# ESTELITE FLOW QUICK ESTELITE FLOW QUICK High Flow

# **TECHNICAL REPORT**



Tokuyama Dental

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#### 1. Introduction

Tokuyama Dental has developed a wide range of light-cured dental filling composite resins based on our unique supra-nano spherical filler technology. Represented by the Palfique® Estelite® paste, Estelite® Sigma, and Palfique® Estelite® LV, all these products have won high acclaim for their outstanding esthetics and surface glossiness.

In 2005, Tokuyama Dental introduced the Estelite® Flow Quick, a flowable composite resin combining the then newly-developed catalyst technology (RAP technology) and our unique filler technology to dramatically reduce curing times (to nearly 1/3) over conventional flowable resins. Thanks to RAP technology, Estelite® Flow Quick features an exceptionally high polymerization rate and boasts class-leading filler content (71 wt. %) among the flowable composite resin products currently available, as well as displaying superior physical and engineering properties not offered by conventional products.

Now we've applied the RAP technology used in Estelite® Flow Quick to produce a new type of composite resin product, the Estelite® Flow Quick High Flow, which we introduced commercially in October 2009. Estelite® Flow Quick High Flow offers excellent esthetics and high polymerization activity, features achieved through an improved matrix polymerization rate and relatively high stability in ambient lighting attributable to this RAP technology, as well as the outstanding esthetics provided by the monodispersing supra-nano spherical filler technology.

Below we give the technical background of the product and describe its features and physical properties.

- 2. Composition of the Estelite® Flow Quick and Estelite® Flow Quick High Flow
- 2.1. Filler Composition

Estelite® Flow Quick

- Silica-zirconia filler (supra-nano spherical filler)
- 0.4 μm and 0.07 μm
- filler content: 71 wt. % (53 vol. %)

#### Estelite® Flow Quick High Flow

- Silica-zirconia filler (supra-nano spherical filler)
- 0.4 μm and 0.07 μm
- filler content: 68 wt. % (49 vol. %)

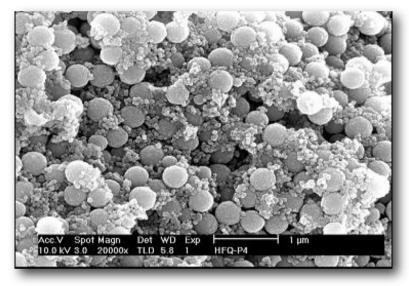


Fig1. EsteliteFlowQuick HighFlow (X20000)

#### 2.2. Matrix

Estelite® Flow Quick •Bis-MPEPP, TEGDMA, UDMA

Estelite® Flow Quick High Flow

Bis-GMA, TEGDMA

- 2.3. Shade Variations (four shades)
  - Estelite<sup>®</sup> Flow Quick

A1, A2, A3, A3.5, A4(Cerv.), B1, B2, B3, B4, C1, C2, C3, OA1, OA2, OA3, BW(Bleaching White) and CE(Inc.)

Estelite® Flow Quick High Flow A1, A2, A3, and OPA2

#### 2.4. Indications

- Direct anterior and posterior restorations (particularly for small/shallow/tunnel shaped cavities)

- Cavity lining

- Blocking out cavity undercuts before fabrication indirect restorations

Repair of porcelain/composite

#### 3. Background Technologies

- 3.1. Radical Amplified Photo-Polymerization (RAP) Technology
- 3.1.1. Initiator mechanism

The catalyst technology adopted for Estelite® Flow Quick High Flow is the radical amplified photo-polymerization initiator (RAP technology) used in Estelite® Flow 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. Figure 2 is a schematic diagram of RAP technology.

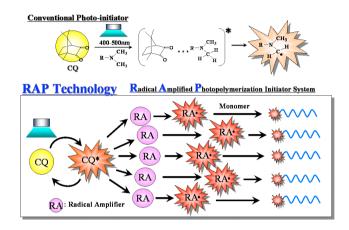


Fig.2 Radical amplified polymerization initiator system

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 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, 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 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 accelerates polymerization, we compared the volume of residual monomers after light curing and the volume of radicals generated for a flowable resin containing RAP initiator (EFQ-RAP) and a flowable resin containing a conventional photo-polymerization initiator of CQ/amine (EFQ-CQ). The results are shown in Figs. 3 and 4.

As Fig. 3 shows, compared to a conventional CQ/amine photo-polymerization initiator system, using RAP initiator for the 10-second and 30-second irradiation groups dramatically reduces the residual monomer fraction. Notably, the RAP initiator system prevails even if we compare 10-second irradiation for the RAP initiator system to 30-second irradiation for the CQ/amine system. 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). This is believed to support the mechanism of activity presented in Fig. 1.

The relationship between light intensity and polymerization speed is schematically depicted in Fig. 5. With the RAP initiator, polymerization proceeds slowly and remains stable at low light intensity – that is, under weak light sources, such as dental lights. However, at higher intensities (e.g., light from irradiation units), polymerization accelerates sharply.

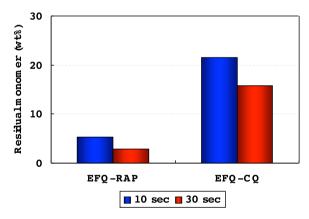


Fig.3 Residual monomer(wt%)

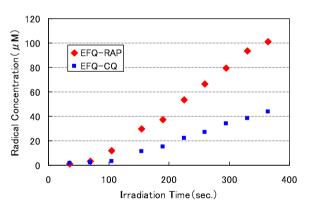


Fig.4 Change of radical concentration



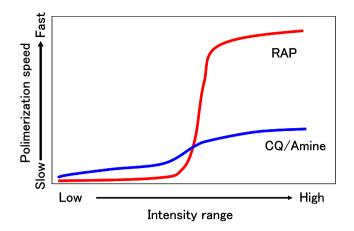
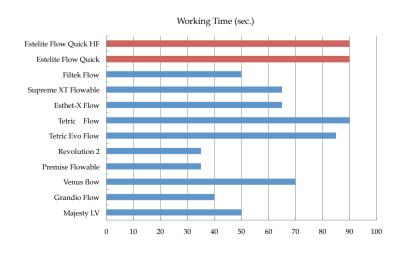
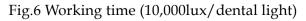


Fig.5 Correlation between intensity range and polimerization speed

#### 3.1.2. Stability in ambient lighting

It has generally believed that more photo-polymerization initiators must be added to achieve high polymerization activity with short exposures. However, increasing catalyst volumes generally means reduced stability in ambient lighting. In clinical settings, this can increase paste viscosity during the filling procedure, sometimes forcing a second filling attempt due to the impossibility of resin sculpting. Large photo-polymerization initiator volumes also have other adverse effects, including significant changes in color from before to after curing. In contrast, RAP technology makes it possible to achieve a balance between polymerization activity and stability in ambient lighting, as described in Section 5.1. Figure 6 presents the ambient light stabilities (under 10,000 lux dental lighting) of commercial flowable composite resins. As Fig. 6 shows, Estelite® Flow Quick and Estelite® Flow Quick High Flow are the most stable of the commercially available flowable composite resins in ambient lighting, despite the fact that it requires such brief irradiation times to cure. This gives the clinical staff time to perform clinical procedure such as filling without undue haste.





#### 3.2. Supra-Nano Spherical Fillers

Tokuyama Dental Corporation synthesizes monodispersing 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. 7).



Fig.7 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. 8 gives the correlation between filler particle size and filler content and compressive strength. Fig. 9 gives the correlation between filler particle size and surface roughness and hardness. From Fig. 8, we see that filler content begins to fall significantly below 0.1  $\mu$ m, but is nearly constant at sizes above that. In addition, we observe maximum compressive strength at particles size ranging from 0.1 to 0.5  $\mu$ m. From Fig. 9, we see that surface roughness decreases with particle sizes down to approx. 0.5  $\mu$ m but remains constant at sizes below that. Surface hardness attains the highest value at particle sizes ranging from 0.1 to 0.5  $\mu$ m. 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.<sup>1</sup>

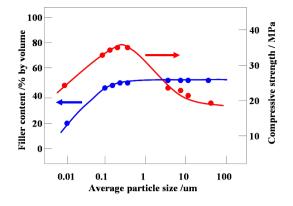


Fig.8 Correlation between particle size, filler content and compressive strength

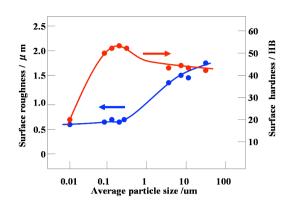


Fig.9 Correlation between particle size, surface roughness and surface hardness

For Estelite® Flow Quick High Flow and Estelite® Flow Quick, we use monodispersing spherical fillers made of silica-zirconia produced by the sol-gel method, with particle sizes of 0.4 µm and 0.07 µm (Fig. 10). 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. 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 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® Flow Quick High Flow and Estelite® Flow Quick High Flow, the silica/zirconia composition is adjusted to prepare fillers with optimal refractive indices.

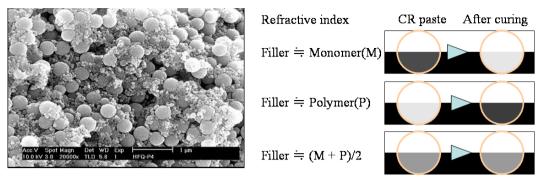
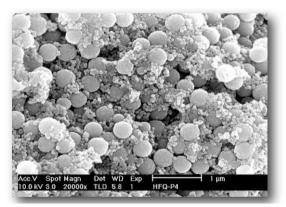


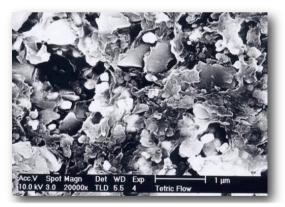
Fig.10 Estelite FlowQuick High

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

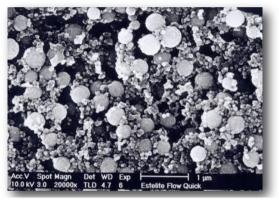
Fig.11



ETELITE Flow Quick High Flow(×20,000)

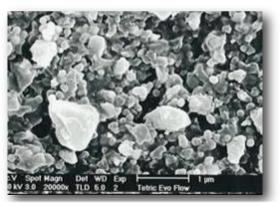


Tetric Flow



Refractive

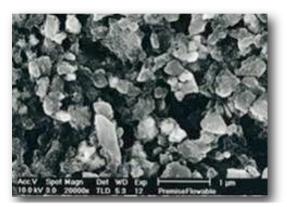
ESTELITE Flow Quick(×20,000)



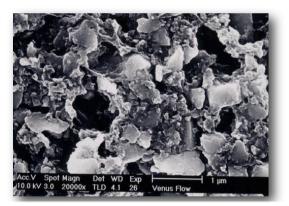
Tetric Evo Flow



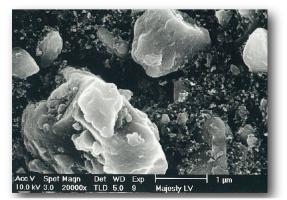
Filtek Flow



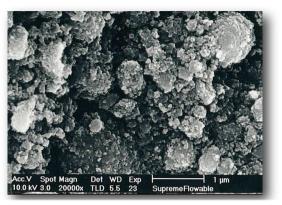
Premise Flowable



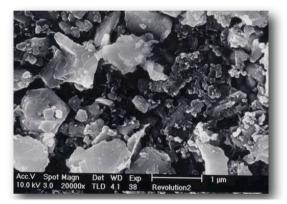
Venus Flow



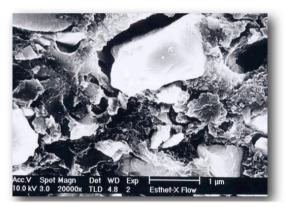
Clearfil Majesty LV



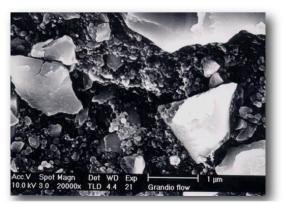
Filtek Supreme XT Flowable



Revolution 2



Esthet X Flow



Grandio Flow



- 4. Material Properties
- 4.1. Polymerization Shrinkage

We assessed polymerization shrinkage rates by our proprietary method. Figure 12 presents a schematic diagram of the measurement system.

Figure 13 shows polymerization shrinkage rates for Estelite® Flow Quick, Estelite® Flow Quick High Flow and other commercially available composite resin products. The graph presents the shrinkage rate at 3 minutes from the start of irradiation.

We see that the shrinkage rate of Estelite® Flow Quick and Estelite® Flow Quick High Flow is average for commercially available composite resin products.

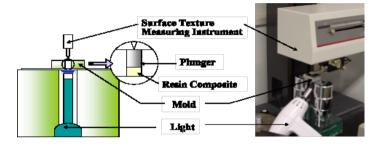
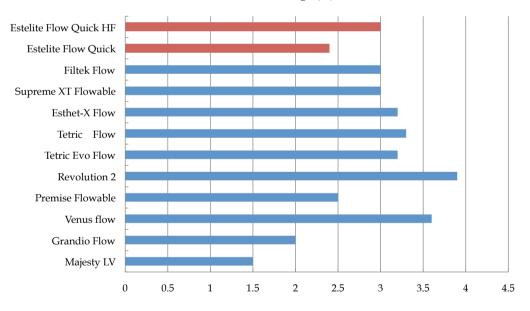
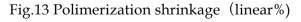


Fig.12 Method of polymerization shrinkage



linear Shrinkage (%)



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#### 4.2. Wear Properties (antagonistic wear test)

We examined the wear resistance of composite resin together with that of human teeth by the method shown in Fig. 14. Fig. 15 shows the results. Estelite® Flow Quick High Flow achieves a good balance between volumetric loss of CR and abrasion of human teeth. Like Estelite® Flow Quick, it is a resin with excellent wear characteristics: Despite its wear-resistance, it does not cause wear in the opposing teeth.

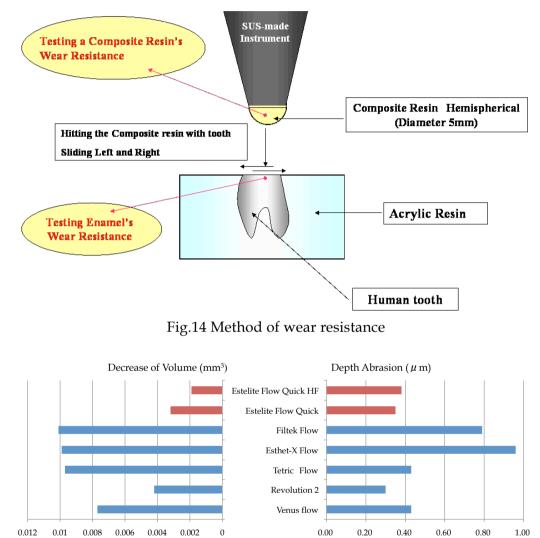


Fig.15 Wear resistance (50000cycle)

#### 4.3. Flexural and Compressive Strength

Figure 16 and Figure 17, respectively, present the flexural and compressive strength of Estelite® Flow Quick and Estelite® Flow Quick High Flow versus other commercially available composite resin products. As with Estelite® Flow Quick, of the commercially available composite resins, Estelite® Flow Quick High Flow falls into the category of products exhibiting high flexural and compressive strength.

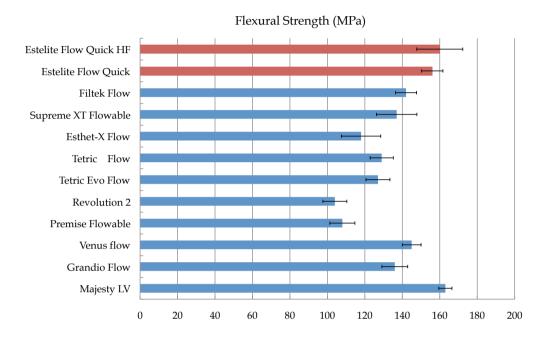
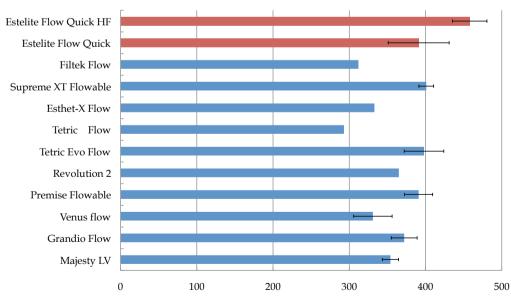
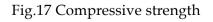


Fig.16 Flexural strength



Compresive Strength (%)



#### 4.4. Surface Glossiness

Figure 18 shows the glossiness of a cured CR surface polished with waterproof abrasive paper (#1500) using Sof-Lex<sup>™</sup> Superfine disc (for 60 seconds under a cooling water spray). Like Estelite® Flow Quick, Estelite® Flow Quick High Flow exhibits extremely high surface glossiness.

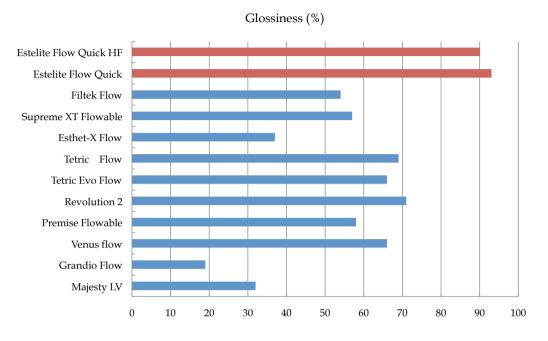


Fig.18 Surface Glossiness

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4.5. Change in Color and Translucency from Before to After Polymerization

Estelite<sup>®</sup> Flow Quick High Flow and Estelite<sup>®</sup> Flow Quick show only minor changes in color and translucency from before to after polymerization. This means the results of color matching can be judged with fair accuracy before curing. Figure 19 presents the changes in translucency and color from before to after polymerization for various commercial composite resin products.

As the figure shows, Estelite® Flow Quick High Flow shows only minor changes in translucency and color from before to after polymerization, facilitating the shade assessment procedure. Estelite® Flow Quick High Flow reduces the potential fordiscovering a color match problem after polymerization is complete.

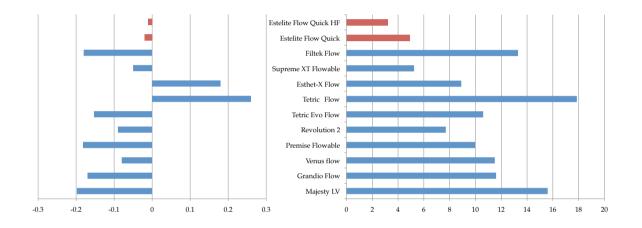


Fig19 Color change before and after polymerization

#### 4.6. Staining by Coffee

Composite resins placed in the oral cavity will degrade over time with exposure to various food and drink substances. When the effects of degradation are pronounced compared to surrounding teeth, it becomes visible and cosmetically significant. Here, we examined the effects of staining by coffee (24-hour immersion test at 80°C). Fig. 20 gives the results.

The extent of the color change following immersion in coffee for Estelite® Flow Quick High Flow is similar to that of Estelite® Flow Quick. Both rank among the best of the composite resin products available commercially. We can safely conclude that in actual clinical applications, the color observed at the time of restoration will be longlasting.

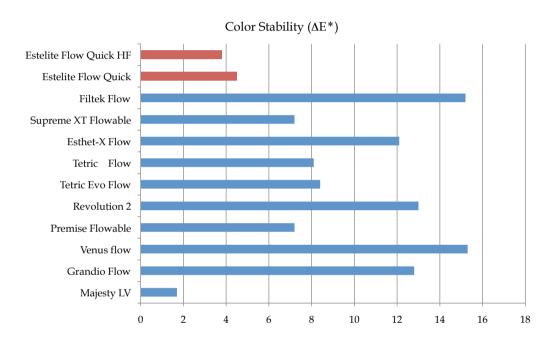


Fig.20 Color stability( $\Delta E^*$ )

#### 4.7. Radiopacity

The composition of the inorganic filler and its filler content determine the radiopacity of composite resins. A resin will exhibit higher radiopacity if it contains more filler and if the filler contains higher proportions of elements with high atomic numbers. However, fillers with greater proportions of elements with high atomic numbers will display high refractive indices and exhibit significant changes in color and translucency from before to after polymerization.

As described in Section 4.5, the inorganic filler used in Estelite® Flow Quick High Flow and Estelite® Flow Quick has been designed to minimize changes in color and translucency from before to after polymerization while achieving the maximum radiopacity for that composition. Figure 21 shows the radiopacity of commercially available composite resins.

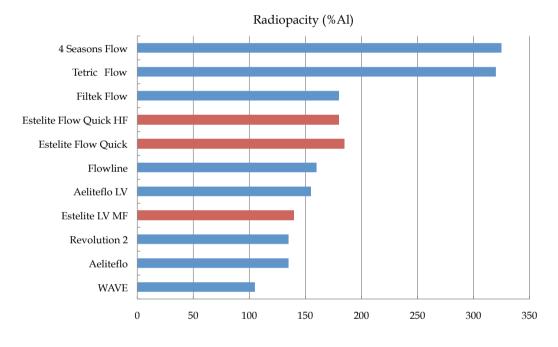


Fig.21 Radiopacity

While the radiopacity of Estelite® Flow Quick is average, it is adequate for follow-up observations of restoration work. Fig. 22 is a clinical X-ray image of Estelite® Flow Quick High Flow.



Fig.22 Radiopacity of EsteliteFlowQuickHF

#### 5. Shade Variations

After determining that Estelite<sup>®</sup> Flow Quick could restore clinical cases similar to universal resins, we decided that having the same shades as the latter is desirable and 17 color shades were prepared. We provide Estelite<sup>®</sup> Flow Quick's entire shade lineup below:

A1, A2, A3, A3.5, A4(Cerv.), B1, B2, B3, B4, C1, C2, C3, OA1, OA2, OA3, BW(Bleaching White) and CE(Inc.)

Estelite® Flow Quick's color shades were adjusted to match that of Estelite® Sigma Quick's shades upon intraoral resin placement in the prepared tooth cavity. Consequently, when comparing Estelite® Flow Quick and Estelite® Sigma Quick shades against a white background, the shades tend to appear somewhat darker. However, as mentioned above, when placed in a prepared tooth cavity Estelite® Flow Quick's shades have been adjusted to match the shades of Estelite® Sigma Quick without disturbing aesthetic harmony.

Estelite® Flow Quick High Flow is available in four shades (A1,A2,A3,and OPA2). The colors corresponding to A1, A2, and A3 are the identical to those for Estelite® Flow Quick (which is available in a total of 17 shades). The new OPA2 shade for Estelite® Flow Quick High Flow has a higher contrast ratio than the OA2 shade of Estelite® Flow Quick and is ideal for masking mild tooth discoloration. As shown in Fig. 24, filling with OPA2 effectively hides black spots. When Estelite® Sigma Quick (A3) is filled incrementally over Estelite® Flow Quick High Flow OPA2, the black spot becomes all but invisible. As this example shows, the OPA2 shade for Estelite® Flow Quick High Flow effectively masks mild tooth discoloration.

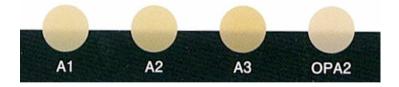


Fig.23 Shade options

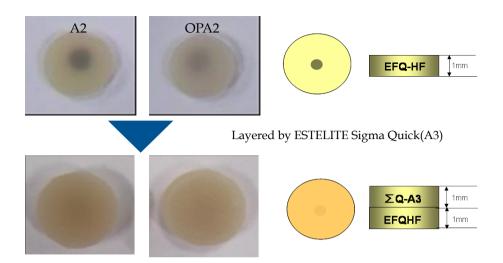


Fig.24 Masking Effect of OPA2

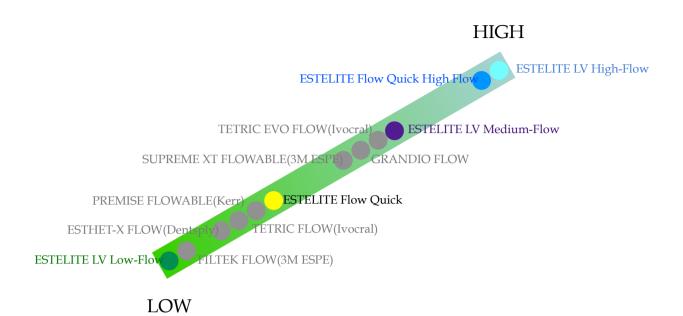
#### 6. Handling

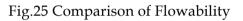
#### 6.1. Paste Characteristics

Estelite<sup>®</sup> Flow Quick High Flow was selected for its high flow paste characteristics to optimize suitability for the lining and restoration of small cavities, such as tunnel cavities. As Fig. 25 shows, Estelite<sup>®</sup> Flow Quick High Flow exhibits high flowability compared to other commercial composite resins. Figures 26 is a photo showing the condition of the Estelite<sup>®</sup> Flow Quick High Flow and Estelite<sup>®</sup> Flow Quick pastes after 0.03 grams of each are measured onto glass plates and held vertically for 1 minute at 37°C. Figure 27 shows the condition of the paste after 0.1 grams of each paste is held horizontally for 2 minutes at 37°C.

As Figs. 25, 26, and 27 show, Estelite<sup>®</sup> Flow Quick High Flow exhibits high flowability compared to the composite resins currently available, making it ideal for lining.







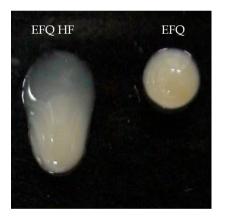


Fig. 26 1min. after vertical position(37°C)

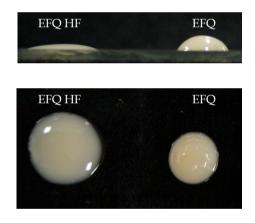


Fig. 27 2min. after horizontal

#### 6.2. Syringes

Flowable composite resins are applied to cavities by directly filling with a syringe to which pre-filled fine syringe tips are fitted. With these syringes, the pressure remaining within the syringe after filling results in paste leaks from the tip. This phenomenon, which we call post-dispensing leakage, leads to various undesirable results, including excess resin that sticks to areas surrounding the cavity during cavity filling and paste spills on tabletops when the syringe is set down.

Estelite® Flow Quick High Flow is used with a uniquely designed syringe (Fig. 28), which controls residual pressure within the syringe after application to prevent postdispensing leaks. The larger grip also allows a surer grip and makes rotating the syringe easier.

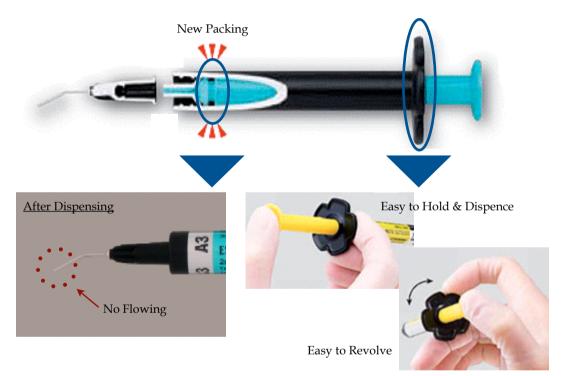


Fig.28 New syringe

#### 7. Summary

Estelite® Flow Quick and Estelite® Flow Quick High Flow is a composite resin that offers a range of outstanding characteristics, including high polymerization activity and high esthetic qualities. These characteristics are attributable to the adoption of the photo-polymerization catalyst technology (RAP technology) and to monodispersing supra-nano spherical filler technology.

(1) Fast Curing

- Cures in approximately 1/3 the time required for conventional composite resins.
- Compatible with a wide range of light-curing units (cures rapidly with halogen,
- LED, and xenon light sources)

• Displays higher stability in ambient lighting than conventional products.

(2) Superior physical properties

• Exhibits outstanding wear resistance, but with reduced opposing teeth abrasion

(3) Superior esthetic qualities

- High surface glossiness achieved with short polish times
- Minimal change in color and translucency from before to after polymerization
- Addition of a new shade of higher opacity (Estelite<sup>®</sup> Flow Quick High Flow OPA2)

(4) Excellent handling

- Estelite<sup>®</sup> Flow Quick : A medium flowability type with minimal slumping and it is good adaptation to cavity walls.
- Estelite® Flow Quick High Flow : High flowability for use in lining and tunnel cavity restoration

• New syringe design to prevent post-dispensing leakage and reduce residual paste in the syringe

Larger grip for a surer grip and easier syringe rotation

8. Reference

1) Shigeki Yuasa, "Spherical Composite Oxide Particle Fillers (in Japanese)", in DE No. 128, pp. 33-36 (1999)