Influence of the volumes of bis-acryl and poly(methyl methacrylate) resins on their exothermic behavior during polymerization

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This study aimed to evaluate the influence of the volumes of a bis-acryl resin (Luxatemp) and a poly(methyl methacrylate) resin (Jet) on their exothermic behaviors during polymerization based on vinyl group conversion. The number of vinyl groups reacted and exotherm were determined based on weight percent of methacrylate groups using FTIR spectroscopy. Temperature changes during polymerization at 23°C were recorded for 20 minutes using a multiple cavity mold overlying a thermocouple. The number of vinyl groups reacted and exotherm of Luxatemp were consistently lower than those of Jet at each resin volume. Mean peak temperature rises of Luxatemp and Jet were in the range of 2.0–6.6°C and 4.2–11.6°C respectively, with Luxatemp and Jet taking 2 and 10 minutes respectively to reach their peak temperatures. As their resin volumes increased, their peak temperatures and total peak areas were also observed to increase significantly ($p < 0.01$).

Keywords: Exothermic behavior, Bis-acryl, Poly(methyl methacrylate)

INTRODUCTION

During tooth structure reduction in a prosthetic treatment, oral tissues are inadvertently injured during tooth preparation. Against this background, provisional restorations are crucial to ensuring predictable clinical success because of the many important roles they play in maintaining a patient's dental health before the definitive restoration is fitted: temporarily protect the pulp and tissue of prepared abutments, promote gingival healing, and defend the abutments from microleakage and chemical injuries^{1,2}. In addition, provisional restorations can assist in the development of occlusal schemes and evaluation of phonetics, occlusal vertical dimension, and mastication³.

Amidst the wide array of polymer-based provisional crown and fixed partial denture materials commercially available in the dental market, polymethyl methacrylate (PMMA) and bis-acryl resins account for a large market share^{4,6}. However, concerns have arisen about the possibility of thermal damage to the pulp with the use of these resins associated with exothermic polymerization reactions. When PMMA resin is used to fabricate provisional restorations directly on teeth using the direct method, heat generated during the exothermic polymerization of PMMA resins can result in traumatic injuries to the dental pulp^{7,8}. To overcome the problems of high exotherm and large shrinkage associated with PMMA resins, the indirect method is highly recommended as it also produces provisional restorations of superior marginal fit^{4,9}. Nonetheless, the direct method remains widely used because of time constraints and inadequate

laboratory support¹⁰.

Apart from the fabrication technique of direct method *versus* indirect method, deficiencies of high exotherm and high shrinkage were alternatively addressed by improving the provisional material, culminating in the introduction of provisional composite resins such as bis-acryl resin¹¹. However, studies have suggested that when used for direct intraoral fabrication of provisional restorations, bis-acryl resins also pose a risk of thermal injury on the pulp⁷. For example, in an experiment by Driscoll *et al.*¹², bis-acryl and PMMA resins were used to make specimens whose sizes were the same as restorations used on maxillary molars. The experiment was performed at 23°C, and the temperature increases produced by bis-acryl and PMMA resins were 14.8°C and 27.3°C respectively¹². In another study by Kim and Watts⁹, the temperature increase of four different bis-acryl resins ranged from 6.9 to 12.7°C when the specimen volume was 115.3 mm³.

Both bis-acryl and PMMA resins cause a temperature rise during polymerization¹², because both resins are cured by an addition polymerization reaction — characterized by the exothermic conversion of vinyl groups containing carbon-carbon double bonds⁹. Exothermic heat release from the resins primarily increases with an increasing number of vinyl groups, and this is directly related to the resin volume¹³. To prevent potential pulpal damage when the resins are cured intraorally, the dual factors of resin volume and residual dentin thickness should be taken into consideration⁹. Numerous studies have compared the exothermic release during polymerization of different

provisional materials, and differences in temperature rise were observed among the materials^{5,9,12}. However, few studies are available on the relationship between the number of carbon-carbon double bonds (C=C) in a resin material and the resulting exothermic behavior during polymerization.

The purpose of this study was to investigate the influence of the volumes of bis-acryl and PMMA resins on their exothermic behaviors in terms of the number of reacted vinyl groups in the materials. The conversion of C=C into carbon-carbon single bonds (C–C) before and after curing was examined using Fourier transform infrared (FTIR) spectroscopy. These results were evaluated in conjunction with the respective changes in temperature during polymerization. A thermocouple was used to measure the respective temperatures from the start of mixing and through the polymerization reaction for a total duration of 20 minutes.

MATERIALS AND METHODS

The bis-acryl and PMMA resins investigated in this study were Luxatemp (DMG, Hamburg, Germany) and Jet (Lang Dental Manufacturing Co., Wheeling, IL, USA) respectively. Details of these provisional materials are given in Table 1.

Monomer conversion calculation

In this study, the degree of conversion (DC) of each material was expressed as a change in the weight percent of methacrylate groups (WPMG) before and after curing¹⁴. Solutions of known dilution of methyl methacrylate (MMA) (Junsei Chemical Co., Ltd., Tokyo, Japan) with spectroscopic-grade hexane were prepared. The molecular weight and density of MMA were used to calculate the number of moles of C=C per mL for each of the standard solutions. The infrared spectrum of each solution was obtained using a FTIR spectroscope (IRPrestige-21, Shimadzu, Tokyo, Japan) with an attenuated total reflectance (ATR) unit (MIRacle, Pike Technologies Inc., Madison, WI, USA) under these experimental conditions: gain=1, 8 scans, 2 cm⁻¹ resolution. Height of the absorbance peak of aliphatic C=C at 1,636 cm⁻¹ was determined by baseline technique. Linear regression analysis was performed on the data to yield the mathematical relationship between the

absorbance of the monomer solutions and C=C concentration per mL^{14,15}.

The mathematical relationship shown in Fig. 1 was used to determine the C=C molar concentrations of the uncured resins—for Luxatemp, it was the resulting paste immediately after mixing the two components; for Jet, it was the liquid monomer. Weight of the methacrylate groups in each mL of the uncured resins was calculated by multiplying the molar concentration by the molecular weight of a methacrylate unit. The density of Luxatemp was determined using a density determination kit (VPG214CN, Ohaus Corp., Pine Brook, NJ, USA) at room temperature. For Jet, its density was measured using a 10 mL volumetric flask and an analytical balance (AP210S, Ohaus Corp.) at room temperature¹⁴. The WPMG of each uncured resin (WPMG_u) was calculated by dividing the weight of the methacrylate groups per mL of the resin by the resin density¹⁵.

Each resin mixed according to its manufacturer's recommendations was placed between two polyester strips and pressed with two plain glass slides on either side of the material. The thin resin film was self-cured for 20 minutes in air at ambient temperature (23°C).

Fig. 1 Calibration curve establishing the relationship between IR absorption and C=C molar concentration per mL.

Table 1 Resin-based provisional materials investigated in this study

Material	Category	Manufacturer	Composition	Lot number
Luxatemp	Bis-acryl	DMG, Hamburg, Germany	Urethane dimethacrylate, Aromatic dimethacrylate, Glycol methacrylate, Glass, Silica filler	576202
Jet	Poly(methyl methacrylate)	Lang Dental Mfg. Co., Wheeling, IL, USA	Powder: Polymethyl methacrylate, Benzoyl peroxide; Liquid: Dibutyl phthalate, Ethyl alcohol, Methyl salicylate, <i>N,N</i> -dimethyl- <i>p</i> -toluidine	Powder: 1410-06ER; Liquid: 144207AS/01AB

Next, thin resin specimens were individually sandwiched between two diamond cell units, which were mounted on a microscope (AIM-8800, Shimadzu) connected to a FTIR spectroscope. Height of the absorbance peak of C=C group at $1,636\text{ cm}^{-1}$ was calculated for each resin. The thickness of each cured specimen was measured using a micrometer. The WPMG of each cured resin specimen (WPMG_c) was calculated using the following equation ($n=10$):

$$\text{WPMG}_c = A/(T \times K)$$

where A =height of C=C absorbance peak at $1,636\text{ cm}^{-1}$, T =thickness of cured film (mm), and K =optical constant of methacrylate group (0.64)^{14,15}.

DC was calculated by determining the proportion of WPMG_c to WPMG_u as follows:

$$\text{DC (\%)} = (1 - \text{WPMG}_c / \text{WPMG}_u) \times 100$$

Thermocouple measurement

Figure 2 is a schematic presentation of the multiple cavity mold used in this study. Grooves (25 mm in length) that allowed the thermocouple to sit under the specimen were prepared in a flat acrylic plate. The grooves originated from the outer edge towards the center of the plate. A Type T thermocouple (Fep-T, Yamari Industries, Ltd., Osaka, Japan) made from 0.65-mm electric wire with an exposed tip of 5 mm was placed inside each groove. A 50- μm -thick polyester film was placed over the thermocouples. To cater to the different resin volumes, a Teflon plate with cavities of 6 mm diameter and of different heights (4, 6, 8, 10, and 12 mm) was prepared and placed over the polyester film. Mold heights and their resultant resin volumes are presented in Table 3. Finally, a polyester film and glass plate were placed over the multiple cavities in the Teflon mold. This setup was connected to a data logger (TDS-102, Sokki, Tokyo, Japan) to record the temperature change and the time taken to reach peak temperature during the polymerization reaction of the materials.

With an automix applicator gun, the Luxatemp mixture was injected directly into each cavity in the Teflon mold immediately after discarding a small amount of initial material. For Jet resin, its powder and liquid components were mixed in a rubber bowl using a plastic spatula for 1 min and then transferred immediately into each cavity (powder:liquid=2:1 by weight). Thereafter, excess material flowing out of each cavity was removed by pressing with a polyester film and a glass plate. Finally, the assembly was held in place using clips.

With the experiment conducted at an ambient temperature of 23°C , temperature changes of the materials were recorded as a function of time (every 2 sec for 20 min from the start of mixing) as their polymerization reactions proceeded. Based on the data obtained for each peak temperature rise (*i.e.*, the difference between actual maximum temperature measured and 23°C) and the time taken to reach the peak temperature, the total peak area was calculated⁹. The test was repeated 10

Fig. 2 Schematic illustration of multiple cavity mold used in this study.

times for each variable ($n=10$).

Filler weight percent of Luxatemp

The filler weight content of Luxatemp was calculated using the standard ash method¹⁶. The initial weight of each specimen (W_0) was measured using an analytical balance. After heating in an electric furnace for 30 min at 600°C , the specimen was re-weighed (W_1). Filler weight fraction (wt%) was determined with the following formula ($n=3$):

$$\text{Filler wt\%} = (W_1 / W_0) \times 100$$

Statistical analysis

Data obtained were statistically analyzed using Student's *t*-test or one-way ANOVA with Scheffe's *post hoc* test at a significance level of 0.01. All statistical analyses were performed using SPSS 14.0 for Windows (SPSS, Chicago, IL, USA).

RESULTS

Table 2 summarizes the C=C molar concentrations per mL of uncured Luxatemp and Jet resins (as determined using FTIR spectroscopic method) and the DC value of each resin (calculated using the proportion of WPMG_c to WPMG_u). Luxatemp had a lower C=C molar concentration than Jet (0.0058 versus 0.0095 mol/mL) and a significantly lower DC after 20 minutes of curing ($53.46 \pm 2.78\%$ versus $81.60 \pm 1.19\%$) ($p < 0.001$).

For each volume of each resin, Table 3 presents the calculated number of moles of C=C before curing, the number of moles of reacted C=C after curing, and the curing exotherm. The values of Luxatemp were consistently lower than those of Jet at each resin volume.

Figure 3 shows the representative temperature increase-time plots of each resin at different volumes. Table 4 presents the means and standard deviations of peak temperature rise, the time taken to reach peak temperature, and the total peak area of each resin at each volume. The mean peak temperature rises for

Luxatemp and Jet were in the range of 2.0–6.6°C and 4.2–11.6°C respectively, whereby the magnitude of temperature rise increased significantly with increasing volume of each resin ($p<0.01$). Time taken to reach peak temperature was approximately 2 min for Luxatemp and 10 min for Jet, and that no significant differences in time were found among the different volumes of each resin ($p>0.01$). When calculating the total peak area, a straight line was assumed between the start of mixing and the start of measuring for each volume of Jet resin. Similarly, the total peak area increased significantly

with increasing volume for each resin ($p<0.01$).

Figure 4 shows the relationships between the number of moles of reacted C=C and peak temperature rise as well as with the total peak area. Strong correlations were found between the number of moles of reacted C=C and peak temperature rise ($R^2=0.9968$ and $R^2=0.9970$ for Luxatemp and Jet respectively). There were also strong correlations between the number of moles of reacted C=C and total peak area ($R^2=0.9623$ and $R^2=0.9793$ for Luxatemp and Jet respectively).

Table 2 Number of moles of C=C per mL in uncured resins and degrees of conversion of cured resins

Resin	IR absorption	Number of moles of C=C per mL	Weight of methacrylate groups per mL	Resin density (g/mL)	Weight percent of methacrylate groups	Percent degree of conversion (SD)*
Luxatemp	0.031197	0.0058	0.4000	1.5184	26.34	53.46 (2.78)
Jet	0.050819	0.0095	0.6559	0.9430	69.55	81.60 (1.19)

*Significant difference was found according to Student's *t*-test ($p<0.001$).

Table 3 Calculated number of moles of reacted C=C and exotherm after curing for each volume of each resin

Mold height (mm)*	Resultant resin volume (mm ³) and code in parentheses	Calculated number of moles of C=C before curing †		Calculated number of moles of reacted C=C after curing ‡		Calculated exotherm (J) after curing §	
		Luxatemp	Jet	Luxatemp	Jet	Luxatemp	Jet
4	113.1 (C1)	0.0007	0.0011	0.0004	0.0009	19.64	49.10
6	169.6 (C2)	0.0010	0.0016	0.0005	0.0013	29.46	73.64
8	226.2 (C3)	0.0013	0.0021	0.0007	0.0018	39.27	98.19
10	282.7 (C4)	0.0016	0.0027	0.0009	0.0022	49.09	122.74
12	339.3 (C5)	0.0020	0.0032	0.0011	0.0026	58.91	147.29

* A Teflon plate with cavities of 6 mm diameter and of different heights was used.

† Each value was calculated from the C=C molar concentration per mL and resin volume.

‡ Each value was calculated by multiplying the number of moles of C=C in uncured resin by the mean monomer conversion fraction (0.5346 for Luxatemp or 0.8160 for Jet).

§ Each value was calculated by multiplying the number of moles of reacted C=C by the theoretic heat release per mole of reacted carbon double bond (56 kJ/mol)¹³.

Table 4 Peak temperature rise, time taken to reach peak temperature, and total peak area of each resin at different volumes [mean (standard deviation)]

Resin volume	Peak temperature (°C)*		Time to reach peak temperature (sec)†		Total peak area (°C sec)*	
	Luxatemp	Jet	Luxatemp	Jet	Luxatemp	Jet
C1	2.0 (0.3)	4.2 (0.4)	115 (9)	643 (31)	357.5 (98.7)	667.7 (103.3)
C2	3.3 (0.3)	6.3 (1.0)	118 (7)	607 (58)	497.8 (127.1)	996.0 (244.4)
C3	4.2 (0.3)	8.2 (1.1)	113 (3)	589 (10)	825.4 (141.0)	1,376.8 (206.7)
C4	5.6 (0.7)	9.8 (1.0)	106 (6)	619 (39)	1,344.8 (214.9)	1,899.6 (176.8)
C5	6.6 (0.6)	11.6 (1.0)	113 (15)	632 (30)	1,499.8 (309.9)	2,040.5 (231.8)

* Values gradually and significantly increased with increasing resin volume according to Scheffe's test at $\alpha=0.01$. At each resin volume, the results obtained for Luxatemp were consistently and significantly lower than those of Jet according to Scheffe's test at $\alpha=0.01$.

† No significant differences were found among all resin volumes within the same material according to Scheffe's test at $\alpha=0.01$.

Fig. 3 Temperature changes of resins with different volumes during polymerization at 23°C: (a) Luxatemp and (b) Jet. In the case of Jet, thermocouple measurements began at 1 min after mixing started.

Fig. 4 Linear regression analysis showing the relationships between the number of moles of reacted C=C (derived from FTIR spectroscopy) and peak temperature rise (derived from thermocouple measurement) as well as with the total peak area.

DISCUSSION

The exothermic behaviors of bis-acryl and PMMA resins during polymerization are related to the ΔH (enthalpy change) values of their monomers. For some monomers, the ΔH values are close to the difference between the bond energies of π -bonds in an alkene and σ -bonds in an alkane (82–90 kJ/mol)¹³. In the case of MMA, its ΔH value is only 56 kJ/mol because of the effect of its monomer structure on polymerization behavior¹³. For Luxatemp and Jet resins investigated in this study, their monomers had the same methacrylate group ($\text{CH}_2=\text{C}(\text{CH}_3)\text{C}=\text{O}$). Therefore, a basic assumption throughout this study was that heat released in the

reaction of carbon-carbon double bond (C=C) conversion to carbon-carbon single bond (C–C) was proportional to the number of reacted vinyl groups ($\text{CH}_2=\text{CH}-$)¹⁷.

Based on their IR absorption values in the calibration curve (Fig. 1), the numbers of moles of C=C in uncured resins were determined (Table 2). Before curing, the numbers of moles of C=C of Luxatemp and Jet were calculated to be 0.0058 and 0.0095 mol per mL of resin respectively. The lower C=C concentration in Luxatemp was expected because of its monomer type — namely, dimethacrylate with higher molecular weight than MMA. Moreover, the inclusion of silica and glass fillers (measured to be 41.5% in this study) in Luxatemp contributed to decreasing the C=C concentration further.

Not all C=C bonds are converted into C–C bonds during polymerization¹⁵. Therefore, the degree of conversion (DC) of each resin (expressed as a change in WPMG before and after curing) was also calculated in this study to determine the number of moles of reacted C=C and exotherm. Results showed that the number of moles of reacted C=C and exotherm after 20 minutes of polymerization were consistently lower in Luxatemp than in Jet at each resin volume (Table 3). These results which were determined using FTIR spectroscopic method were consistent with the findings of previous studies^{11,12}, where bis-acryl resins exhibited lower exotherm than PMMA resins.

Similarly, temperature rise data obtained from thermocouple measurements indicated that the peak temperatures of Luxatemp were consistently lower than those of Jet at each resin volume, and that the peak temperature increased significantly with increasing volume for both resins (Table 4). However, the mean peak temperature rise (2.0°C) generated by 113.1 mm³ (C1 volume) of Luxatemp in this study was lower than the 6.9°C temperature rise generated by a similar specimen volume of 115.4 mm³ in a study by Kim and Watts⁹. This discrepancy could be attributed to variations in experimental design and procedures, including the multiple cavity mold setup. Apart from the

critical effect of peak temperature on heat-induced pulpal damage when provisional restorations are cured intraorally, the total peak area is another important consideration⁹. In this study, the results of total peak area for both resins showed similar trends as those of peak temperature.

On the time taken to reach the peak temperature, no significant differences were found among all the resin volumes within the same resin (Table 4). For the Jet resin, Fig. 3b revealed that it required a longer induction period than Luxatemp. In addition, unlike Luxatemp, Jet did not present a clear end point of induction, but exhibited a gradual rise in temperature followed by a marked increase to the peak temperature. This phenomenon could be partly explained by its unique polymer-monomer interaction¹⁸. In the case of Jet, prepolymerized PMMA powder particles were dissolved in MMA monomer by mixing. A residual amount of benzoyl peroxide initiator which remained after the initial preparation of PMMA would form free radicals when brought into contact with the activator, *N,N*-dimethyl-*p*-toluidine. On the other hand, a polymerization inhibitor delayed the onset of the polymerization reaction of PMMA-MMA mixture¹⁸. Consequently, the gradual and slow polymer-monomer interaction, followed by the action of initiators and inhibitors, resulted in a not-so-clear initiation of the polymerization reaction. Figure 3b also implied that exothermic heat was most probably generated during dough formation¹⁹.

The patterns of temperature increase shown in Fig. 3 also provided useful guidelines on the use of Luxatemp and Jet for direct intraoral fabrication of provisional restorations. In the case of Luxatemp, not only is immediate water-cooling highly recommended⁹, an increased cooling time for the intraoral use of Luxatemp would minimize potential pulpal damage. In the case of Jet, the intraoral cooling measure (air-blowing or water-cooling) must be adequately applied for 6 to 7 minutes after mixing⁹. Apart from the use of direct technique which must be accompanied with appropriate and adequate intraoral cooling, provisional restorations can be fabricated using the reline technique or indirect technique^{20,21}.

For each resin volume, peak temperature rise and total peak area results were found to have strong correlations with the number of moles of reacted C=C determined using the FTIR spectroscopic method (Fig. 4). However, it must be pointed out that apart from the number of reacted vinyl groups, peak temperature is also a function of accelerator concentration and rate of polymerization^{9,22}. On this ground, it is probable that for the same calculated exotherm, different peak temperature and total peak area results were yielded by two different resins. In this study, C4 volume of Luxatemp and C1 volume of Jet exhibited the same number of moles of reacted C=C and hence similar exotherms (Table 3). However, the corresponding peak temperatures, and especially the total peak area results, were substantially different from each other (Table 4).

These results showed that although the number of reacted vinyl groups could be a major determining factor, it was not the single factor which determined the observed exothermic behaviors of resin-based provisional materials in this study.

During polymerization, the heat dissipation efficiency of a resin material also affects its exothermic behavior⁹. Heat generated within a resin medium, which is very viscous, becomes increasingly difficult to remove with increasing volume of the resin material²³. Moreover, different surface-to-volume ratios of resin materials could result in different heat dissipation efficiencies, although heat generated within the resins is assumed to be the same⁹. In the current study, Luxatemp was in a cavity of greater height (C4 volume) than Jet (C1 volume) when both resins exhibited similar numbers of moles of reacted C=C and exotherms. It was probable that the greater height of C4 volume cavity resulted in a higher peak temperature and total peak area. This implied that the design of resin-based provisional restorations could be a more critical factor than volume itself in adequately controlling the heat produced by a resin material. Indeed, from clinical standpoint, heat formation on the resin surface and its dissipation at the interface between resin and dentin might be more important than the peak temperature within the resin⁹.

In the case of Luxatemp, another factor that could have affected its exothermic behavior might be the influence of the incorporated fillers. This aspect was not examined in the present study, but it certainly and obviously warrants further investigation.

Results of the present study should not be applied directly and undiscerningly to the clinical environment. First, the number of moles of reacted C=C and resulting exotherm were determined solely based on the FTIR spectroscopic method. Therefore, these herein-obtained data still need to be validated by other analytical test methods. Secondly, although the findings of this study were comparable to those of previous studies^{11,12}, only two materials were investigated in this study. Thirdly, actual pulpal response to exothermic heat released from the two resin materials was not examined in this study, but which obviously mandates investigation in a future study.

CONCLUSIONS

Within the limitations of this study, the results obtained suggested that the polymerization exotherm of a resin-based provisional material was primarily proportional to the number of reacted vinyl groups, which was directly related to the resin volume. However, in the context of resin-tooth interaction in an oral environment, the number of reacted vinyl groups would not be the single determiner to the actual temperature change in the resin material. Other factors that might influence pulpal injury when resin-based provisional materials are used for direct intraoral fabrication of restorations include the condition of the tooth to be restored provisionally, heat dissipation efficiency as a

function of the provisional restoration design, and adequacy of intraoral cooling measure (such as air-blowing or water-cooling).

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