiting a broader diameter range. According to this discovery, the solution has a higher resistance to stretching brought on by the forces acting on the polymer jet when there are too many GF particles present. Prior research indicates that a change in fibre diameter during pressured gyration is caused by a combination of the effects of polymer concentration, rotational speed, and operating pressure, however flow into the vessel may also be regulated. All of these factors were, however, held constant throughout the experiment, indicating that GF loading was the underlying factor. (21)

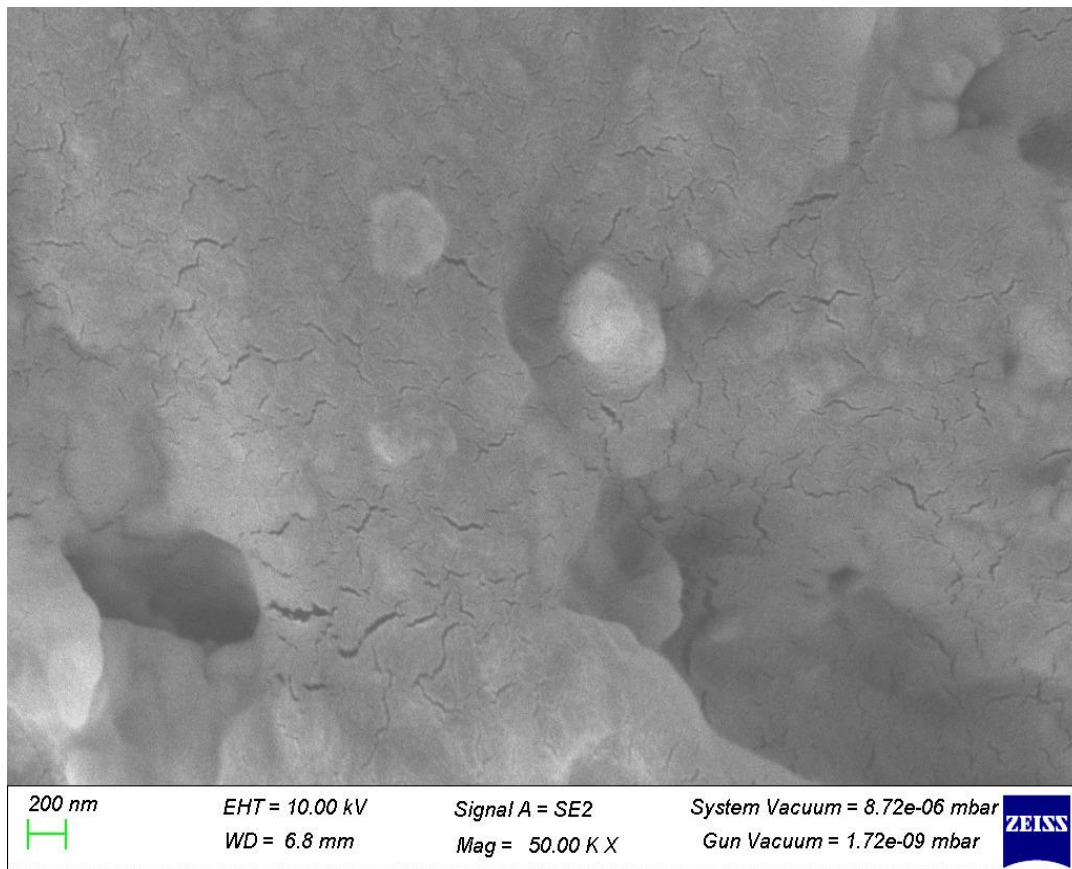


Figure 5. SEM test of the PMMA material with GF fibre.

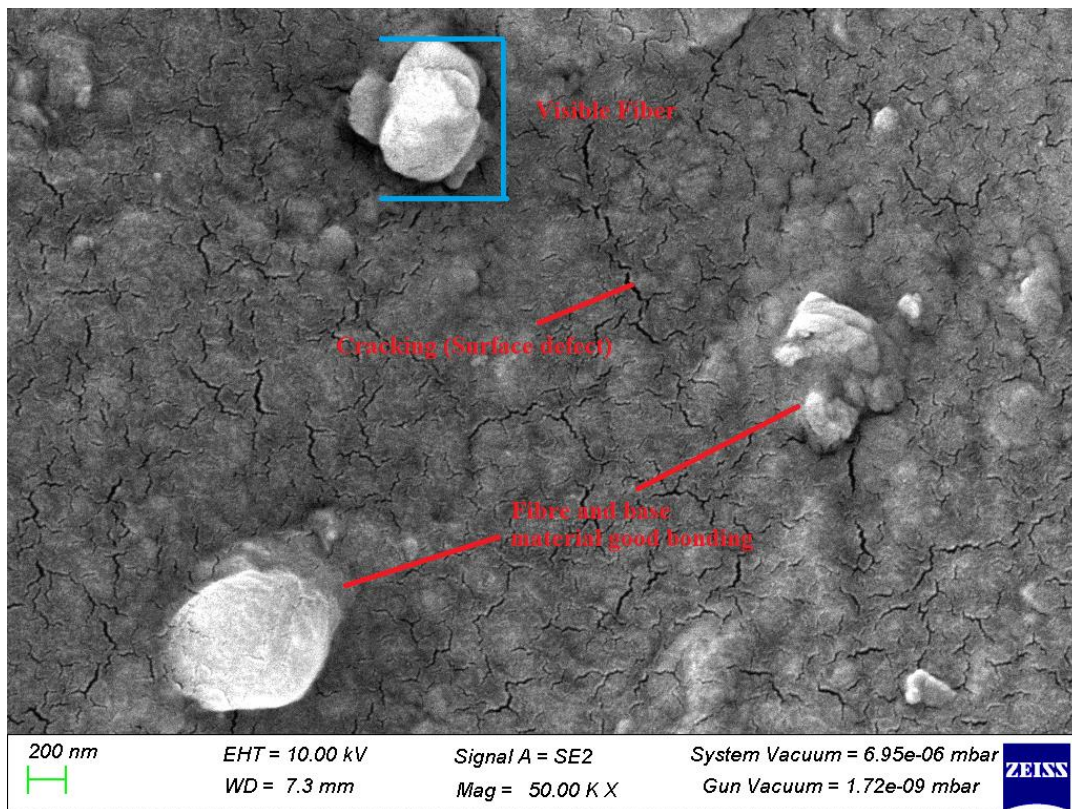


Figure 6. SEM test of the PMMA material with GF and WF fibre.

A composite formulation of different weight percent GF, WF with SBR rubber was proposed in this work, and it showed a balanced combination of mechanical and thermal characteristics. Ferreira et al. used graphite oxide (GrO) with many agglomerated flakes as a filler (0.1 wt%) to the brittle PS matrix. Rather of serving as flaws, the agglomerates strengthened the PS matrix. Next, the amorphous polymer displays mechanical characteristics that are similar to those of the semicrystalline polymer, leading to greater deformations and a lower modulus. As we can see in above fig. 5 and 6 the WF and NF can be visible in fig. 6 more frequently as its wt.% composition is increases in Fig 6 but in fig 5 only glass fiber can be dopped with PMMA base material. due to mixing in low wt. percentage its visibility is low at 50.00 KX magnification scale also.

Hardness Test

The combination of GF/WF/PMMA produced the composite's maximum hardness value. The primary factors believed to significantly impact the successful bonding of the fiber/matrix interface and the thorough dispersion of fibers within the matrix are considered the primary driving forces behind the formation of hardness in polymer composites. When the distribution of fibers is uneven across a region, the corresponding hardness value of that area will differ from that of a composite specimen surface where the fiber distribution is uniform. Put differently, a surface of the specimen possessing a compact or densely structured composition will exhibit greater hardness compared to one with a porous structure. (22)

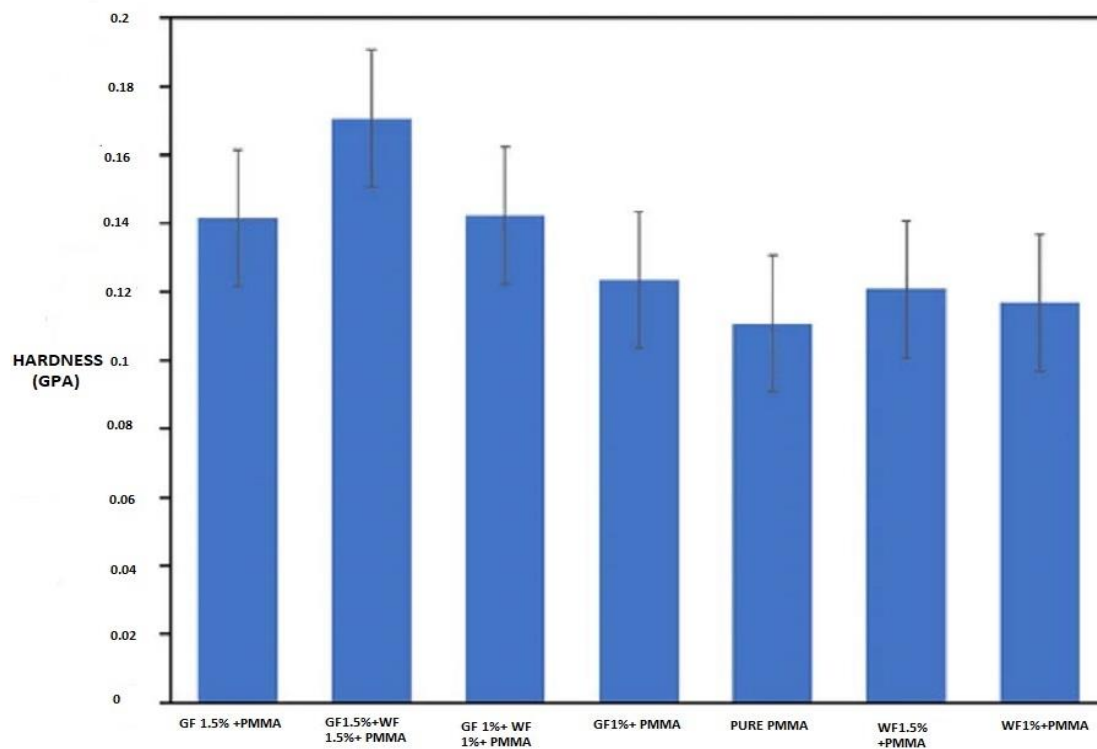


Figure 7. Hardness Test of samples.

Highest surface hardness can be shown by the GF 1.5% and woolen fiber 1.5% and rest PMMA as shown in Figure 7 above and the lowest hardness can be shown by the pure PMMA strips as GF and WF composition increases Hardness also increases and maximum at 1.5% composition of GF and WF afterwards it starts decreasing lowest hardness can be shown in pure PMMA samples. As we decrease the fiber composition the hardness of the samples decreases. When the proportion of glass fibers was raised within the GF/PMMA composites, the flexural modulus showed an almost straight-line increase. This trend followed the principle of blending materials. The flexural strength, however,

displayed the highest value in accordance with glassfibre content. Because of a significant presence of empty spaces, this phenomenon occurs. The greatest capacity for absorbing impact energy within the GF/PMMA composites was noted when the glass-fiber content was at 30%. However, this effectiveness declined as the concentration of glass fibers increased further. This pattern originates from changes in the impact mechanisms caused by different amounts of fibers. GP-1, featuring external layers abundant in glass fibers, demonstrated higher flexural strength and modulus compared to the uniform composite. Even though the various GF/PMMA FGM composites had almost equivalent impact absorption energies, they displayed distinct patterns of failure. (23)

SCOPE FOR FUTURE WORK

Since saliva is always present in the oral cavity, denture bases are typically vulnerable to a humid oral environment. Filling denture acrylic with nanoparticles might enhance its qualities; in particular, the integration of nanofibers or nanotubes had a considerable positive impact on PMMA's properties relative to nanoparticle fillers. This mainly occurs because nanotubes and nanofibers possess a greater surface area-to-volume ratio compared to nanoparticle fillers. Moreover, hybrid mixing materials generate an ideal combination in the resulting composite by offering improved balance and stability between the qualities of the combined fillers. Hence, it is recommended to use natural and vegetable fibres; nevertheless, more is necessary. (24)

CONCLUSION

By analyzing all the results, It is concluded that PMMA reinforced with 1.5% GF and 1.5% WF fibre shows the maximum impact strength and flexural strength values. These types of samples shows the great mixing ability to fiber and PMMA. Lowest value of result can be shown by pure PMMA strips. GF/WF/PMMA received the highest result in terms of the composite's hardness. The main reasons thought to have the greatest impact on increasing the hardness of polymer composites are the strong connection formed at the interface between the fibers and the matrix, as well as the thorough spread of fibers throughout the matrix material. If fibers are unevenly distributed in a certain region, the hardness value will deviate from that calculated for the composite specimen surface. In simpler terms, a specimen surface characterized by a compact or dense structure will exhibit higher hardness compared to a surface with a porous structure as the composition of GF and WF increases. The mechanical properties of the samples show an initial increase as the composition of GF and WF increases, reaching a maximum at 1.5%, after which they begin to decline. High amount of GF can also be harmful so optimum wt. percentage composition used in testing procedure.

Declaration of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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