Effect of Deep Freezing of High Impact Heat Activated Polymethylmethacrylate Resin on Its Flexural Strength and Impact Strength– An In Vitro Study

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Abstract

Aim: The aim of this study is to evaluate the conventional and deep freezing of PMMA on flexural and impact strength of commercially available high impact heat cure PMMA.

Materials and Methods: Fabrication Of High Impact Heat Cure Acrylic Resin Samples was done with the help of a metal die with 65mmx10mmx3 mm dimensions metal moulds. Curing of samples was performed by using an Acrylizer PB-042, (delta) at 74°c for 2 hours following Finishing of samples using tungsten carbide burs and fine abrasive without altering the specimen surfaces and dimensions. The material in its initial dough stage transferred into an aluminium cover and placed in a plastic airtight container and stored in deep freezer at -18°C for 1 month. Total 80 samples are fabricated and divided into two groups and each group will contain 40 samples. Each group was further subdivided into two subgroups and each subgroup will contain 20 samples. The prepared 80 samples were subjected to flexural strength test and impact strength test. Test for flexural strength was conducted using Universal Testing Machine at AMS material testing lab, Hyderabad. The test for impact strength was conducted using izod impact tester.

Statistical Analysis and Results: Statistical Analysis was done with Statistical Package for Social Sciences [SPSS] for Windows Version 22.0. The flexural strength of the control group had shown a mean value of 50.062 ± 10.042 MPa, whereas the experimental group had demonstrated a higher mean value of 68.822 ± 12.290 MPa. The mean difference between the two groups was -18.761 MPa, with the 95% confidence interval ranging from -25.945 to -11.576. The impact strength of the control group had shown a mean value of 4.909 ± 0.261 kJ/m^2, whereas the experimental group had demonstrated a higher mean value of 7.630 ± 0.511 kJ/m^2. The mean difference between the two groups was -2.721 kJ/m^2, with the 95% confidence interval ranging from -2.980 to -2.461. The difference was statistically significant ($p<0.001$)

Conclusion: Authors concluded that freezing protocol had imparted structural modifications within the resin that enhanced its ability to resist deformation under bending forces and improved its resilience against sudden impacts. The results also highlighted that deep freezing of polymethylmethacrylate dough at -18°C had a positive effect on the mechanical performance of the resin. So, the incorporation of deep freezing into the preparation protocol could be considered a beneficial modification, thereby improving its overall reliability for clinical application

Keywords: Deep Freezing, High Impact Heat Activated Polymethylmethacrylate Resin, Flexural Strength, Impact Strength

Introduction

The pursuit of an optimal denture base material has been a persistent challenge in the field of dentistry throughout history. In the absence of an ideal material, researchers in this field have endeavoured to find suitable alternatives such as ivory, shellac, vulcanite, and others. The rapid evolution of materials since 1900 has introduced newer alternatives; with polymethylmethacrylate emerging as one of them.[1] Heat cured acrylic resins are the most commonly used materials for the fabrication of denture bases in dentistry, since its introduction by Dr. Walter Wright in 1937. It continues to be material of choice for denture fabrication because of its ease of processing, dimensional stability, accuracy of fit and improved aesthetics. They are usually composed of pre polymerized polymethylmethacrylate (PMMA) powder particles, which are mixed with methyl methacrylate (MMA) and cross-linking agents such as ethylene glycolmethacrylate. The polymerization reaction (curing process) results in conversion of MMA into PMMA during which monomer molecules are converted into polymers.[2] A significant challenge in prosthodontics is regulating the working time of resin materials, especially polymethyl methacrylate (PMMA), during denture fabrication. Polymerization begins almost immediately after mixing, thereby limiting the available processing time and reducing the feasibility of fabricating multiple dentures simultaneously.[3] To address this issue, deep freezing has been explored as a preservation technique. This method involves storing materials at extremely low temperatures, to significantly slow or halt chemical and metabolic activity. Deep freezing is widely used in biomedical sciences for the long-term preservation of biological specimens such as cells, tissues, and organs, as it prevents degradation and maintains structural integrity.[4] In dentistry, this approach has gained attention for its potential to improve the handling characteristics and overall performance of denture base resins.[5] Impact strength refers to the amount of energy a material can absorb before fracturing under sudden force. This property is vital for denture base materials, as dentures regularly encounter various stresses during everyday use—such as chewing pressure, accidental drops, or biting hard substances. The majority of dentures are crafted from heat-cured PMMA or rubber-reinforced PMMA, materials known for their enhanced impact resistance and extended durability under functional stress.[6-8] Flexural strength is the measure of stiffness and resistance of a material till its fracture Repeated flexing from chewing ultimately fatigues the prosthesis. Flexural failure of a material is considered the primary mode of clinical failure. Any probable breakage of prostheses can lead to functional and aesthetic problems.[8] Hence, this study was conceptualized to evaluate the influence of deep freezing on the flexural strength and impact strength of commercially available high impact heat activated PMMA.

Aim and Objectives

Aim

The aim of this study is to evaluate the conventional and deep freezing of PMMA on flexural and impact strength of commercially available high impact heat cure PMMA.

Objectives

1. To evaluate the flexural strength of high impact heat cure PMMA samples prepared at room temperature.
2. To evaluate the flexural strength of high impact heat cure PMMA samples frozen to -18^{o} C.

3. To evaluate the impact strength of high impact heat cure PMMA samples prepared at room temperature.
4. To evaluate the impact strength of high impact heat cure PMMA samples frozen to -18^{o} C.
5. To compare the obtained flexural strength and impact strength values between control and experimental group.

Materials and Methods

This study was planned, abstracted and executed in the department of Prosthodontics of the institute. A metal die with 65mmx10mmx3 mm dimensions metal moulds (fig no 1) was prepared as per ISO (20795-1) standardization. Four metal sample analogues were placed in each flask ensuring sufficient space between investing medium and metal analogues. The metal sample analogues were lifted from investing medium, thereby creating mould space into which acrylic resin was packed at room temperature and separating medium (DPI) was applied. The material used was high impact heat cure acrylic denture base material in a powder-liquid form. (triplex hot Ivoclar) was used for sample fabrication. Approximately after the onset of mixing, the mixture was probed gently with a gloved finger. For Packing Acrylic resin into the mould space, the assembled flask was then positioned in a hydraulic press, where pressure was applied gradually in incremental steps. Then the flask was opened, and the cellophane sheet was removed in a single, smooth motion. Any excess resin on the flat peripheral areas surrounding the mould space was carefully trimmed, ensuring that the resin within the mould cavity remained undisturbed. Curing of samples was performed by using an Acrylizer PB-042, (delta) at 74°c for 2 hours. Following this, denture flask was allowed to cool to room temperature.

Deep freezing the High Impact Heat Cure Acrylic Resin

After reaching the initial dough stage, the high impact heat cure acrylic denture base material is transferred into an aluminium cover and placed in a plastic airtight container (fig no 2,3) and stored in deep freezer at -18°C for 1 month.[10] Similarly same procedure is mentioned for control group fabrication was followed for the deep-freezing samples fabrication. The plastic container is removed after 1 month from the freezer and packed in a dental plaster mould space in a dental flask, which is prepared at the time of deep freezing and followed by curing, finishing of the samples and storing the samples in water. A total 80 samples are fabricated and divided into two groups and each group will contain 40 samples. (fig no 4,5,6,7,8) Each group will be further subdivided into two subgroups and each subgroup will contain 20 samples;

Group I: Control Group (40 Samples)
- **Sub Group IA:** 20 samples for evaluation of Flexural strength of PMMA sample at room temperature.
- **Sub Group IB:** 20 samples for evaluation of impact strength of PMMA samples at room temperature.

Group Ii: Deep Freezer Test Group (40 Samples)
- **Sub Group IIA:** 20 samples for evaluation of Flexural strength of PMMA dough frozen at -18^{0}C.
- **Sub Group IIB**: 20 samples for evaluation of impact strength of PMMA dough frozen at -18^{0}C.

Test for flexural strength was conducted using Universal Testing Machine (fig no 9) at AMS material testing lab, Hyderabad. The test for impact strength was conducted using izod impact tester (fig no 10) at AMS material testing lab, Hyderabad. Testing procedure was as follows.

Testing For Flexural Strength

40 samples, 20 from each group (Group IA and Group IIA) were tested for flexural strength using Instron universal testing machine and flexural strength was evaluated. Fracture of the specimen was indicated by a sharp crackling sound accompanied by a sudden drop in the digital signal. The amount

of load required to induce failure was recorded in MPa (N/mm2). The results are recorded through the three-point bending test.

Testing For Impact Strength: (IZOD Impact Tester)

40 samples, 20 from each group (Group IB and Group IIB) were tested for impact strength using Izod impact tester Model IT504, USA. The fabricated samples were notched in the middle, with a notch cutter as in compliance with ISO 179:1993 to a depth of 0.2mm leaving a residual depth of 2.8mm beneath the notch. In this Izod impact test, the test piece was a cantilever i.e. the specimen is clamped upright in the anvil with a V-notch at the level of clamp. The dimensions of the sample were entered in the display box. The pendulum was released onto the test specimen which was aerodynamically controlled. The impact strength of notched specimen was calculated in KJ/mm2 as given by the following equation:

Figure 1: Mould Space Created For Sample preparation

Figure 2: Transferred Dough to the Airtight Plastic Container

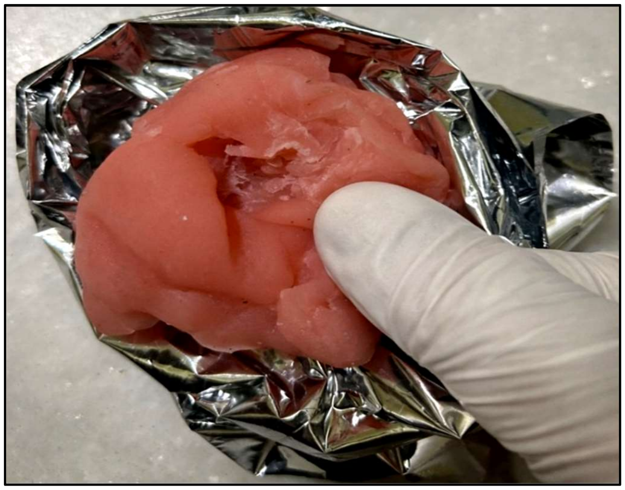


Figure 3: Assessing the Dough With Finger Probing

Figure 4: Packed In Mould Space

Figure 5: Trial Closure Done

Figure 6: Control and Test Group Samples Stored In Water

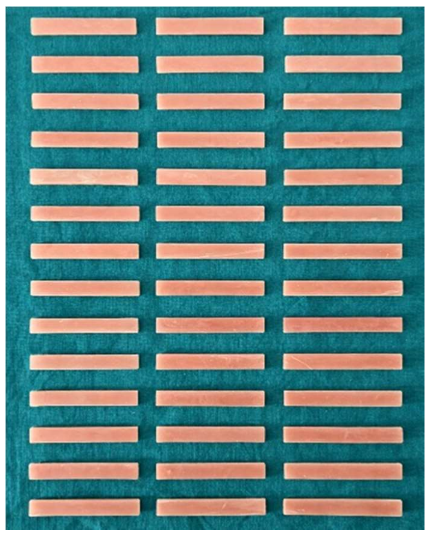


Figure 7: Control Group

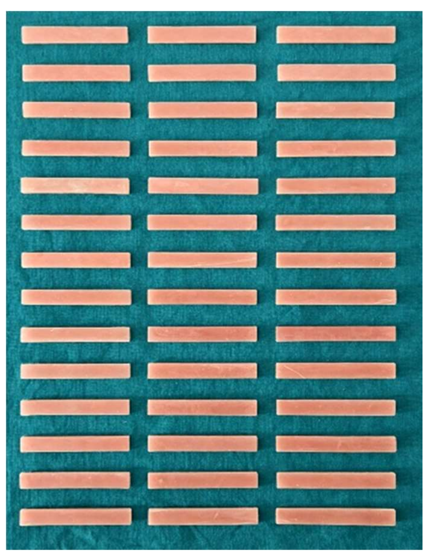


Figure 8: Experimental Group

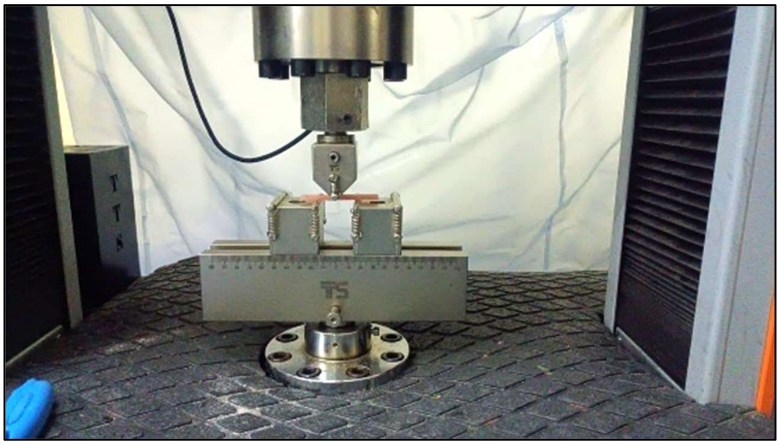


Figure 9: Universal Testing Machine

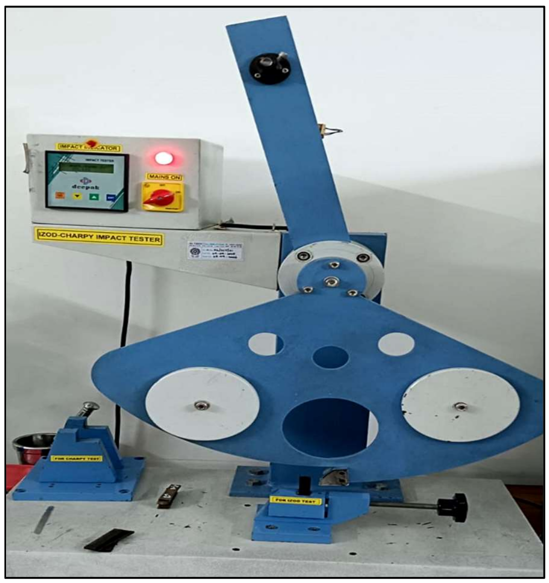


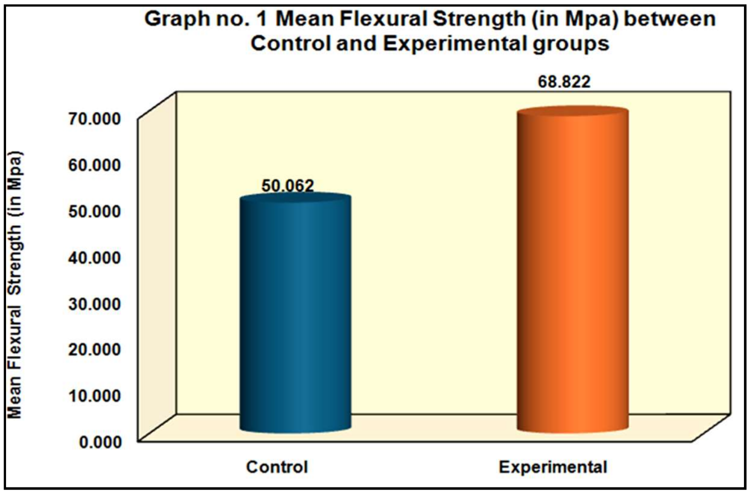
Figure 10: IZOD Impact Tester

Statistical Analysis

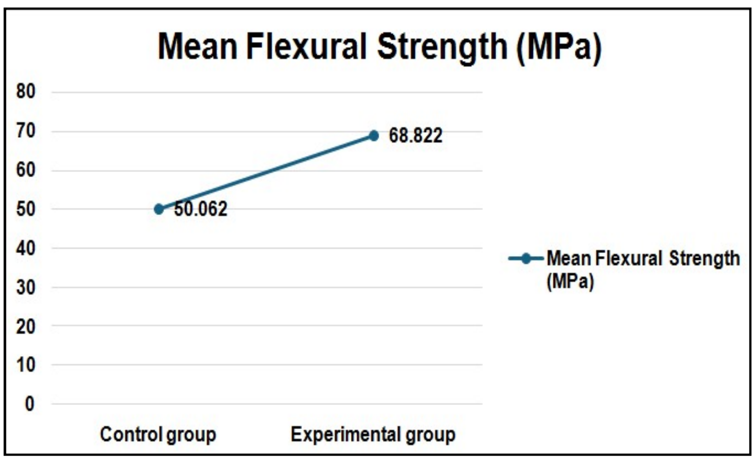
Statistical Package for Social Sciences [SPSS] for Windows Version 22.0 Released 2013. Armonk, NY: IBM Corp., was used to perform statistical analyses. Descriptive analysis includes expression of Flexural Strength and Impact Strength in terms of Mean & SD for each group. Independent Student t test was used to compare the mean Flexural Strength and Impact Strength between Control and Experimental groups. Level of significance was set at $p<0.05$.

Results

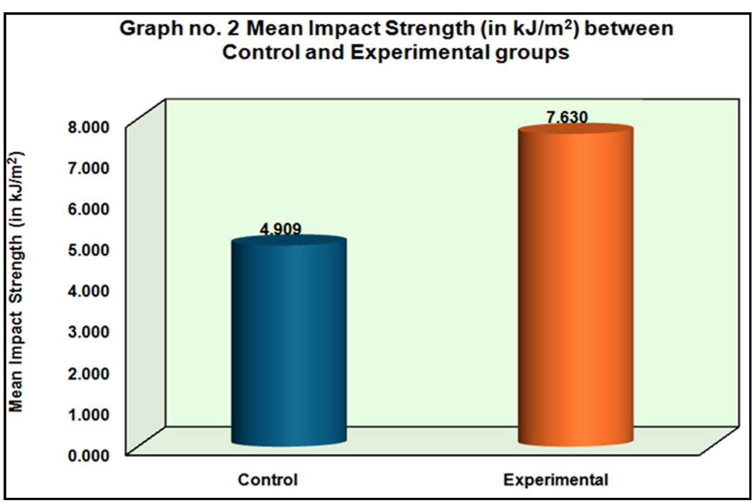
Null Hypothesis was Deep freezing of polymethylmethacrylate dough at -18°C before processing produces an improvement in flexural strength and impact strength compared to dough processed at room temperature. The flexural strength of the control group had shown a mean value of 50.062 ± 10.042 MPa, whereas the experimental group had demonstrated a higher mean value of 68.822 ± 12.290 MPa. The mean difference between the two groups was -18.761 MPa, with the 95% confidence interval ranging from -25.945 to -11.576. The difference was statistically significant ($p<0.001$), indicating that the specimens subjected to deep freezing had exhibited a considerable increase in flexural strength compared to the control group. [Graph no. 1& 2] The impact strength of the control group had shown a mean value of 4.909 ± 0.261 kJ/m², whereas the experimental group had demonstrated a higher mean value of 7.630 ± 0.511 kJ/m². The mean difference between the two groups was -2.721 kJ/m², with the 95% confidence interval ranging from -2.980 to -2.461. The difference was statistically significant ($p<0.001$), indicating that the specimens subjected to deep freezing had exhibited a marked increase in impact strength compared to the control group. [Graph no. 3 & 4]



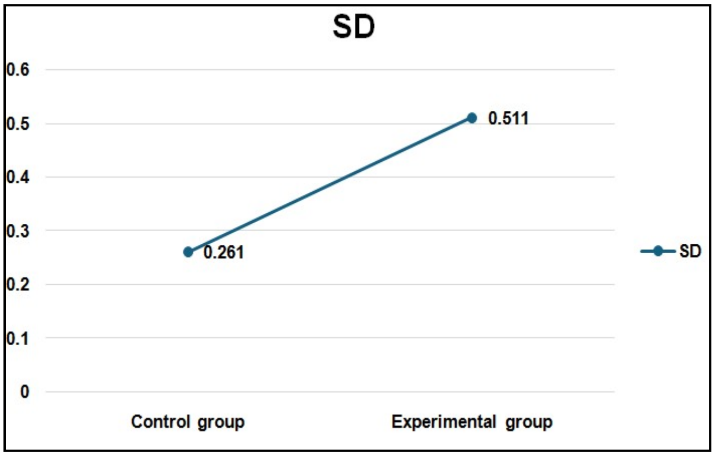
Graph No. 1



Graph No. 2



Graph No. 3



Graph No. 4

Clinical Implications

The clinical implications of this study suggested that the mechanical performance of polymethylmethacrylate resin could be significantly improved by modifying the processing protocol. The control group, in which the dough was maintained at room temperature, consistently demonstrated lower mechanical properties. In contrast, the experimental group, in which the dough was frozen at -18°C prior to processing, showed superior flexural strength and impact strength. This indicated that the freezing process had enhanced the resin's ability to resist deformation and fracture, thereby improving its durability under functional stresses. From a clinical perspective, these findings implied that incorporating deep freezing into the preparation of polymethylmethacrylate resin could lead to prostheses with greater longevity and reliability. The enhanced flexural strength would reduce the risk of fracture under masticatory forces, while the improved impact strength would provide better resistance against accidental drops or sudden forces. Together, these improvements could contribute to more predictable outcomes in prosthodontic practice, offering patients prostheses with enhanced mechanical resilience and extended service life.

Discussion

The introduction of acrylic resins in dentistry marked a turning point in prosthodontics, particularly after the development of polymethyl methacrylate (PMMA), which became widely accepted due to favourable mechanical properties and processing characteristics. Despite these advantages, PMMA is not an ideal material. Denture fracture continues to be a common clinical complication, often resulting from fatigue failure, flexural stresses, and impact forces experienced during mastication or accidental dropping of dentures.[11] Arnold et al described Flexural strength is considered one of the most clinically relevant mechanical properties because dentures are subjected to continuous bending forces during function.[6,12] Flexural fatigue has been identified as a major contributor to clinical failure of denture base polymers.[11,13] Investigations comparing different denture base resins revealed significant variations in mechanical performance depending on polymer composition and processing conditions. Impact strength represents another critical parameter determining clinical durability. Dentures frequently experience sudden forces during accidental falls or abrupt occlusal contacts.[14,15] Surface defects and internal voids have been shown to reduce impact resistance considerably.[16] Jagger DC stated Various reinforcement strategies, including woven fibers and elastomer modifications, have been evaluated to enhance energy absorption capacity.[17,18,19] High-impact acrylic resins were developed through the incorporation of rubber graft copolymers, allowing crack deflection and energy dissipation mechanisms that reduce brittle fracture behavior.[7,20,21] The present study explored deep freezing of PMMA dough at −18°C prior to processing as a novel approach to modify material behavior.[5,22] Deep freezing is widely utilized in biological sciences for preservation of structural integrity by reducing molecular activity and slowing chemical reactions.[4] Application of this principle to denture base resins aims to prolong working time and potentially influence polymerization kinetics.[3,23] Sato. Y et al., investigated on deep freezing and it stated that low-temperature storage could preserve material stability and influence mechanical outcomes.[24] The aim of this investigation was to evaluate the effect of deep freezing on flexural and impact strength of high-impact heat-cured PMMA, aligning with the stated objectives of comparing specimens processed at room temperature and those frozen at −18°C before polymerization. The experimental design incorporated standardized sample fabrication, ASTM-compliant specimen dimensions, and controlled curing cycles to ensure reproducibility and reliability of results. Impact strength results similarly demonstrated statistically significant enhancement in the frozen specimens. Increased impact strength suggests improved toughness and energy absorption capacity.[25,26] Jagger DC et al stated that, Rubber-modified PMMA relies on crack-stopping mechanisms, and uniform dispersion of elastomer particles enhances these effects. Deep freezing may stabilize the dough stage, promoting better distribution of polymer and elastomer components before curing.[5] Another possible explanation relates to reduction in residual monomer content. Residual monomer acts as a plasticizer that weakens polymer chains and reduces mechanical strength.[2] The present findings align with investigations demonstrating that modification of processing protocols can significantly enhance denture base performance. Studies evaluating fiber reinforcement, polymer blending, and curing alterations consistently report improvements in mechanical strength when microstructural integrity is optimized.[21,27] The statistical analysis confirmed significant differences between groups of both flexural and impact strength values showed marked improvement in the frozen specimens, demonstrating that pre-processing thermal modification positively influences mechanical performance. The magnitude of improvement suggests that freezing alters polymer chain arrangement rather than producing minor experimental variation.[5,18,28] The results also support the theoretical concept that prolongation of workable dough stage improves packing plasticity.[3] Adequate adaptation during packing reduces void formation, which is known to improve themechanical properties.[8] Improved mould adaptation minimizes internal defects, thereby enhancing structural integrity and resistance to fracture.[16] This study contributes valuable evidence supporting modification of conventional processing techniques. Deep freezing represents a simple and cost-effective method that does not require alteration of material composition. By improving both stiffness and toughness, the technique may enhance prosthesis longevity without increasing manufacturing complexity.

Limitations of the Study

The study was conducted under controlled laboratory conditions, which may not fully replicate intraoral environments involving temperature fluctuations, saliva exposure, and cyclic loading. Only one commercially available high-impact heat cure PMMA material was evaluated, limiting generalization to other brands or formulations. The investigation assessed only two mechanical properties (flexural strength and impact strength), without evaluating fatigue resistance, hardness, surface roughness, surface hardness. The freezing protocol was restricted to −18°C for 1 month only, without comparison of other different freezing temperatures. Long-term aging conditions such as thermocycling or prolonged water immersion were not performed prior to testing. Manual procedures such as dough stage assessment and packing technique may introduce operator-dependent variability. The sample size, although adequate for statistical testing, may still limit broader extrapolation to clinical scenarios. Clinical performance factors such as fracture behaviour under cyclic functional loading and real denture design variables were not assessed.

Conclusion

In light of the constraints inherent in our research, the authors conducted a thorough investigation and drawn following conclusions:

1. The use of deep freezer for polymethylmethacrylate resin was influenced by the processing conditions of the dough.

2. The control group, in which the dough was maintained at room temperature, consistently exhibited lower values for both flexural strength and impact strength. (50.062±10.042 & 4.909±0.261)
3. In contrast, the experimental group, in which the dough was subjected to deep freezing at -18°C prior to processing, showed a marked improvement in bothflexural strength and impact strength. (68.822 ± 12.290 MPa&7.630 ± 0.511 kJ/m²)
4. This indicated that the freezing protocol had imparted structural modifications within the resin that enhanced its ability to resist deformation under bending forces and improved its resilience against sudden impacts.
5. Overall, the results highlighted that deep freezing of polymethylmethacrylate dough at -18°C had a positive effect on the mechanical performance of the resin.
6. The experimental group outperformed the control group across both tested properties, suggesting that the freezing process had strengthened the material and improved its durability.
7. These findings provided evidence that the incorporation of deep freezing into the preparation protocol could be considered a beneficial modification, as it enhanced both the stiffness and toughness of the resin, thereby improving its overall reliability for clinical applications.

Nevertheless, the authors expect further future studies and research those are essential to have deepen understanding and validation of current perspectives and norms.

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