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ASSESSMENT OF MARGINAL INTEGRITY AND FRACTURE RESISTANCE OF NANOGRAPHENE-REINFORCED POLYMETHYL METHACRYLATE VERSUS CONVENTIONAL POLYMETHYL METHACRYLATE SINGLE CROWNS

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ABSTRACT

Problem: CAD/CAM PMMA, as a provisional material, needs physical, mechanical, and biological improvements.

Purpose: To compare graphene-reinforced PMMA crowns' marginal adaption and fracture resistance to CAD/CAM PMMA crowns under simulated oral conditions.

Methods: A typodont mandibular first molar was prepared for an all-ceramic crown. Duplicating it yielded 20 dental epoxy resin dies. A 3D scanning was performed for each die to design 20 crowns. Ten crowns were milled from PMMA blanks (P) and 10 from graphene-reinforced PMMA blanks (G). Next, resin cement luted each crown to its die. Each specimen was digitally imaged for marginal gap assessment. Four equidistant landmarks at each specimen's cervical circumference were measured. A chewing simulator aged tested crowns for 120,000 cycles. The marginal gap evaluation was repeated after aging. Each crown was evaluated for fracture resistance in a universal testing machine that determined fracture load in Newton. All data were statistically analyzed.

Results: The tested materials did not differ statistically before or after thermocycling. Group (P) showed no statistically significant difference in mean gap distance after thermocycling, while Group (G) showed a statistically significant rise (P-value = 0.033, Effect size = 0.719). The fracture resistance of the two groups was not significantly different (P-value = 0.839, Effect size = 0.132). **Conclusions:** The mean marginal gap between PMMA and G-PMMA was clinically acceptable. Neither material fractured beyond posterior maximal masticatory stresses, demonstrating clinical resistance. The clinical uses of graphene-reinforced PMMAs are similar to CAD/CAM PMMAs, considering them effective long-term interim materials rather than permanent restorative alternatives.

KEYWORDS: CAD/CAM PMMA crowns, graphene-reinforced PMMA, marginal fit, fracture resistance, and nanographene.

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INTRODUCTION

Provisional crowns are necessary to safeguard the prepared teeth' vitality and periodontium.[1] Additionally, they preserve both the oral function and esthetic appearance.[1,2] The crucial factor for the long-term clinical effectiveness of any restorations is the marginal adaptation.[3-6] Inadequate marginal fit can exacerbate microleakage and plaque buildup, resulting in the dissolution of cement, recurring decay, and the formation of periodontal inflammation, ultimately leading to tooth loss.[7,8] Besides marginal adaptation, the fracture resistance of interim restorations is of great concern, mainly in long-term temporization with patients having parafunctional habits or long-span fixed dental prostheses.[9]

Computer-aided design and computer-aided manufacturing (CAD/CAM) technology has currently been employed as an indirect method in producing provisional crowns.[10] CAD/CAM PMMA has superior flexural strength to bis-acrylic resin and conventional PMMA.[11] Based on the available evidence, CAD/CAM PMMA has been identified as a suitable material for long-term usage as a provisional prosthesis.[12-15]

An extremely strong and elastic flat carbon atoms monolayer organized into a two-dimensional honeycomb lattice is called "graphene". It has been extensively used to regenerate periodontal tissue by coating the implant surface to enhance osseointegration.[16-18] It possesses strong antibacterial characteristics and has demonstrated exceptional biocompatibility. Graphene's advanced properties have led to its integration with dental materials, including metals, ceramics, and polymers.

Blending graphene-linked materials and polymers creates composites with increased mechanical characteristics. Remarkably, the enhancements remain discernible even when the amount of filler added to the polymer matrix is limited. The use of graphene and carbon fillers has been demonstrated to substantially improve the flexural strength of PMMA polymers, leading to significant enhancements in their physicomechanical properties.[19,20]

This in vitro study evaluated the marginal adaptation and fracture resistance of provisional crowns manufactured from CAD/CAM PMMA and graphene-reinforced PMMA. By comparing the two different types of provisional crowns, it was possible to make an informed choice of materials based on their marginal adaptation features and fracture resistance. The current research hypothesis was that there would be a significant difference in the marginal fit and fracture resistance of graphene-reinforced PMMA and PMMA polymers that have been CAD/CAM milled.

MATERIALS AND METHODS

Sample size calculation

Via sample size calculation software (G*Power; Version 3.1.9.2, HHUD, Germany), fracture resistance was used as the primary power analysis outcome. On the basis of Reeponmaha T et al. results,[11], and using an alpha (α) level of (5%) and β level of 0.8 (Power = 80%), the Effect size (d) was 4.12. The minimum anticipated sample size was 3 specimens per group, which was increased to 10 specimens per group.

Specimens' preparation

A mandibular first molar typodont tooth was prepared to receive a ceramic crown.[21] After that, 20 dental epoxy resin dies were made by duplicating it. A 3D dental scanner (Identica Hybrid T500; MEDIT Corp., Seoul, Korea) was used to scan each prepared die. Designing of crowns (exocad Dental CAD; Exocad GmbH, Germany) was performed, and then 20 crowns were milled (K5; vhf camfacture AG, Germany); 10 crowns from PMMA blanks (PMMA DISK; Yamahachi Dental MFG., Aichi Pref., Japan), and the other 10 crowns were milled from graphene-reinforced PMMA blanks (G-CAM; Graphenano Dental, Valencia, Spain). The cement space was set at 50 µm. All crowns were abraded with 50-μm Al_2O_3 particles at a 10-mm distance and 45°angle for 20 seconds (basic Quattro IS; Renfert, Hilzingen Germany). Then, the crowns were polished on their exterior surface following the manufacturer's instructions. They were then subjected to ultrasonic cleaning (Sonorex Super; Bandelin, Berlin, Germany) and distilled water for 5 min. Lastly, oil-free compressed air drying was applied, allowing specimens to dry at room temperature for at least 4 weeks.

Sandblasting (CEMAT NT4; Wassermann Dental Maschinen GmbH, Hamburg, Germany) was used to prepare the dies and crowns for cementation.[22] Afterwards, applying 250 g of load for a minute, the crowns were seated and cemented on the corresponding dies using a self-adhesive resin cement (G-CEM; GC, IL, USA).[23]

Marginal gap distance assessment before aging

Each specimen was imaged using a stereomicroscope with an integrated camera (Nikon Eclipse E600, Tokyo, Japan). Three Mega Pixels digital camera was positioned vertically at 2.5 cm from the specimen. The angle formed by the lens axis and the emitting light sources was roughly 90 degrees. Using a fixed 35× magnification, the images were captured at their highest resolution of 2272 × 1704 pixels and then linked to a computer. The photos were captured at a minimum resolution of 1280 × 1024 pixels for each image. The gap width was measured and analyzed using a computerized image analysis system (Image J 1.43U, National Institute of Health, USA). All limits, sizes, frames, and measurable parameters were expressed in pixels. Consequently, the system underwent calibration to convert the pixels into precise real-world units. In this work, calibration was conducted by comparing a ruler of known size with a scale produced by image analysis software (ImageJ; National Institutes of Health, NY, USA).

Each specimen was photographed by capturing shots of its margins (Fig. 1). For each image, morphometric measurements were conducted using four equidistant landmarks along the cervical circumference of each surface. The measurement at every point was conducted three times.

Artificial ageing

A dual-axis chewing simulator (four-station multimodal ROBOTA chewing simulator; ROBOTA Co., Giza, Cairo, Egy Technology Co.) performs the artificial aging on the crowns. A servomotor (model ach-09075dc-t; AdTech technology co., Shenzhen, China) was equipped with an inbuilt thermo-cyclic protocol to run this simulator. Both groups of specimens were mounted and subjected to sequential testing under a weight of 5 kg and a chewing force of 49 N for 120,000 cycles.

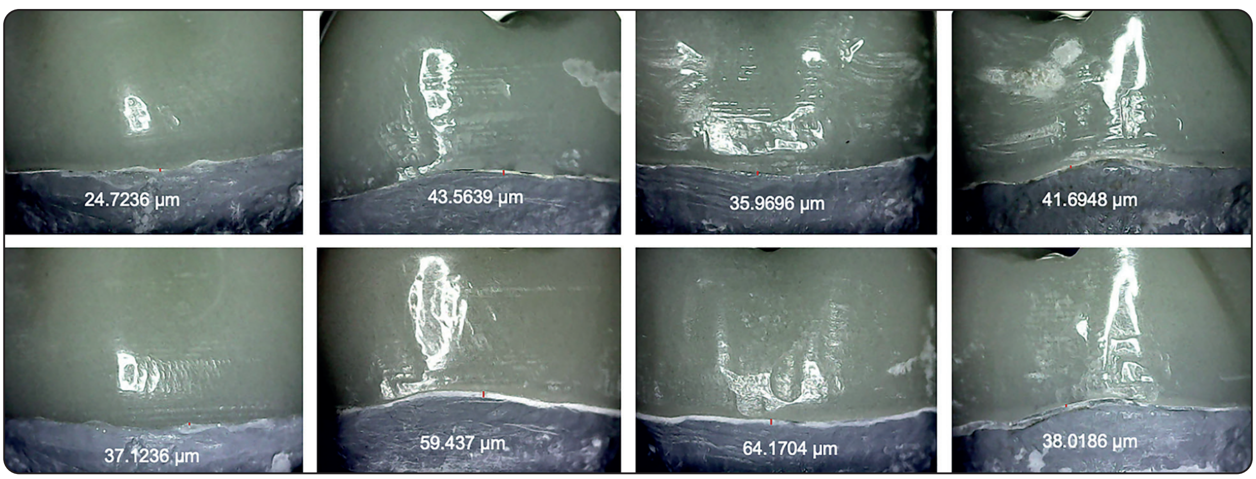

Fig. (1): Marginal gap measurements of PMMA (A) and G-PMMA (B) crowns at each surface using a stereomicroscope.

This simulation replicated a 6-month clinical service period.[23,24] Following that, the specimens were immersed in distilled water for 3 min to undergo ultrasonic cleaning.

Marginal gap distance assessment after aging

The specimens were assessed for marginal gap distance, as mentioned before.

Fracture Resistance

For fracture resistance, every specimen was tested on a computer-controlled materials testing machine with a 5 kN load cell (Model 3345; Instron, MA, USA). The specimens were mounted on the testing machine's lower fixed compartment. A compressive mode of the load was applied occlusal through a rounded tip metallic rod (8.6 mm diameter) attached to the top moveable compartment of the testing machine. To guarantee consistent stress distribution and lessen the transmission of local force peaks, a tin foil sheet was positioned between the rod and specimen (Fig. 2). The rod was moved at a cross-head speed of 1 mm/min. Coinciding with an audible crack and marked fracture (Fig. 3) and an abrupt decrease in the load-deflection curve occurrence. The load at failure was recorded in Newtons as all data was collected by computer software (Bluehill Universal; Instron, MA, USA).

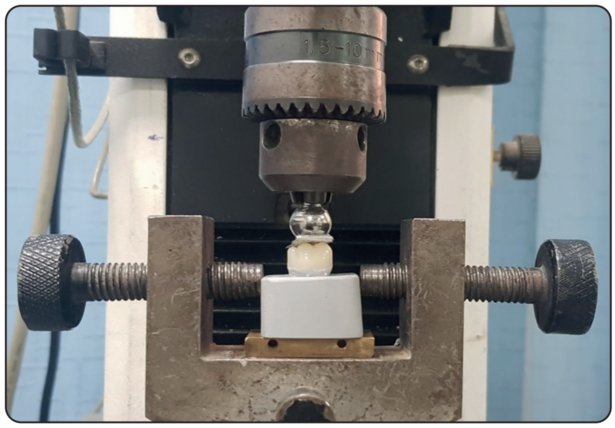

Fig. (2): A specimen under testing on the universal testing machine to evaluate its fracture resistance.

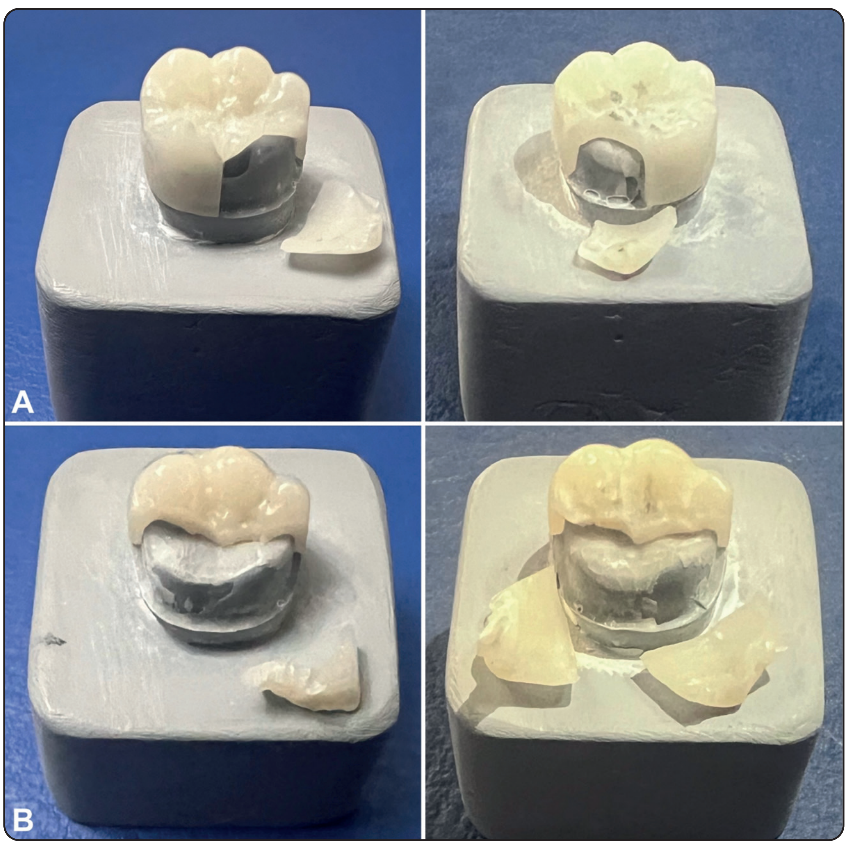

Fig. (3) Fractured specimens; PMMA crowns (A) and graphene-reinforced PMMA crowns (B).

Statistical Analysis

By exploring the data distribution and applying normality tests (Kolmogorov-Smirnov and Shapiro-Wilk tests), numerical data showed a normal (parametric) distribution using statistical software (IBM SPSS Statistics for Windows: Version 23.0; IBM Corp., NY, USA). Data were shown as mean standard deviation (SD) values. Student's t-test was used to compare the fracture resistance of the two material types. Repeated measures ANOVA test was used to study the effect of material type, thermocycling, and their interactions on mean gap distance. Bonferroni's post-hoc test was used for pair-wise comparisons in the significance of ANOVA. The significance level was set at $P \leq 0.05$.

RESULTS

Marginal gap (μm)

Regardless of thermocycling, material type had no statistically significant effect on mean gap distance (P-value = 0.887, Effect size = 0.006). Similarly, there was no statistically significant change in mean gap distance after thermocycling (P-value = 0.051, Effect size = 0.654). Additionally, the interaction between variables had no statistically significant effect on mean gap distance (P-value = 0.151, Effect size = 0.440), leading to the independence of variables as shown in Table 1.

Regarding Group (P), there was no statistically significant change in mean gap distance after thermocycling (P-value = 0.527, Effect size = 0.107). On the other hand, Group (G) revealed a statistically significant increase in the mean gap distance after thermocycling (P-value = 0.033, Effect size = 0.719), as shown in Table 2.

Fracture resistance (N)

No statistically significant difference in fracture resistance between the two groups (P-value = 0.839, Effect size = 0.132) was found as shown in Table 3.

TABLE (1) The mean, standard deviation (SD) values and results of repeated measures ANOVA test for main effects of different variables on gap distance (μm).

Source of variation		Type III Sum of Squares	df	Mean Square	Mean (μm)	SD	*F*-value	*P*-value	Effect size *(Partial eta squared)*
Material type	**Group (P)**	0.551	1	0.551	38.9	4.1	0.023	0.887	0.006
	Group (G)				39.3	4.5			
Thermocycling	**Before**	44.998	1	44.998	37.2	4.3	7.559	0.051	0.654
	After				42.5	1.8			
Material type x Thermocycling interaction		18.686	1	18.686			3.139	0.151	0.440

df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05.

TABLE (2) The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between gap distance (μm) with different interactions of variables.

Thermocycling	Group (P)		Group (G)		*P*-value	Effect size *(Partial eta squared)*
	Mean	SD	Mean	SD		
Before thermocycling	38.2	5.2	36.1	4	0.616	0.069
After thermocycling	39.6	3.6	42.5	1.8	0.275	0.285
***P*-value**	0.527		0.033*			
Effect size *(Partial eta squared)*	0.107		0.719			

: Significant at $P \le 0.05$.

Table (3): Descriptive statistics and results of Student's t-test for comparison between fracture resistance (N) of the two groups.

Group (P)		Group (G)		*P*-value	Effect size *(d)*
Mean (N)	**SD**	**Mean** (N)	**SD**		
1539.2	80.6	1574.5	368.1	0.839	0.132

: Significant at $P \le 0.05$.

DISCUSSION

Polymeric materials, particularly PMMA, are essential in fabricating temporary oral prostheses, veneers, and crowns in the dental sector.[25,26] This synthetic polymer satisfies the prosthetic criteria in esthetics, mechanical characteristics, chemical stability, corrosion resistance, and biocompatibility for potential application as a dental material.[27-34] Technological progress in digital dentistry has led to the introduction of CAD/CAM PMMA-based polymers. A strongly cross-linked structure of these materials may provide mechanical advantages over conventionally polymerized PMMA resins, making them suitable for alternative materials in long-term interim prostheses.[27]

Still, some researchers have documented particular limitations of the material, including discoloration, hydrolytic degradation, and low fracture resistance.[35] Consequently, many researchers are currently devoted to enhancing the material's physical, mechanical, and biological characteristics by integrating nanoparticles, such as graphene oxide, into its composition to broaden its applications.[36-39] Some authors argue that as chemical and mechanical reinforcements using comparable materials have demonstrated significant enhancements in the mechanical characteristics of PMMA, it remains difficult to avoid compromising other features such as color, translucency, or biocompatibility. One possible explanation for why PMMA continues to be the preferred material for long-term provisionlization is as indicated.[40]

Graphene is a honeycomb lattice composed of a single layer of sp2 hybridized carbon atoms with remarkable properties and viability.[41-44] Graphene possesses a distinctive combination of various exceptional characteristics, including a large specific surface area of 2630 $m^2\ g^{-1}$, remarkable thermal conductivity of 5000 $Wm^{-1}\ K^{-1}$, high intrinsic mobility of 200,000 $cm^2\ v^{-1}\ s^{-1}$, and Young's modulus of Y < 1.0 Tpa.[45-50] Regarding biomedical applications, graphene presents various benefits, including its biocompatibility and biodegradability, strength, flexibility, and antibacterial properties.[51-54] Graphene-based materials can be classified into four fundamental groups: single-layer and few-layered graphene, graphene oxide (GO), and reduced graphene oxide (rGO).[55,56]

Horizontal discrepancies, such as over-contoured margins, permit some clinical modifications. Nevertheless, according to Holmes et al.,[57] vertical marginal gaps are more detrimental and more difficult to rectify, potentially leading to the gradual dissolution of the cement and consequent complications and failure.[58] Thus, this study quantified solely the vertical marginal gap.

In vitro techniques such as direct visualization, cross-sectioning, and the silicone replica technique have been employed to quantify the marginal gap of restorations. However, each method has its limitations.[59,60] A direct observation technique utilizing a stereomicroscope was selected as it can subject the specimen to cementation and occlusal loading, as well as simulations that mimic intraoral conditions and ensure the preservation of each specimen following measurement. Additionally, it can analyze the slight difference between the results before and after aging. In addition, this approach enables measurements to be conducted in several positions around each die, minimizing the risk of mistakes due to the selection of measurement areas.

Marginal discrepancies of interim restorations are influenced by several factors, such as material types, fabrication procedures, thermal and mechanical aging, and the interval duration in which the restorations are used.[35,61-63] The CAD/CAM PMMA exhibited high marginal accuracy values (38.2±5.2 μm) owing to its uniform and extensively cross-linked structure, as well as a polymerization process conducted under ideal conditions of high pressure and temperature,[40,64,65] which resulted in reduced water solubility and sorption.[65] The study by Abdullah et al. revealed that provisional crowns produced using CAD/CAM technology exhibit a superior fit compared to direct provisional crowns.[66] Nevertheless, G-PMMA exhibited a marginal gap distance of 36.1±4 μm, which was not statistically significant compared to PMMA. Hence, the hypothesis was rejected.

The thermomechanical aging process influences the marginal gap of interim restorations through several mechanisms, such as polymerization stresses, residual unreacted monomer, voids in resins, and water sorption.[61,64,67-69] Thermal fluctuations induce expansion and contraction of the resin, particularly in the thin margin region, resulting in the beginning and spread of cracks through weak or porous resin areas, potentially leading to an increase in the marginal gap.[61,64] Moreover, the water employed in the process of simulated aging has the potential to infiltrate and reduce the length of the polymer chain, therefore causing fatigue of the resin at the margin. This degradation would result in releasing the remaining tension and the remaining marginal integrity.[61,67,69] The presence of moisture in the surroundings can also cause the leaching of the remaining monomer, leading to a higher concentration of voids near the margin and a higher probability of fracture.[67] Subjecting to repeated occlusal pressure, stress can be evenly spread over the temporary crowns. Whenever the stress is above the elastic threshold of the resin, plastic deformation can take place, distorting the marginal region where the plasticizing influence of water has already weakened.[64,69]

The hypothosis regarding the marginal gap was rejected. Both tested materials exhibited an increase in the marginal gap distance, with non-significant effects in PMMA crowns (39.6±3.6 μm). However, a significant difference in G-PMMA crowns' marginal gap distance (42.5±1.8 μm) was observed. This occurrence may be attributed to the presence of residual monomers and a delay in the polymerization reaction caused by graphene interference.[70]

The impact of aging on the marginal gap of CAD/CAM interim restorations has mostly concentrated on CAD/CAM milling interim restorations and typically only includes thermocycling.[71] Hence, it is still necessary to compare with prior research. The current investigation found that the marginal gap, although increased significantly in G-PMMA crowns following aging treatment, remained within a clinically acceptable range. Therefore, according to the available evidence, McLean et al.[72] indicated that a maximum margin gap of 120 µm is regarded clinically acceptable. Boening et al.[73] also suggested that a marginal gap between 100 µm and 200 µm may fall within the clinically allowed range. The results indicate that the materials chosen for the investigation maintained a satisfactory, marginal fit even after undergoing thermomechanical aging, similar to the effects observed after 6 months of oral service.

Concerning fracture resistance, the hypothesis was rejected. The fracture resistance of tested G-PMMA crowns (1574.5+368.1 N) showed no significant improvement relative to PMMA crowns (1539.2+80.6 N). The observed behavior could be attributed to the extremely low concentration of graphene. This is consistent with the findings of Punset M et al., who documented that the concentration of graphene, similar to the material investigated in this study, which the manufacturer does not reveal, was examined using Raman spectra and determined to be 0.027% by weight using the phase rule.[43]

Nevertheless, several studies have demonstrated that the mechanical characteristics of polymers were enhanced when the graphene concentration was low.[74,75] Alamgir et al. discovered that graphene was incorporated into the PMMA resin at a concentration of 0.0025 wt%, which was very low, yet it led to enhanced resistance to deformation and a higher Young's modulus in the nanocomposite compared to PMMA.[76]

However, graphene's use as a reinforcement in composites exhibits remarkable efficacy in terms of mechanical characteristics. Various applications employ a 2.5% weight concentration of the composite material.[77] These ingredients are unsuitable for prosthetic materials that require excellent aesthetics. However, evidence suggests that when the graphene concentration in PMMA surpasses 0.35%, it has a significantly dark color, deemed unsuitable for maintaining good dental aesthetics.[78]

One crucial factor to consider is that the literature reports average occlusal forces of 250 and 350 N in the incisor and molar regions, respectively. Nevertheless, individuals suffering from bruxism may incur far greater forces, and existing evidence indicates that these values might rise to 720–900 N.[79-81] The fracture values, which surpassed the maximal masticatory forces of around 900 N, indicate that all the materials investigated possess the capacity to withstand the clinically encountered forces.

Regarding the clinical significance, contrary to a recent study,[11] the current results indicate that the specific graphene-reinforced PMMA polymer being studied differs from traditional CAD/CAM PMMA polymers, particularly in its mechanical responses to compressive stresses. Hence, the current data do not provide evidence to expand the clinical uses of graphene-reinforced PMMAs beyond those of traditional CAD/CAM PMMAs. Therefore, it is recommended that they continue to be considered efficient long-term interim materials rather than alternating materials for permanent restorations. Nevertheless, some manufacturers assert that their graphene-reinforced PMMA-based material is suitable for definitive prostheses.[27] However, the evidence regarding the mechanical dominance of dental restorative materials reinforced with graphene remains limited.[27,43,82]

An inherent limitation of the current study is that it exclusively utilized commercially accessible dental materials. Furthermore, the manufacturers refrained from revealing the graphene amount incorporated into the graphene-reinforced polymer under investigation as a patent safeguards the composition. The method to include or chemically bind the graphene to the PMMA polymer must be clearly defined. Enhanced details regarding the manufacturing process, including the specific amount of graphene, should be revealed to understand better any potential relationship between the real graphene concentration and the material's final mechanical qualities and biocompatibility. Another area for improvement of the current work was the absence of any attempt to examine graphene's composition and grain size incorporated into PMMA using Raman spectroscopy or scanning electron microscopy (SEM)/X-ray diffractometry (XRD) analysis.[43,83,84] Exploring this aspect could be a captivating area for future investigation.

CONCLUSIONS

The mean marginal gap between PMMA and G-PMMA fell within the clinically acceptable range. Neither material exhibited fracture values beyond the maximal masticatory forces in the posterior area, showing the ability to withstand the clinically encountered forces. The clinical uses of graphene-reinforced PMMA align with those of traditional CAD/CAM PMMAs. It is recommended that they continue to be considered efficient long-term interim materials rather than being selected as substitutes for permanent restorations.

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