ESTELITE UNIVERSAL FLOW

Technical Report

Ver. 2.2

Tokuyama Dental

Contents

1. Introduction

Tokuyama Dental has developed various light-curing dental restorative composite resins that take advantage of its proprietary Supra-Nano Spherical filler technology. Represented by Palfique Estelite® Paste, Estelite® Σ, and Palfique Estelite® LV, these products have acquired a reputation for outstanding aesthetics and gloss.

In 2005, Tokuyama Dental launched Estelite Flow Quick®, a new flowable composite resin, based on a new catalyst technology (RAP technology™) and a proprietary filler technology. This approach results in remarkably fast curing compared to conventional flowable resins (requiring approximately 1/3 the time). Due to RAP technology™, Estelite Flow Quick® features high conversion and leading levels of filler content (71 wt%) among flowable composite resins. It offers outstanding scientific and engineering properties not found with conventional flowable composite resins.

This material has been used frequently in clinical applications in recent years, not only for the clinical convenience of the flowable resin, but also thanks to improved reliability (e.g., strength) and diverse range of flowability.

Tokuyama Dental has developed Estelite Universal Flow® through the application of the Supra-Nano Spherical filler, RAP technology™, and a new composite filler. The technological backgrounds, features, and material properties of this new composite are described below.

2. Materials

2.1 Components

- Bis-GMA, Bis-MPEPP, TEGDMA, UDMA
- Supra-Nano Spherical filler (200nm spherical SiO_2-ZrO_2)
- Composite Filler (include 200nm spherical SiO_2-ZrO_2)
- Filler loading

  Super Low: 70 wt% (56 vol%)

  Medium: 71 wt% (57 vol%)

  High: 69 wt% (55 vol%)

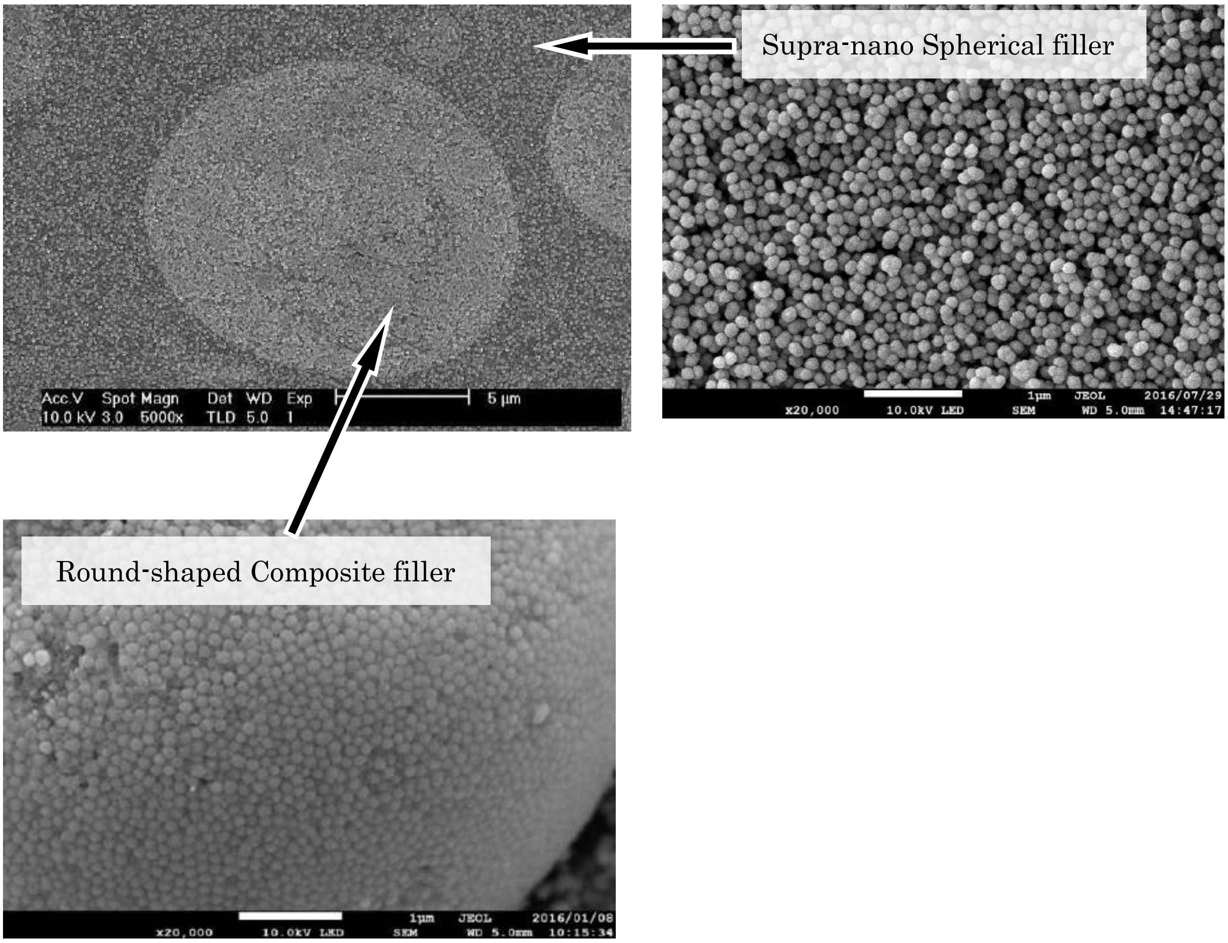


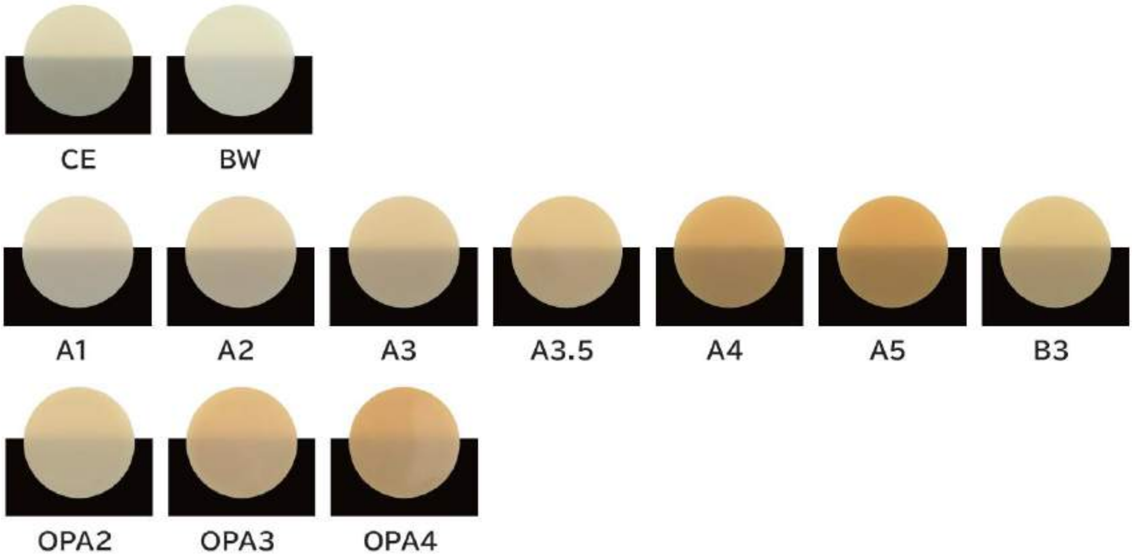
Fig.1 SEM images of Estelite Universal Flow®

2.2 Flowability

The flowability of Estelite Universal Flow® can be chosen from the following three types, according to the patient and purpose of use: Super Low, Medium and High.

2.3 Shades

Estelite Universal Flow® is available in six shades for Super Low (A1, A2, A3, A3.5, A4, A5), in twelve shades for Medium (CE, BW, A1, A2, A3, A3.5, A4, A5, B3, OPA2, OPA3, OPA4), and in seven shades for High (A1, A2, A3, A3.5, OPA2, OPA3, OPA4). The curing time is 10 seconds of light exposure for all shades (800 mW/cm^2 or greater).



2.4 Concept and features

Estelite Universal Flow® is the universal flowable composite for all cavity classes with excellent esthetic properties, physical-mechanical properties and handling properties with selectable three-flowability. The main features are showen below:

1. Excellent esthetic properties
   - ✓ wide color matching and blending
   - ✓ high polishability
   - ✓ high gloss retention
2. Excellent physical- mechanical properties
   - ✓ high strength
   - ✓ low wear resistance
   - ✓ minimal polymerization shrinkage
   - ✓ staining resistance
3. Excellent handling properties
   - ✓ three flowability options and the good handling
   - ✓ short curing time and enough working time
   - ✓ minimal shade shift through curing

2.5 Indications

- Direct anterior and posterior restorations
- Cavity base or liner
- Blocking out cavity undercuts before fabrication indirect restorations
- Repair of porcelain/composite

3. Technical background

3.1 Radical Amplified Photo-Polymerization Initiator (RAP technology™)

3.1.1 Mechanism

The catalyst technology adopted for Estelite Universal Flow® is the Radical Amplified Photo-polymerization initiator (RAP technology™) used in Estelite Σ Quick®. As a major feature, the initiator balances the high polymerization activity needed to cure the resin with short exposure times (1/3 of that required by conventional products) and stability in ambient lighting. These two traits are often regarded as mutually conflicting, since shorter curing times tend to reduce stability. However, this unique catalyst technology achieves a balance of these two factors. Fig.2 is a schematic diagram of RAP technology™.

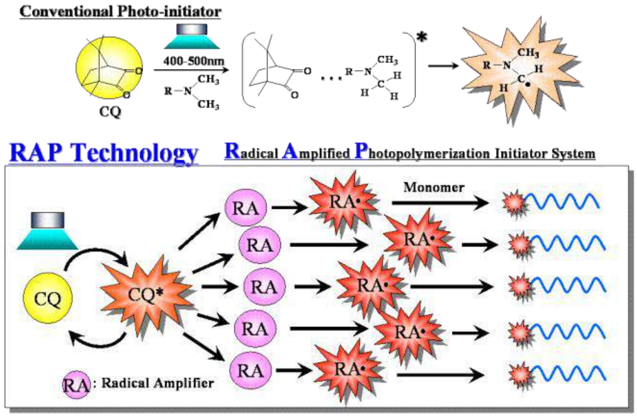


Fig.2 Radical amplified polymerization initiator system

Conventional photo-polymerization initiators consist of camphorquinone (hereafter abbreviated CQ) and amines. The mechanism of action involves the excitation of CQ by irradiation, followed by the abstraction of hydrogen in the alpha-position of amine by the excited CQ, producing amine-derived radicals. The amine-derived radicals function as the polymerization initiator and react with monomers to generate polymers, ultimately producing the curing effect. In this catalyst system, CQ is consumed as it changes to CQ-H in polymerization initiator generation. Unlike CQ, CQ-H is not excited by light. This means a single molecule of CQ can only produce a single polymerization initiator molecule.

With the radical amplified photo-polymerization initiator, the initial stage of CQ excitation by light is the same as in conventional systems. However, energy is transferred to the radical amplifier (hereafter abbreviated RA); the RA is subsequently excited, and then allowed to decompose to produce RA-derived radicals. Theses radicals act as the polymerization initiator and react with monomers to generate polymers, producing the curing effect. After transferring energy to RA, the excited CQ returns to the ground state and is once again excited by irradiation and contributes to the reaction for polymerization initiator species generation. In other words, with RAP technology™, CQ is recycled within the polymerization initiator generation reaction, and a single CQ molecule can produce multiple initiator radicals. Thus, in addition to being highly active, RAP initiators can be used with smaller CQ volumes than conventional catalysts and improve stability in ambient lighting, including dental and fluorescent lights. The present initiator system is also free of chemical reactions between two molecule species, such as hydrogen abstraction in conventional systems, allowing shorter times from the photo-excitation of CQ to initiator radical generation.

To confirm that RAP technology™ increases polymerization rates, we compared the amount of residual monomers after a light cure for two different flowable composite resins: EFQ-RAP, which contains a radical amplified photo-polymerization initiator, and EFQ-CQ, which contains a conventional photo-polymerization initiator composed of CQ and amines. Fig.3, 4 shows the results. Fig.3 indicates that the radical amplified photo-polymerization initiator significantly reduces residual monomers compared to the conventional CQ-amine photo-polymerization initiator for both 10-second and 30-second exposures. This holds true even when comparing EFQ-RAP after 10-seconds of exposure to EFQ-CQ after 30-seconds of exposure. We also found that more radicals are generated in the RAP initiator system than the conventional CQ/amine system (radical concentrations greater by a factor of approximately 2.5) according to Fig.4. These results support the mechanism of action shown in Fig. 2.
RAP technology facilitates a control of polymerization rate. Polymerization rate is slow and material is stable under small light intensity (ambient light such as a dental light), however, polymerization rate becomes quick under large light intensity (light irradiation unit) (Fig.5).

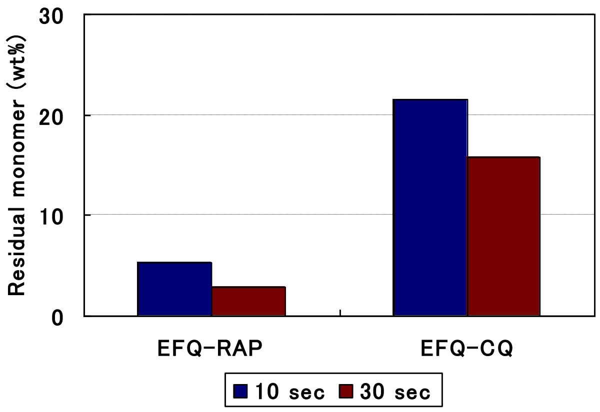


Fig.3 Residual monomer(wt%)

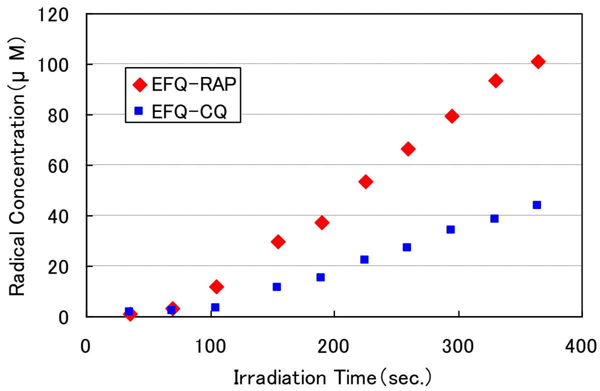


Fig.4 Change of radical concentration

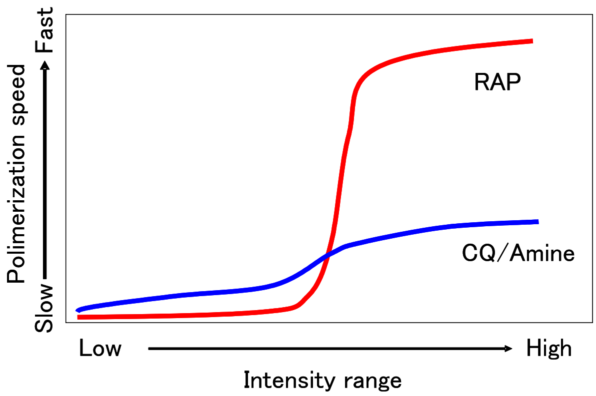


Fig.5 Correlation between intensity range and polymerization speed

3.2 Supra-Nano Spherical Filler Technology

Tokuyama Dental synthesizes mono-dispersing Supra-Nano Spherical fillers by a special technique called the sol-gel method. Unlike the conventional filler manufacturing method, which involves crushing glass materials, fillers with the present method are produced by creating filler cores in organic solvent and allowing the filler to grow gradually from the cores. This method makes it possible to produce uniform, spherical fillers (Fig.6)。

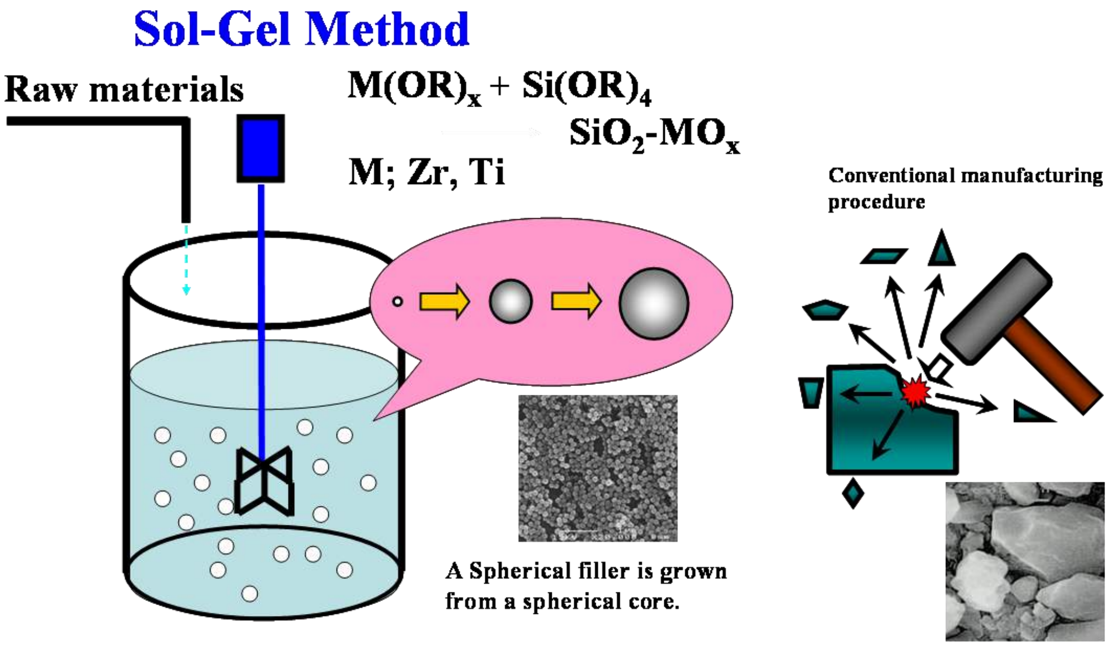


Fig.6 Summary of sol-gel method

A major feature of the sol-gel method is that it allows the filler size to be controlled by adjusting reaction times. In composite resins, filler size significantly affects the physical characteristics of the cured body and its esthetic aspects. Smaller filler sizes produce a superior surface glossiness, but make it difficult to increase filler content, leading to problems such as increased polymerization shrinkage and poor physical characteristics such as reduced flexural strength.

Fig.7 gives the correlation between filler particle size and filler content and compressive strength. Fig.8 gives the correlation between filler particle size and surface roughness and hardness. From Fig.7, we see that filler content begins to fall significantly below 100 nm, but is nearly constant at sizes above that. In addition, we observe maximum compressive strength at particles size ranging from 100 to 500 nm. From Fig.8, we see that surface roughness decreases with particle sizes down to approx. 500 nm but remains constant at sizes below that. Surface hardness attains the highest value at particle sizes ranging from 200 to 300nm. Based on the above results, we conclude that the best balance between esthetics and physical characteristics can be achieved by using Supra-Nano sized particles (200nm).[1]。

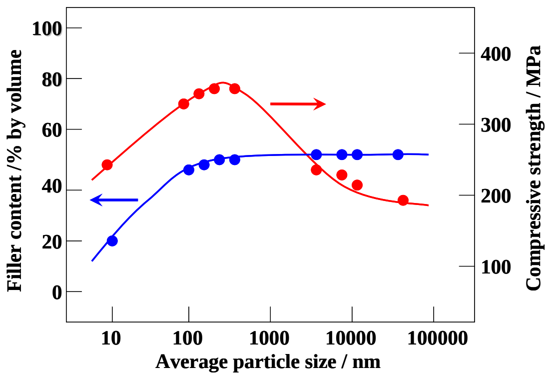


Fig.7 Correlation between particle size, filler content and compressive strength

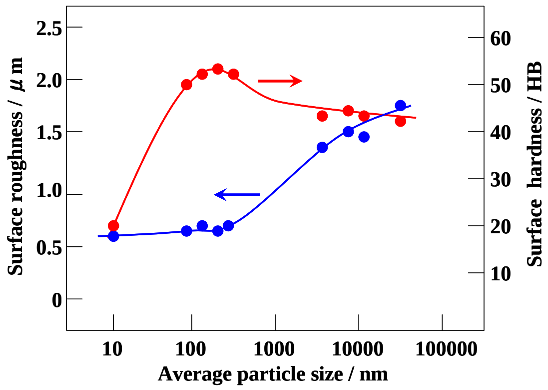


Fig.8 Correlation between particle size, surface roughness and surface hardness

For Estelite Universal Flow®, we use Supra-Nano Spherical fillers made of silica-zirconia produced by the sol-gel method, with particle sizes of 200 nm (Fig.9)。

Another major feature of the sol-gel method is that the refractive index of the filler can be controlled by changing the type and fraction of the additive.

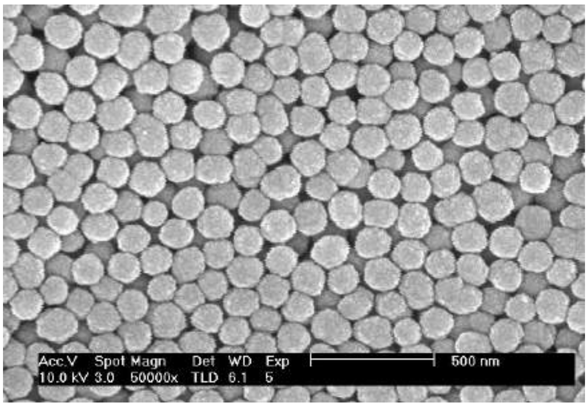


Fig.9 Supra-nano spherical filler

Composite resins tend to show a strong relationship between the filler refractive index and that of the matrix organic resin. To reproduce the semi-translucent quality of natural teeth using composite resins, we must control the difference between the refractive indices of the filler and the organic resin. Composite resins

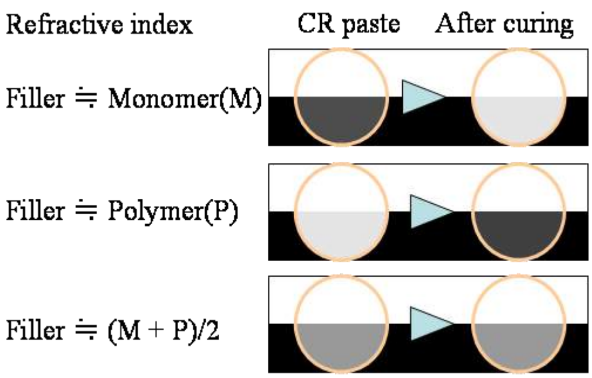
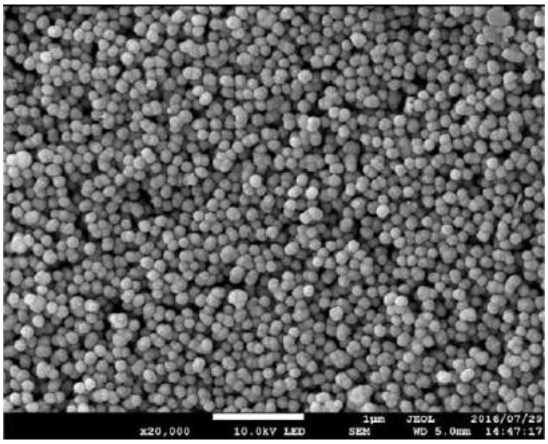


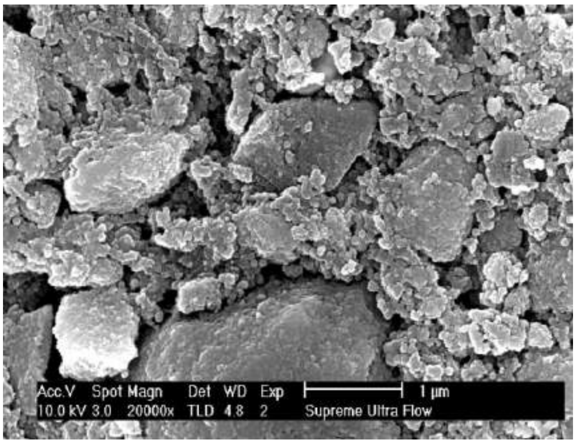
Fig.10 Relationship with refractive index and translucency

consist of fillers and organic resins containing catalysts. When the refractive indices of both materials are equal, the composite resin is highly translucent; when they differ significantly, the resin is opaque. The refractive index of resins tends to change from before to after polymerization; the refractive index of the cured resin (polymer) tends to be higher than that of the resin (monomer) before curing. To suppress changes in translucency from before to after polymerization, we must maintain the same difference between the refractive indices of the resin and filler from before to after polymerization. This means maintaining the refractive index of the filler close to the intermediate value of the refractive indices of the monomer and the polymer. In Estelite Universal Flow®, the silica/ zirconia composition is adjusted to prepare fillers with optimal refractive indices.

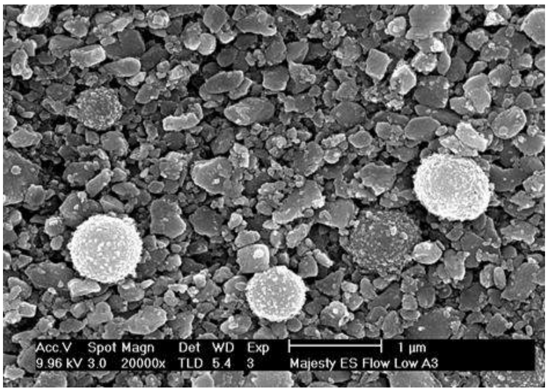
Below are SEM images of fillers used in Estelite Universal Flow® and in flowable composite resins from other manufacturers.



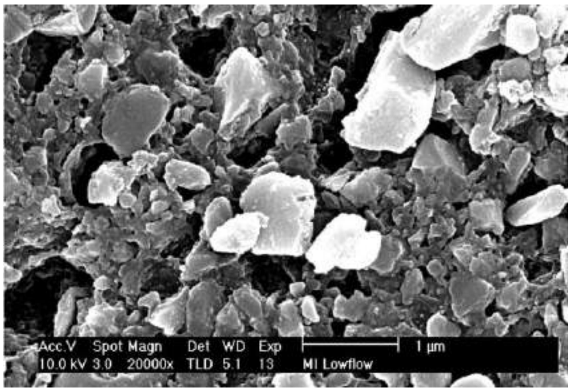
Estelite Universal Flow®



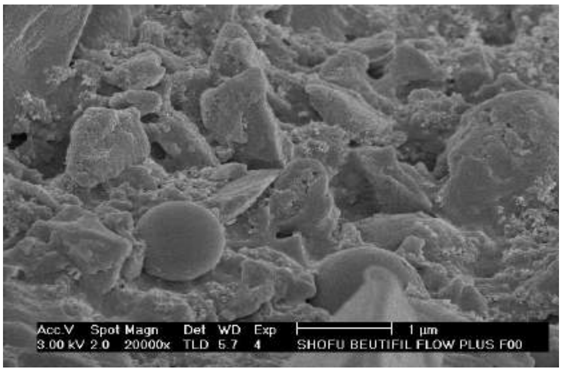
Filtek Supreme Ultra Flowable



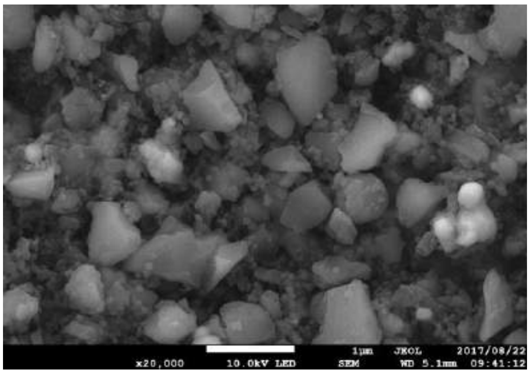
Clearfil Majesty ES Flow



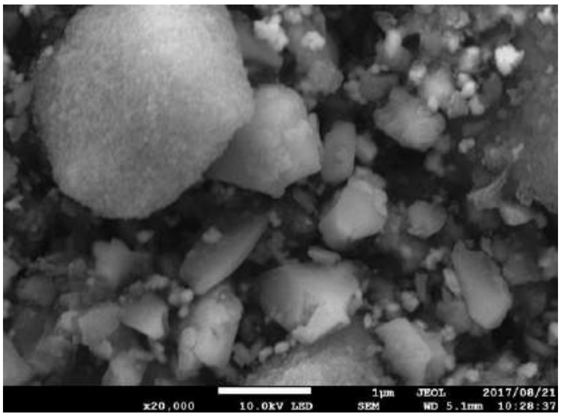
G-aenial Universal Flo



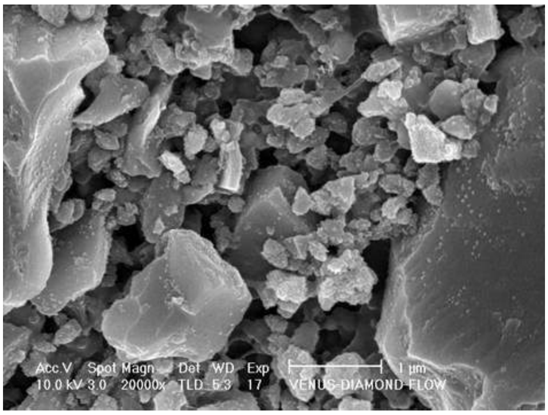
Beautifil Flow Plus F00



Herculite Ultra Flow



Tetric Evo Flow



Venus Diamond Flow

3.3 Round shaped Composite Filler Technology

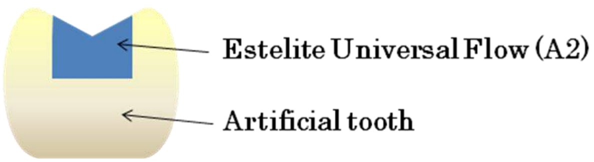
As described previously, the controlled particle size of Supra-Nano Spherical filler lends aesthetic properties to composite resins, including polishability, gloss retention, and wear resistance. On the other hand, composite pastes consist of a single particle size filler tend to have low flowability, which makes it difficult to increase filler content while maintaining satisfactory handling. Tokuyama Dental has developed a proprietary composite filler containing Supra-Nano Spherical filler and combined it with Supra-Nano Spherical filler to simultaneously achieve esthetic properties, handling, and mechanical properties of a composite resin.

A newly developed organic-inorganic composite filler "Round-shaped Composite Filler" was adopted for Estelite Universal Flow®. This composite filler is designed to be round-shaped having a characteristic texture on the filler surface and controlled which the average particle size is approximately 10μm. It can be expected that the filler having the characteristic structure leads to a ball-bearing effect. The adoption of the novel composite filler to flowable composite can be contributes to the various properties such as gloss retention, high strength, excellent wear resistance, and low polymerization shrinkage, comparable to universal composites without sacrificing any other properties.

4. Material properties

4.1 Color matching and blending

Estelite Universal Flow® is basically designed to make it possible that most cases can be restored by limited mono shade thorough its excellent shade matching/ color blending effect. Fig.11 shows the color compatibility of Estelite Universal Flow® A2 shade when it is filled in each artificial tooth (B1, A1, B2, A2, C1, A3) after creating a cavity 4 mm in diameter and 2 mm deep, and Fig.12 shows a table of color compatibility for each shade of natural teeth. Estelite Universal Flow® provides a strong blending effect, which allows it to fit a wide range of color tones of natural teeth with a single shade and to be natural margin.



- Each cavity on artificial tooth (B1, A1, B2, A2, C1, A3) is filled with Estelite Universal Flow (A2).
- Cavity : Φ = 4 mm, depth = 2 mm

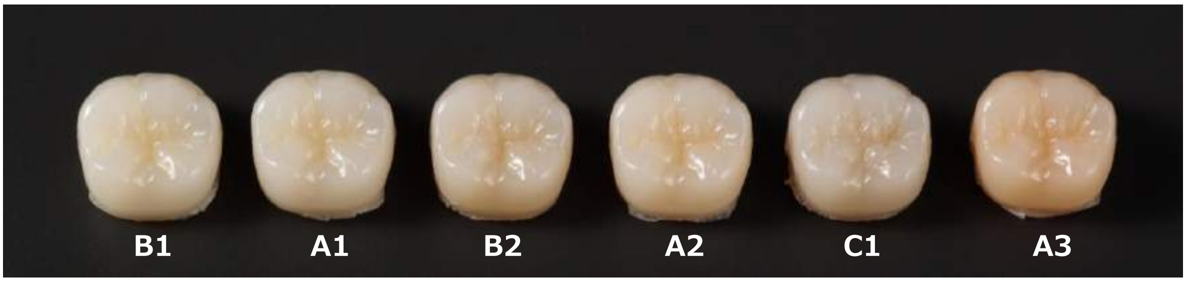


Fig.11 Shade matching test using artificial teeth

	Whiter	B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3.5	B4	C3	A4	C4	Darker
A1	E	E	E	E	E	E	E	G	G	V	V	G	—	—	—	—	—	—
A2	G	E	E	E	E	E	E	V	G	E	E	V	G	G	G	—	—	—
A3	—	V	V	V	E	E	V	E	V	E	E	E	E	E	V	V	G	—
A3.5	—	—	—	G	—	G	—	V	V	E	E	E	E	E	E	E	E	V
A4	—	—	—	—	—	—	—	—	G	G	V	E	E	E	E	E	E	E
A5	—	—	—	—	—	—	—	—	—	G	G	V	E	E	G	E	E	E
B3	—	G	G	E	V	V	V	V	E	E	E	E	E	E	V	G	—	—
BW	E	E	E	V	—	—	—	—	—	—	—	—	—	—	—	—	—	—
OPA2	G	E	E	E	—	E	V	—	—	G	—	—	—	—	—	—	—	—
OPA3	—	G	G	—	—	E	—	G	G	E	V	E	E	V	G	—	—	—
OPA4	—	—	—	—	—	—	—	—	V	V	V	E	E	E	V	E	G	—
CE	transparent area (e.g. incisal edge)																	

E = Excellent, V = Very Good, G = Good, — = Poor

Fig.12 Shade matching chart of Estelite Universal Flow®

4.2 Polishability

Fig.13 shows surface gloss after each surface of cured composite is polished with #1500 sandpaper, followed by Sof-Lex™ superfine (3M-ESPE) for 60 seconds. Fig.14, 15 shows the relationship between polishing time and surface gloss. The results show that Estelite Flow Quick®, Estelite Universal Flow® produces extremely high gloss in short polishing sessions.

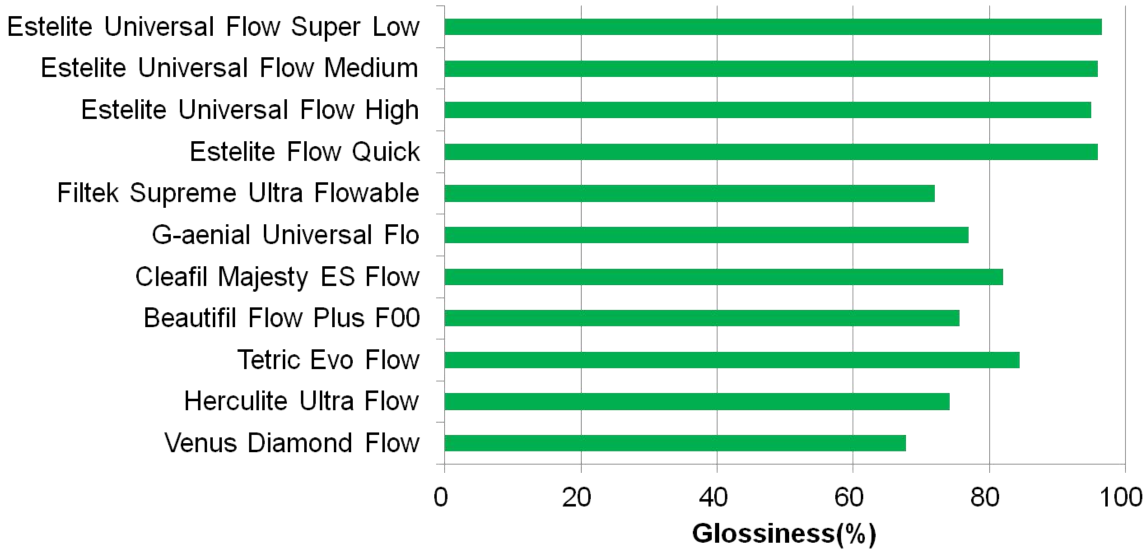


Fig.13 Surface Glossiness

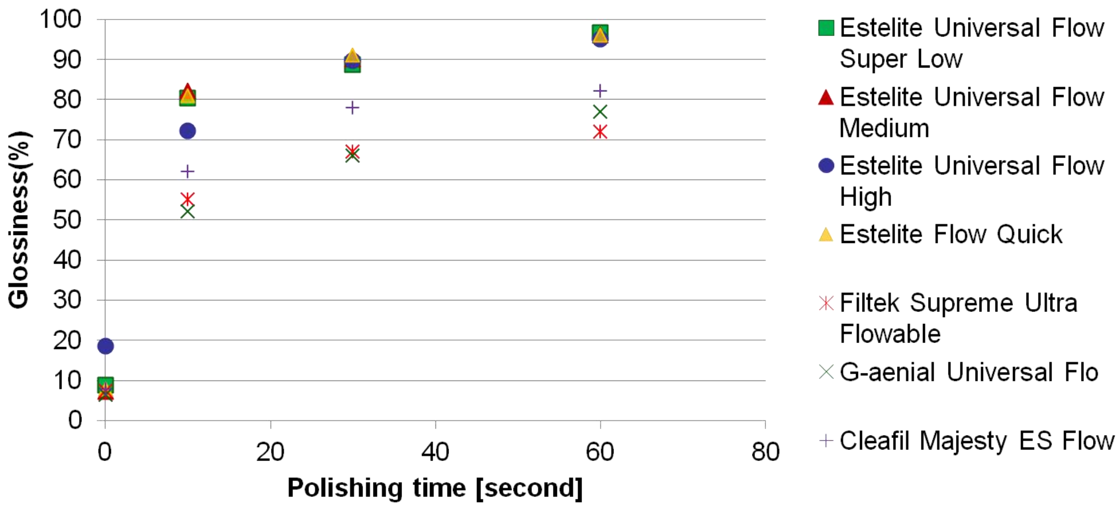


Fig.14 Relationship of glossiness to polishing time

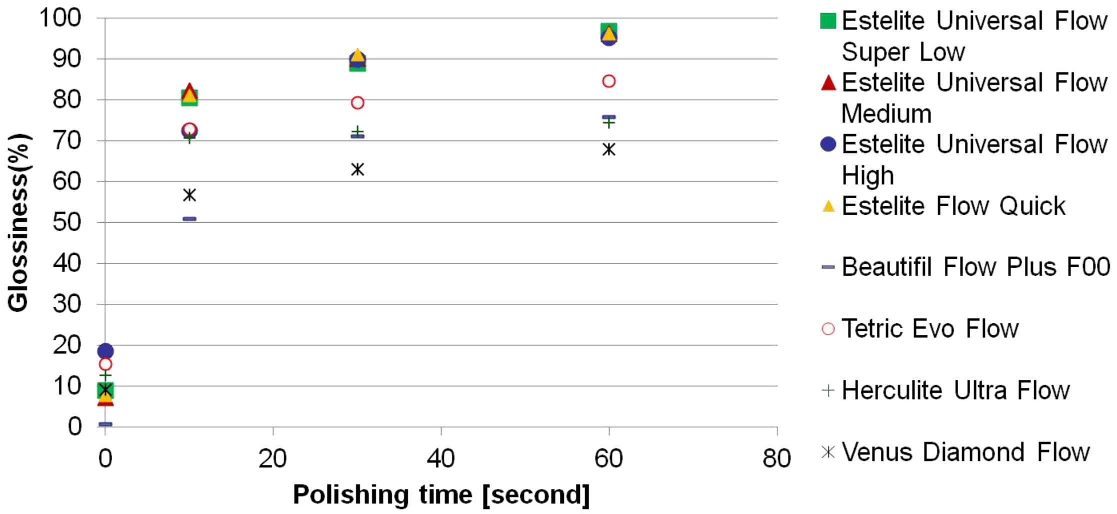


Fig.15 Relationship of glossiness to polishing time

4.3 Gloss retention

In addition to exhibiting extremely high gloss with relatively short polishing, Estelite Universal Flow® features a remarkably persistent gloss.

Fig.16 shows the surface glossiness of cured resin after 0, 3000, 5000, 10000 times thermal cycle test (5℃/ 55℃). This result shows that Estelite Universal Flow® keeps its surface smoothness, resulting in glossing over time.

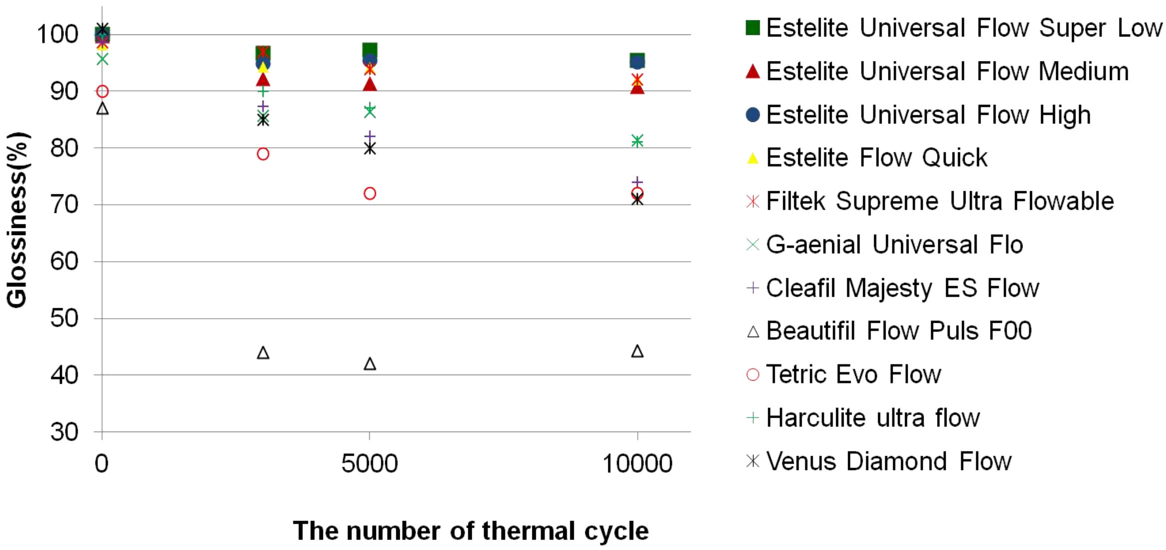


Fig.16 Gloss retention

4.4 Strength

Fig.17 presents the flexural strength and Fig.18 presents the compressive strength of Estelite Universal Flow® and other commercially available flowable composite resins.

The flexural strength and the compressive strength of Estelite Universal Flow® are high class among commercially available flowable composite resins and comparable to recent flowable composites claiming universal-use such as G-aenial universal Flo, Beautifil flow Plus and Cleafil Majesty ES flow.

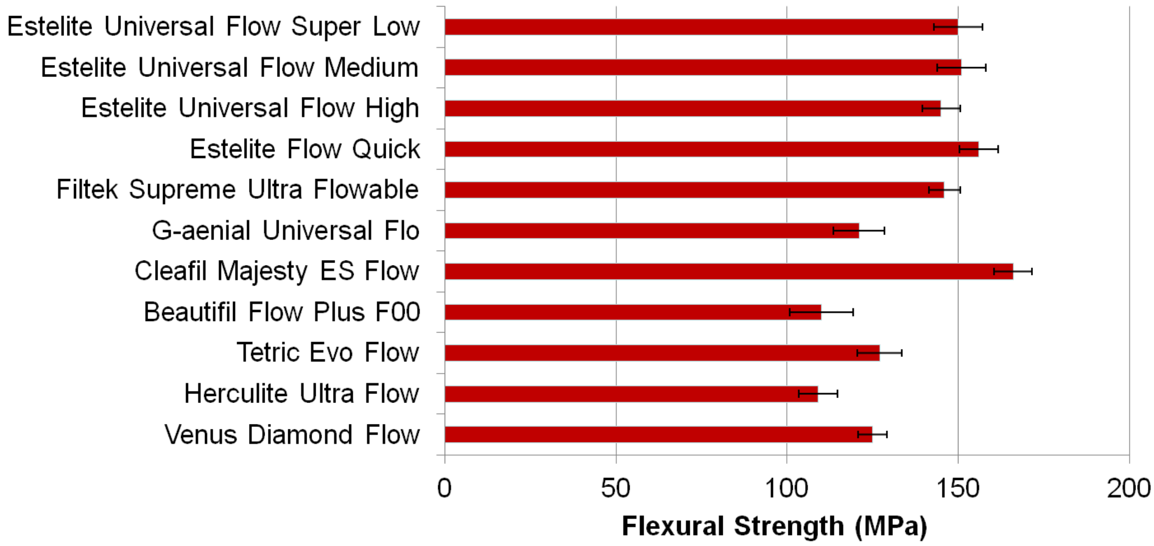


Fig.17 Flexural Strength (MPa)

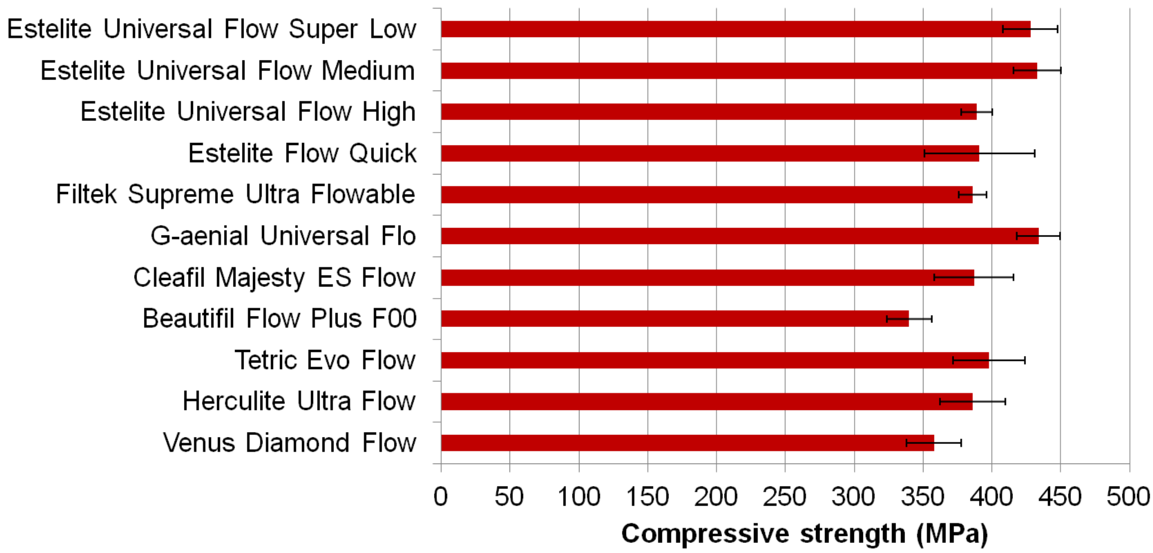


Fig.18 Compressive Strength (MPa)

The flexural and compressive strength of Estelite Universal Flow® compared to commercially available universal (sculpt-able) composites are shown in Fig.19 and Fig.20. The flexural strength and compressive strength of Estelite Universal Flow® are equal to or superior to those of the leading universal composites including hybrid composites.

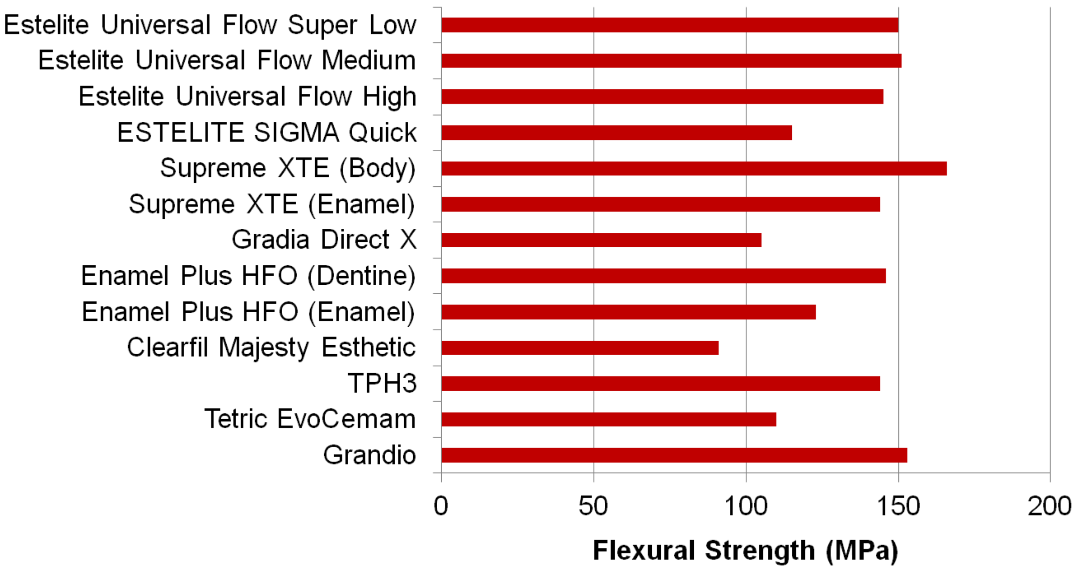


Fig.19 Flexural Strength (MPa) compared with universal composites

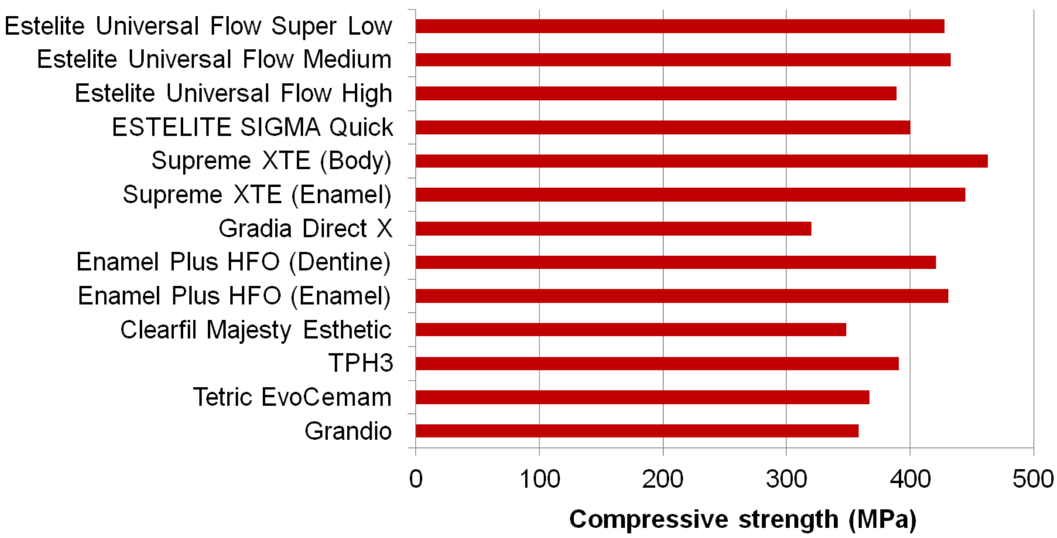


Fig.20 Compressive Strength (MPa) compared with universal composites

4.5 Wear characteristics

Wear resistance of the composite resin against a human tooth was examined using the method shown in Fig. 21. The comparison results are shown in Fig. 22. The results shows that Estelite Universal Flow® exhibits an excellent balance between volume loss of the composite resin and wear of the human tooth, which leads to the conclusion that Estelite Universal Flow® is a composite resin that is less likely to abrade opposing teeth, while not easily becoming abraded, as in the case of Estelite Flow Quick®.

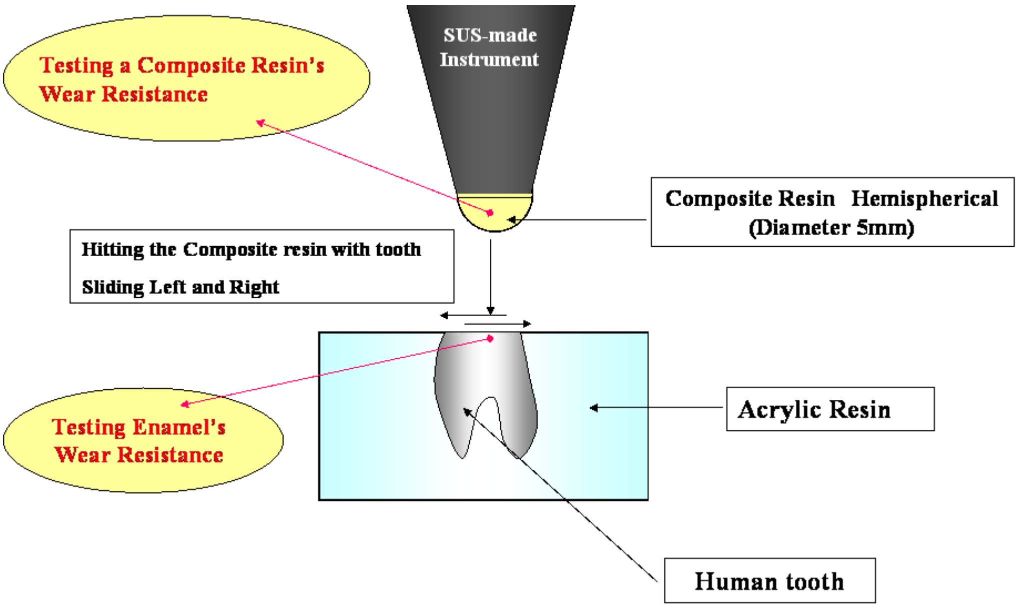
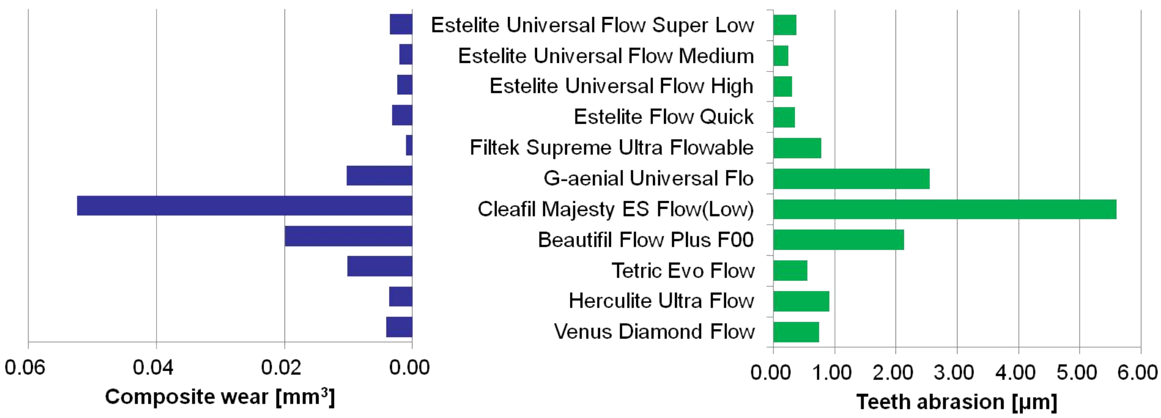


Fig.21 Method of wear resistance



Composite wear
(Decrease of composite volume)

Opposing tooth abrasion
(Abrasion depth of opposing teeth)

Fig.22 Wear characteristics

The wear resistance of Estelite Universal Flow® compared to commercially available universal (sculpt-able) composites is shown in Fig.23. The composite wear of Estelite Universal Flow® is equal to or superior to those of the leading universal composites.

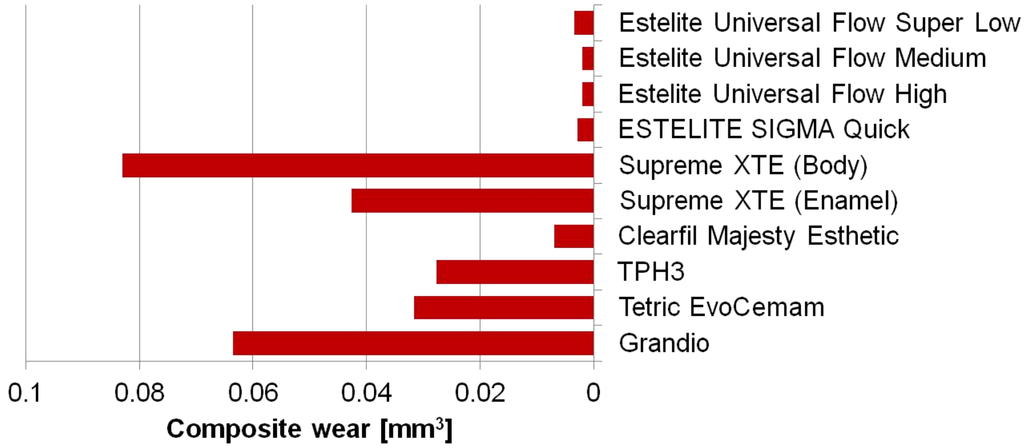


Fig.23 Wear characteristics compared with universal composites

4.6 Polymerization shrinkage

We measured polymerization shrinkage by our original method. Fig.24 is a schematic diagram of the measurement method. This method can measure shrinkage in the cavity floor (interface between the composite resin and plunger in Fig.24) when the composite resin is placed into a cavity and exposed to light in a clinical procedure. This permits evaluation of shrinkage under conditions closer to those encountered in actual clinical settings.

Fig.25 shows the polymerization shrinkage (%linear) of Estelite Universal Flow® and other commercially available flowable composite resins. The figure indicates shrinkage 3 minutes after the start of light exposure.

The polymerization shrinkage (%linear) of Estelite Universal Flow® (Super Low, Medium and High) is 2.3%. This is the minimum level among commercially available flowable composite resins. This result is due to the high filler volume content made possible by the combination of Supra-Nano Spherical filler and newly developed composite filler.

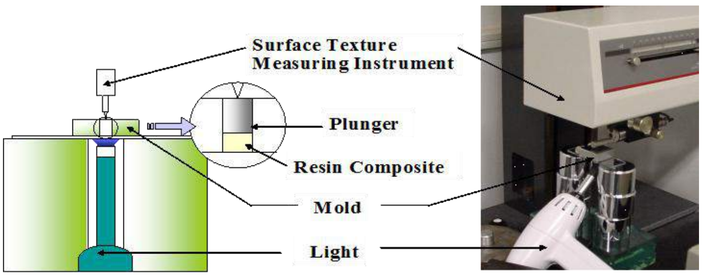


Fig.24 Method of polymerization shrinkage

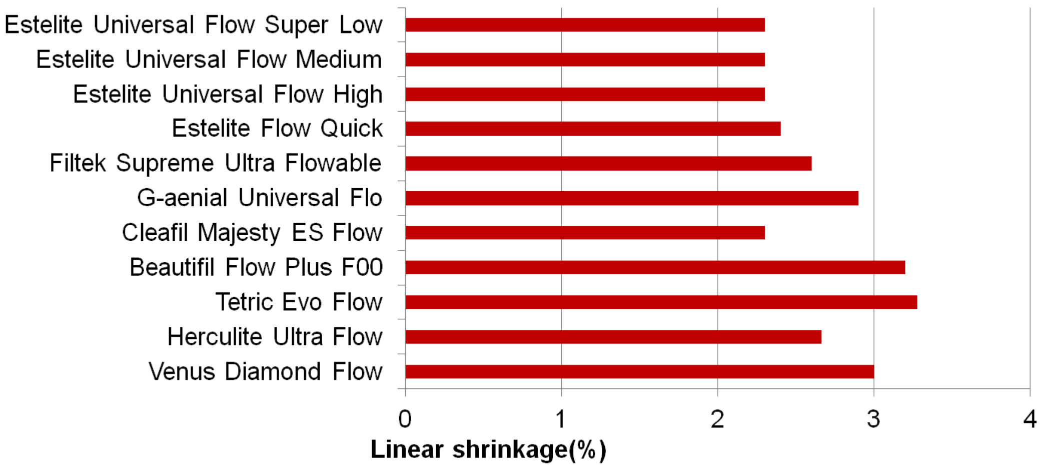


Fig.25 Polymerization shrinkage (%linear)

4.7 Staining resistance

A composite resin used in an oral cavity degrades over time due to the effects of various foods and drinks. If such changes are greater than those of the dentition, the composite resins will be judged as lacking aesthetics when evaluated visually. For this, we examined the degree of staining by coffee (immersed for 24 hours at 80°C). The results are shown in Fig. 26.

The extent of staining for Estelite Universal Flow® after soaking in coffee was relatively low among commercially available flowable composite resins. Estelite Universal Flow® can retain its color at the time of restoration over a long term.

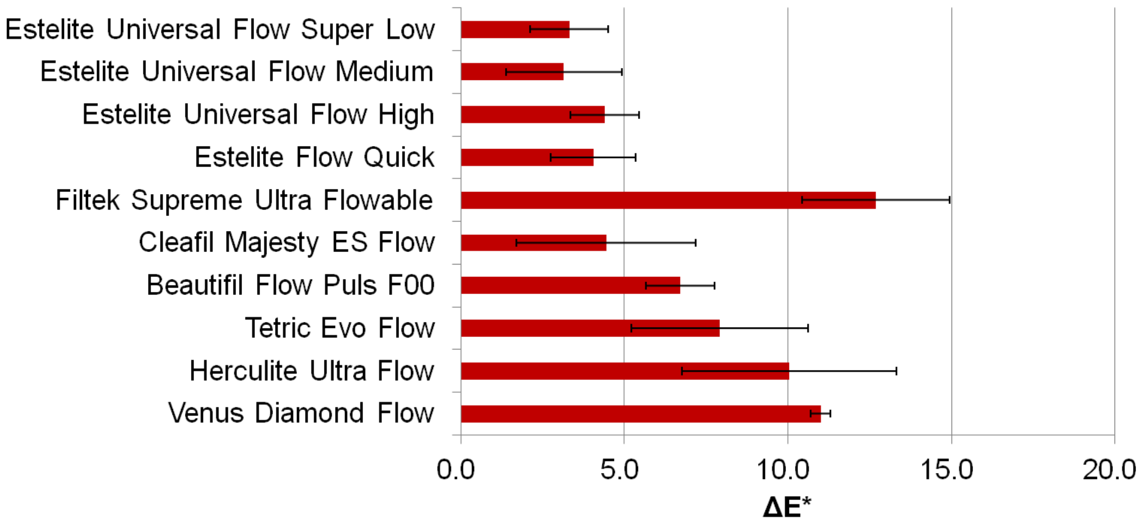


Fig.26 Color Stability (ΔE*)

The staining resistance of Estelite Universal Flow® compared to commercially available universal (sculpt-able) composites is shown in Fig.27. The color stability of Estelite Universal Flow® is equal to or superior to those of the leading universal composites.

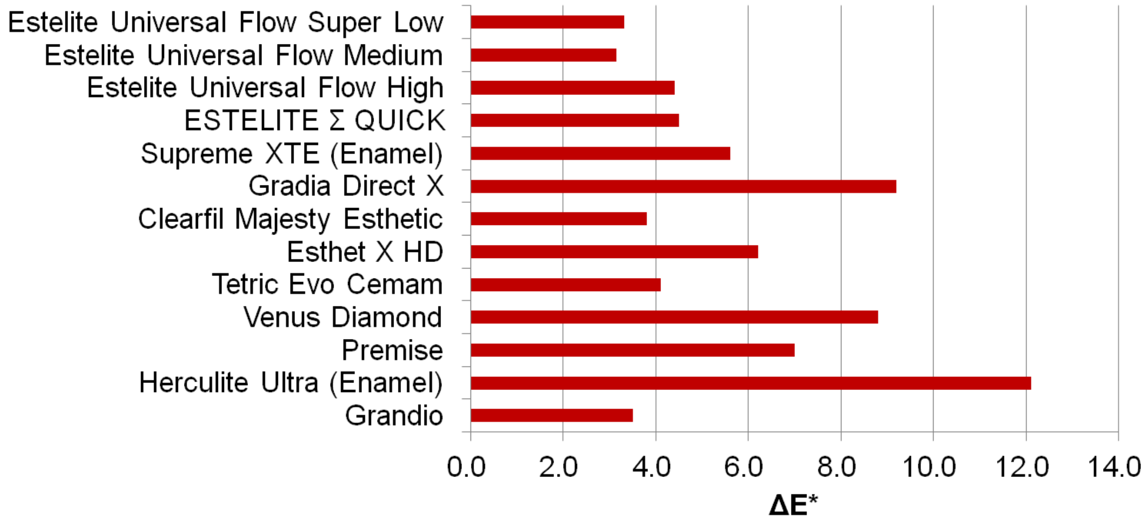


Fig.27 Color Stability (ΔE*) compared with universal composites

4.8 Flowability options and the handling

Estelite Universal Flow® offers three flowability options, optimized for all cavity classes, including Class I, II, III, IV, V, incisal margin, and cavity liner. The best flowability for a patient and a case can be chosen. “Super Low” has low flowability, non-slumping, non-running and precision stacking properties, especially suitable for class III, IV and occlusal restoration. “Medium” has medium flowability, high versatility, less slumping and less running properties, suitable for all restoration. “High” has high flowability, easy placement property, especially suitable for small cavities and serving as a cavity liner. All flowability options have common handling characteristics of less flow from nozzle-tip after dispensing, low stickiness and easy extrusion (Fig. 28).

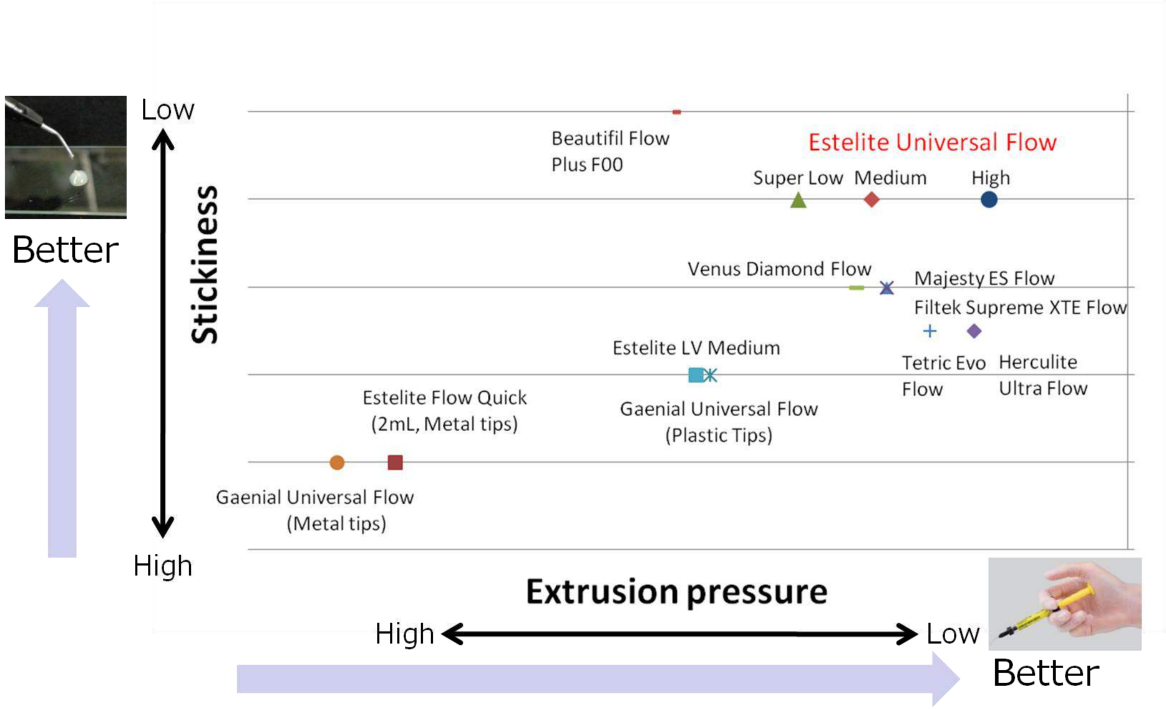


Fig. 28 Comparison of handling properties

4.9 Curing time and stability in ambient light

In the past, high polymerization activity with short exposures could only be achieved by increasing the amount of photo-polymerization initiator used. However, increasing the amount of the catalyst decreases the stability of the resin in ambient light. Additionally, the viscosity of the paste may increase during the filling step in clinical services, making the resin impossible to fill and requiring a second filling attempt. In addition, increasing the amount of catalyst can also exacerbate changes in color before and after polymerization. While increasing the amount of photo-polymerization initiator is believed to result in various undesirable effects, RAP technology™ can provide both polymerization activity and stability in ambient light, as described in detail in 3.1. Fig.29 compares light curing time and Fig.30 compares stability under ambient light (10,000 lx of dental light) measured in accordance with ISO 4049 between Estelite Universal Flow® and other commercially available flowable composite resins.

As shown in Fig.29 and Fig.30, Estelite Universal Flow® offers good stability in ambient light compared with products from other manufacturers, in spite of curing in less exposure time. This gives clinicians more time to perform filling and other steps.

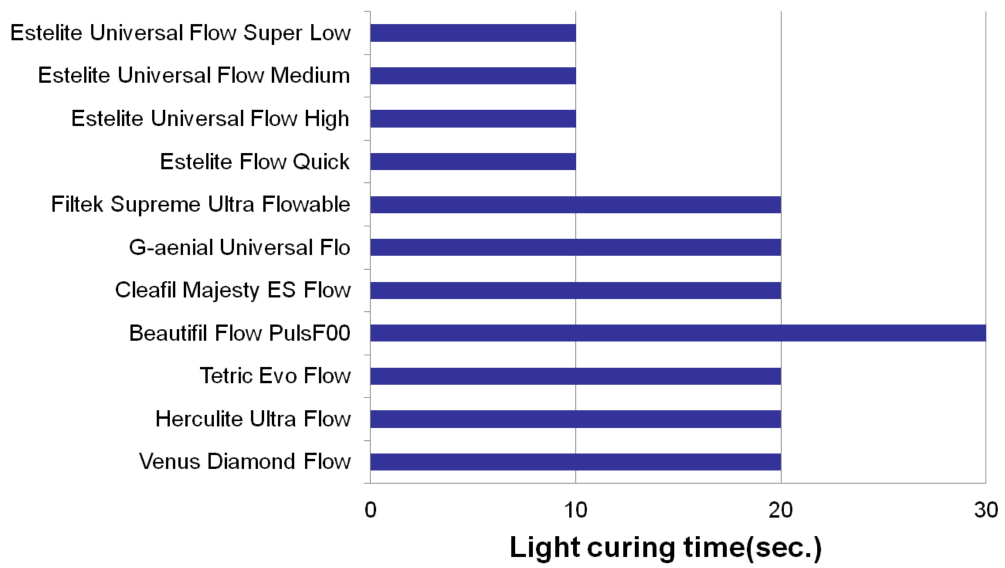


Fig.29 Light curing time according to manufacturers' recommendation

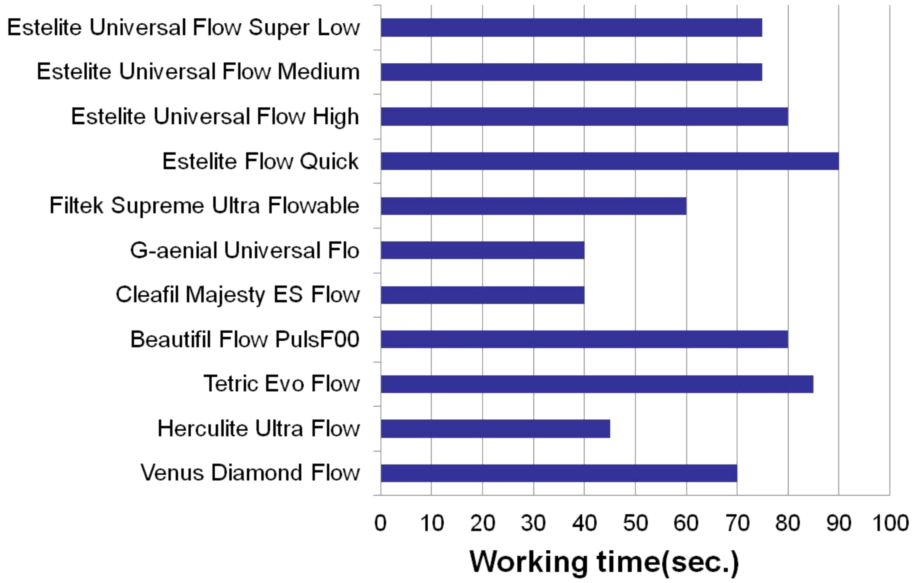


Fig.30 Working time under ambient light

4.10 Color and translucency before and after curing

Estelite Universal Flow® features relatively low changes in color and translucency before and after polymerization, permitting rough color matching before polymerization. Fig. 31 shows the changes in color and translucency for Estelite Universal Flow® and other commercially available flowable composite resins. As indicated in the figures, Estelite Universal Flow® offers low changes in both color and translucency, making shade-taking for Estelite Universal Flow® especially easy. Estelite Universal Flow® can reduce failures caused by colors that diverge significantly after curing.

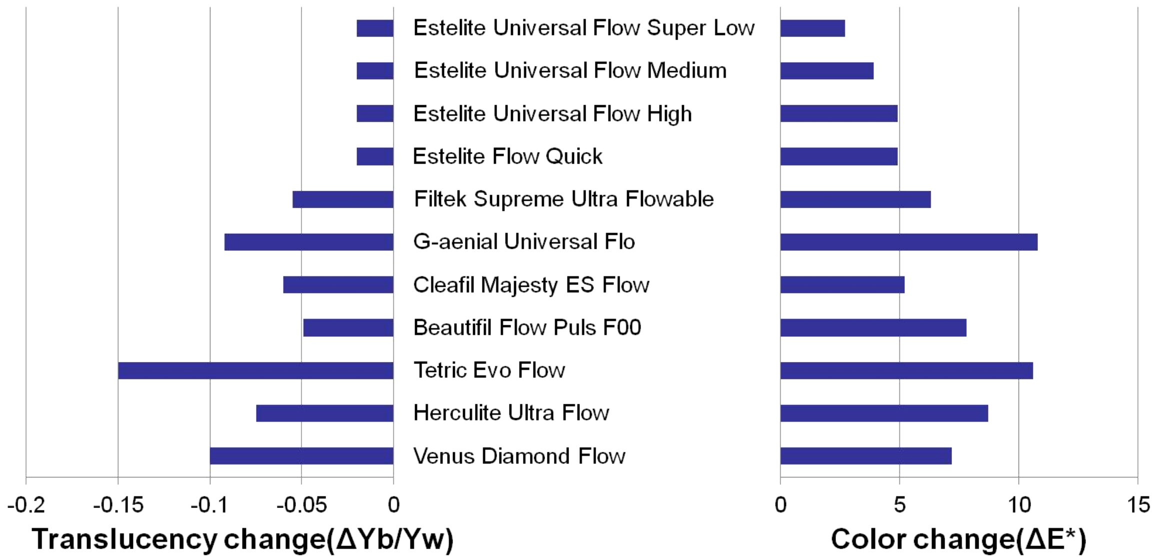


Fig.31 Change of color and translucency before and after polymerization

4.11 Radiopacity

Radiopacity is determined by the composition of the inorganic filler and its filler content. The radiopacity of a resin is higher if the composition of the resin includes larger amount of elements with high atomic numbers at higher filler content. However, a filler containing large amounts of elements with high atomic numbers is associated with large refractive indices.

Fig.32 shows the radiopacity of commercially available flowable composite resins.

The radiopacity of Estelite Universal Flow® is of average level, but sufficient for prognosis observations.

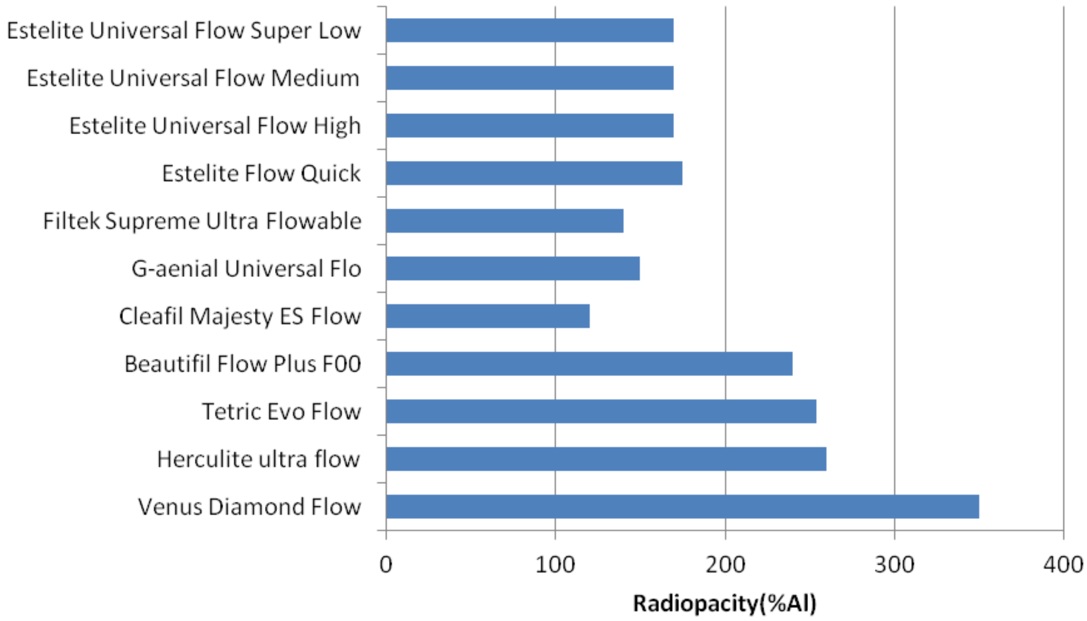


Fig.32 Radiopacity

5. Summary

Estelite Universal Flow® is the universal flowable composite resin having various features of excellent esthetic, physical-mechanical and handling properties as summarized below:

1. Excellent esthetic properties
   - ✓ wide color matching and blending
   - ✓ high polishability
   - ✓ high gloss retention
2. Excellent physical- mechanical properties
   - ✓ high strength
   - ✓ low wear resistance
   - ✓ minimal polymerization shrinkage
   - ✓ staining resistance
3. Excellent handling properties
   - ✓ three flowability options and the good handling
   - ✓ short curing time and enough working time
   - ✓ minimal shade shift through curing

These features are achieved by adopting original Radical-Amplified Photo-polymerization initiator technology (RAP technology), Supra-nano Spherical filler and Round-shaped Composite filler.

6. References

1) Shigeki Yuasa, "Composite oxide spherical particle filler"
DE, No.128, 33-36 (1999)

2) Shigehisa Inokoshi,
Dental Outlook, Vol.88, No.4, 785-821, 1996

3) Shigehisa Inokoshi,
DE, No.163, 5-8 (2007)