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Fracture Surface Evaluation of Heat-Cured Acrylic Resin Cured at 100°C in Different Duration – A SEM Analysis

Prof. Dr. Kazi Ziaul Islam[1*], Dr. Ali Afzal Khan[2], Prof. Dr. Md. Mahbubur Rahman[3], Dr. Newaz Mohsina[4]

[1]Professor and Principal, Department of Prosthodontics, Saphena Women's Dental College and Hospital, Dhaka, Bangladesh
[2]Associate Professor and Head, Department of Prosthodontics, Update Dental College and Hospital, Dhaka, Bangladesh
[3]Professor, Department of Prosthodontics, Faculty of Dentistry, Bangladesh Medical University, Dhaka, Bangladesh
[4]Dental Surgeon and In-charge Casualty, Department of Oral and Maxillofacial Surgery, Dhaka Dental College and Hospital, Dhaka, Bangladesh

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***Corresponding author:** Prof. Dr. Kazi Ziaul Islam
Professor and Principal, Department of Prosthodontics, Saphena Women's Dental College and Hospital, Dhaka, Bangladesh

Abstract **Original Research Article**

Background: Heat-cured polymethyl methacrylate (PMMA) acrylic resin is widely used as a denture base material, yet fracture remains a common clinical problem. Processing variables, particularly curing duration at high temperature, may influence internal defects and fracture behavior. Scanning electron microscopy (SEM) provides valuable insight into fracture surface morphology and failure mechanisms. ***Aim:*** The aim of this study was to evaluate and compare the fracture surface characteristics of heat-cured PMMA acrylic resin cured at 100 °C for different durations using SEM analysis. ***Methods:*** In this in vitro experimental study, 60 PMMA secimens were prepared and cured at 100 °C for 20, 40, and 60 minutes. Specimens were fractured using a three-point bending test, and the fracture surfaces of 30 samples were examined under SEM at 50× and 100× magnifications. ***Results:*** Specimens cured for 20 minutes showed predominantly brittle fractures with smooth surfaces and frequent microvoids. The 40-minute group exhibited mixed fracture patterns, while the 60-minute group demonstrated predominantly ductile fracture characteristics, with reduced porosity and increased crack deflection and branching. ***Conclusion:*** Longer curing duration at **100 °C** improves fracture surface characteristics of heat-cured PMMA, indicating enhanced resistance to crack propagation.

Keywords: PMMA; heat-cured acrylic resin; curing duration; fracture morphology; SEM.

INTRODUCTION

Polymethyl methacrylate (PMMA)–based heat-cured acrylic resin remains the most widely used denture base material because it is inexpensive, esthetic, easy to process, and clinically acceptable in strength and handling. However, fractures of acrylic denture bases are still common in practice, typically resulting from repeated flexural fatigue during mastication, accidental impact (dropping), stress concentration around notches, and processing-related defects.[1,2] Because many of these failures originate from microscopic flaws created or amplified during polymerization, understanding how processing variables influence the internal structure and the resulting fracture pattern is essential for improving clinical performance and reducing repair/remake rates.[3]

Heat polymerization of denture base acrylic resin is initiated by free radicals generated from benzoyl peroxide when sufficient temperature is applied. The polymerization reaction is exothermic, and the temperature within the resin mass can rise above the surrounding water-bath temperature depending on specimen thickness and the curing schedule. A long, controlled heating cycle is traditionally recommended to promote more complete conversion of methyl methacrylate (MMA) monomer to polymer while minimizing internal stresses and porosity. In contrast, "short-cut" or accelerated cycles often used to save laboratory time may leave higher residual monomer, increase internal defects, and adversely affect mechanical behavior. Harrison and Huggett demonstrated that different curing cycles can produce a wide range of residual monomer values, and they identified an "optimum" cycle (7 hours at 70 °C followed by 1 hour at 100 °C) that yielded low residual monomer across many commercial materials. [4] This is clinically relevant because residual monomer is not only a biocompatibility concern but is also associated with reduced material properties and increased susceptibility to crack initiation and propagation.

The influence of time–temperature schedules on residual monomer has been investigated for decades. Early work showed that varying short curing cycles can substantially change residual monomer levels, implying that polymerization duration at high temperature (including terminal boiling phases) matters for monomer reduction and overall material quality. [5] Later studies confirmed that additional post-processing heat cycles can further reduce residual monomer concentrations in denture-base acrylic resin, again highlighting that extended exposure to heat can drive polymerization forward or facilitate monomer diffusion. [6] These findings align with ISO requirements for denture base polymers, which specify maximum allowable residual methyl methacrylate content (e.g., 2.2% mass fraction for Type 1, 3, 4, and 5 materials) and minimum flexural property thresholds, underscoring that processing must be adequate to meet standardized performance criteria. [7]

A key processing concern during curing at or near 100 °C is porosity formation. Traditionally, it has been suggested that rapid heating may cause monomer boiling and vapor entrapment, creating internal voids that weaken the resin and act as crack initiators. However, the actual behavior depends strongly on flask clamping pressure and the internal pressure–temperature environment during polymerization. Yau and colleagues measured pressure and temperature changes during processing. They showed that clamping pressure can substantially elevate the boiling point of the monomer above the maximum temperature reached, even during a fast boiling-water cycle. They reported no porosity when adequate pressure was maintained. [8] This indicates that "duration at 100 °C" does not act in isolation; rather, the combined effects of curing time, heating rate, specimen geometry, and pressure control determine whether voids form and whether the final microstructure is homogeneous.

While bulk mechanical testing quantifies strength, fracture surface evaluation can reveal *why* a specimen failed. Scanning electron microscopy (SEM) fractography is particularly valuable because it can identify features such as voids, unreacted regions, craze patterns, river lines, hackle markings, and mirror–mist–hackle transitions, which collectively indicate the fracture mode (brittle vs. more ductile behavior), crack origin, and the presence of processing defects. In denture acrylic resins, SEM-based fracture morphology has been used to relate polymerization methods and microstructural characteristics to impact performance, and observations commonly show predominance of brittle fracture features with recognizable surface patterns. [9]

Therefore, evaluating fracture surfaces of heat-cured acrylic resin polymerized at 100 °C for different durations using SEM can provide mechanistic insight into how curing time influences defect population (e.g., porosity or microvoids), matrix continuity, and crack propagation pathways. By linking SEM features to processing schedules and known effects on residual monomer and porosity, such an analysis can help optimize curing protocols that balance laboratory efficiency with material integrity and long-term clinical durability. [4]

OBJECTIVES

The main objective was to evaluate the effect of different curing durations at 100 °C on the fracture surface characteristics of heat-cured acrylic resin using scanning electron microscopy (SEM).

METHODOLOGY & MATERIALS

Study Design and Setting:

This study was designed as a comparative in vitro experimental study. The study period extended from January 2009 to December 2010. The research was carried out through collaborative efforts involving the Department of Prosthodontics, Dental Faculty, Bangladesh Medical University, Bangladesh Council of Scientific and Industrial Research (BCSIR), and the Center for Advanced Research in Sciences (CARS), University of Dhaka, Dhaka, Bangladesh.

Study Materials and Sample Size:

The study samples consisted of heat-cured polymethyl methacrylate (PMMA) acrylic resin denture base material. A total of 60 test specimens were fabricated according to standardized dimensions. The specimens were divided into three groups based on curing duration at 100 °C:

- **Group I:** 20 specimens cured at 100 °C for 20 minutes
- **Group II:** 20 specimens cured at 100 °C for 40 minutes
- **Group III:** 20 specimens cured at 100 °C for 60 minutes

For scanning electron microscopic (SEM) evaluation of fracture surfaces, 10 specimens from each group were selected, resulting in a total of 30 fractured surfaces examined under SEM.

Specimen Preparation:

All test specimens were fabricated using an experimental metal die conforming to ASTM specifications, with final dimensions of 65 × 12.7 × 2.5 mm.

Preparation of Molds:

Dental stone was used to invest the metal die. Prior to investing, the metal die was coated with a thin layer of petroleum jelly to facilitate easy removal after setting of the stone. To prevent fracture of the mold during retrieval, space was created on one side of the metal die during the first pour of dental stone. This

technique allowed uncomplicated removal of the metal die after complete setting of the second pour.

Packing Procedure:

Polymethyl methacrylate polymer and methyl methacrylate monomer was mixed in a clean mixing jar according to the manufacturer's instructions. Once the acrylic resin reached the dough stage, it was packed into the mold space. Trial closure was performed, and excess material was removed prior to final closure.

Processing Procedure:

The flask containing the packed acrylic resin was processed using the compression molding technique. The flask was immersed in a water bath, and the temperature was monitored using a thermometer fixed with a clamp. The temperature of the water bath was gradually increased until it reached 100 °C. Once this temperature was achieved, curing time was recorded using a stopwatch according to the assigned group (20, 40, or 60 minutes). After completion of the curing cycle, the flask was allowed to bench-cool before deflasking.

Retrieval and Storage of Specimens:

After bench cooling, the flask was opened carefully, and the intact acrylic resin specimens were retrieved without distortion. The specimens were then stored in bottles containing water until further processing.

Trimming, Finishing, and Polishing:

Excess flash was trimmed using a laboratory micromotor with appropriate burs. The specimens were finished on a flat surface using sandpaper and polished with pumice powder. Final dimensions of all specimens were verified using a Vernier caliper to ensure conformity with the standardized size of 65 × 12.7 × 2.5 mm.

Mechanical Testing and Fracture Induction:

All 60 specimens were mounted on the designated platform of a Hounsfield Universal Testing Machine. A load was applied at the center of each specimen using a three-point bending test until fracture occurred. The fractured specimens were then collected for fracture surface analysis.

Fracture Surface Morphology Analysis:

Initial classification of fracture patterns was performed by visual inspection. Fractures were categorized as brittle when the fractured fragments could be repositioned accurately along the fracture line with smooth fracture surfaces. Fractures showing plastic deformation with rough and jagged surfaces were classified as ductile, following the criteria described by Fernanda Faot *et al.,* (2009). [10]

Scanning Electron Microscopy (SEM) Evaluation:

A total of 30 fractured surfaces (10 from each group) were subjected to SEM analysis using a JEOL scanning electron microscope (Model: JSM-6490LA). SEM photomicrographs of the fracture surfaces were obtained at 50× and 100× magnifications. From each curing-duration group, five fractured surfaces were randomly selected and evaluated in detail for fracture surface morphology, including porosity, crack initiation sites, and crack propagation patterns.

RESULT

Table 1. Distribution of Fracture Type According to Curing Duration at 100 °C

Curing duration at 100 °C	Number of specimens examined (SEM)	Brittle fracture	Ductile fracture
20 minutes	10	Predominant	Absent
40 minutes	10	Moderate	Moderate
60 minutes	10	Minimal	Predominant

Table 1 shows that specimens cured for 20 minutes predominantly exhibited brittle fractures, those cured for 40 minutes showed a mixed brittle–ductile pattern, and specimens cured for 60 minutes demonstrated predominantly ductile fracture behavior, indicating a progressive change with increasing curing duration at 100 °C.

Table 2. SEM Fracture Surface Topography at Different Curing Durations (100 °C)

Fracture surface feature	20 minutes	40 minutes	60 minutes
Surface smoothness	High	Moderate	Low
Surface roughness	Low	Moderate	High
Homogeneity of fracture surface	Uniform	Moderately uniform	Non-uniform
Evidence of tearing ridges	Absent	Occasionally present	Frequently present

Table 2 demonstrates that specimens cured for 20 minutes exhibited smooth and uniform fracture surfaces with no tearing ridges. Specimens cured for 40 minutes showed moderate surface roughness with occasional tearing ridges. In contrast, specimens cured for 60 minutes presented high surface roughness, non-uniform fracture surfaces, and frequent tearing ridges, indicating increased plastic deformation with longer curing duration at 100 °C.

Table 3. Porosity and Defect Characteristics Observed under SEM

Parameter	20 minutes	40 minutes	60 minutes
Microvoid frequency	High	Moderate	Low
Void size	Small to moderate	Mostly small	Rare / absent
Void distribution	Uniformly scattered	Irregular	Minimal
Association with crack origin	Frequent	Occasional	Rare

Table 3 shows that specimens cured for 20 minutes exhibited a high frequency of microvoids, often uniformly distributed and frequently associated with crack initiation. Specimens cured for 40 minutes demonstrated a moderate number of mostly small voids with occasional association with crack origin. In contrast, specimens cured for 60 minutes showed minimal or absent porosity, with rare involvement of voids in crack initiation, indicating improved structural integrity with longer curing duration at 100 °C.

Table 4. Crack Initiation and Propagation Patterns

Crack behavior	20 minutes	40 minutes	60 minutes
Primary crack origin	Pores and voids	Mixed sites	Matrix discontinuities
Crack path	Straight	Slightly deflected	Highly deflected
Crack branching	Absent	Occasional	Frequent
Crack arrest features	Absent	Mild	Evident

Table 4 shows that specimens cured for 20 minutes had cracks originating from pores with straight paths, while 40-minute specimens showed mixed initiation sites and slight deflection. Specimens cured for 60 minutes exhibited highly deflected, frequently branched cracks with evident crack arrest features.

Table 5. SEM Observations at Different Magnifications

SEM magnification	20 minutes	40 minutes	60 minutes
50×	Smooth fracture plane with voids	Moderately rough surface	Rough, irregular surface
100×	Clear pores and river lines	Reduced pores and river lines	Plastic deformation and tear ridges

Table 5 shows that at 50× magnification, specimens cured for 20 minutes exhibited smooth fracture planes with visible voids, while 40-minute specimens showed moderately rough surfaces and 60-minute specimens demonstrated rough, irregular surfaces. At 100× magnification, clear pores and river lines were evident in the 20-minute group, reduced in the 40-minute group, and replaced by features of plastic deformation and tear ridges in the 60-minute group.

DISCUSSION

This in vitro SEM-based study evaluated fracture surface morphology of heat-cured PMMA denture base resin processed at 100 °C for 20, 40, and 60 minutes. The results demonstrated a clear curing-duration–related trend: specimens cured for 20 minutes showed predominantly brittle fracture, smoother fracture planes, and more frequent microvoids; the 40-minute group showed a transitional pattern; and the 60-minute group showed predominantly ductile fracture features, rougher and more irregular surfaces, reduced porosity, and more crack deflection/branching with evident crack arrest characteristics. A key observation in the present study was the higher microvoid frequency in the 20-minute group with frequent association of voids with crack initiation, which progressively reduced as curing time increased to 40 and 60 minutes. This supports the concept that processing-related defects (voids/microvoids) act as stress concentrators, enabling rapid crack initiation and brittle failure. Previous investigations have emphasized that polymerization variables strongly influence defect formation and internal quality of denture base acrylic resins. For example, investigations into processing conditions have shown that the pressure–temperature environment during curing can influence whether monomer boiling and porosity occur, and that adequate pressure can elevate the monomer boiling point and reduce porosity risk even in boiling-water cycles.[8] In addition, porosity has been repeatedly highlighted as a major contributor to weakened acrylic resin and premature fracture. Studies examining porosity under different polymerization methods (including heat-activated processing) have shown that polymerization cycle selection impacts porosity formation and, consequently, mechanical behavior.[11] In the current study, the shift from frequent void-associated crack origins (20 minutes) to rare void involvement (60 minutes) is consistent with the understanding that longer curing at the target temperature may improve matrix continuity and reduce internal defect population, thereby improving resistance to crack initiation. The present SEM findings also showed a progressive change in fracture topography:

high smoothness and uniformity (20 minutes) → moderate roughness (40 minutes) → high roughness, non-uniformity, and tearing ridges (60 minutes). In SEM fractography, smoother and more planar surfaces commonly correlate with brittle fracture, whereas rougher surfaces with tearing ridges are consistent with greater energy dissipation during fracture and more complex crack propagation. Comparable SEM-based fracture morphology observations have been reported in denture acrylic resin studies evaluating processing-related differences, where brittle fractures tend to show more organized/defined features while intermediate fractures show more disorganized patterns. [7] Although the Faot *et al.,* investigation primarily compared polymerization methods (microwave versus water-bath), its fracture morphology framework helps interpret the present curing-duration effect: the 20-minute group in our study resembled a defect-driven brittle pattern, while the 60-minute group showed features consistent with greater fracture resistance and more "ductile-like" morphology.[7] The intermediate behavior observed at 40 minutes in our samples is also consistent with a transitional state where processing improvements reduce major defects but may not fully optimize the internal microstructure. With advances in polymerization cycles aimed at improving laboratory techniques, Vasconcellos *et al.,* (2003) compared an alternative polymerization cycle incorporating a pause period with the conventional manufacturer-recommended cycle. [12] They reported that transverse strength and surface microhardness were not adversely affected by the alternative cycle. These findings support the present observations, suggesting that modifications or extensions of polymerization protocols such as increased curing duration at 100 °C can improve internal structural quality without compromising mechanical performance. Fracture surface evaluation is a valuable tool for understanding failure behavior, as it allows prediction of crack initiation and propagation pathways. Halahan (1997) emphasized that fracture analysis plays an important role in identifying crack propagation planes and underlying failure mechanisms. [13] According to fracture mechanics principles, four major fracture modes may be identified in polymeric materials: microvoid coalescence (dimple rupture), transgranular cleavage, fatigue fracture, and decohesive rupture. In the present study, the predominance of microvoid-associated brittle fracture features in the 20-minute group and the increasing evidence of crack deflection, branching, and plastic deformation in the 60-minute group suggest a transition from defect-driven brittle failure toward more energy-absorbing fracture mechanisms with longer curing duration. One plausible mechanism underlying these findings is that longer exposure at high temperature promotes more complete polymerization and/or reduces residual monomer, which may alter microstructure and improve overall integrity. Harrison and Huggett demonstrated that "short cut" curing cycles can produce substantially higher residual monomer values than more controlled schedules, and that curing cycle selection has a major influence on residual monomer levels in denture base polymers.[4] Lamb *et al.,* also described how process variables influence residual monomer, and emphasized that residual monomer can adversely affect properties through a plasticizing effect and altered deformation behavior. [14] At first glance, a decrease in residual monomer might be expected to make PMMA more rigid; however, the fracture morphology in this study suggests that structural integrity and defect reduction likely dominated the fracture behavior. In the 20-minute group, the presence of voids and straight crack paths suggests that fracture occurred rapidly once cracks initiated, producing brittle, smooth fracture planes. With extended curing (60 minutes), fewer defects meant cracks were less likely to initiate at pores; once initiated, cracks were more likely to be deflected or branched, producing a rougher surface and crack arrest features that indicate higher resistance to crack propagation. This interpretation aligns with the concept that internal defects control crack initiation in acrylic resin and that improved processing reduces defect-driven brittle failure.[8] Support for the influence of additional heat exposure on polymer conversion/residual monomer reduction is also seen in studies where an additional heat-cure cycle reduced residual monomer concentrations. [6] Although our study did not quantify residual monomer, the observed reduction in void-related crack initiation and the more complex crack paths with longer curing are consistent with improved material integrity expected from better polymerization conditions. The current study observed a progression from straight crack paths with no crack arrest features (20 minutes) to highly deflected and frequently branched cracks with evident crack arrest (60 minutes). Crack deflection and branching generally indicate that the crack encounters microstructural barriers or heterogeneity, requiring additional energy to advance. This observation is consistent with SEM studies of denture acrylic resins in which fracture behavior patterns correlate with processing and microstructural quality. [8] Clinically, this implies that increasing curing duration at 100 °C may improve the resistance of heat-cured acrylic resin to catastrophic crack growth, even when fracture is induced in a standardized three-point bending setup. Even though the present work focused on fracture surface evaluation rather than numerical flexural strength reporting, published mechanical studies reinforce the importance of processing on fracture resistance. For example, work evaluating flexural and impact strength of PMMA denture base materials confirms that PMMA fracture resistance is sensitive to material and processing variables. [15] Thus, the morphological improvements observed in our 60-minute group (reduced porosity, increased crack deflection/branching, and plastic deformation features) are consistent with the broader literature indicating that optimized polymerization improves performance and failure resistance.

Limitations of the study

This in vitro study does not fully simulate clinical oral conditions such as cyclic masticatory loading, thermal changes, and moisture variations. Fracture analysis was qualitative and based on SEM observations without quantitative measurements or residual monomer estimation. Only one curing temperature and material were evaluated, which may limit generalization of the findings.

CONCLUSION

Within the limitations of this in vitro SEM-based study, it can be concluded that curing duration at 100 °C has a marked influence on the fracture surface morphology of heat-cured PMMA denture base resin. Short curing duration (20 minutes) was associated with predominantly brittle fracture, smoother fracture surfaces, and higher microvoid frequency, whereas extended curing duration (60 minutes) resulted in reduced porosity, increased crack deflection and branching, and predominantly ductile fracture characteristics. The findings suggest that longer curing durations at 100 °C improve internal structural integrity and resistance to crack propagation, which may contribute to enhanced durability of heat-cured acrylic denture base materials.

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Ethical approval: The study was approved by the Institutional Ethics Committee.

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