Basic and Clinical Studies on Polyfunctional Composite Resin Material

—Polymerization Shrinkage and Tensile Strength of the Polyfunctional Based Monomer

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Abstract: This study was conducted to observe the polymerization shrinkage and tensile strength of the polyfunctional based composites containing various types of diluent monomer systems at several concentrations. Fifteen matrix-monomers systems were prepared and four experimental polyfunctional composites were made by filling a hybrid filler with a constant weight of 86.5%. The rate of polymerization shrinkage was calculated by the specific gravity of the monomer and the polymer by using a picnometer and Hubbard type hydrometer. Tensile strength was measured by using an Instron Universal Testing Machine. The results of this study indicated that polymerization shrinkage of unfilled polyfunctional resin ranged from a volume percentage of 3.88 to 8.56 and the tensile strength of the unfilled polyfunctional resin showed more significant results than Bis-GMA unfilled resin. The volume percentage of polymerization shrinkage of the filled resin ranged from 1.23 to 1.89; the best and most significant value was obtained from the polyfunctional monomer diluted with Bis-MPEPP 2.6E. Tensile strength of the filled polyfunctional experimental resin ranged from 71.58 to 80.92 MPa, which indicates that M-4 resin showed significantly higher tensile strength compared to the M-6 resin formulation.

1. Introduction

Important factors in the marginal adaptation of resin based restorative materials to dental cavity walls include polymerization shrinkage, viscosity and polymerization time of the material, thermal expansion and other mechanical properties of the resin. These factors lead to the gap formation and therefore, marginal leakage between the tooth and the restoration. Several studies have shown that the marginal gap caused by initial polymerization shrinkage is often not eliminated by the acid etching technique and usage of the bonding agent or cavo surface designs1-4).

Thus, although the relationship between all the above factors and the long term adaptation is not completely clear, the new types of composite resin materials have shown good results on thermal expansion and mechanical properties. Consequently, the elimination or reduction of polymerization shrinkage is considered of major importance.

Composite resin formulations must contain a diluent to reduce the viscosity of the liquid ingredient to clinically suitable consistency.

The objective of this study was to determine the polymerization shrinkage and tensile strength of the polyfunctional-based unfilled resin containing various types of diluent monomer systems at several concentrations.
2. Materials and Methods

Fifteen matrix monomers and various types of diluent monomers at several concentrations were experimentally prepared as listed in Table 1; Figures 1 and 2, shows the molecular structures of the resin based monomers used in this study.

As shown in Table 2, a photosensitizer, camphorquinone was used for initiation of the polymerization and referred to as a photosensitizer as well as N,N-dimethylaminoethylmethacrylate tertiary amine.

Figure 3, shows the molecular structures of camphorquinone and DMAEM tertiary amine.

Four matrix monomer formulations were prepared for the experimental filled polyfunctional resin, as listed in Table 3. A hybrid filler were added to each resin formulation that contained 83.0 weight percent silica filler and 3.5 weight percent microfiller.

1) Polymerization shrinkage

The rate of polymerization shrinkage was calculated by the specific gravities of the monomer and the polymer using the following equation:

\[ S_\gamma = \frac{1}{d_m} - \frac{1}{d_p} \times 100\% \]

where \( d_m \) is the specific gravity of the monomer, \( d_p \) is the specific gravity of polymer and \( S_\gamma \) is the polymerization shrinkage.

The specific gravities of the monomers were determined by using a picnometer at a constant room temperature of 20°C and humidity of 30%.
The specific gravities of the polymers were determined by using a Hubbard type hydrometer at a constant room temperature of 20 °C and humidity of 30% using the equation:

$$S = \frac{W_m - W_b}{W_w - (W_T - W_m)} \times \left(\frac{D_w - D_a}{D_a}\right) / \rho$$

where,
- $S$: Specific gravity ($t_1/4$°C)
- $D_w$: Density of water at the temperature $t_1$°C (g/cm$^3$)
- $D_a$: Density of dry air at the room temperature (g/cm$^3$)
- $\rho$: Density of water at the temperature 4°C (g/cm$^3$)
- $W_w$: Water equivalence of Hubbard bottle at the temperature $t_1$°C (g)
- $W_b$: Weight of bottle and plug (g)
- $W_m$: Weight of the plugged bottle half filled with sample (g)
- $W_T$: Weight of plugged bottle filled with sample (lower half) and water (upper half) (g)

2) Tensile strength

To measurement of direct tensile strength, a piece of flat-base composite resin measuring 2 x 2 x 20 mm, and all samples were loaded into a metal mould to make the tensile specimen. Ten specimens were made for each trial experimental resin. Each specimen was light cured through the matrix in five aspects for 20 seconds each, using the Optilux (Demetron, CT., USA) light curing unit. The specimens were removed from the mould and stored in 100% humidity for 15 minutes at 37°C. The specimens were then immersed into distilled water for 24 hours at 37°C. After that, the specimens were removed from the distilled water and placed at room temperature of 20°C for 10 minutes. Prior to testing, each specimen was dried and tested by an Instron Universal Testing Machine, with a cross head speed of 1 mm/min.

3. Results and Discussion

1) Polymerization shrinkage of the unfilled resin

Results for the polymerization shrinkage of the unfilled resin are presented in Figure 4. From these data it is considered that the polymerization shrinkage of the unfilled polyfunctional based resins ranged from a volume percentage of 3.88 to 8.56 and the unfilled Bis-GMA based resin ranged from 4.12 to 9.46. The polymerization shrinkage of M-6 with the Bis-MPEPP 2.6E...
formulation showed the least shrinkage and the Bis-GMA with 3G formulation showed the greatest value, which can be attributed to the smaller molecular weight of 3G (M.W. = 286) compared to the other tested resin. Shimomura (1987) also indicated that the degree of polymerization of 3G was low due to its smaller molecular weight.

All means of polymerization shrinkage of the unfilled polyfunctional resin of M-4 and M-6 showed lower significant values that the means for the Bis-GMA resin tested ($p<0.005$). It also can be seen that the resin with a larger molecular weight, such as M-4 (M.W. = 596) and M-6 (M.W. = 856) should exhibit a lower shrinkage than the Bis-GMA resin (M.W. = 512).

Unfortunately, dilution of the monomer to achieve a workable viscosity for the monomer system also results in an increase of polymerization shrinkage$^{5-7)$. Thus, polymerization shrinkage should be higher for the material which contains a high proportion of low molecular weight diluents. The size of the reactive monomer is a resin system has also been ascribed much importance in determining the level of polymerization shrinkage$^9)$. It is generally believed that the greater the amount of small reactive monomer in a system, the shrinkage will be larger.

2) Tensile strength of the unfilled resin

The tensile strength of the unfilled resin is shown in Figure 5, which illustrates the quantitative significant superiority of the polyfunctional resin over the Bis-GMA resin formulation ($p<0.05$). Tensile strength of the unfilled polyfunctional based ranged from 11.35 to 44.68 MPa, which indicates that M-6 with 3G formulation showed the greatest value of tensile strength.

The relationship between polymerization shrinkage and tensile strength of the unfilled resin formulation tested is shown in Figure 6. The figure shows that the best value of polymerization shrinkage (degree of polymerization) and tensile strength was obtained from the polyfunctional monomer diluted with Bis-MPEPP 2.6E.

3) Polymerization shrinkage and tensile strength of the filled experimental resin

The polymerization shrinkage and tensile strength of the experimental resin tested is presented in Table 4. The polymerization shrinkage of the filled resin tested ranged from a volume percentage of 1.23 to 1.89, which showed that the resin with the Bis-MPEPP 2.6E diluent exhibited significantly less shrinkage compared to the resin without the Bis-MPEPP 2.6E diluent formulation ($p<0.05$).

It can be seen that the tensile strength of the
filled polyfunctional resin ranges from 71.58 to 80.92 MPa. The tensile strength of the M-4 resin was significantly higher than the tensile strength of the M-6 resin ($p<0.05$).

### 4. Conclusions

Based on the results of the investigation, the following conclusions can be drawn:

1. Polymerization shrinkage of the unfilled resin tested in this study ranged from a volume percentage of 3.88 to 9.46 depending on the diluent used, the Bis-MPEPP 2.6E diluent monomer showed the least amount of shrinkage. The same results were also obtained from the filled resin.

2. The polyfunctional unfilled resin with every diluent exhibited higher tensile strength compared to the Bis-GMA unfilled resin.

3. The tensile strength of the filled polyfunctional experimental resin ranged from 71.58 to 80.92 MPa, which indicates that the M-4 resin showed a significantly higher value of tensile strength than M-6 resin formulation.

4. A new composite resin which has a higher value of mechanical properties and less polymerization shrinkage compared to the conventional resin of the Bis-GMA and the 3G system could be made with a polyfunctional monomer diluted with the Bis-MPEPP 2.6E formulation.

5. Further laboratory and clinical studies must be carried out to confirm the physical and mechanical properties of the polyfunctional resin material.

### Acknowledgements

The authors wish to thank Prof. Toshiyuki Miyaji, Prof. Ei Suzuki, Ass. Prof. Takashi Miyazaki and Department of Dental Technology, Showa University.

### References


(Received January 20, 1991)