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Introduction

Building upon over 40 years of innovation in the field of restorative dentistry, 3M ESPE created, at the turn of this century, a new category of dental material—the nanocomposite. Through precise manipulation of the filler architecture at the nanoscale, 3M ESPE developed a breakthrough composite filling material that significantly advanced the clinical performance of universal composites.

In 2002, 3M ESPE launched Filtek™ Supreme Universal Restorative. This was the first product that utilized nanotechnology to provide the esthetics of a microfill and the strength of a hybrid. All of the filler particles in this novel composite are surface-modified, bonded nanofillers.

Incorporation of this filler technology into a flowable restorative was realized in 2005, with the introduction of 3M™ ESPE™ Filtek™ Supreme Plus Flowable Restorative. This product was formulated to blend the strength and esthetics offered in 3M™ ESPE™ Filtek™ Supreme Plus Universal Restorative, while still maintaining the “flow-on-demand” handling of 3M™ ESPE™ Filtek™ Flow Flowable Restorative.

Through global customer research and discussions with general dentists and opinion leaders, usage and areas of potential improvements for flowables were identified. Flowable usage has increased over the past 5 years because of new indications, better physical properties, better handling characteristics and, finally, their ease of use. In order for this trend to continue, enhancements were needed. Based on customer feedback, the following improvements are realized with the launch of Filtek™ Supreme Ultra Flowable Restorative.

Improvements

- Lower shrinkage
- Improved esthetics
  - Added fluorescence
  - Improved polish retention
- Delivery
  - Easier-to-read shade designation
  - Capsule and syringe delivery

These improvements were made without compromising the strengths of Filtek Supreme Plus flowable restorative:
- “Flow-on-demand” handling
- Wear-resistance
- Physical properties
- Shade match to the tooth and to Filtek™ Supreme Ultra Universal Restorative
Product Description
Filtek™ Supreme Ultra Flowable Restorative is a low-viscosity, visible light-cured, radiopaque flowable nanocomposite. Bonding to the tooth structure is accomplished through the use of dental adhesive systems, either total-etch or self-etch, that are designed to be compatible with methacrylate composites. Filtek Supreme Ultra flowable restorative can be used alone, after placing a compatible adhesive, or in conjunction with a methacrylate-based restorative such as Z100™ Restorative, Filtek™ Z250 Restorative, Filtek™ P60 Restorative or Filtek™ Supreme Ultra Universal Restorative.

The restorative is packaged in capsules and syringes. It is available in a variety of tooth-colored shades. The shades offered with Filtek Supreme Ultra flowable restorative were designed to coordinate with shades offered with Filtek Supreme Ultra universal restorative.

Indications for Use
Filtek Supreme Ultra flowable restorative is indicated for:
• Base/liner under direct restorations
• Undercut blockout
• Class III and V restorations
• Restoration of minimally invasive cavity preparations
  (including small, nonstress-bearing occlusal restorations)
• Repair of small defects in esthetic indirect restorations
• Pit and fissure sealant
• Repair of resin and acrylic temporary materials

Composition
Filtek Supreme Ultra flowable restorative contains BisGMA, TEGDMA and Procrylat resins. The fillers are a combination of ytterbium trifluoride filler with a range of particle sizes from 0.1 to 5.0 microns, a non-agglomerated/non-aggregated surface-modified 20 nm silica filler, a non-agglomerated/non-aggregated surface modified 75 nm silica filler, and a surface-modified aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles). The aggregate has an average cluster particle size of 0.6 to 10 microns. The inorganic filler loading is approximately 65% by weight (46% by volume).

Filtek Supreme Ultra flowable restorative is a modification of Filtek™ Supreme Plus Flowable Restorative based on customer needs. In a global market research study on flowable restoratives, overall esthetics and shrinkage were identified as key features that would impact product desirability. Also, many respondents believed that materials, made by the same manufacturer and utilizing similar components, work better together. The preference for flowables in capsules (unit dose delivery) was determined to be growing globally.

The formulation improvements of Filtek Supreme Ultra flowable restorative take advantage of the advancements made in filler processing and resins since the introduction of Filtek Supreme Plus flowable restorative. The formulation was modified to improve properties, such as shrinkage, fluorescence and polish retention.
Shades
Filtek Supreme Ultra flowable restorative is available in the same shades as Filtek Supreme Plus flowable restorative. These shades are matched to the VITAPAN® Classical Shade Guide and correspond to shades of Filtek Supreme Ultra universal restorative. The opacity of the shades corresponds to the Body shade opacity, except for the Opaque shade which has the opacity of the Dentin shades in Filtek Supreme Ultra universal restorative.

Shades: A1, A2, A3, A3.5, A4, Opaque A3
B1, B2
C2
D2
W (White), XW (Extra White)

Fluorescence
One additional esthetic property of natural dentition is fluorescence. Fluorescence occurs when energy is absorbed and emitted at a longer wavelength. In teeth, this means the absorption of light in the UV region (350-365 nm) and emitting light in the visible region (400-600 nm with a peak maximum of 440 nm). It is thought that this property contributes to the vitality and lifelike appearance of dentition. In natural teeth, dentin (due to amino acids contained in the collagen) exhibits higher fluorescence than enamel. The fluorescence of Filtek Supreme Ultra flowable restorative is similar to Filtek Supreme Ultra universal restorative. Both fluoresce more than the disc of Filtek Supreme Plus flowable restorative (Figure 1).

Figure 1: Fluorescence
Background
Fillers
Nanocomposites

3M ESPE manufactures many of its fillers using a sol gel process. The sol gel process is a route wherein fillers are made from liquids precursors, or a “sol.” These liquids are chemically and mechanically processed to produce particles. One aspect of this process results in sintering which effectively coalesces primary particles together to form larger filler particles. Sintering can be viewed as a type of melting process whereby the particles are softened, creating a surface which can attach to neighboring particles resulting in a particle-to-particle bond. The sintering process can produce fillers that are highly densified or compacted, as found in 3M™ ESPE™ Z100™ Restorative and Filtek™ Z250 Universal Restorative (Figure 2). In 2002, 3M ESPE discovered a way to modify the sintering process to produce loosely agglomerated nanoparticles, i.e., nanoclusters. Although structurally different from densified particles, these nanoclusters behaved similarly to the densified particles found in other composites in terms of providing a range of particle sizes which allow for high filler loading. This resulted in a material with the strength and wear of hybrids with significantly improved polish retention and optical properties. This technology advance was first used in 3M™ ESPE™ Filtek™ Supreme Universal Restorative (Figures 3 and 4).

Filtek™ Supreme Plus Flowable Restorative was formulated using the same surface-modified zirconia/silica nanocluster filler found in the Filtek Supreme universal restorative Dentin, Enamel and Body shade composition. The nanocluster filler particles consist of loosely bound aggregates of engineered zirconia and silica nanofiller particles. Two non-agglomerated/non-aggregated nanofillers were added to the formulation to reduce the interstitial spacing of the filler particles leading to higher filler loadings. One was the 75 nm non-agglomerated/non-aggregated silica previously found in Filtek Supreme Universal Restorative Translucent shades. The other was a 5-10 nm zirconia particulate which was unique to the flowable composition, and enhanced the radiopacity provided by the nanoclusters. This optimized filler system contributed to the excellent physical, wear and polish properties of Filtek Supreme Plus flowable restorative, while still allowing for the desired flowable handling.
**Filler Improvements in Filtek™ Supreme Ultra Flowable Restorative**

This filler technology was improved again. The manufacturing process, where the clusters are formed, was modified to produce less sintering. Once again, the zirconia/silica nanoclusters are produced in a broad range of sizes enabling high filler loading and are surface-treated to chemically bond them to the resin. As the particles are not as strongly sintered, the cluster size range could be broadened (vs. Filtek Supreme Plus flowable restorative) without adversely affecting properties such as polish retention. These nanoclusters still have the structural integrity to provide strength and fracture resistance. The bonded nanoclusters wear at a similar rate as the surrounding resin matrix. In the SEM (Figure 5), note the shape of the primary nanoparticles are still evident in the clusters. Two additional surface-treated engineered nanoparticles are added that, upon photoinitiation, will crosslink with the resin matrix. These fillers were previously used in Filtek™ Supreme Plus Universal Restorative. The 20 nm non-agglomerated/non-aggregated silica was used in Dentin, Enamel and Body shades. The 75 nm non-agglomerated/non-aggregated silica was used in the Translucent shades and Filtek Supreme Plus flowable restorative formulations.

The fourth filler in this system, ytterbium trifluoride, is added to enhance radiopacity.
Resin system

The key feature of flowable restoratives is their flow properties. The ability to wet the surface of the cavities quickly and with minimal instrumentation defines this filling material category. These materials are used as liners, as well as actual filling materials for small restorations. Besides flowable handling, these materials must possess adequate physical properties, such as strength and wear resistance, to ensure clinical success in a variety of indications.

The challenge to creating these materials is achieving a clinically acceptable balance of properties. Frequently modifications that produce a flowable restorative (lower viscosity) adversely affect strength, wear resistance and shrinkage.

Both resin composition and filler loading contribute to shrinkage. Methacrylate composites inherently shrink during polymerization. The amount of shrinkage is impacted by the monomers used. Generally speaking, low viscosity monomers are low molecular weight. Low molecular weight monomers can cause the polymerized resin matrix of the composite system to be harder because the higher number of double bonds per unit of weight enables higher conversion and crosslinking. However, it can also lead to higher shrinkage.

Typically, in order to reduce shrinkage in a composite, manufacturers increase filler loading. The lower the viscosity of the resin system, the more filler can be added to the resin before the viscosity becomes stiff. In the case of flowable restoratives, increasing filler loading is not usually a viable option to reduce shrinkage because the flow properties diminish.

The challenge the Filtek™ Supreme Ultra Flowable Restorative development team faced was how to reduce the shrinkage without adversely affecting the final composite hardness and the flow. The resin system of Filtek™ Supreme Plus Flowable Restorative is a combination of two (2) high-molecular weight monomers BisGMA (2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane) and BisEMA(6) (2,2-Bis[4-methacryloxypropoxyphenyl]propane), and one low-molecular weight monomer TEGDMA (Tri[ethylene glycol]dimethacrylate).

![BisEMA(6) Molecule](image)

![TEGDMA Molecule](image)

TEGDMA is a low-viscosity monomer which can contribute to higher shrinkage but helps create a hard resin matrix. TEGDMA is present in a relatively high concentration to keep the viscosity of Filtek Supreme Plus flowable restorative low enough to flow, while containing a high enough filler load to impart good strength and wear resistance. The shrinkage of Filtek Supreme Plus flowable restorative, while higher than universal restoratives, was similar to other flowables.
To reduce the shrinkage, a substitute for TEGDMA needed to be found. The team identified a monomer, Procrylat (2,2-bis[4-(3-methacryloxypropoxy)phenyl]propane), which is high molecular weight but low viscosity. The high-molecular weight monomer is similar to BisGMA. The difference between BisGMA and Procrylat is the lack of pendant hydroxyl groups. The lack of hydroxyl groups reduces the viscosity of this monomer due to decreased hydrogen bonding potential.

In Filtek Supreme Ultra flowable restorative, the new monomer replaces all of the BisEMA(6) and a portion of the TEGDMA and BisGMA monomers. This optimized combination of monomers results in a low-viscosity resin system which allows for a similar filler loading as Filtek Supreme Plus flowable restorative. While the strength and wear resistance is similar for the two products, the shrinkage of Filtek Supreme Ultra flowable restorative is significantly less than its predecessor.

In addition, a dimethacrylate polymer is added as rheology modifier to produce the “flow-on-demand” feature as in Filtek Supreme Plus flowable restorative. This additive enables the viscosity of Filtek Supreme Ultra flowable restorative to change when manipulated, offering greater control during placement. Many flowable composites will run when subjected to gravitational force. This property makes control of the restorative difficult in certain indications such as Class V restorations or liner indications. Filtek Supreme Ultra flowable restorative holds its shape under these conditions. However, when a shear force is applied to Filtek Supreme Ultra flowable restorative, such as during extrusion through the dispensing tip and manipulation on the preparation, the viscosity of the restorative decreases. This means Filtek Supreme Ultra flowable restorative becomes more fluid, allowing for easy adaptation to the preparation. Once this shear force is removed, such as when manipulation stops, Filtek Supreme Ultra flowable restorative returns to its original, non-slumping viscosity.
Physical Properties

Volumetric shrinkage

A method for determining polymerization shrinkage was described by Watts and Cash (Meas. Sci. Technol. 2(1991), 788-794). In this method, a disc-shaped test specimen and uncured paste are sandwiched between two glass plates and light cured through the lower rigid plate. The flexible upper plate is deflected during the polymerization of the test specimen. The less the flexible plate bends, the lower the shrinkage. Deflection is measured and recorded as a function of time. Although this process actually measures linear shrinkage, volumetric shrinkage was closely approximated due to the fact that the dimensional changes were limited to the thickness dimension. The lower the value, the less the shrinkage.

In this test, samples were exposed for 60 seconds to a 3M™ ESPE™ Visilux™ 2 Visible Light Curing Unit. The final shrinkage was recorded 4 minutes after the end of light exposure.

Polish retention

**Toothbrush abrasion**

Composite materials were shaped into tiles and thoroughly cured. The surfaces were polished wet using a Beuhler variable-speed grinder-polisher to remove the air-inhibited layer and to ensure a uniform surface. They were stored in water at 37°C for 24 hours. Gloss was measured. The samples were brushed with toothpaste and a toothbrush that was mounted on an Automatic Toothbrush Machine. Gloss measurements were taken after 500 cycles and then every 1000 cycles. The test was terminated after 6000 toothbrush strokes.

In-vitro, 3-Body wear

The wear rate was determined by an in-vitro 3-body wear test. In this test, composite (1st body) is loaded onto a wheel, which contacts another wheel, which acts as an “antagonistic cusp” (2nd body). The two wheels counter-rotate against one another dragging abrasive slurry (3rd body) between them. Dimensional loss during 200,000 cycles is determined by profilometry at regular intervals (i.e., after every 40,000 cycles). As the wear in this method typically follows a linear pattern, the data is plotted using linear regression. The wear rates, i.e., the slope of the lines, are determined. The comparison of rates reduces some of the variability in the test due to sample preparation and can be predictive of anticipated wear beyond the length of the actual test.

Fracture toughness

The values reported for fracture toughness (K1c) are related to the energy required to propagate a crack. In this test, a short bar of material is cured. A notch is cut into it. The bar is placed on a fixture that supports either end, and an anvil is positioned above the notch. The anvil presses down until the bar breaks. This is similar to 3-point bend (similar to the fixture that provides flexural strength and modulus data).


Fracture Toughness
Source: 3M ESPE internal data
Compressive and diametral tensile strength

Compressive strength is particularly important because of chewing forces. Rods are made of the material and simultaneous forces are applied to the opposite ends of the sample length. The sample failure is a result of shear and tensile forces.


Diametral tensile strength is measured using a similar apparatus. Compressive forces are applied to the sides of the sample, not the ends, until fracture occurs.

The diametral tensile strength of Filtek Supreme Ultra flowable restorative is higher than Revolution Formula 2, Premise Flow, Heliomolar Flow, SureFil SDR Flow and Tetric EvoFlow.

Flexural strength and modulus

Flexural strength is determined in the same test as flexural modulus. Flexural strength is the value obtained when the sample breaks. This test combines the forces found in compression and tension.


Flexural Strength

Flexural modulus is a method of defining a material’s stiffness. A low modulus indicates a flexible material. The flexural modulus is measured by applying a load to a material specimen that is supported at each end.

The flexural modulus of Filtek Supreme Ultra flowable restorative is comparable to Filtek Supreme Plus flowable restorative, Premise Flow and Grandio Flow.

Flexural Modulus
Field evaluation

A field evaluation was conducted in two countries, Germany and Sweden, to confirm the clinical performance and acceptability of Filtek™ Supreme Ultra Flowable Restorative. Data detailed in this section includes the 133 respondents.

Recruitment criteria were designed to obtain 80% competitive flowable users and 20% Filtek™ Supreme Plus Flowable Restorative users. Approximately 43% of the former were Tetric EvoFlow users. Evaluators received a selection of 6 shades in either capsules or syringes depending on their delivery preference.

Current flowable usage

Sixty-three percent of the respondents use more than one flowable in their practice. Dentists are using flowable for a variety of indications, both as an interior surface material and as a final filling material. The frequency that these types of restorations are done in a week and the frequency a flowable is used in these indications varies significantly. Therefore, the number of times a flowable is used in each indication varies from a high of 12 liner/base applications per week (mean), to a low of 2 porcelain or composite repair restorations per week.

The primary reasons cited for using a flowable composite as a liner/base include:

• Stress relief
• Cavity and marginal adaptation (including the ability of the material to wet the surface, flow, the ability to seal dentin, and to produce a gap-free restoration)
• Handling
• Ease of use
• Shades (either a layer that can be discriminated from dentin, or to have a material that matches surrounding tooth or universal composite)

The primary reasons cited for using flowables as filling materials include:

• They provide a smooth transition from restoration to tooth
• Handling in terms of flow, ability to adapt to the cavity and margins, and the wetting characteristics
• The procedure is faster—finishing and polishing time is reduced
• The procedure is simple—frequently dentists don’t need to touch the material to adapt, and they are easy to apply
Evaluation of Filtek Supreme Ultra Flowable Restorative

There were 5,063 placements made with Filtek Supreme Ultra flowable restorative during the field evaluation.

Thirty percent of the placements were internal surface applications. The remaining 70% of the placements were as a filling material. This distribution did not change for capsule users vs. syringe users. Capsule evaluators accounted for 1,426 placements.

Capsule users found the Filtek Supreme Ultra flowable restorative capsule design acceptable. In all design attributes (i.e., tip access into the prep, ease of dispensing, control of dispensing, visibility into the prep, and overall delivery satisfaction), the new Filtek Supreme Ultra flowable restorative capsule was rated comparable to their current capsule.

Dentists, including Tetric EvoFlow users, rated their satisfaction with the following features significantly higher for Filtek Supreme Ultra flowable restorative than their current, most frequently used flowable.

**Esthetic features:**
- Ease of polish
- Shade blend with surrounding tooth structure
- Shade match with the universal (no matter which universal composite was used)
- Final polish
- Chameleon effect
- Overall esthetic results

**Handling features:**
- Ability to hold shape (stays where I put it, doesn’t slump)
- Cavity adaptation
- Stickiness

**Overall satisfaction with the product:**
More than 50% of the dentists rated the “Flow” and the “Overall esthetic results” as better with Filtek Supreme Ultra flowable restorative than their current, most frequently used flowable. In all features compared, more than 80% of the dentists rated Filtek Supreme Ultra flowable restorative the same or better than their most frequently used flowable.
Questions and Answers

Q. What is the difference between Filtek™ Supreme Plus Flowable Restorative and Filtek™ Supreme Ultra Flowable Restorative?
A. There have been changes made in the filler, pigments and resin system. These changes have provided for lower shrinkage, better polish retention and fluorescence.

Q. Do the shades of Filtek Supreme Ultra Flowable Restorative match Filtek™ Supreme Ultra Universal Restorative?
A. Yes. In addition, the shades of Filtek Supreme Ultra flowable restorative are the same as its predecessor, Filtek Supreme Plus flowable restorative.

Q. What is the source of the almost 20% shrinkage reduction in Filtek Supreme Ultra Flowable Restorative compared to its predecessor?
A. The changes in the resin system components have provided a low viscosity and lower shrinking framework.

References

3. SEMs courtesy of Dr. Jorge Perdigao, University of Minnesota, Division of Operative Dentistry, Department of Restorative Sciences.
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SDev = standard deviation