Tokuyama Dental

Resin-based Dental Restorative Material

PALFIQUE ASTERIA

Technical Report

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(=PALFIQUE LX5), 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.

The RAP technology™ used in Estelite Flow Quick® was applied to universal composite resins. Estelite Σ Quick® (=PALFIQUE LX5), released in 2007 and Estelite Omega®, released in 2011. Estelite Σ Quick® (=PALFIQUE LX5)and Estelite Omega® provide outstanding esthetics and high polymerization activity based on Supra-nano Spherical filler technology and RAP technology.

Palfique Asteria universal composite which applied these original technologies is focused on simplified layering restorative therapy and outstanding esthetic results. The subsequent sections describe the technical background, features, and properties of Palfique Asteria.

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: 82 wt% (71 vol%)

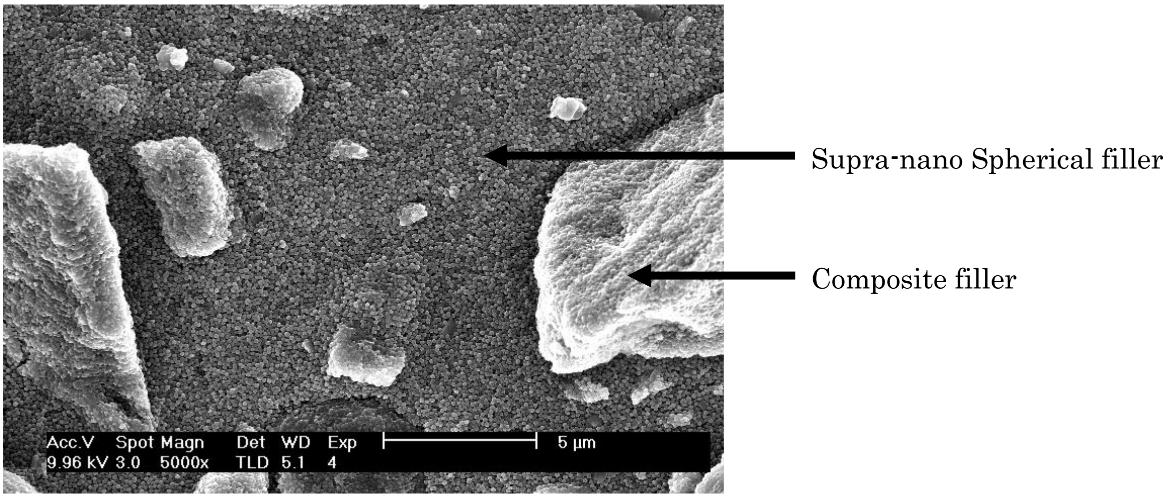


Fig1. PALFIQUE Asteria (5000x)

2.2. Indications

- Direct anterior and posterior restorations including occlusal surfaces
- Direct bonded composite veneer
- Diastema closure
- Repair of porcelain/composite

2.3. Shades

Palfique Asteria introduces a new simplified 2-step layering concept. This comprehensive system is comprised of minimal shades; just 7 Body shades and 5 Enamel shades cover the entire dental shade range. Esthetic result is obtained with simple 2-layering concept of Palfique Asteria, because the Body shades replicate chroma and hue and Enamel shades replicate value.

The Body shades have excellent blending ability with less width of margin bevel thanks to their state-of-the-art optical properties. The Body shades provide some translucency with sufficient opacity to avoid shining through without the use of opaque or dentin shades. Therefore, an invisible margin is achieved by covering a margin with Body shades (except the incisal area)(Fig.6). A1-A4 Body shades blend with most natural dentition. BL is designed for high value bleached teeth and B3B for yellowish teeth (Fig. 5).

The Enamel shades have compatible translucency as a substitute for natural enamel. The primary use of the NE shade is for anterior teeth and the OcE is for the posterior occlusal area (Fig. 6, 7). The 3 supplemental Enamel shades (substitutes for NE) may be used for the following applications: TE is for high translucent anterior teeth, WE is for whitish enamel and YE is for discolored or orangish enamel (Fig.5, 8).

Layering concept and shading system of Palfique Asteria are designed by Dr. Noboru Takahashi.

- **Body Shades** **A1B, A2B, A3B, A3.5B, A4B, B3B, BL**
- **Enamel Shades** **NE, YE, TE, WE, OcE**

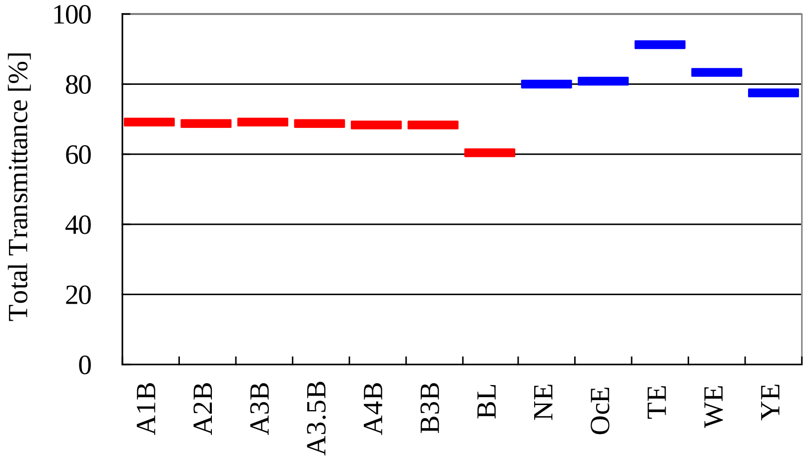


Fig.2 Total transmittance

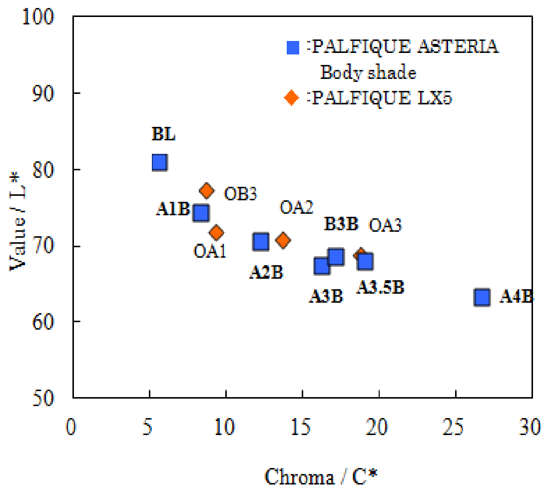


Fig.3 Correlation between Value and Chroma (Body shade)

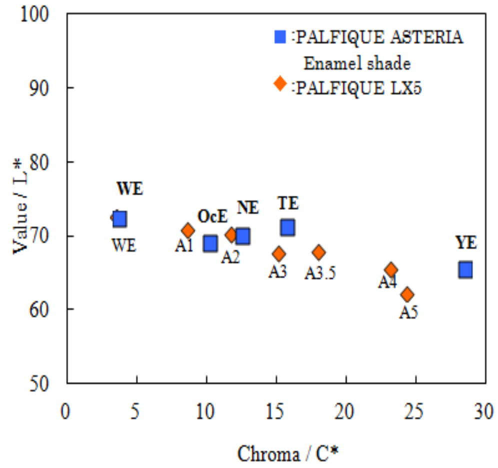
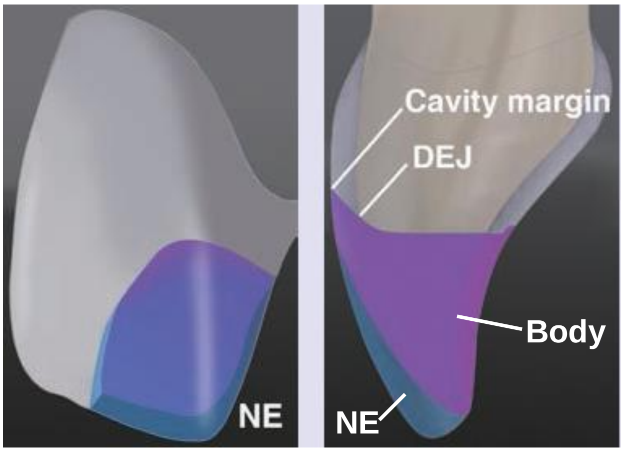


Fig.4 Correlation between Value and Chroma (Enamel shade)

Component	Shade	Tip
BODY	A1B, A2B, A3B, A3.5B, A4B, B3B, BL	**Body shades** are designed for reconstructing the dentin layer The Body shades should cover all enamel margins except the incisal area.
ENAMEL	Natural Enamel (NE)	**NE** is reccomended to restore translucency in the incisal area in most cases.
	White Enamel (WE)	**WE** is recommended for the proximal wall. WE is suggested as an alternative to NE in whiter cases.
	Yellow Enamel (YE)	**YE** is designed to mimic discolored enamel.
	Trans Enamel (TE)	**TE** is the most translucent in this system. This shade is suggested as an alternative to NE in highly translucent cases.
	Occlusal Enamel (OcE)	**OcE** is recommended for the occulusal surface. OcE has exceptional sculptability to shape occlusal cusps and fissures.

Fig.5 Shade structure



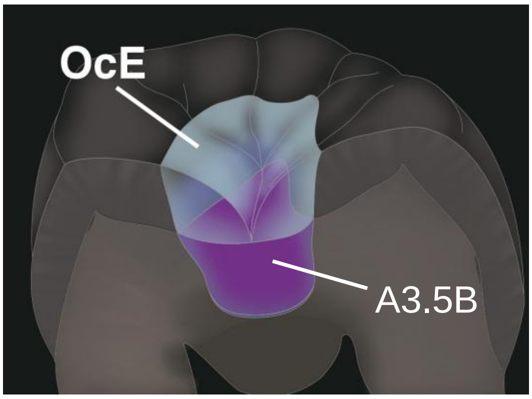


Fig.6 Class IV restoration

Fig.7 Class I restoration (3D layering)

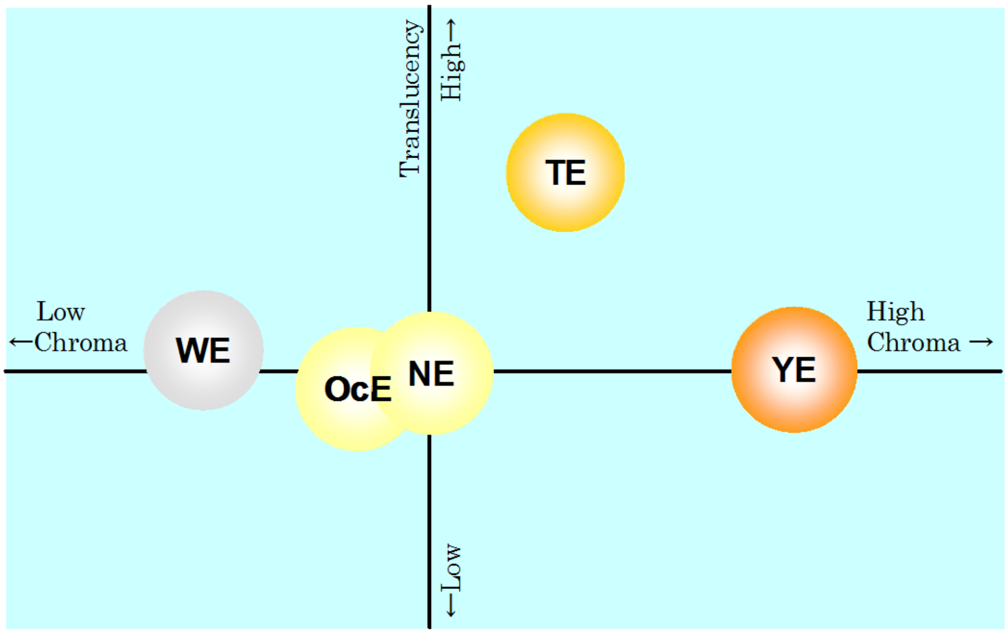


Fig.8 Color and translucency map of the Enamel shades

3. Background technology

3.1. Radical Amplified Photopolymerization initiator (RAP technology™)

3.1.1. Mechanism

The catalyst technology adopted for Palfique Asteria is the Radical Amplified Photo-polymerization initiator (RAP technology™) used in Estelite Σ Quick®(=PALFIQUE LX5). 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.9 is a schematic diagram of RAP technology™.

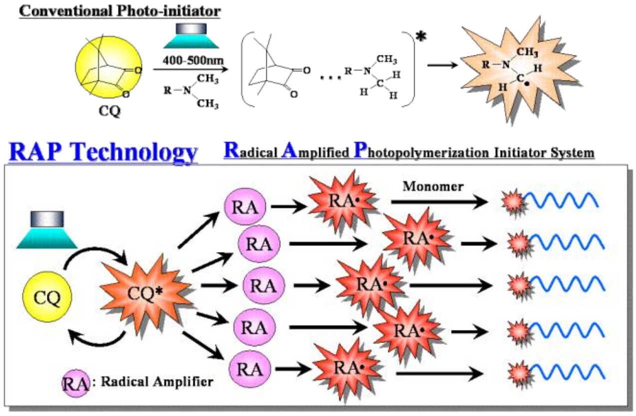


Fig.9 Radical amplified polymerization initiator system

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, 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 composite resins: Estelite Flow Quick®, which contains a radical amplified photopolymerization initiator, and flowable composite, which contains a conventional photopolymerization initiator composed of CQ and amines. Fig.10, 11 shows the results. Fig. 10 indicates that the radical amplified photopolymerization initiator significantly reduces residual monomers compared to the conventional CQ-amine photopolymerization initiator for both 10-second and 30-second exposures. This holds true even when comparing Estelite Flow Quick® after 10-seconds of exposure to conventional flowable composite after 30-seconds of exposure. These results support the mechanism of action shown in Fig. 9.

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.12).

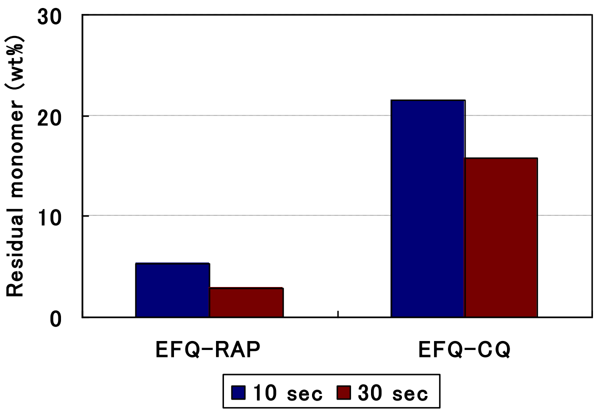


Fig.10 Residual monomer (wt%)

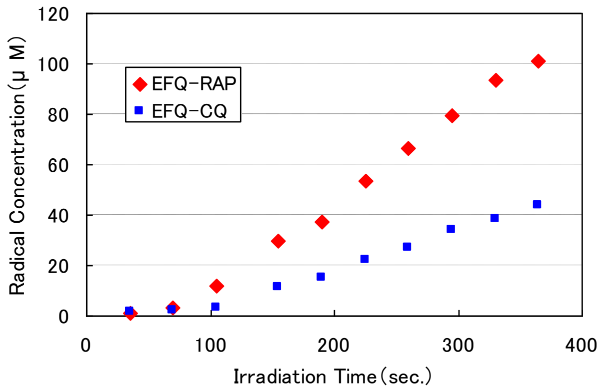


Fig.11 Change of radical concentration

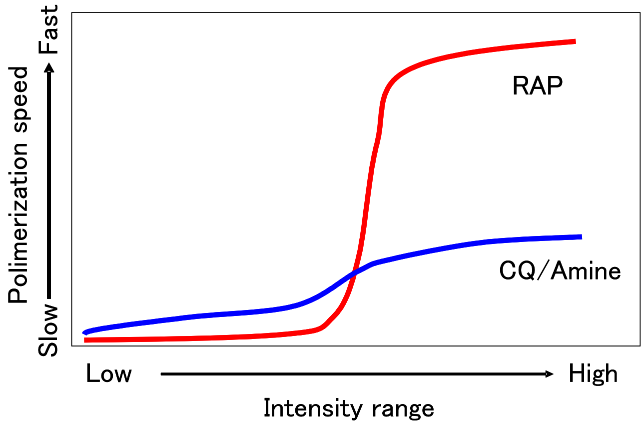


Fig.12 Correlation between intensity range and polymerization rate

3.1.2. Stability in ambient light

In the past, high polymerization activity with short exposures could only be achieved by increasing the amount of photopolymerization 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 sculpt 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 photopolymerization 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 3.1.1. Fig.13 compares stability under ambient light (10,000 lx of dental light) between Palfique Asteria and other commercially available composite resin.

As shown in Fig. 13, Palfique Asteria offers stability in ambient light equivalent to products from other manufacturers, with working times slightly longer than average. This gives clinicians more time to perform filling and other steps.

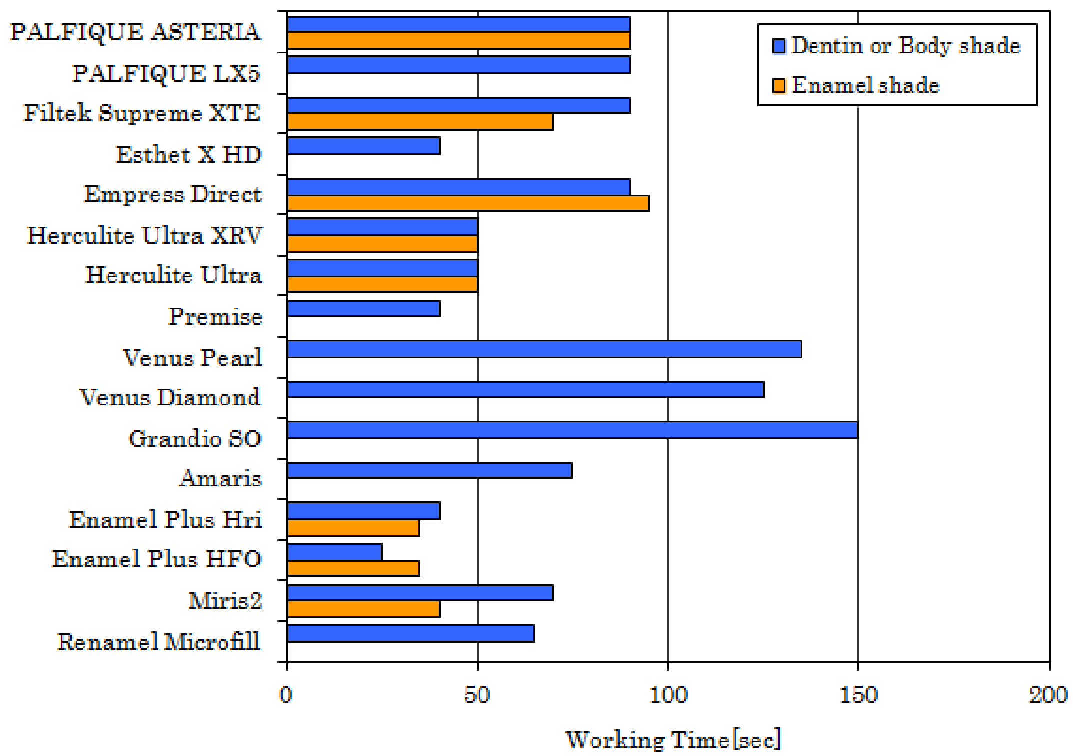


Fig.13 Insensitivity to light (10,000lux/dental light)

3.2. Supra-nano Spherical Filler Technology

Tokuyama Dental 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. 14).

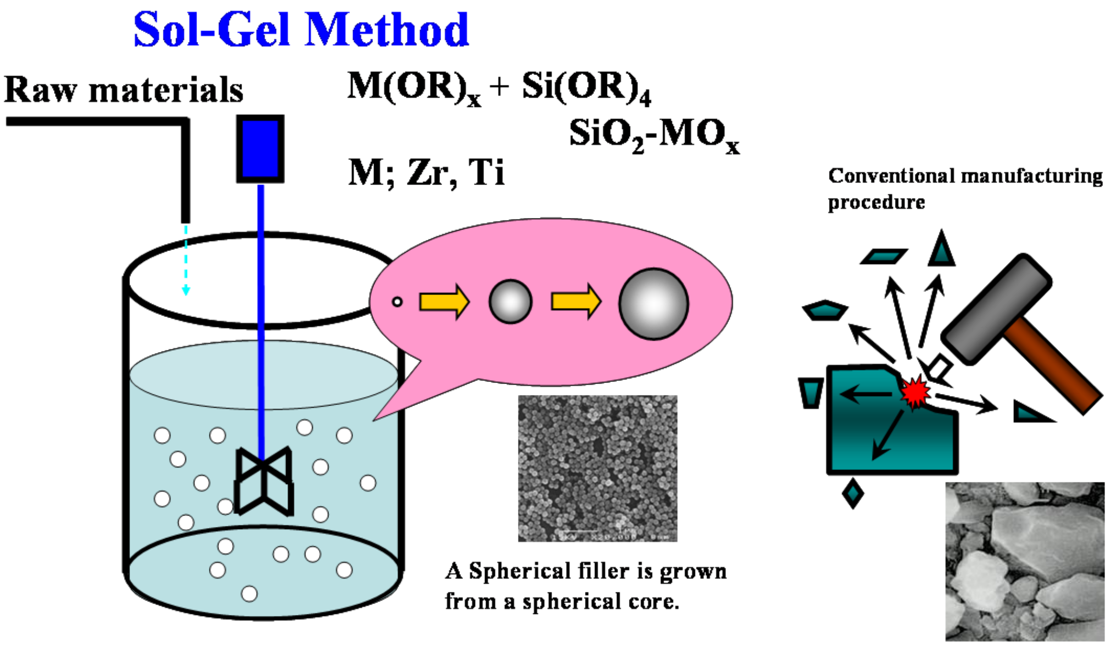


Fig.14 Summary of sol-gel method

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.15 gives the correlation between filler particle size and filler content and compressive strength. Fig.16 gives the correlation between filler particle size and surface roughness and hardness. From Fig.15, 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. 16, 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 100 to 500 nm. 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.[1)]

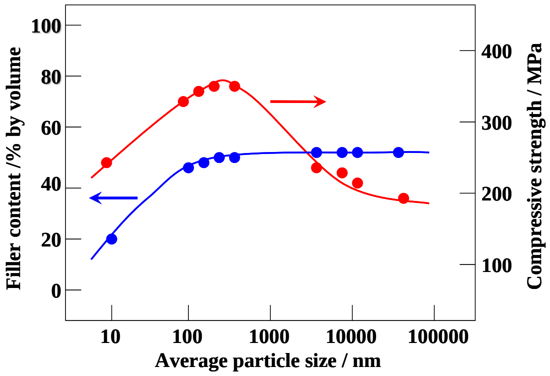


Fig.15 Correlation between particle size, filler content and compressive strength

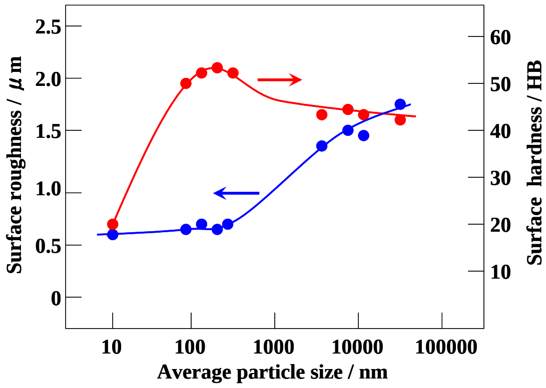


Fig.16 Correlation between particle size, surface roughness and surface hardness

For Palfique Asteria, we use monodispersing spherical fillers made of silica-zirconia produced by the sol-gel method, with particle sizes of 200 nm (Fig. 17).

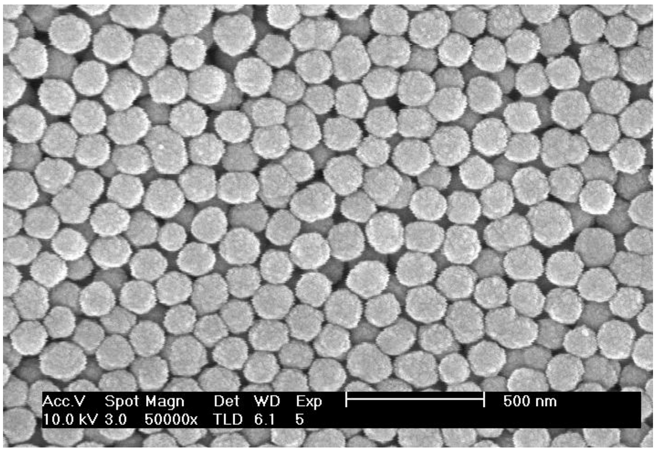


Fig.17 Supra-nano Spherical filler in Estelite ®Asteria

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 (Fig. 18)

In PALFIQUE Asteria, the silica/zirconia composition is adjusted to prepare fillers with optimal refractive indices.

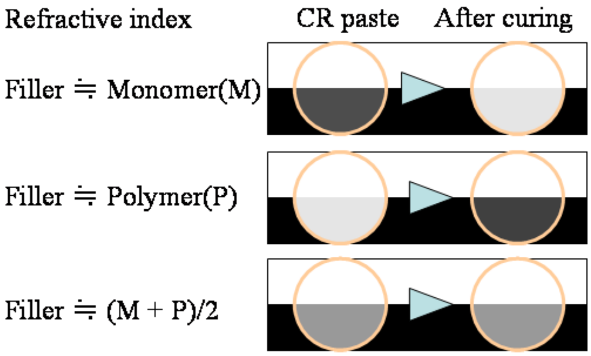
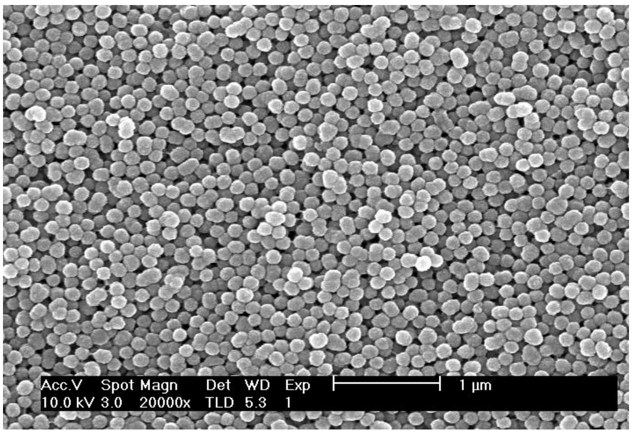
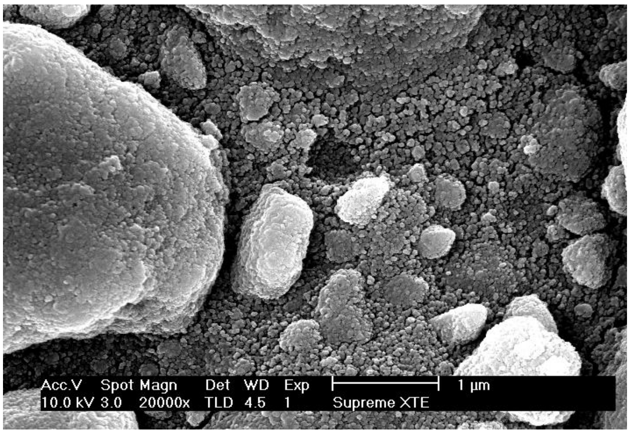


Fig. 18 Refractive Index

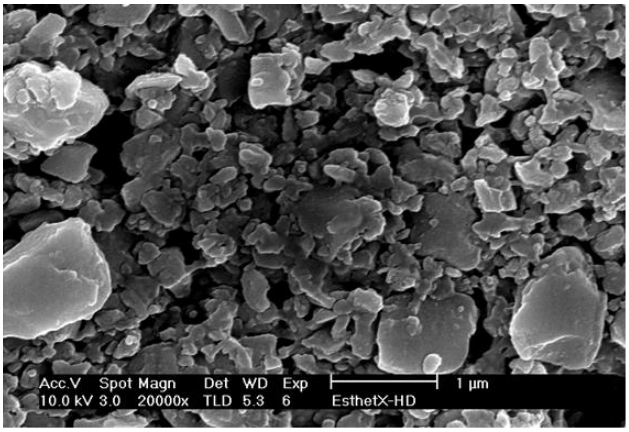
Below are SEM images (20,000X) of fillers used in Palfique Asteria and in composite resins from other manufacturers.



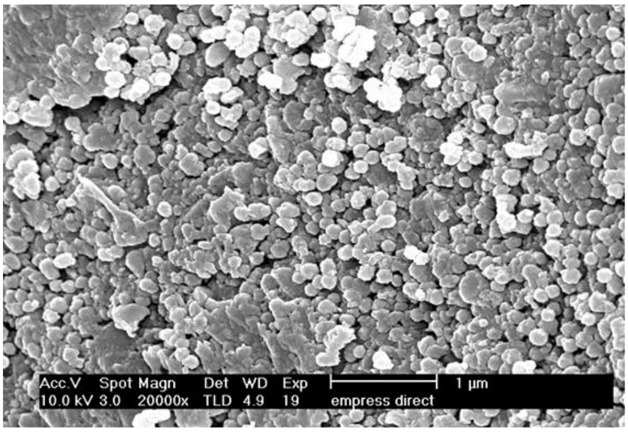
Palfique Asteria



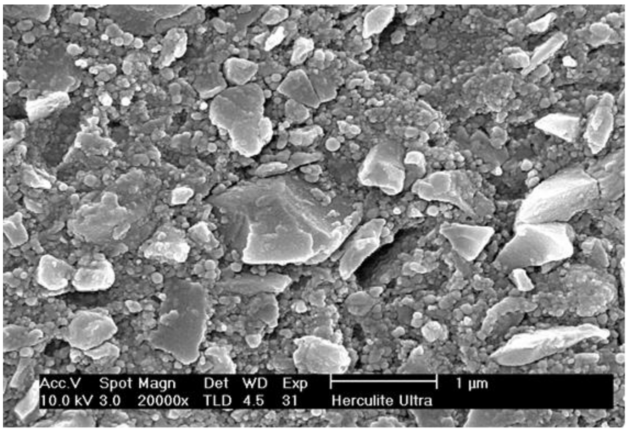
Filtek Supreme XTE



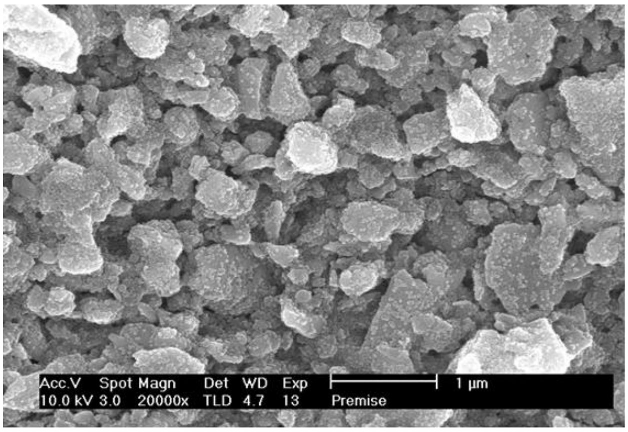
Esthet-X HD



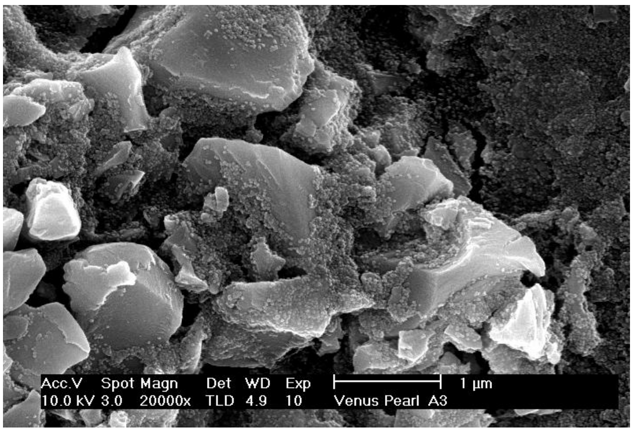
Empress Direct



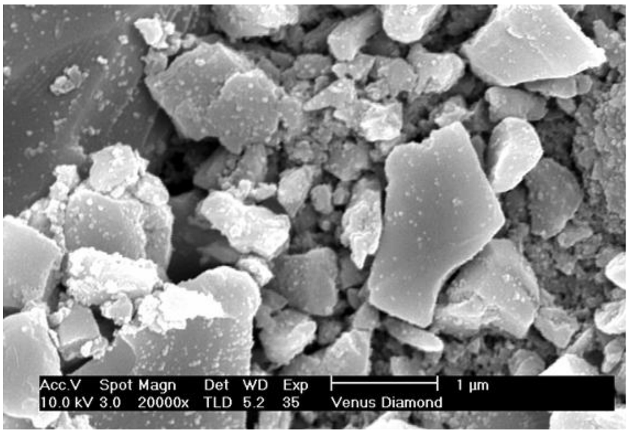
Herculite Ultra



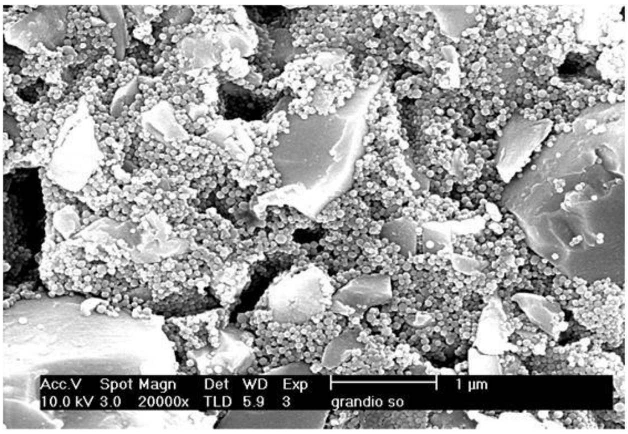
Premise



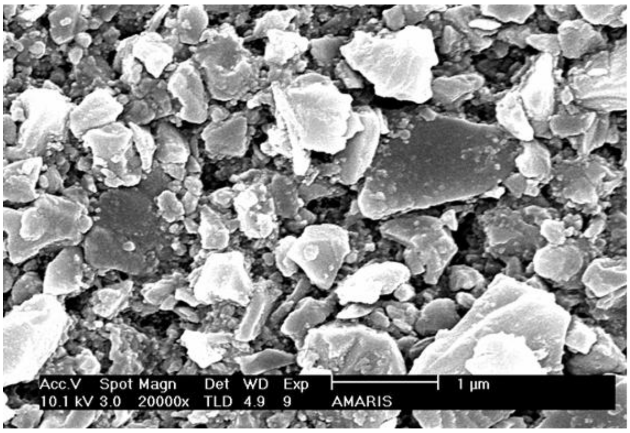
Venus Pearl



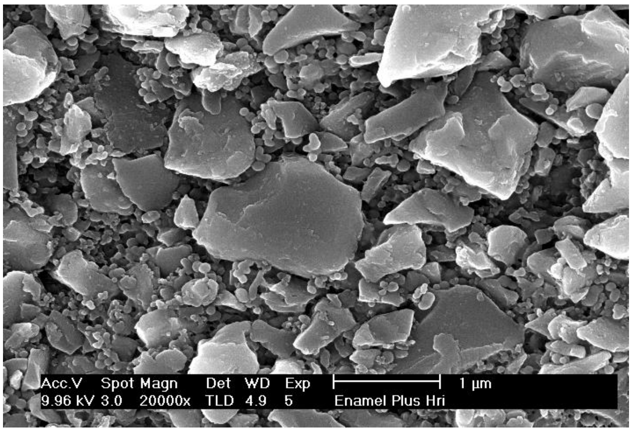
Venus Diamond



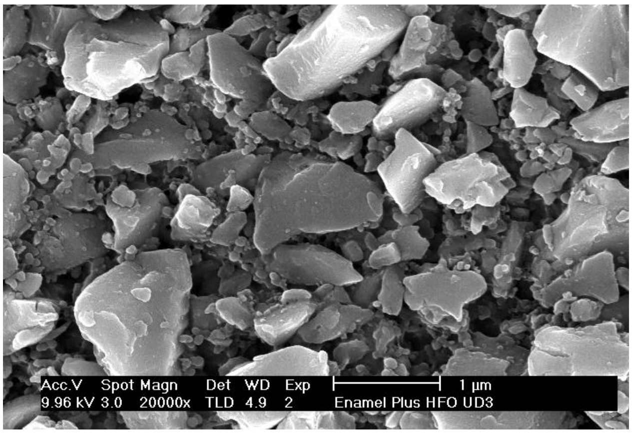
Grandio SO



Amaris

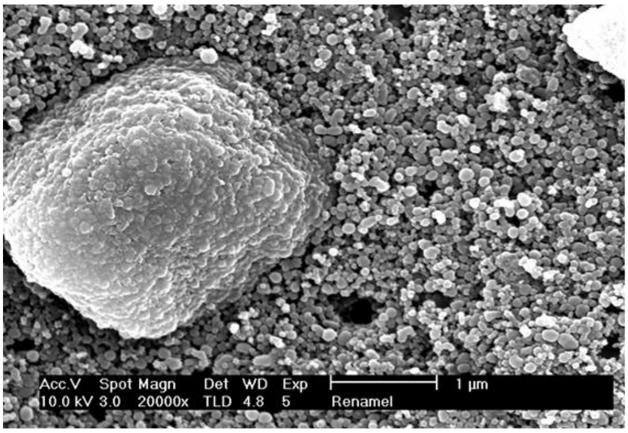


Enamel Plus Hri



Enamel Plus HFO

Miris2



Renamel Microfill

4. Material properties

4.1. Polymerization shrinkage

We measured polymerization shrinkage by our original method. Fig.19 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.19) 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.20 shows the polymerization shrinkage of Palfique Asteria and other commercially available composite resins. The graph indicates shrinkage 3 minutes after the start of light exposure.

The polymerization shrinkage of Palfique Asteria is 1.3%, or the same as for Estelite Σ Quick®(=PALFIQUE LX5). This is the minimum level among commercially-available composite resins. This result is due to the high filler volume content made possible by the combination of Supra-nano Spherical filler and composite filler.

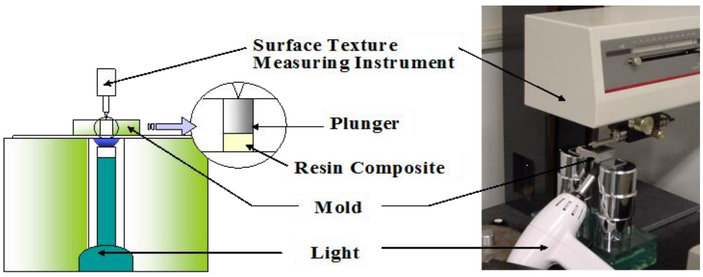


Fig.19 Method of polymerization shrinkage

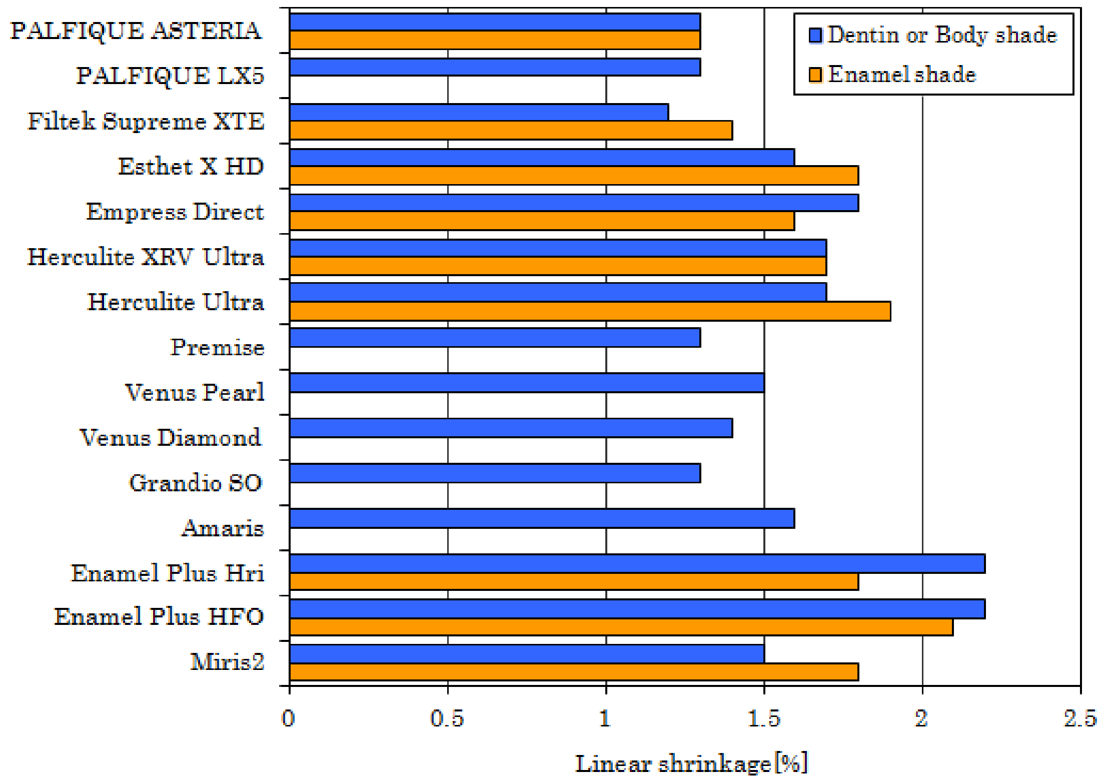


Fig.20　Polymerization shrinkage（linear%）

4.2. Wear Properties (Antagonistic wear test)

We examined the wear characteristics of composite resins in terms of wear resistance of the resin and the human tooth by the method shown in Fig.21.

Fig.22 gives the results. Palfique Asteria demonstrated a good balance between volume loss of CR and wear on human teeth. As with Estelite Σ Quick®(=PALFIQUE LX5), Palfique Asteria itself resists wear without causing unusual wear in opposing teeth.

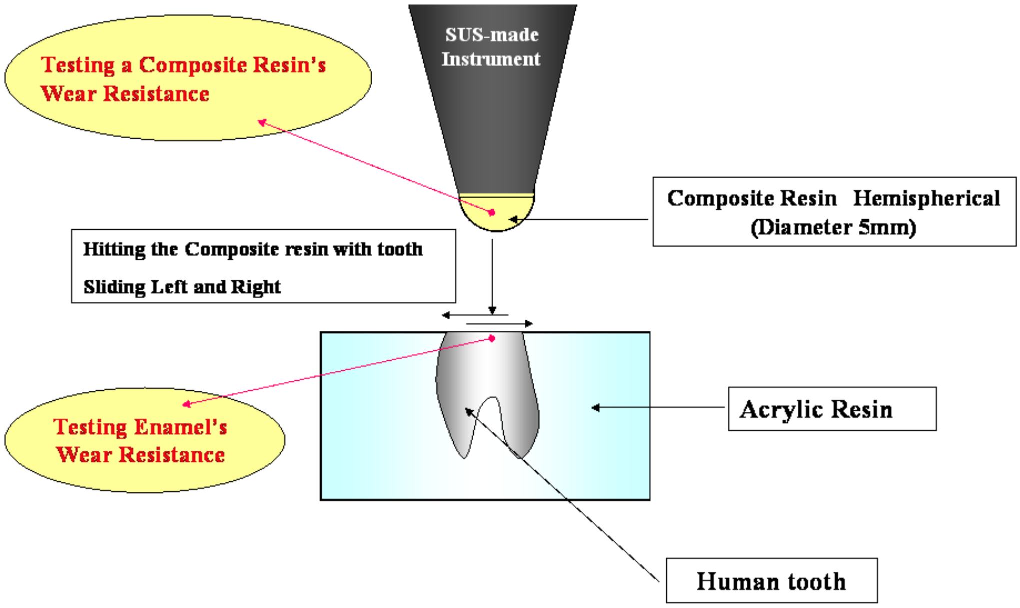
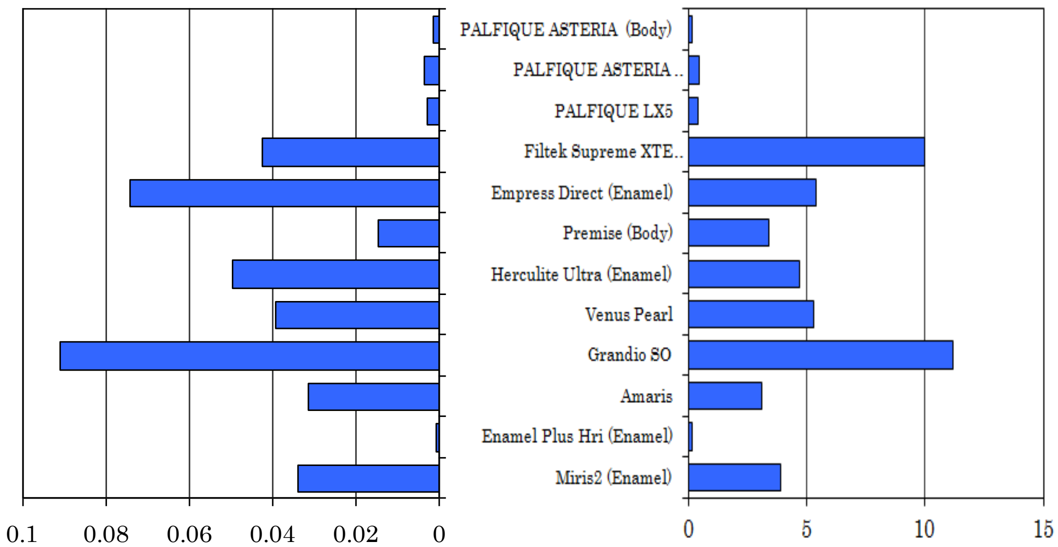


Fig.21 Method of wear resistance



Decrease of Volume (mm³) Depth Abrasion (μ m)

Fig.22 Wear resistance (50000cycle)

4.3. Flexural strength and Compressive strength

Fig.23 presents the flexural strength and Fig.24 presents the compressive strength of Palfique Asrteria and other commercially available composite resins.

The flexural strength and the compressive strength of Palfique Asteria are ranked as average among commercially available composite resins.

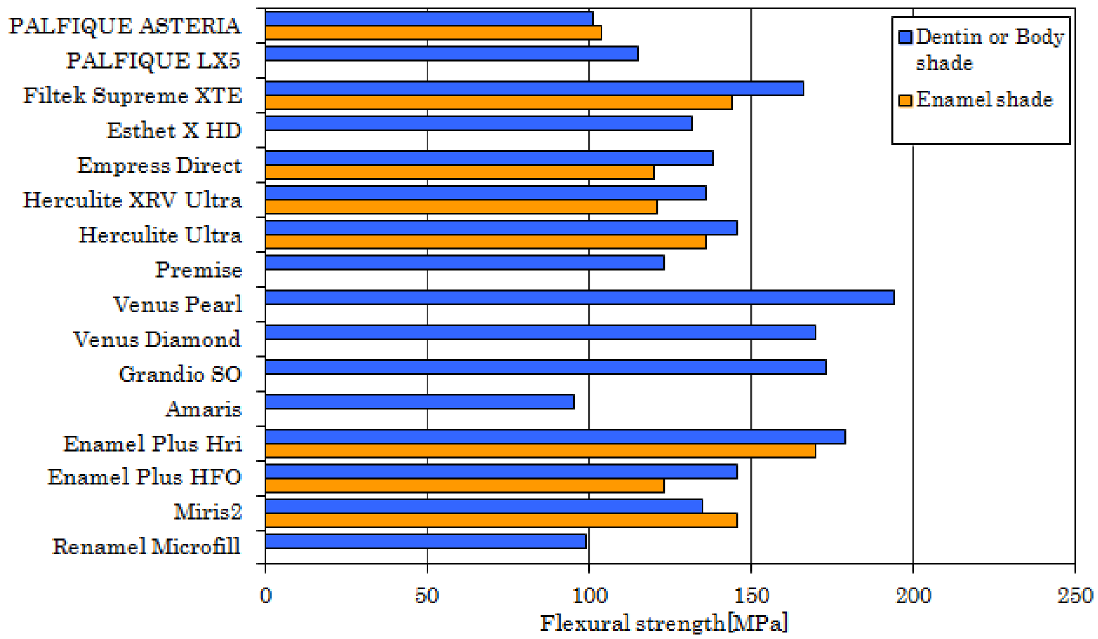


Fig.23 Flexural strength

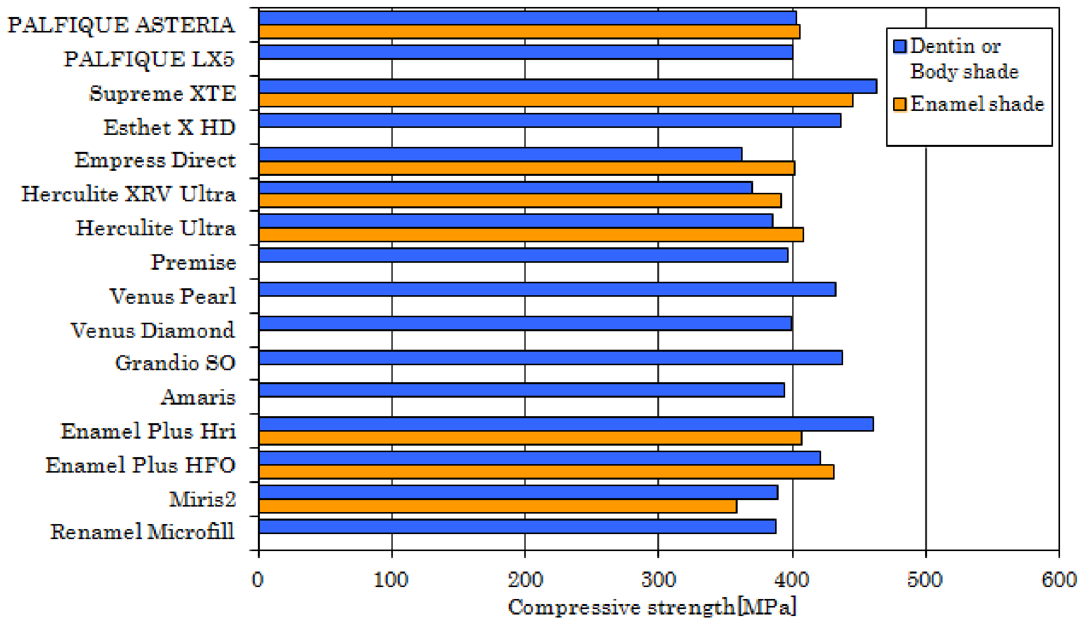


Fig.24 Compressive strength

4.4. Surface Glossiness

Fig.25 shows surface gloss after the surface of cured CR is polished with waterproof abrasive paper (#1500) followed by Soflex super fine (for 60 seconds under running water). Fig.26 shows the relationship between polishing time and surface gloss. The results show that like Estelite Σ Quick®(=PALFIQUE LX5), Palfique Asteria produces extremely high gloss in short polishing sessions.

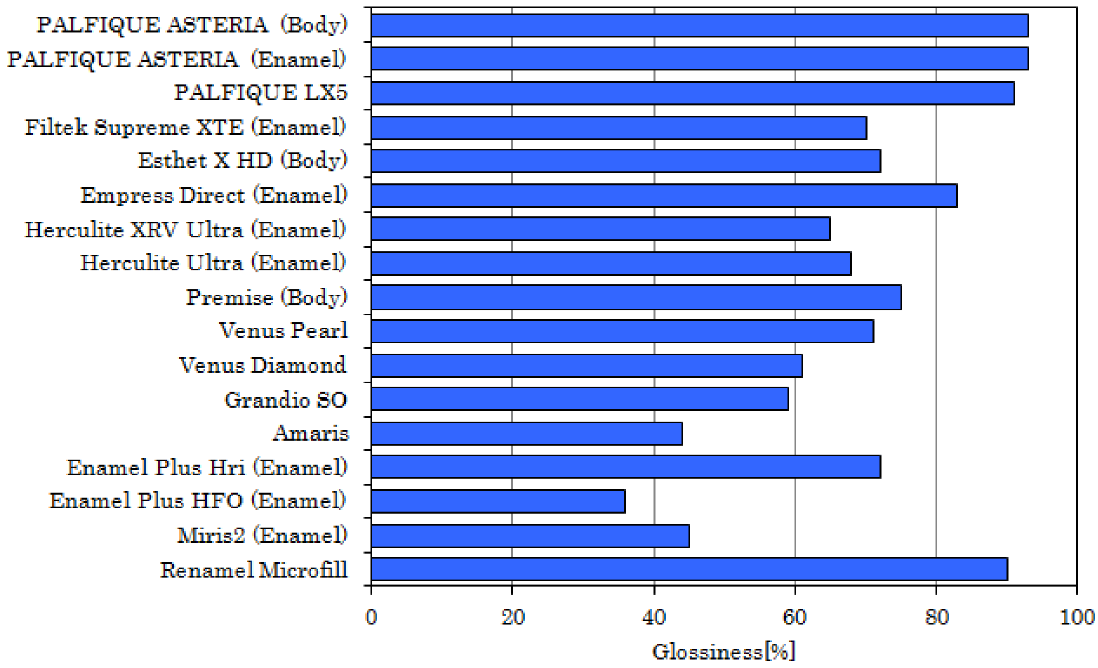


Fig.25 Surface Glossiness

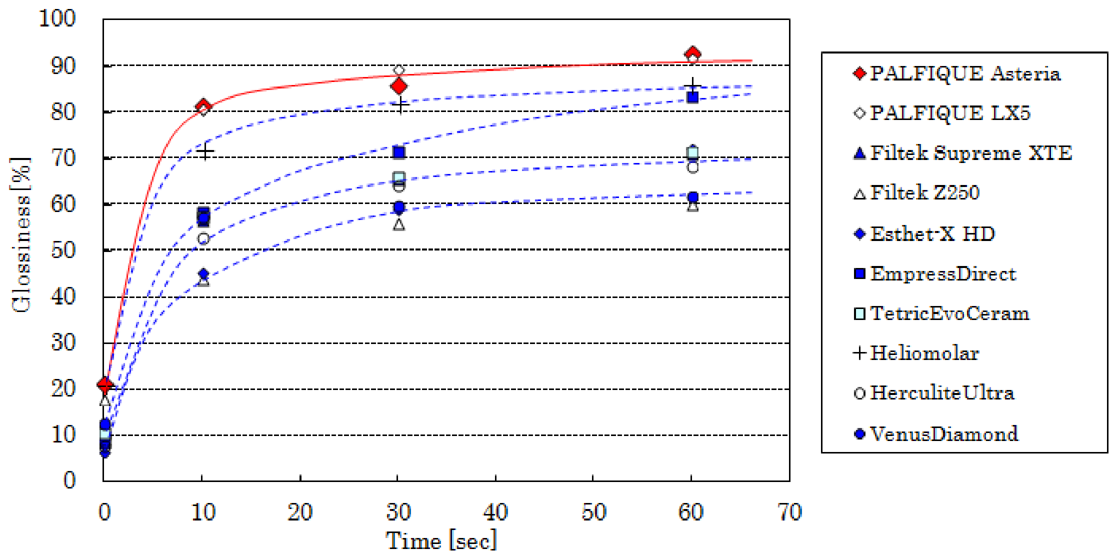
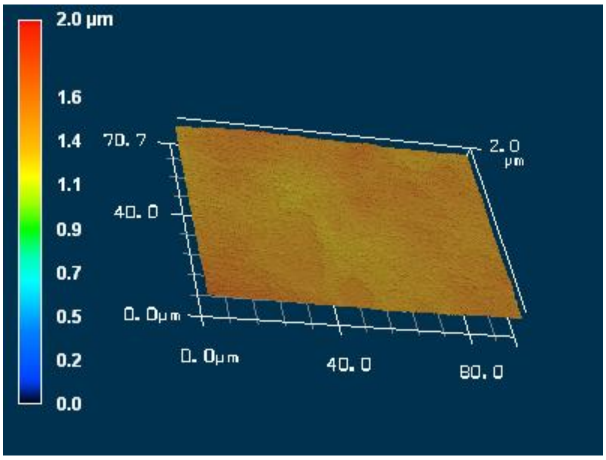


Fig.26 Relationship of glossiness and polishing time（Soflex/superfine）

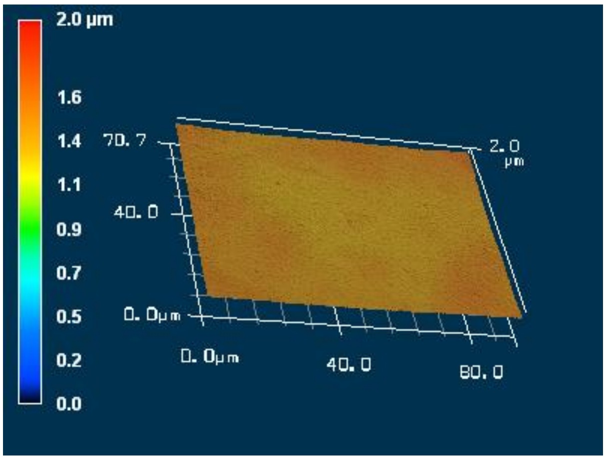
4.5. Gloss Retention

In addition to exhibiting extremely high gloss with relatively short polishing, Palfique Asteria features a remarkably persistent gloss.

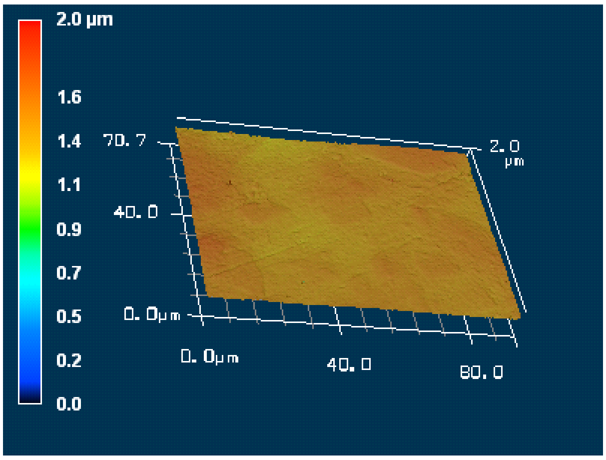
These figures show 3D-images of the surface of cured resin after 10,000 times thermal cycle test (4℃- 60℃). These pictures show that Palfique Asteria keeps its surface smoothness, resulting in glossiness over time (self-shining effect).



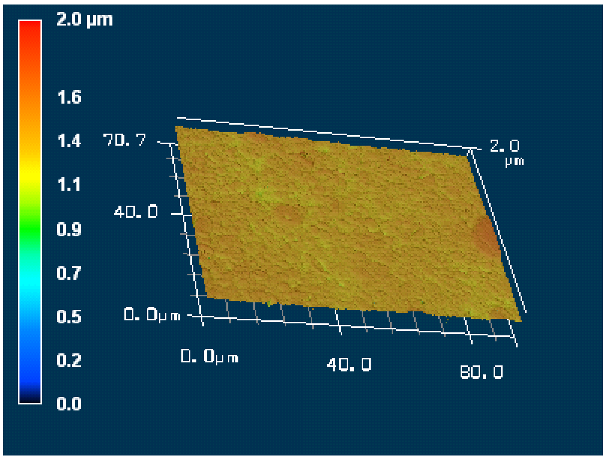
Palfique Asteria (Body)



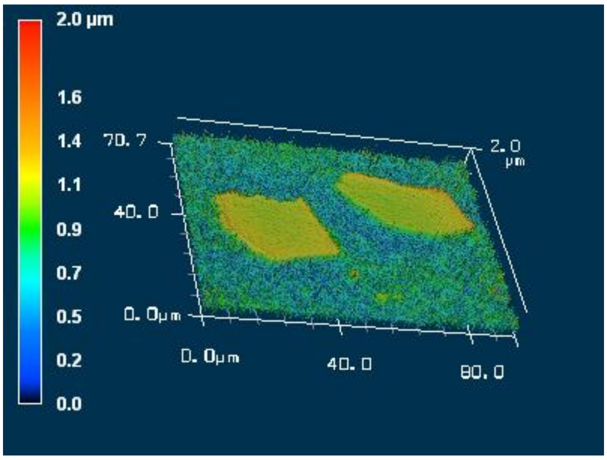
Palfique Asteria (Enamel)



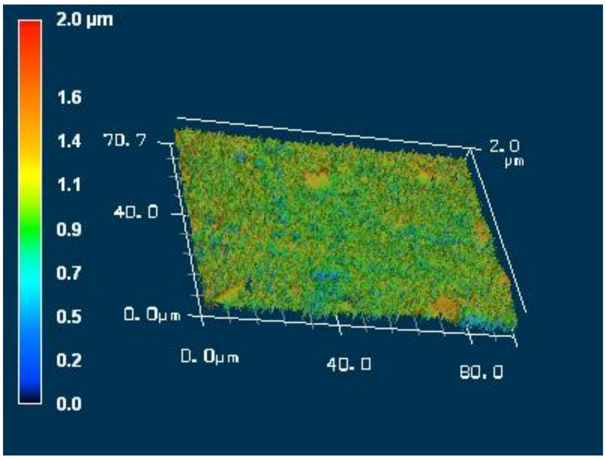
Estelite Σ Quick(=PALFIQUE LX5)



Filtek Supreme XTE



Venus Pearl



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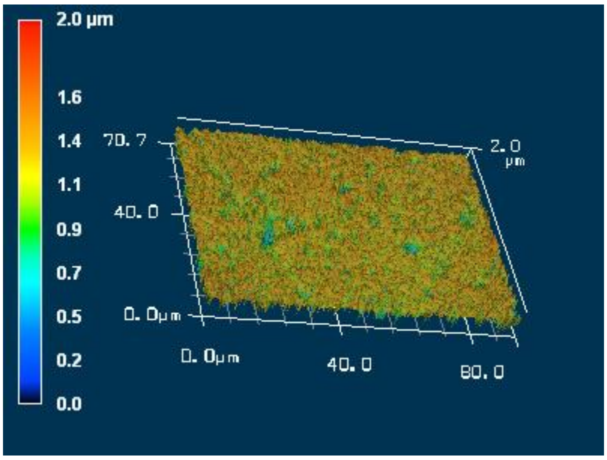
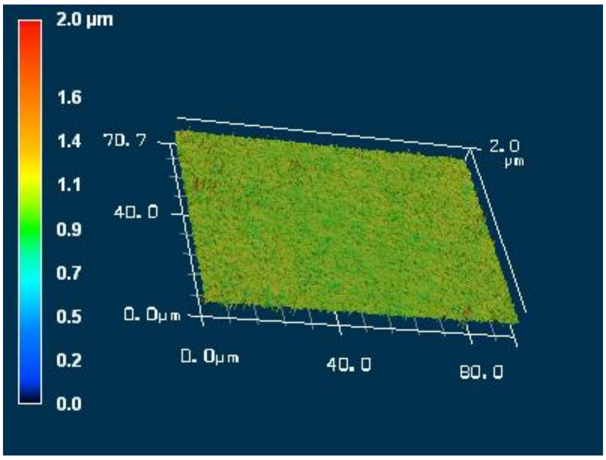


Grandio SO



Enamel Plus Hri

4.6. Changes in color and translucency before and after polymerization

With respect to the shade matching of a composite resin, a resin associated with significant color changes before and after polymerization can present significant restoration issues for color matching, since the actual tooth and the resin cannot be assessed before polymerization. If the color of the composite resin fails to match the color of the tooth substance, the filling must be removed and refilled, a labor-intensive procedure.

Palfique Asteria features relatively low changes in color and translucency before and after polymerization, permitting rough color matching before polymerization. Fig.27, 28 and 29 show the changes in color and translucency for Palfique Asteria and other commercially available composite resins.

As indicated in the figures, Palfique Asteria offers low change in both color and translucency, making shade-matching for Palfique Asteria especially easy. Palfique Asteria can reduce failures caused by colors that diverge significantly after curing.

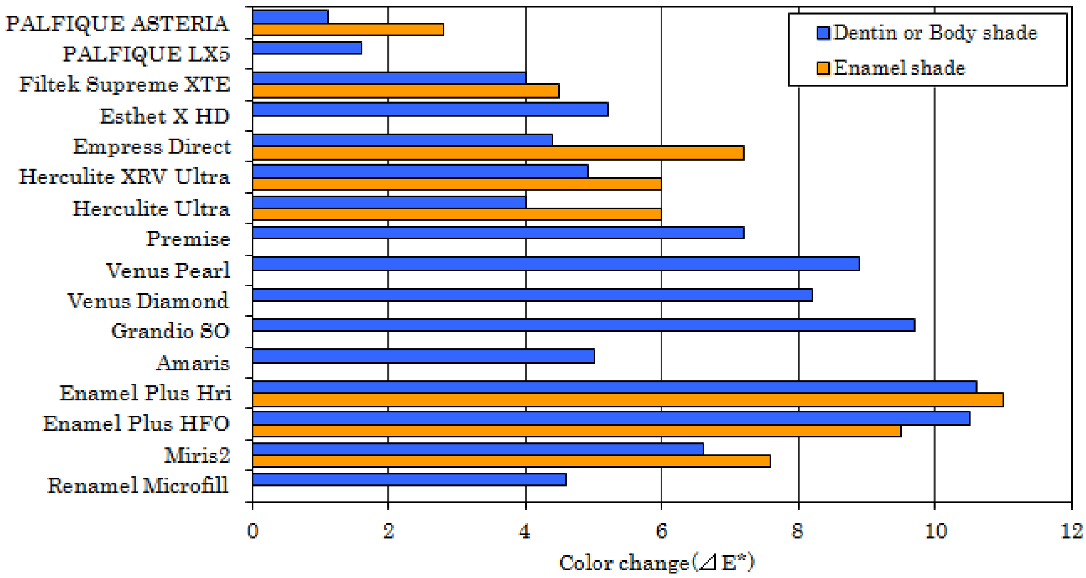


Fig.27 Variation of color tone before and after polymerization

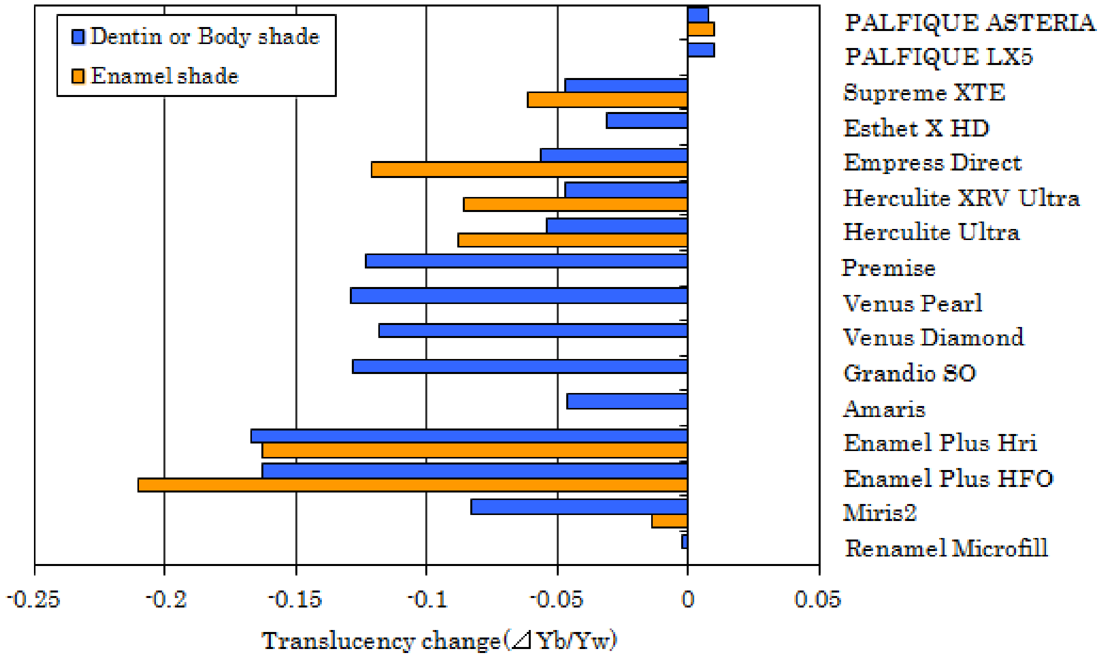


Fig.28 Variation of translucency before and after polymerization

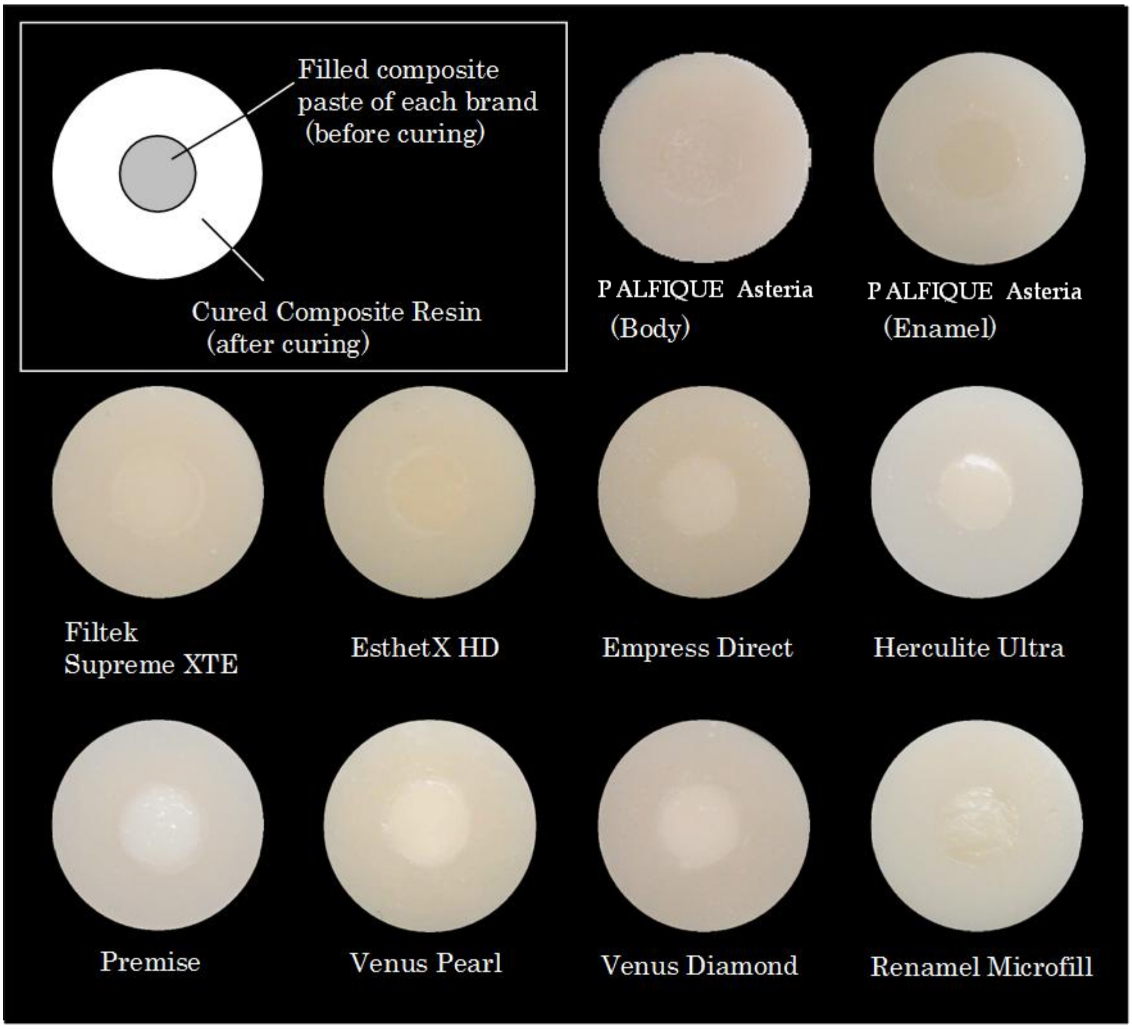


Fig.29 The images of color change before and after polymerization

4.7. Staining by Coffee

A composite resin used in the oral cavity degrades over time due to exposure to various food and drink substances. If this change is pronounced relative to actual teeth, the effect is noticeable and unsightly. Here, we examined potential staining by coffee (24 hours soaking at 80 degrees Celsius). Fig. 30 shows the results.

The extent of staining for Palfique Asteria after soaking in coffee was relatively low among commercially available composite resins. We believe Palfique Asteria will retain its color at the time of restoration over a long term.

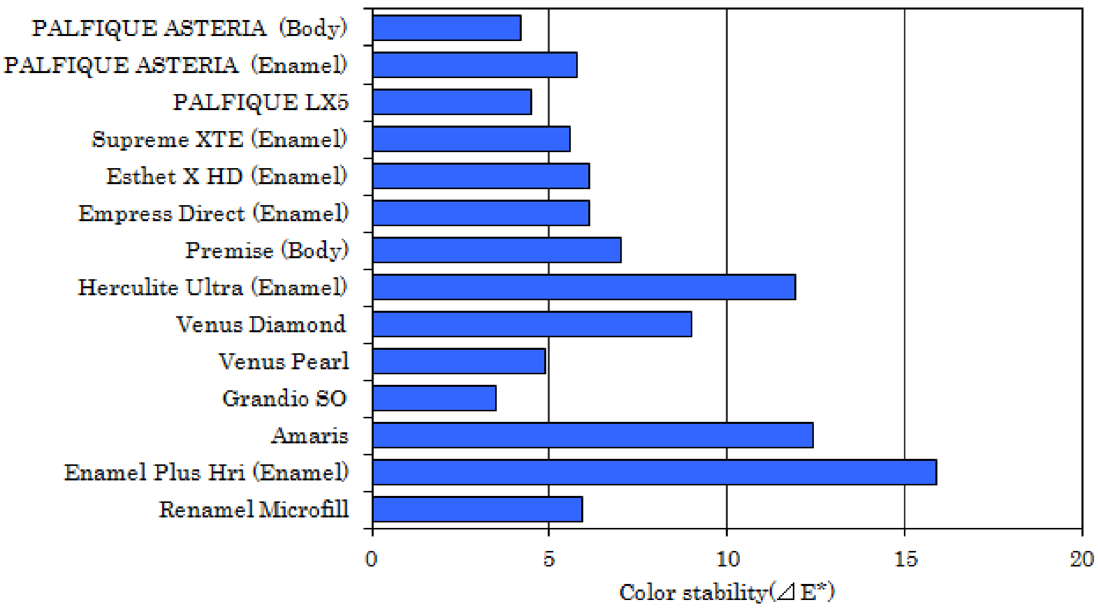


Fig.30 Color stability(⊿E*)

4.8. 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 and significant changes in color and translucency before and after polymerization.

As indicated in 3.2., the inorganic filler used in Palfique Asteria is designed to minimize changes in color and translucency from before to after polymerization and to maximize radiopacity under this constraint. Fig.31 shows the radiopacity of commercially-available composite resins.

The radiopacity of Palfique Asteria is ranked as average among commercially available composite resins, and it meets the levels required to observe prognoses.

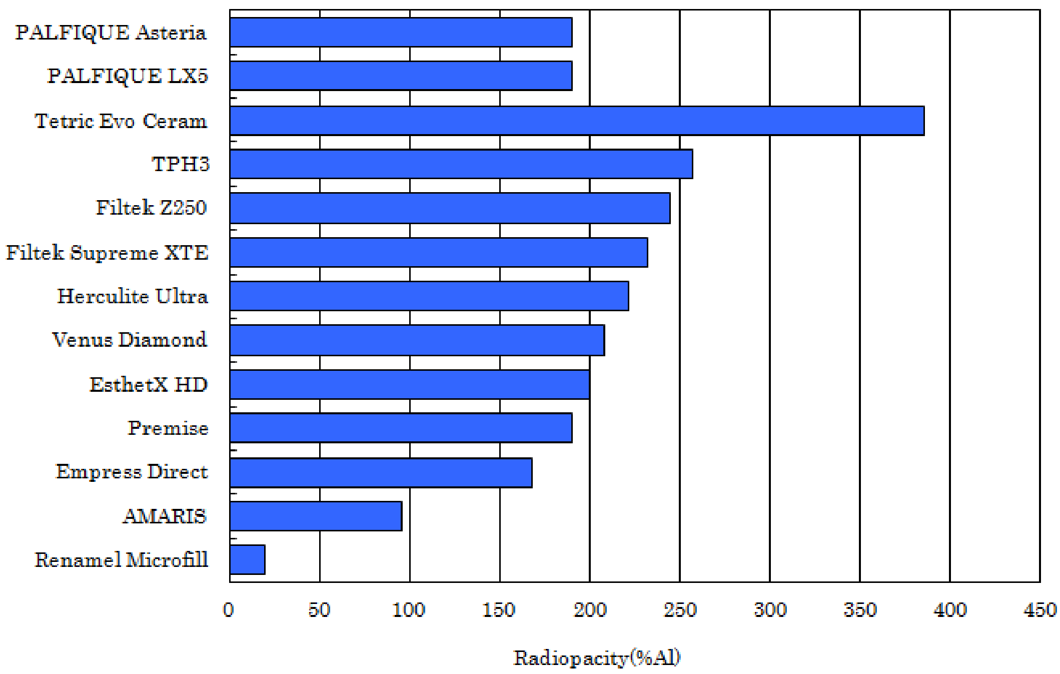


Fig.31 Radiopacity

5. Custom Shade Guide

Palfique Asteria Custom Shade Guide is a shade guide kit for making your own custom shade guides. Since the shade guide of two kinds of form is producible with this kit, a shade can be checked with the form similar to clinical case.

One is deep and narrow form and is suitable for production of the body shade. Another is shallow and large form and is suitable for production of the enamel shade.

Moreover, the produced shade guide tip can be equipped and saved in a holder.

Fig.31 Custom shade guide

Fig.32 Example(NE, A2B)

6. Summary

Palfique Asteria is a composite resin offering various outstanding traits, including desirable levels of polymerization activity and cosmetics thanks to the polymerization catalyst technology (RAP technology) and the Supra-nano Spherical filler technology. Moreover, Palfique Asteria introduces a new simplified 2-step layering concept.

(1) Outstanding esthetics

- Palfique Asteria has optimal shades for 2-step layering concept.
- Palfique Asteria provides high gloss with little polishing.
- Palfique Asteria exhibits high gloss retention.
- Palfique Asteria exhibits minimal changes in translucency and color before and after polymerization.

(2) Fast curing

- Palfique Astria cures in approximately 1/3 the exposure time required for conventional composite resins.
- Palfique Asteria does not require a specific type of light source for the light-curing unit; it cures rapidly under halogen, LED, or Xenon light sources.

(3) Excellent mechanical properties

- Palfique Asteria features low shrinkage.
- Palfique Asteria offers superior characteristics with respect to wear resistance and opposing tooth wear.

(4) Ease of use

- Palfique Asteria is less sensitive to ambient light than conventional products.
- Readily sculpted

7. References

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