Effect of flowability on the flow rate, polymerization shrinkage, and mass change of flowable composites

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As a lining or restoration material, flowable composites are popular due to their ease in use and agreeable marginal adaptation of tooth cavity. The purpose of the present study was to evaluate various properties of flowable composites which were claimed to be of high or low flowability by the manufacturers. For the study, twelve flowable composites from six different manufacturers were used. Based on the manufacturers’ claim, they were divided into two groups: high or low flowability. The products grouped in high flowability showed significantly higher \((p<0.05)\) flow rate (flow distance), polymerization shrinkage, and microhardness (on the top surface), regardless of light-curing unit, than those grouped in low flowability. On the other hand, water sorption and solubility showed non-significantly different values regardless of flowability. Through the study, flow rate had low correlation with microhardness \((R<0.23)\), polymerization shrinkage \((R<0.60)\), and water sorption and solubility \((R<0.36)\) regardless of light-curing unit.

**Keywords**: Flowable composites, Flowability, Flow rate, Polymerization shrinkage, Mass change

INTRODUCTION

As a dental restorative material, light-curing resin composites have inherited many favorable characteristics to compatible with host teeth. They are easy to use, immediately become a rigid rock, enough to serve, from a sticky paste by a external blue light, and can be repaired anytime if the restorative makes functional or structural problems. Since resin composites are commixture of monomers and inorganic fillers, the mechanical properties of resin composites are influenced by these constituents.

Functionally, resin composites are needed to have high strength and surface hardness with high wear resistance to endure mastication stress if they are restored as a part of tooth structure\(^1\)-\(^2\). To sustain wet oral environment while serving as restorative material, they must have low water sorption and solubility to make the restored material intact and to retain the initial integrated structure, whereas they sometimes must allow the dissolution of inorganic constituents such as fluoride to treat or prevent caries\(^3\)-\(^7\). Also, to meet the diverse aesthetic requirements, resin composites are needed to have excellent color matching for diverse tooth color conditions, translucency, and color stability against broad pH and temperature ranges in the oral cavity due to the complicated oral environments by foods, beverages, and oral acids\(^8\)-\(^11\). Through the long-term progress and improvement, recently available resin composites have many favorable and agreeable mechanical properties and aesthetics. However, despite many such excellences, some clinical issues such as polymerization shrinkage, restoration fracture, and recurrence of secondary caries are still inevitable in the monomer-based resin composites due to the shrinkage-carried polymerization process\(^12\)-\(^14\).

Unlike most ordinary resin composites, the outstanding feature of flowable composites is the flowable nature (flowability). This remarkable feature allows the flowable composites to have an easy and swift handling during restoration with agreeable wetting and marginal adaptation in the tooth cavity and to bond orthodontic brackets with acceptable bond strength\(^15\)-\(^17\). Generally such flowable nature was achieved by compromising the content of fillers and diluents; as a result, reduction of inorganic filler content decreases microhardness and increase of diluents content increases the polymerization shrinkage. However, since each basic monomer [ethoxylated bisphenol A glycidyl methacrylate (Bis-EMA), bisphenol A glycidyl methacrylate (Bis-GMA), triethyleneglycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA)] has much different degree of conversion, modulus, viscosity, water sorption, and solubility\(^18\)-\(^20\), flowable composites may have much different mechanical properties depending on the combination and ratio of such monomers and fillers. Recently, flowable composites of different flowabilities (claimed high or low by the manufacturers) are available to meet diverse needs for easier handling and working, however, due to the recent introduction, studies for investigating the effect of flow rate are rare.

The purpose of the present study was to investigate various properties (flow rate, polymerization shrinkage, microhardness, and water sorption and solubility) of flowable composites which were claimed by the manufacturers to have high or low flowability. The hypothesis to be tested is that flowable composites of high flowability claimed by the manufacturers have
consistently different various properties compared with those of low flowability.

**MATERIALS AND METHODS**

*Specimens and light-curing units (LCUs)*

For the study, flowable composites of two different flowabilities (high or low) were used: claimed high flowability [AeliteFlo LV (AL), Beautifil Flow-High Flow (BH), Estelite Flow Quick HF (EH), G-aenial Flo (GF), Palifique Estelite LV-High Flow (PH), UniFil Flow (UF)] and claimed low flowability [AeliteFlo (AF), Beautifil Flow-Low Flow (BL), Estelite Flow Quick (EQ), G-aenial Universal Flo (GU), Palifique Estelite LV-Low Flow (PL), UniFil LoFlo Plus (UL)]. Details of the tested products were summarized in Table 1.

For the light curing, a conventional QTH [Optilux 501 (OP), Kerr, Danbury, CT, USA] and a LED [L.E.Demetron (DE), Kerr, Danbury, CT, USA] light-curing units (LCUs) were used as light sources. The output light intensity of OP and DE was 900 mW/cm² according to the radiometer (Demetron radiometer, Kerr, Danbury, CT, USA).

*Flow distance*

To measure the flow distance of each product, 0.1 g of resin was uniformly squeezed on the plate to make a circle (diameter: approximately 6 mm). To squeeze 0.1 g, a mark that can make 0.1 g mass was made on the syringe wall after the repeated exercises. The plate was stand at 90° and let the resin flow for 1 min at 23±1°C environment. After that, the resin was immediately light cured for 20 s to stop the flow. The flowed distance was measured using a Vernier caliper (series 530; Mitutoyo, Tokyo, Japan).

### Table 1  Materials tested in the present study

| Code | Composition | Filler content vol%/wt%/wt%² | Manufacturer |
|------|-------------|-----------------------------|--------------|
| AF   | Bis-EMA, TEGDMA, Barium glass, glass fillers | 42/60/56.2 | Bisco Inc. Schaumburg, IL, USA |
| AL   | Bis-EMA, TEGDMA, Barium glass, glass fillers | 37/60/58.8 |  |
| BH   | Bis-GMA, TEGDMA, Al-F-B-silicate glass, glass filler, Al₂O₃ | 46.8/53.5/47.4 | Shofu Kyoto, Japan |
| BL   | Bis-GMA, TEGDMA, Al-F-B-silicate glass, glass filler, Al₂O₃ | 48.3/54.5/48.9 |  |
| EH   | Bis-GMA, TEGDMA, silica-zirconia filler | 49/68/65.8 |  |
| EQ   | Bis-EMA, TEGDMA, UDMA, silica-zirconia, silica-titania filler | 53/71/68.7 | Tokuyama Tokyo, Japan |
| GF   | Bis-EMA, UDMA, glycerol dimethacrylate, strontium glass, LaF₃, SiO₂ | /69/63.6 | GC Tokyo, Japan |
| GU   | Bis-EMA, TEGDMA, UDMA, strontium glass, SiO₂ | 50/69/62.4 |  |
| PH   | Bis-GMA, TEGDMA, silica-zirconia, silica-titania filler | 50/68/66.5 | Tokuyama Tokyo, Japan |
| PL   | Bis-EMA, Bis-GMA, TEGDMA, UDMA, silica-zirconia, silica-titania filler | 50/65/63.5 |  |
| UF   | TEGDMA, UDMA, AL-F-silicate glass, silica | 45/67/62.8 | GC Tokyo, Japan |
| UL   | TEGDMA, UDMA, AL-F-silicate glass, silica | 42.7/63/43.3 |  |

AF: AeliteFlo; AL: AeliteFlo LV; BH: Beautifil Flow- High Flow; BL: Beautifil Flow- Low Flow; EH: Estelite Flow Quick HF; EQ: Estelite Flow Quick; GF: G-aenial Flo; GU: G-aenial Universal Flo; PH: Palifique Estelite LV- High Flow; PL: Palifique Estelite LV- Low Flow; UF: UniFil Flow; UL: UniFil LoFlo Plus

wt%¹: weight percent provided by the manufacturers

wt%²: weight percent determined by ash method
Filler weight
The filler weight (wt%) of each resin product was determined using standard ash method in addition to the nominal weight which was provided by the manufacturers. The weight (approximately 50 mg) of each resin specimen was measured using an analytical balance after light curing for easy handling. Then the specimen (n=5) was heated in an electric furnace at 650°C for 30 min to burn out the organic matrix. The wt% was determined by dividing the weight of original specimen to that of the remaining specimen after heating in furnace.

Polymerization shrinkage
The polymerization shrinkage (µm) of the specimens during and after the light-curing process was measured (n=5 for each product) using a linometer (RB 404, R&B Inc., Daejon, Korea). A resin of cylinder shape (diameter: 4 mm, thickness: 2 mm) was placed over the aluminum disc (the specimen stage of the linometer) and its top surface was secured using a glass slide. Since resin can flow downward, placing resin over the aluminum disc was performed as quickly as possible after repeated exercise. The end of the light guide was placed in contact with the glass slide. Before light curing, the initial position (the distance between the aluminum disc and inductive shrinkage sensor) of the aluminum disc was set to zero and then the light from the LCU was irradiated for 40 s. The amount of disc displacement from the sensor that occurred due to polymerization shrinkage was measured automatically for 130 s using the sensor. The resolution and measurement range of the sensor were 0.1 µm and 100 µm, respectively.

Microhardness
To measure the surface microhardness (Hv) of the specimens, resin was filled into a metal mold (4×2×3 mm), both the top and bottom surfaces were covered with glass slides, and light cured for 40 s using the LCU. The cured specimen was removed from the mold and aged for 24 h in a 37°C dry and dark chamber. The microhardness of the top (z=0) and bottom (z=3 mm) surfaces was measured using a Vickers hardness tester (MVK-H1, Akashi, Tokyo, Japan) by evaluating the size of microindentations (n=12 for each test condition). To make the microindentation, a 200-gf load and 10-s dwell time conditions were applied.

Water sorption and solubility
For the measurements of mass change (water sorption and solubility), the specimens (n=7 for each case from each product) were prepared according to the ISO 4049 guidelines. A ring-type metal mold (inner diameter: 8 mm, thickness: 2 mm) was placed over the thin glass slide. Each resin was placed in the mold, and the top surface was covered with a thin glass slide, pressed to make the surface flat, and light cured for 40 s. The specimens were removed from the mold and placed in a desiccator for 22 h at 37°C and then for 2 h at 24°C. Subsequently, the weight was measured to an accuracy of ±0.001 mg (according to ISO 4049, an accuracy of ±0.1 mg is sufficient) by using an analytical balance (Sartorius CP2P, Goettingen, Germany). This cycle was repeated until a constant mass m2 was obtained (in this study, a deviation of less than 0.005 mg was assumed to be constant). After the completion of the m2 measurement, specimens were then immersed in the distilled water for 2 weeks. At this time, each specimen was placed in a tube filled with 15 mL of distilled water. After 2 weeks, the specimens were removed from the tube, and the solution on the surfaces was blotted away until there was no visible moisture. The specimen was then waved in the air for 15 s, and weighed (m3) 1 min after removal from the tube. Subsequently, the specimens were placed in a 37°C desiccator and weighed every 24 h until a constant mass (m4) was obtained. The specimen volume (V) was determined by measuring the specimen diameter at right angles to each other and the thickness at the center of the specimen as well as at four equally spaced points on the circumference using Vernier calipers (series 530, Mitutoyo, Tokyo, Japan) with an accuracy of 0.02 mm. Water sorption (Wsp) and solubility (Wsl) were calculated using the formulas, Wsp=(m5−m4)/V and Wsl=(m5−m3)/V, respectively, where V (mm³) is the specimen volume and m1, m2, and m3 are the masses of the specimen before immersion, after immersion for 2 weeks at 37°C, and after immersion and drying, respectively.

Statistical analysis
The results of each test were analyzed using one-way ANOVA for resin products. A post-hoc Tukey test was performed for a multiple-comparison. t-Test was performed to find the statistical difference between LCUs. All tests were analyzed at α=0.05.

RESULTS
The flow distance (cm) and flow rate (mm/s) of the tested products are shown in Fig. 1 and Table 2. Among the specimens, products of high flowability (low viscosity) showed significantly different flow distance (0.88–3.43 cm) and flow rate (0.15–0.57 mm/s) compared to those of low flowability (high viscosity) products (0.63–1.12 cm; 0.11–0.19 mm/s) (p<0.05). The correlation (R) between the filler content and flow distance was negligibly low (R<0.23 in any cases).

Table 3 shows the polymerization shrinkage of the tested specimens. The products of high flowability showed mostly greater polymerization shrinkage (for DE: 30.3–36.2 µm; for OP: 29.0–36.8 µm) than that of low flowability products (for DE: 21.8–35.1 µm; for OP: 22.9–35.4 µm). The flow distance linearly correlated with polymerization shrinkage at R=0.45 and 0.60 for DE and OP, respectively. The measured polymerization shrinkage was significantly different for different flowabilities regardless of LCU (p<0.05).
Fig. 1 Specimens flowed on the plate for 1 min.

Table 2 Flow distance and flow rate of the tested specimens

| Flow distance (cm) | Flow rate (mm/s) |
|--------------------|------------------|
| ALb                | 0.88±0.14        |
| BH                 | 2.77±0.06        |
| EH                 | 3.33±0.31        |
| GF                 | 1.21±0.11        |
| PH                 | 3.43±0.30        |
| UF                 | 2.46±0.36        |
| AFb                | 0.86±0.10        |
| BLb                | 0.90±0.12        |
| EQb                | 0.87±0.02        |
| GUb                | 1.12±0.24        |
| PLb                | 0.63±0.05        |
| ULb                | 0.78±0.04        |

HF: High flowability; LF: Low flowability
* Statistically significant difference on flowability and resin products after t-test and ANOVA is shown by superscript letters. Same letters are not significantly different (p>0.05).

Table 3 Polymerization shrinkage (μm) of the tested specimens (n=5)

| LCU  | DE  | OP  |
|------|-----|-----|
| AL   | 34.5±2.2ab | 32.6±0.9ab |
| BH   | 36.2±2.0a  | 36.8±2.7a  |
| EH   | 33.5±1.9a  | 36.6±1.4a  |
| GF   | 30.3±0.3c  | 29.0±0.2d  |
| PH   | 30.5±1.5d  | 31.9±2.1a  |
| UF   | 31.7±1.2d  | 30.4±1.0d  |
| AF   | 35.1±1.9ab | 35.4±1.6ac |
| BL   | 31.2±0.6b  | 30.7±1.0b  |
| EQ   | 23.7±0.8e  | 23.2±0.6e  |
| GU   | 28.6±0.3ef | 28.4±1.3df |
| PL   | 21.8±1.5e  | 22.9±0.9f  |
| UL   | 26.1±1.0e  | 26.0±0.9ef |

HF: High flowability; LF: Low flowability
* Statistically significant difference on flowability, resin product, and LCU after t-test or ANOVA is shown by superscript letters or number. Same letters or number are not significantly different (p>0.05).

Table 4 Microhardness (Hv) of the tested specimens (n=12)

| DE   | Top  | Bottom |
|------|------|--------|
| AL   | 25.9±0.8  | 19.2±1.3a |
| BH   | 28.7±0.8b | 25.3±1.1b |
| EH   | 44.3±1.2  | 38.4±0.9f |
| GF   | 36.3±1.4c | 25.7±0.8b |
| PH   | 36.6±2.2e | 16.7±1.4f |
| UF   | 30.3±0.6e | 27.7±0.8b |
| AF   | 25.0±0.8  | 19.3±1.7a |
| BL   | 28.7±0.9b | 25.7±0.9b |
| EQ   | 48.8±0.9d | 36.2±2.1d |
| GU   | 47.5±1.7d | 40.4±1.4f |
| PL   | 9.6±0.7f  | 8.2±0.4f  |
| UL   | 14.6±1.1e | 18.9±0.9g |

| OP   | Top  | Bottom |
|------|------|--------|
| AL   | 24.0±0.4c | 20.0±1.3a |
| BH   | 27.7±0.8b | 24.2±0.6b |
| EH   | 44.6±0.8c | 37.4±1.9a |
| GF   | 38.2±1.1c | 24.7±1.2b |
| PH   | 36.7±1.2f | 15.8±0.8g |
| UF   | 29.6±1.8e | 26.2±0.6f |
| AF   | 25.1±0.7b | 22.1±0.6f |
| BL   | 28.1±1.1b | 24.5±0.9b |
| EQ   | 47.6±1.0d | 35.4±1.5e |
| GU   | 47.9±0.9f | 38.9±0.8g |
| PL   | 10.0±1.0f | 9.5±1.3i  |
| UL   | 16.2±1.0e | 18.1±0.8j |

* Statistically significant difference on flowability, resin product, and LCU after t-test or ANOVA is shown by superscript letters or number. Same letters or number are not significantly different (p>0.05).
**DISCUSSION**

The advantage of flowable composites over most resin composites is an agreeable adaptation to tooth cavity with easy handling due to the flowable nature. To endow flowable nature, composition of resin composites can be modified by lowering filler content or increasing diluents content. However, such modifications of filler and diluents contents can result in the change of general and mechanical properties of flowable composites. The present study tested how the difference of flowability, based on the manufacturers’ claim, affects various properties of flowable composites. The hypothesis tested has to be rejected because various properties (flow rate, polymerization shrinkage, microhardness, and water sorption and solubility) were not consistently different despite much difference of flowability.

Recently, flowable composites are mainly available with two types according to the manufacturers claim: high flowability (low viscosity) and low flowability (high viscosity). From the compositional aspect, flowability can be controlled by handling the filler content and diluents content. According to the disclosed data, AF, AL, BH, and BL are products of lower filler content (less than 60 wt%) compared to the other products (63–71 wt%), based on the nominal wt%. In the present study, the filler contents showed negligibly low correlation with the flow distance of the tested products. Low correlation may indicate the possibility of high dependence on the amount of each monomer and their combinations. Among the monomers contained in the tested products (basically, Bis-EMA, Bis-GMA, TEGDMA, and UDMA), TEGDMA showed the lowest viscosity (approximately 0.01 Pa•s) and greatest shrinkage due to low molecular weight (286 g/mol)\(^{20}\). Also, it showed the highest water sorption due to hydrophilic nature, however, solubility was the lowest among them. Generally, the more densely cross-link network forms, the more heterogeneously the structure becomes\(^{20,24,25}\). Since TEGDMA has higher flexibility than Bis-GMA, it can swell by water and fill up more water into the space within the heterogeneous network.

Each flowable composites of high flowability scored much higher flow distance and flow rate (0.86–3.43 cm; 0.15–0.57 mm/s) compared to their counter products (low flowability) (0.63–1.12 cm; 0.11–0.19 mm/s), so some products (BH, EH, PH, and UF) showed much distance...
difference (2.46–3.43 vs 0.63–0.90 cm) compared to their counter products (BL, EQ, PL, and UL). AL and GF showed low flow distance even though they were claimed to be of high flowability. Since products of the same manufacturer show similar filler content, the content of monomers may act crucial role in the resultant properties even though the exact content of each monomers is not fully disclosed.

Polymerization shrinkage is an inevitable process during the light curing due to the change of a governing molecular bonding from van der Walls to covalent. Shortening of molecular distance during the polymerization process is reflected as a form of shrinkage. The products of high flowability showed significantly higher polymerization shrinkage (29.0–36.8 μm vs 21.8–35.4 μm) than that of counter products despite their low difference in their filler content. EH-EQ and PH-PL products showed high polymerization shrinkage difference (15.1–36.6%), whereas GF-GU products showed low difference (0.3–5.6%). The highest shrinkage of BH would be due to the lowest filler content among the products. Another high shrinkage of EH would be related to high TEGDMA content despite its highest filler content. According to a linear fit, polymerization shrinkage was less correlated both with filler content and flow rate, that may be why some products (EH, PH, and UF) have high polymerization shrinkage even with their high filler contents.

Microhardness had a low (R<0.36 for the top and bottom surfaces) and not high (R<0.49) correlation with flow rate and filler content, respectively, of the products. Low microhardness is basically attributable to the low filler content. However, the inclusion of TEGDMA to the resultant microhardness values is not consistent. AL-AF and BH-BL products have much less microhardness difference compared to the rest products. In the case of EH-EQ and GF-GU products, high flowability products (EH and GF) showed lower microhardness compared to their counter products, whereas, PH-PL and UF-UL products were the counter case. Among the products, PH showed the greatest flow rate and PL showed the lowest microhardness even with their high filler content.

The highest water sorption in BH and BL is attributable to their lowest filler content (in other word, highest monomer content) among the products. The lowest microhardness and flow rate of PL with high filler content may indicate incomplete polymerization and less TEGDMA content, respectively. Also, exceptionally high solubility would be related with much release of uncured residual monomers. Low flow rate of GF and GU would be probably due to the high inclusion of UDMA in addition to their high filler content (UDMA has approximately 10 times greater viscosity than TEGDMA)20. High solubility of GF among the products can be related with the release of fluoride from LaF$_3$ (lantanoid fluoride). According to a simple calculation, LaF$_3$ solubility in pure water can reach to $4 \times 10^{-8}$ mol/L (approximately 9 ppm). Negative solubility (increase of the final specimen weight) in GU would be due to the formation of any new compound. Strontium can form strontium hydroxide when it reacts with water (Sr$+2$H$_2$O$\rightarrow$Sr(OH)$_2$+H$_2$), so such forming a new compound can increase the resultant weight.

**CONCLUSION**

Within the limitations of the present study, flow rate, polymerization shrinkage, and microhardness (on the top surface) of flowable composites, which were claimed to have different flowabilities (high or low) by the manufacturers, had a correlation with the flowability. However, as a degree of flowability, flow rate had low correlation with microhardness, polymerization shrinkage, and water sorption and solubility regardless of the light-curing unit tested.

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