Tetric® N-Ceram Bulk Fill

The nano-optimized 4-mm composite

Scientific Documentation

ivoclar vivadent®
passion vision innovation

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1. Introduction

1.1 Direct composites

Composite materials became available in dentistry in the 1960s [1]. First, they were mainly used in the anterior region, where the colour of amalgam was deemed unesthetic, however since effective bonding agents became available at the beginning of the 1990s, composites have found increasingly broad use as universal restorative materials. Demand has grown for invisible esthetic restorations in both the anterior and posterior regions of the mouth, which has led to consistent increases in the demand for composite materials.

Developments in restorative dentistry were only possible due to the simultaneous development of clinically reliable enamel/dentin adhesives, thus the composite success story is driven not only by patient demands for more esthetic filling materials but by continual industry-led product development and improvement with regard to the physical, esthetic and handling qualities of both adhesives and composites.

1.1.1 A short history

As the name suggests, composites are comprised of at least two different materials. In most cases this involves inorganic or organic fillers which are embedded in an organic matrix. The first step in the development of composite materials was achieved by Bowen in 1962 with the synthesis of a Bis-GMA monomer-formulation filled with finely ground quartz. [1] At the time, only chemically-cured two-component resin-based materials were available. With the advent of photo-polymerisation, UV-cured systems were introduced [2] and in the late 1970s, the first report on a dental filling material that could be cured with visible blue light was published. [3] Direct composites were historically somewhat limited with regard to large posterior restorations due to accelerated wear and polymerisation shrinkage issues. Thus in the 1980s the first generation of indirect (lab-based) composites was introduced. These were/are modelled and cured extra-orally in units capable of delivering higher intensities of light/heat than would be possible intra-orally. The bulk-fill type of direct composites specifically designed for large posterior restorations, represent a new era in direct-filling- technology and a paradigm shift away from the traditional 2 mm increment system.

1.1.2 Monomer technology

Monomers, together with initiators, catalysts and other additives, form the reactive part of a dental composite restorative. The monomers compose the matrix of a composite material. They must be stable in the oral environment, exhibit shade stability and low polymerisation shrinkage (high molecular weight). High molecular, multi-functional (mostly bi-functional) methacrylate compounds have proven most suitable for this purpose.

Bis-GMA (bisphenol-A-diglycidyl-dimethacrylate) was first synthesised and introduced in the early sixties and is one of the most frequently used monomers. Due to a propensity for water absorption, which can lead to swelling and discoloration, it tends to be used in relatively small amounts and mixed with other methacrylates. Most resin matrices consist of dimethacrylate mixtures. Dimethacrylate refers to methacrylates with two polymerisable methacrylate groups. UDMA (urethane dimethacrylate) is another common compound, and has the advantage of having a lower viscosity than Bis-GMA. It can therefore be used undiluted and as UDMA has no hydroxyl side groups (OH groups), it exhibits low water absorption. Modern composite materials usually consist of low viscous dimethacrylates in combination with Bis-GMA.[4] The overall monomer content of a composite, accounts for approximately 12 - 40% of the mass, depending on the characteristics of the product.

1.1.3 Filler technology

Fillers are responsible for imparting composites with the adequate strength to withstand the stresses and strains of the oral cavity and to achieve acceptable clinical longevity. Composite restoratives tend to be classified according to their filler composition i.e. macrofilled, microfilled or hybrid composites. Macrofilled composites predominantly contain glass fillers with a mean particle size of >3 µm. Microfilled composites mainly contain filler particles with a mean diameter of less than 100 nm and today such fillers are called nanofillers. In hybrid composites, the spaces between the coarse filler particles, which usually have a diameter of less than 1 µm, are occupied by microfillers. Fillers in differing types, sizes and concentrations determine the translucency, strength, opalescence and radiopacity of a material and are crucial for reducing wear and polymerisation shrinkage as their inclusion enables the reduction of the monomer content.

Macrofillers
The first composites contained just macrofillers. These macrofilled composites exhibited favourable shrinkage behaviour and flexural modulus, but their surface properties were inadequate and their wear properties poor. In essence, they were clinically unsuccessful. [5]

Microfillers
In 1974 a patent was granted to Ivoclar Vivadent for a composite employing microfillers.[6] Microfilled composites heralded a breakthrough as they were the first materials to be sufficiently wear resistant whilst maintaining good surface quality in the mouth. Microfillers could not however overcome two essential problems: Firstly, they could not reinforce a composite material as effectively as macrofillers, resulting in low flexural strength and low flexural modulus; and secondly, microfillers severely increase the viscosity of a composite due to their high specific surface area, meaning only limited amounts can be used. As a result, microfilled composites exhibit high polymerisation shrinkage. This can however be largely overcome by preparing an initial microfilled composite which is pre-polymerised and then ground to a fine powder and employed as a filler in the final dental material. These organic polymer fillers can be termed “Isofillers”. Ivoclar Vivadent used this filler technology as early as the development of Heliomolar. Microfilled composites typically demonstrate higher wear resistance than other types of composite materials because of the smaller size of the particles.[7]

Hybrid-fillers
Hybrid composites represented the next logical step in composite development. As the term 'hybrid' suggests, a variety of different fillers are employed to optimally combine the properties of all types of fillers, further improving the mechanical properties of the final material. This allows for a very high filler load, resulting in high physical strength and reduced polymerisation shrinkage. This technology was employed in the creation of the micro-hybrid products Tetric and Tetric Ceram and the nano-hybrid materials Tetric N-Ceram and Tetric N-Ceram Bulk Fill.

1.1.4 Bulk fill composites

Composites with improved depth of cure and reduced shrinkage characteristics for bulk fill purposes have been around for some years. In 2008, Polydorou et al [8] published an *in vitro* study in which the depth of cure of two translucent composites were evaluated. Independent of the light source (LED or halogen) they showed that adequate curing of QuiXfil/Dentsply samples was possible to a depth of 3.5 to 5.5 mm. Using the same method with micro-filled composites they achieved a depth of cure of just 2.5 mm.

It is important to note that bulk fill materials do not constitute a uniform class of materials. While the ability to apply the material in thick increments is a common theme, there are differences in clinical application and the way in which the fillings are built up. A selection are summarised in the table below:

Product	**Manufacturer**	**Consistency**	**Increment Thickness**	**Application**
Tetric N-Ceram Bulk Fill	Ivoclar Vivadent	Sculptable	4 mm	Single layer possible
QuiXX	Dentsply	Sculptable	4mm	Single layer possible
x-tra Fil	Voco	Sculptable	4 mm	Single layer possible
Beautifil Bulk Restorative	Shofu	Sculptable	4 mm	Single layer possible
Venus Bulk Fill	Heraeus Kulzer	Flowable	4 mm	Over-layered with conventional composite
SDR	Dentsply	Flowable	4 mm	Over-layered with conventional composite
SonicFill	Kerr	Flowable, sound activated, sculptable	5 mm	Single layer possible
x-tra base	Voco	Flowable	4 mm	Over-layered with conventional composite
Filtek Bulk Fill	3M Espe	Flowable	4 mm	Over-layered with conventional composite
Beautifil Bulk Flowable	Shofu	Flowable	4 mm	Over-layered with conventional composite

Table 1: Summary characteristics of various bulk fill composites

All bulk fill composites need to exhibit low shrinkage stress and thus good marginal integrity, adequate resistance to chewing forces in the posterior region, adequate working time in ambient light, adequate radiopacity, plus good polishing properties and aesthetics.

The sculptable (non-flowable) materials can be applied in one increment and moulded and sculpted to mimic the natural tooth topography. Flowable materials are unsuitable for single layer fillings however as they cannot be sculpted at the surface, they need to be over-layered by a conventional composite in order to model cusps and create life-like morphology.

2. Tetric N-Ceram Bulk Fill

Taking composite technology to the next level, Tetric N-Ceram Bulk Fill was developed to follow on from the clinically reliable, universal composite Tetric N-Ceram. Tetric N-Ceram Bulk Fill is a light-cured, hybrid composite for direct restorations (Class I and II) in posterior teeth, and may also be used for Class V restorations, extended fissure sealing in molars and premolars and for reconstructive build-up. Tetric N-Ceram Bulk Fill can be applied in “bulk” increments of up to 4 mm without any adverse effect on the material's polymerisation behaviour or mechanical properties. It can be cured with conventional LED curing lights and also in just 10 seconds using a light source with > 1000 $mWcm^2$, such as Bluephase N®.

For years the accepted state of the art method has been to apply thin layers (up to 2 mm) of composite on top on one another, which are successively cured. This was deemed necessary to avoid unnecessary polymerisation shrinkage. The curing of 4 mm increments represents a paradigm shift in dentistry. Tetric N-Ceram Bulk Fill achieves this via incorporating advanced composite-filler technology, a pre-polymer shrinkage stress reliever, the photo initiator Ivocerin® (polymerisation booster), and a light sensitivity filter.

2.1 Monomer technology

Tetric N-Ceram Bulk Fill contains the same dimethacrylates as Tetric N-Ceram: Bis-GMA, Bis-EMA and UDMA. As with all composites, these are converted into a cross-linked polymer matrix during the polymerisation process. The organic matrix of Tetric N-Ceram Bulk Fill accounts for approximately 21% of the mass. Bis-GMA, Bis-EMA and UDMA exhibit low polymerisation shrinkage by volume. Both Tetric N-Ceram and Tetric N-Ceram Bulk Fill are the result of a coordinated optimised mixture of monomer matrix and fillers.

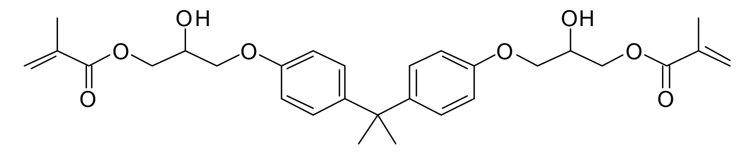
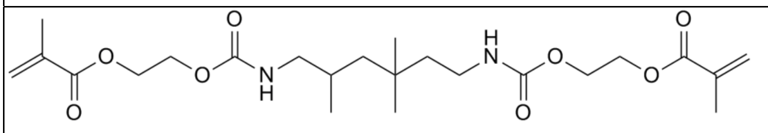
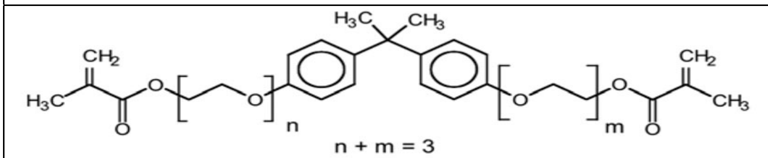
	**Bis-GMA** Bisphenol A-diglycidyl dimethacrylate
	**UDMA** Urethane dimethacrylate
	**Bis-EMA** Ethoxylated bisphenol A dimethacrylate

Table 2: Table illustrating the structural formulae for the monomers used in Tetric N-Ceram Bulk Fill

2.2 Filler technology

The filler technology behind Tetric N-Ceram Bulk Fill is also based on that of the clinically proven Tetric N-Ceram. Tetric N-Ceram Bulk Fill incorporates several different types of filler (barium aluminium silicate glass with two different mean particle sizes, an „Isofiller“, ytterbium fluoride and spherical mixed oxide) in order to achieve the desired composite properties. Tetric N-Ceram Bulk Fill has an overall standard filler content of approximately 61% (vol.) and 17% polymer fillers or „Isofillers”. Illustrations of the various fillers contained in Tetric N-Ceram Bulk Fill are shown below:

Glass fillers

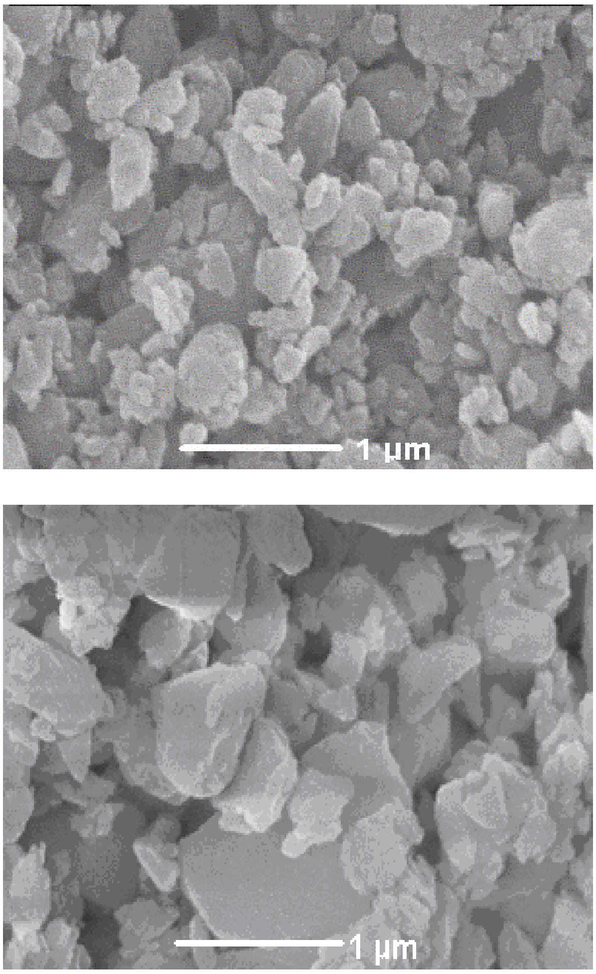


Fig. 1a,b: Barium aluminium silicate glass fillers with a mean particle size of 0.4 µm (top picture) and 0.7 µm (bottom picture) as used in Tetric N-Ceram Bulk Fill.

Isofiller

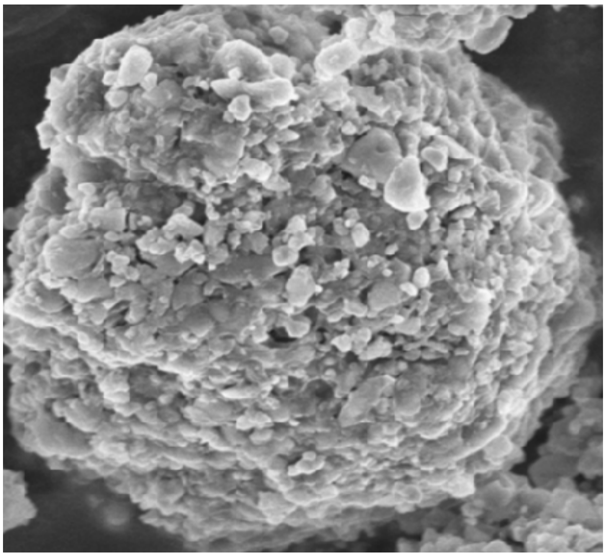


Fig. 2: *Isofiller* composed of cured dimethacrylates, glass filler and ytterbium fluoride

Ytterbium fluoride

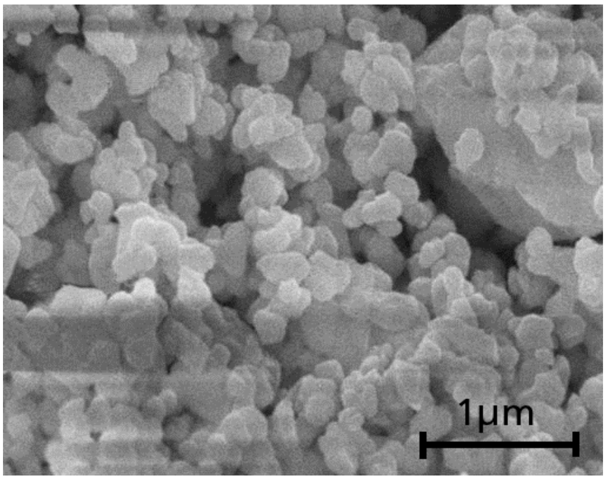


Fig. 3: Ytterbium fluoride with a mean particle size of 200 nm

Spherical mixed oxide

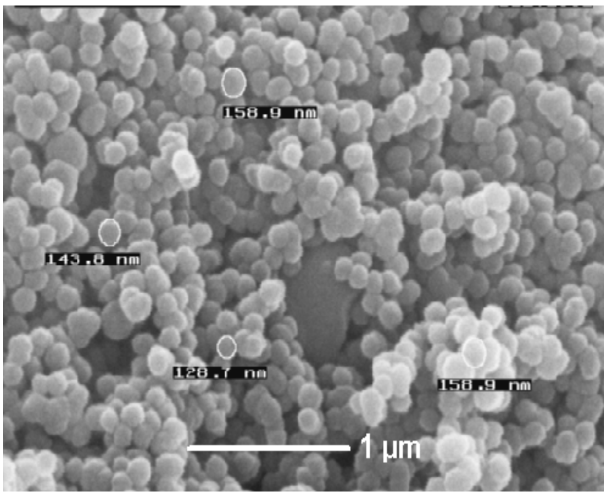


Fig 4: Mixed oxide with a mean particle size of 160 nm

Glass fillers result in low wear and favourable polishing properties i.e. low surface roughness and high gloss. “Isofillers” are instrumental in lowering the shrinkage and shrinkage stress. Tetric N-Ceram Bulk Fill utilises a specially designed shrinkage stress relieving “Isofiller” which is discussed in more detail in section 2.2.2. Ytterbium fluoride confers high radiopacity to dental materials and is capable of releasing fluoride. Spherical mixed oxide provides the basis for reduced wear and favourable consistency. The spherical particles minimise the thickening effects of fillers, as they provide the largest volume with the smallest surface area possible. Primary particles, (individual bodies) and secondary particles (agglomerates) combine to form the ideal consistency. Mixed oxide also provides aesthetic advantages, as the refractive index is matched to that of the matrix (polymer) meaning light can pass through the medium (restoration) unhindered. This results in restorations that are virtually indiscernible from the surrounding tooth structure.

Fig. 5: Illustration of different refractive indices in water (left) and monomer mixture with coordinated refractive index (right)

The picture above illustrates the principle of coordinating the refractive indices of the fillers and the matrix. The glass on the left contains water with a refractive index of 1.33. The glass on the right contains a monomer mixture with a refractive index set at 1.51 i.e. the same as the glass rod. Thus if the refractive index of the fillers corresponds to that of the matrix as in the right hand glass, the structure is virtually invisible as the light is not refracted differently.

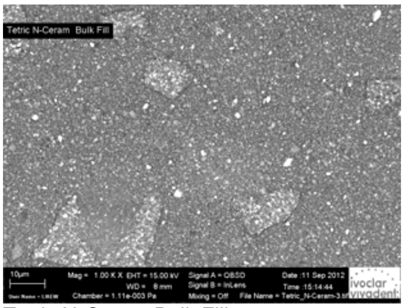
The pictures below show the aesthetic results possible with Tetric N-Ceram Bulk Fill, the bulk fill restorations shown in Fig. 6b that replaced the amalgam restorations of Fig. 6a, are virtually indiscernible from the surrounding tooth structure.

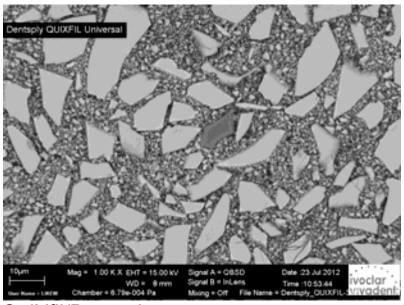
Fig 6a, b: Replacement of original Class I posterior amalgam fillings (left) with Tetric N-Ceram Bulk Fill restorations (right).

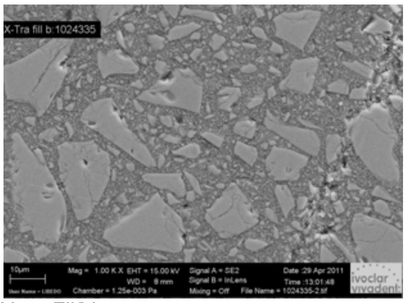
(Dr Eduardo Mahn, Santiago, Chile)

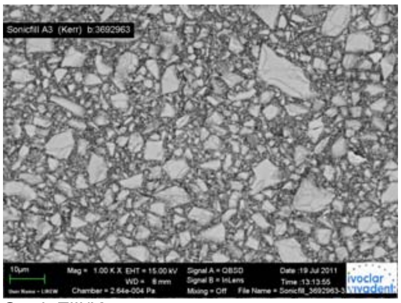
2.2.1 Filler size and polishability

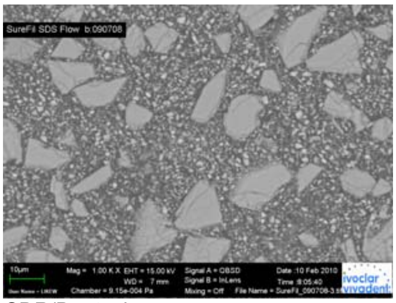
The mix and size of the fillers are responsible for the excellent polishability and high gloss of Tetric N-Ceram Bulk Fill. It comprises fillers of comparatively small size as large fillers are unable to produce the same smooth, glossy surface as small fillers. The scanning electron microscope (SEM) images below show the clear differences in filler size of Tetric N-Ceram Bulk Fill (top left) compared to other composite materials.

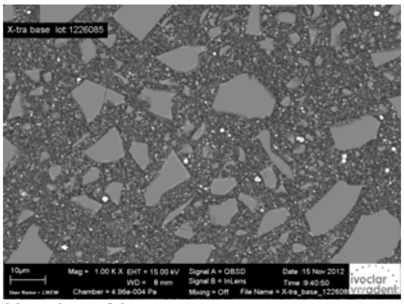

Tetric N-Ceram Bulk Fill

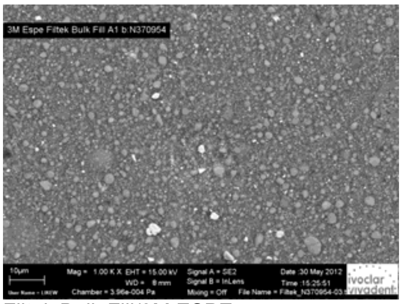

QuiXfil/Dentsply


X-tra Fil/Voco


SonicFill/Kerr


SDR/Dentsply


X-tra base/Voco


Filtek Bulk Fill/3M ESPE

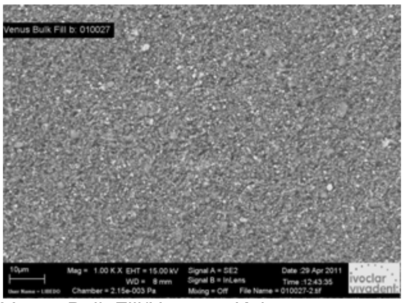

Venus Bulk Fill/Heraeus Kulzer

Fig. 7a-h: SEM images of various composite materials. *(R&D Ivoclar Vivadent, 2011)*

Apart from Filtek Bulk Fill/3M Espe (bottom, left) and Venus Bulk Fill/Heraeus Kulzer (bottom middle), all the other materials contain relatively large fillers. This correlates with the polishing results shown in section 4.5.

2.2.2 Shrinkage stress reliever

Tetric N-Ceram Bulk Fill can be applied in increments of up to 4 mm. Clearly reducing polymerisation shrinkage is one of the most important issues here. Composite resins shrink during polymerisation which was the original rationale behind applying composites in 2 mm increments with successive polymerisation intervals. Problems associated with polymerisation shrinkage include marginal discoloration, marginal gaps, secondary caries, cracking and hypersensitivity. Shrinkage stress in Tetric N-Ceram Bulk Fill is however kept to a minimum. A special patented filler which is partially functionalised by silanes, acts as a unique shrinkage stress reliever. The following diagram illustrates this mechanism:

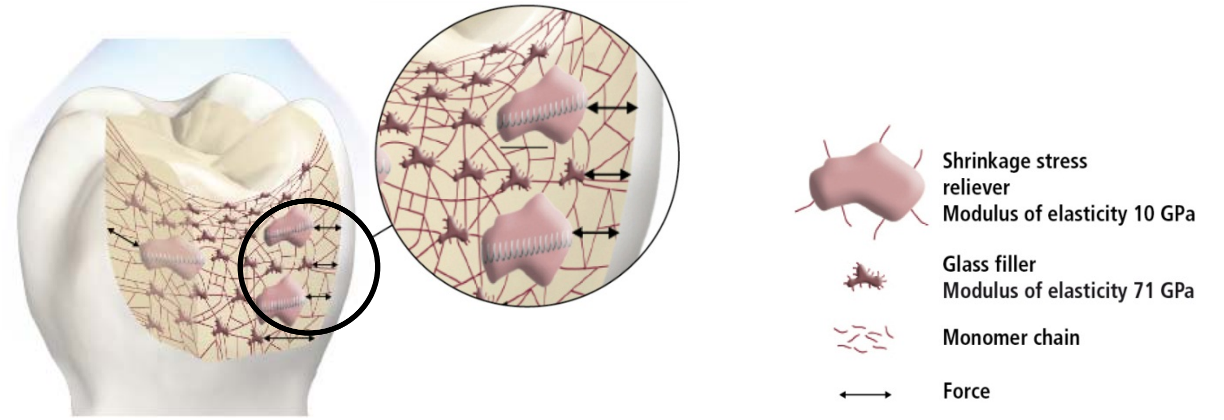


Fig. 8: Schematic representation of the shrinkage stress reliever in a Tetric N-Ceram Bulk Fill restoration acting like a spring and reducing stress within the restoration

When the composite is cured, the monomer chains located on the fillers together with the silanes begin a cross-linking process and forces between the individual fillers come into play and place stress on the cavity walls. This stress is influenced by both volumetric shrinkage and the modulus of elasticity of the composite. A high modulus of elasticity denotes inelasticity and a low modulus of elasticity denotes higher elasticity. Due to its low elastic modulus (10 GPa) the shrinkage stress reliever within Tetric N-Ceram Bulk Fill acts like a spring (expanding slightly as the forces between the fillers grow during polymerisation) amongst the standard glass fillers which have a higher elastic modulus of 71 GPa. The shrinkage stress reliever essentially “holds on” to the cavity walls along with the matrix and the adhesive. The silanes bonded to the filler particles improve the bond between the inorganic filler (glass and quartz particles) and the monomer matrix as they are able to establish a chemical bond between the glass surface and the matrix. Ultimately, the volumetric shrinkage and shrinkage stress in Tetric N-Ceram Bulk Fill are reduced during polymerisation – allowing increments of up to 4 mm to be placed whilst ensuring a tight marginal seal.

2.3 Polymerisation technology

Light-curing composites “set” by way of free radical polymerisation. Incoming photons from the curing light are absorbed by photoinitiators. The energy absorbed excites the molecules, and enables the formation of free radicals (if one or several activators are present) and this triggers polymerisation. The darker and/or the more opaque a material is, the shallower the depth of cure because less light can reach the initiators within the composite. It is often not possible to polymerise thick increments reliably unless the material is highly translucent or contains somewhat limited amounts of light-refracting fillers. Conventional initiator systems alone are unable to cure increments exceeding 2 mm reliably.

Initiator molecules are only able to absorb photons within a specific spectral range. Camphorquinone is an initiator widely used in polymer synthesis. It is always used together with a co-initiator – usually I the form of a tertiary aromatic amine. Camphorquinone has a light absorption spectrum of approximately 390 nm to 510 nm, with a peak sensitivity at 470 nm. Camphorquinone reacts to visible light in the blue range and has an intense yellow hue due to its absorption properties. Other initiators such as Lucirin TPO an acyl phosphine oxide which bleaches out entirely after polymerisation, tend therefore to be preferred for composite bleach shades or colourless protective varnishes. Acyl phosphine oxide has a considerably lower sensitivity peak than camphorquinone.

2.3.1 Light initiator Ivocerin®

Tetric N-Ceram Bulk Fill utilises the initiators: camphorquinone plus an acyl phosphine oxide, together with a recently patented initiator Ivocerin. It is standard in dentistry to apply composites in individually cured, 2 mm increments, as larger layers would negatively affect the depth of cure. In order to increase the possible increment depth, all parameters influencing depth of cure such as translucency, colour, initiator types and concentration; plus curing time and light intensity have to be considered. The new light initiator Ivocerin - a dibenzoyl germanium derivative [9,10] plays an important role here. It allows the application and curing of posterior restorations in larger increments of up to 4 mm, without compromising the optical properties of the composite such as translucency or colour.

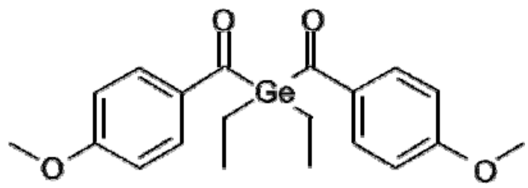


Fig. 9: Structural formula of germanium based photoinitiator Ivocerin®

Ivocerin® and light absorption

The standard initiator system plus Ivocerin results in a material featuring an absorption maximum in the blue light range from around 370 to 460 nm.[10] The initiator absorption spectra are depicted in figures 10 and 11.

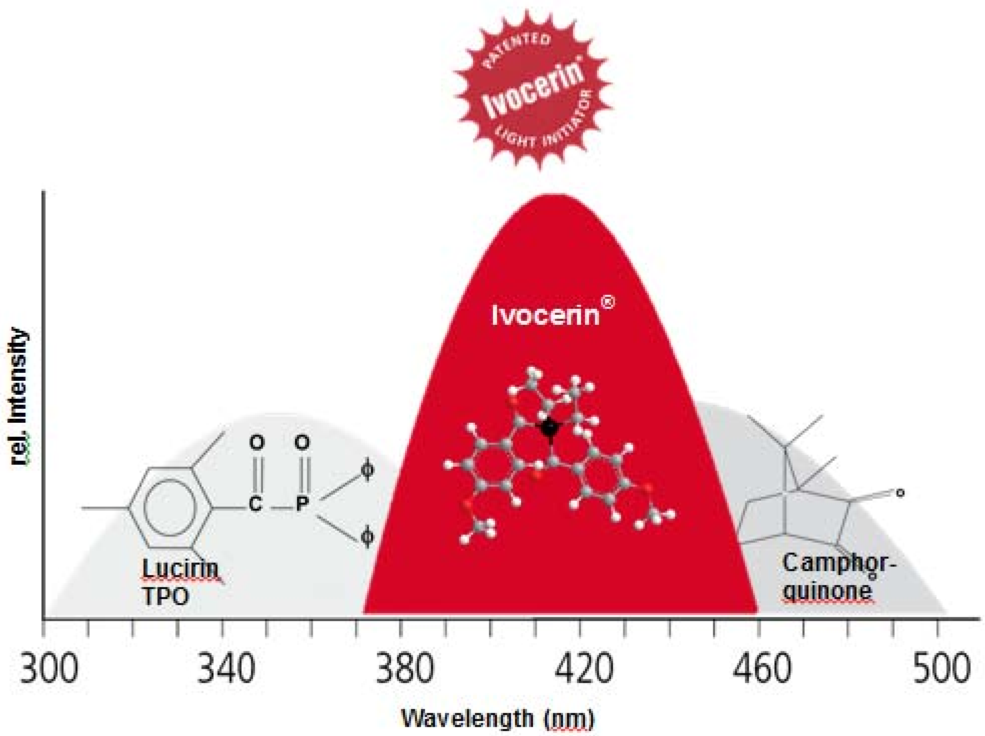


Fig. 10: Schematic representation of the absorption spectra of acyl phosphine oxide (e.g. Lucirin TPO), camphorquinone and Ivocerin

Ivocerin features a high absorption coefficient (higher than camphorquinone) allowing for increased quantum efficiency. The initiator is far more light-reactive than camphorquinone or acyl phosphine oxide (e.g. Lucirin TPO), enabling the material to polymerise more rapidly and with a greater depth of cure. In this sense it acts as a polymerisation booster. The graph below compares the absorption spectra of the three initiators using an equal concentration of initiator (0.2%) dissolved in monomer.

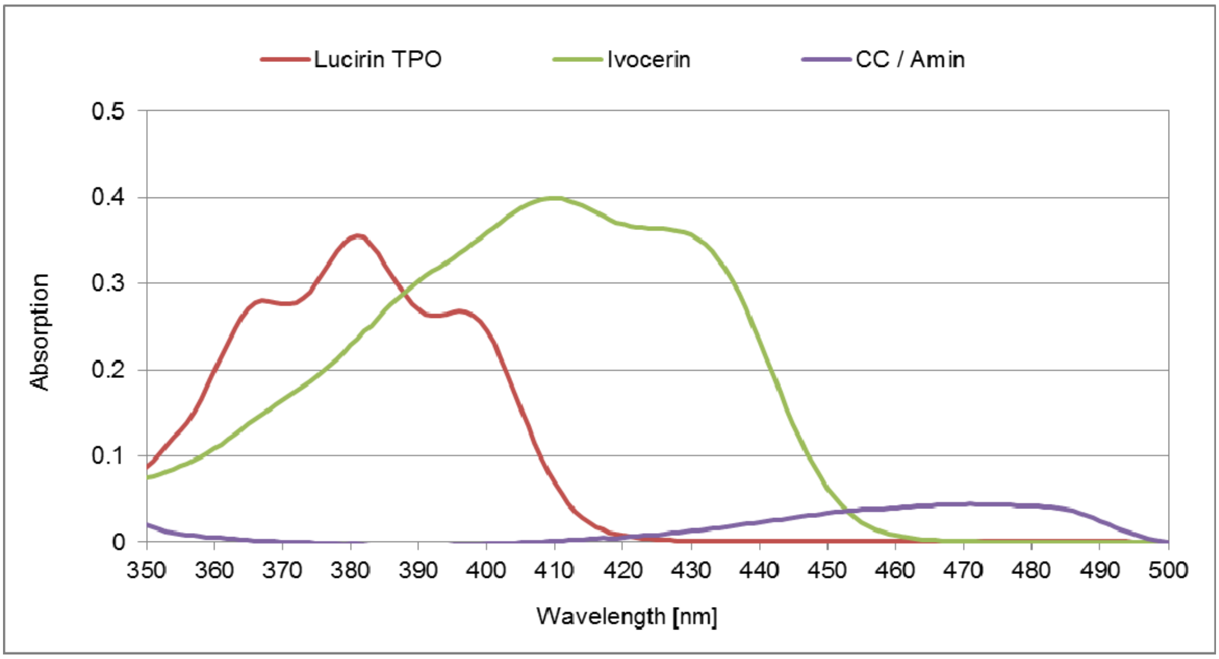


Fig. 11: Absorption spectra of equal concentrations (0.2%) of acyl phosphine oxide (e.g. Lucirin TPO), camphorquinone (CC / Amin) and Ivocerin, as measured in the laboratory. *(R&D Ivoclar Vivadent, 2014)*

Ivocerin® and aesthetics

All standard initiators are yellow, as this is the complementary colour to blue light with which all standard composites are polymerised. Although the yellow colour largely disappears during curing, a slight hue will always remain. This is however deemed acceptable as natural teeth are also slightly yellow in colour. Acyl phosphine oxide absorbs light largely in the UV-area and thus has just a very slight yellow colour making it highly suitable for composite bleach shades.

Ivocerin®

Acyl phosphine oxide

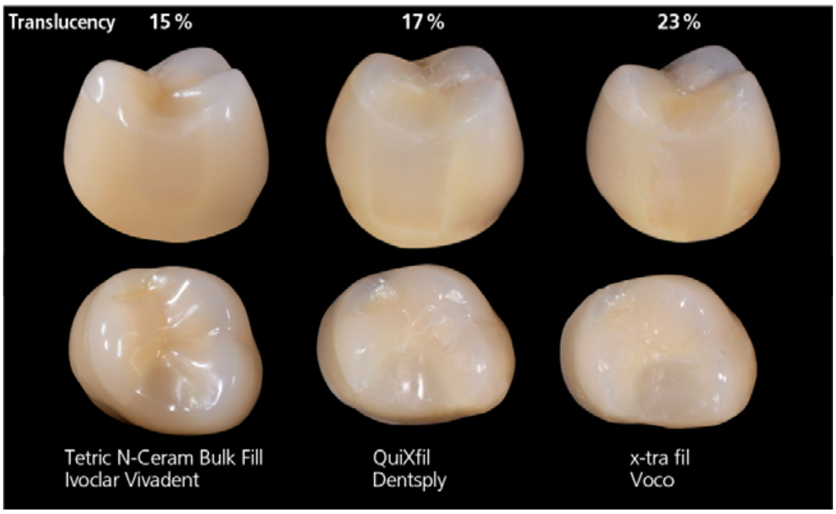
Camphorquinone

Fig. 12a-c: Light initiators contained in Tetric N-Ceram Bulk Fill in their pure form:

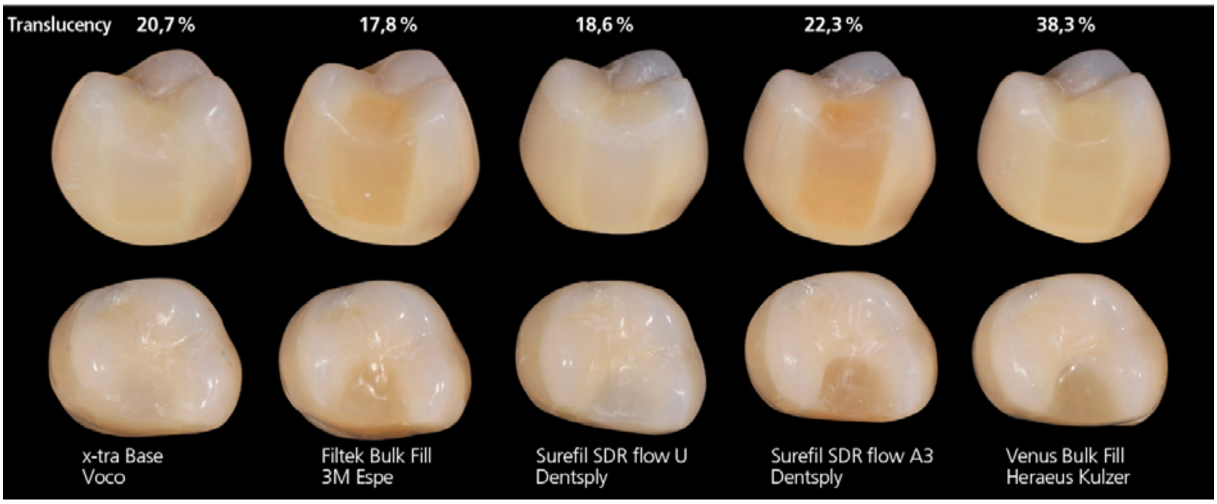
Ivocerin is also yellow in colour, but can be used in relatively small quantities due to its enhanced reactivity. This is useful as it means its properties can be used without negatively affecting the optical properties of tooth-coloured pastes with enamel-like translucency.

The following photographs illustrate the optimal translucent properties of Tetric N-Ceram Bulk Fill as compared to various other (sculptable and flowable) bulk fill composites. Typical Class II cavities were prepared with an approximate size of 3 mm in width, 3 mm in length and 4 mm in depth. The tooth on the left in the top photo (13a) is filled with Tetric N-Ceram Bulk Fill showing a translucency of 15%. It is the most aesthetic i.e. the least distinct from the surrounding tooth structure. Several of the other fillings are visibly transparent and distinct from the surrounding tooth structure i.e. they are less esthetic.

Sculptable bulk fill composites



Flowable bulk fill composites



Figs. 13a,b: Sculptable and flowable bulk fill composites showing varying translucency and corresponding aesthetics. Photos: Dr E. Mahn, Santiago Chile. Values: *(R&D Ivoclar Vivadent, 2014)*

Ivocerin® and depth of cure

The polymerisation booster Ivocerin allows Tetric N-Ceram Bulk Fill to be set to an enamel-like translucency of 15%. This is sufficient, such that when exposed to the light of a high energy curing unit such as Bluephase N, the restoration cures reliably. Whilst the number of photons that reach the cavity floor is significantly lower than the number that reach the surface, it is sufficient for Ivocerin to trigger polymerisation at a depth of 4 mm.

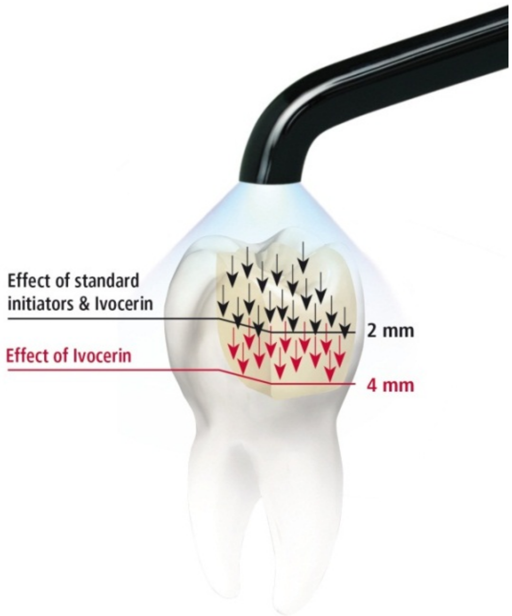


Fig. 14: Effect of Ivocerin polymerisation booster on light curing (10s $\geq$ 1000 mW/cm^2)

2.3.2 Light sensitivity filter

A material that is applied in 4 mm increments and subsequently contoured needs to provide sufficient working time before the product begins to polymerise. The longer the working time the more user-friendly the product. As composite materials generally contain photoinitiators that react to blue light, both ambient light and dental operating lights (which contain blue light) are capable of triggering premature polymerisation.

Tetric N-Ceram Bulk Fill incorporates a patented light sensitivity filter to prevent premature polymerisation. This provides a working time of more than three minutes under defined light conditions of 8000 lux. (See section 4.2, figure 20).

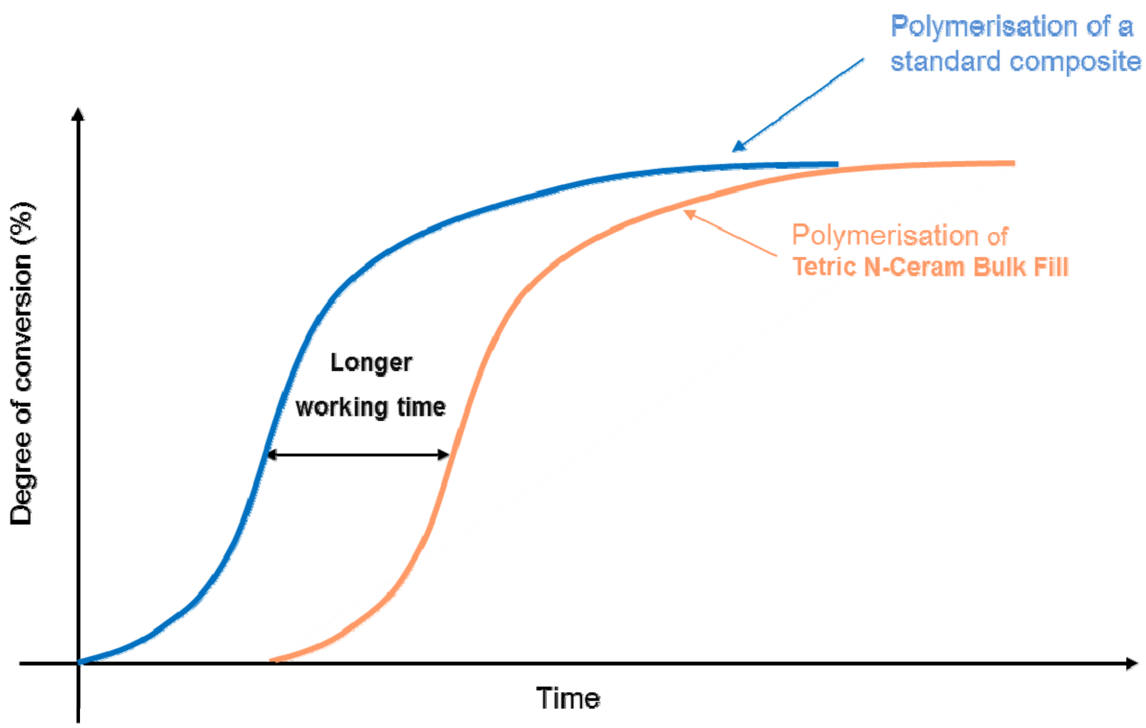


Fig. 15: Schematic representation: Light sensitivity filter delays polymerisation in ambient light.

Importantly whereas the stabiliser/inhibitor delays the polymerisation process in the presence of “low level” blue light, it does not impair curing under the intensive blue light of a polymerisation unit.

2.4 Conclusion: Paradigm shift from 2 mm to 4 mm increments

Before the introduction of bulk fill composites, standard dental teaching recommended a maximum layer thickness for composite fillings of 2 mm.[11,12] This was in order to minimise shrinkage stress and to ensure adequate depth of cure. Notably in deep cavities, placing such restorations can be time consuming and with many layers involves the not insignificant risk of incorporating air bubbles.[8]

Due to the incorporation of the polymerisation booster Ivocerin, a light sensitivity filter and a shrinkage stress reliever, a real paradigm shift in dentistry is now possible.

Tetric N-Ceram Bulk Fill is an esthetic time-saving composite that can be applied efficiently in 4 mm increments.

3. Technical Data

Tetric N-Ceram Bulk Fill

Standard composition (in weight %)

Dimethacrylates	21.0
Polymer Filler	17.0
Barium glass filler, Ytterbium trifluoride, Mixed oxide	61.0
Additive, Initiators, Stabilisers, Pigments	< 1.0

Physical properties

In accordance with:

EN ISO 4049:2009 Dentistry – Polymer-based restorative materials (ISO 4049:2009)

		Specification	Example value
Flexural strength	MPa	≥ 80	120
Water sorption (7 days)	µg/mm³	≤ 40	24.8
Water solubility (7 days)	µg/mm³	≤ 7.5	< 1.0
Radiopacity	% Al	≥ 100	260

Other physical properties

Vickers hardness HV 0.5/30	MPa		620
Flexural modulus	MPa		10000
Layer thickness (IV Method)	mm		4.0
Transparency: (depending on opacity)	%		15 - 17

4. Materials Science Investigations / In Vitro

4.1 Depth of Cure

Assuming correct adequate curing with a suitably functioning curing unit, translucency and shade have the most significant effect on the curing depth. The darker and more opaque a composite, the lower the curing depth,[13] but if manufacturer instructions are followed closely, a good degree of cure is usually obtained on the surface of a composite, irrespective of translucency or shade.[14] Assessing cure across the entire thickness of a restoration *in vivo,* is however impossible.

ISO 4049: Depth of cure
The international standard ISO 4049 for polymer based restorative materials suggests measuring depth of cure via preparing cylindrical specimens 6 mm long and 4 mm wide, or if a depth of cure greater than 3 mm is claimed, the length should be at least 2 mm longer than twice the claimed depth of cure. After curing according to the manufacturer's instructions, the material is removed from its mould, the inhibition layer and other uncured material is scraped away and the height of the remaining material is measured. This value divided by 2 is considered to be the depth of cure. This method does not account for post-irradiation polymerisation.

Vickers/Knoop hardness: Depth of cure
There are a number of *in vitro* test methods for establishing depth of cure. Vickers hardness and Knoop hardness profiles of the cured material are suitable and can be conducted some time after curing, allowing for post-irradiation polymerisation.

Cured specimens are usually prepared in cylindrical moulds and the hardness at the top and bottom of the cylinder is measured to obtain a simple single hardness measure. For a hardness profile throughout the material, cured specimens are cut vertically into two pieces. The cut surfaces are polished and the hardness is determined at intervals from the top to the bottom. Hardness is often expressed as a percentage of the surface hardness which is considered 100%.[14] Experience has shown that the simple hardness measures (top and bottom) correspond well to the more thorough hardness profile measurements.[15] According to research carried out by Professor David Watts of the University of Manchester, UK, an acceptable curing depth is achieved, if the bottom hardness corresponds to at least 80% of the surface hardness.[16]

The following internal investigation using Vickers hardness testing confirms the adequate depth of cure at 4mm with Tetric N-Ceram Bulk Fill.

Depth of cure of bulk fill composites – ISO 4049 vs. Vickers Hardness Test. S. Singhal, D. E. Antonson, S. A. Antonson, C. A. Munoz. University of Buffalo, New York, USA. Poster, ADM 2013.

The objective of this study was to compare the depth of cure values obtained with various bulk fill materials using the ISO 4049 method and Vickers hardness tests.

Method
Four bulk fill products: Tetric N-Ceram Bulk Fill, QuiXX/Dentsply, x-tra fill/Voco and SonicFill/Kerr and two conventional composites: Tetric N-Ceram and Filtek Z350 XT/3M ESPE were tested according to the depth of cure methods mentioned above. Five specimens per product were prepared by condensing resin composites in a rubber mould (6 x 6 x 10 mm). A Bluephase Style curing light was used to cure each specimen according to the manufacturers' instructions.

ISO 4049: The measurements were carried out according to the norm. Three measurements were made per specimen and the mean was considered the final value.

Vickers Hardness: Specimens were polished using 1200 grit SiC paper under water spray. Vickers hardness was measured at a distance of 0.5, 2, 4 and 5 mm from the top surface 1 hour after curing. Specimens were stored in an incubator at 37°C under distilled water for 24 hours and hardness measurements were repeated to evaluate post-irradiation polymerization. Ratios of bottom/top (B/T) hardness vales were calculated (2/0.5 mm, 4/0.5 mm, 5/0.5 mm). Adequate depth of cure was assumed if the bottom hardness corresponded to at least 80% of the top surface hardness.

Results

As per ISO 4049 and B/T hardness ratio (1-hour), all bulk fill composites achieved the manufacturers' claimed depth of cure i.e. 4mm; except for SonicFill (5 mm). The conventional composites showed significantly lower depth of cure compared to the bulk fill composites at 4mm and 5mm depth. Tetric N-Ceram Bulk Fill showed the highest B/T hardness ratio at 4 mm after 1 hour, whereas x-tra fill showed the highest ratio after 24 hours, with Tetric-N-Ceram the second highest. After 24 hours, bulk fill products exhibited a significant increase in B/T hardness ratio at 5 mm.

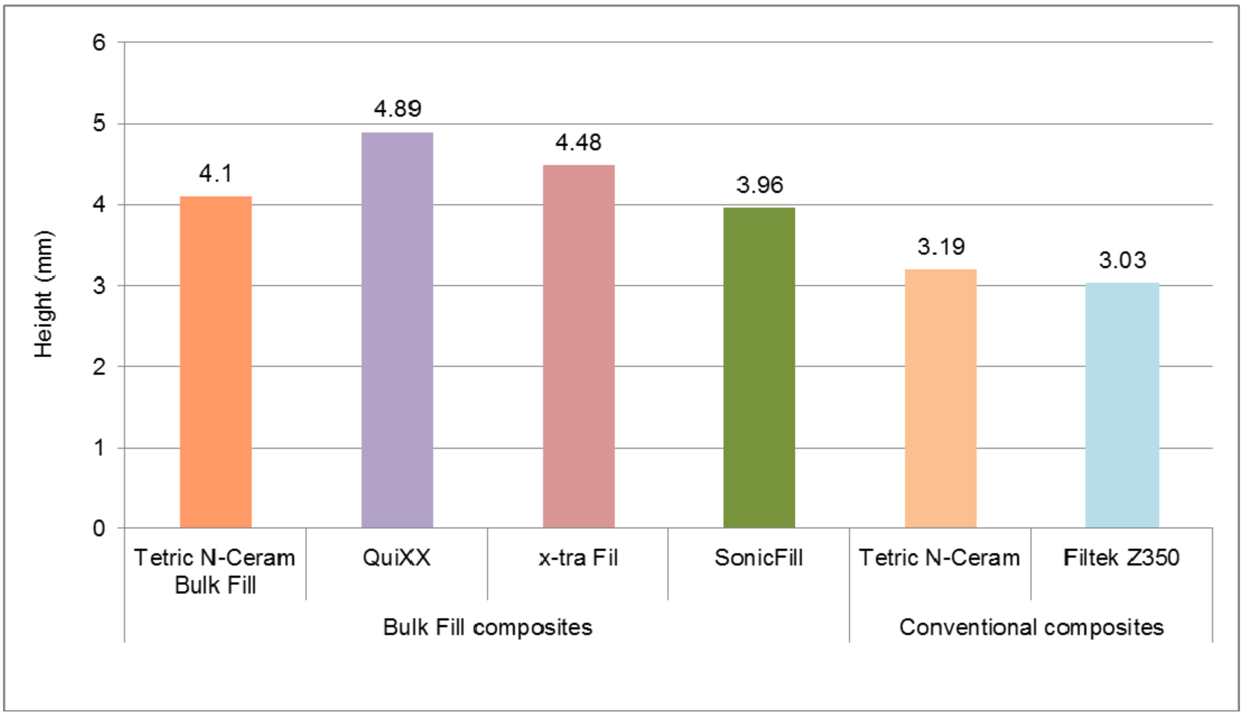


Fig. 16: Depth of cure in mm of various composites, according to ISO 4049 testing. *(Uni Buffalo, 2013)*

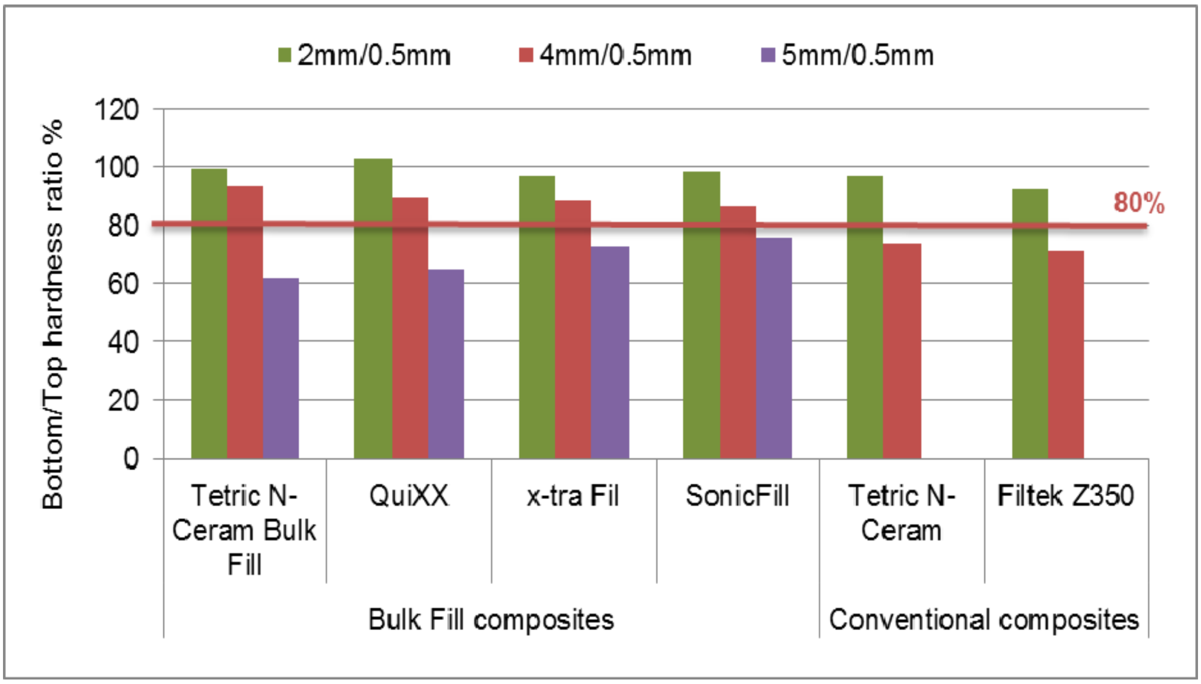


Fig. 17: Percentage Bottom/Top Vickers hardness ratios for various bulk fill and conventional composites 1 hour following polymerisation. (Uni Buffalo, 2013)

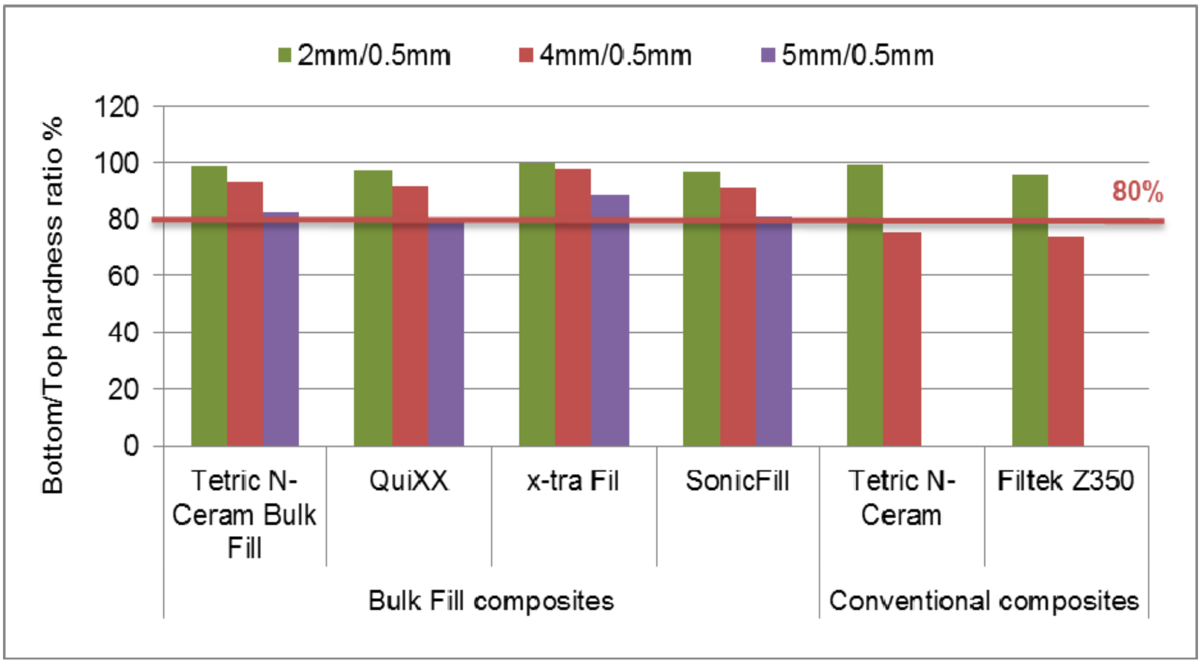


Fig. 18: Percentage Bottom/Top Vickers hardness ratios for various bulk fill and conventional composites 24 hours following polymerisation. (Uni Buffalo, 2013)

Conclusion
All the bulk fill products achieved the depth of cure levels claimed by the respective manufacturers except for SonicFill (5 mm) (Fig.17). All the products achieved a bottom/top hardness ratio in excess of 80% (apart from SonicFill at 5 mm) after one hour (Fig. 18) and all the products achieved a ≥ 80% bottom/top hardness ratio after 24 hours (Fig. 19). The testing carried out according to ISO 4049 showed lower depth of cure compared to the hardness testing.

4.2 Sensitivity to ambient light

The time available to apply and contour a composite material before it starts to polymerise plays an important role in determining its user friendliness.

Composite materials normally contain photoinitiator systems that react to the blue light portion of the visible light spectrum. The source of that blue light is immaterial. As both daylight and dental operating lights comprise a certain amount of blue light, they can contribute to the (premature) polymerisation of composite materials. The higher the intensity of the ambient light, the shorter the working time before the material begins to polymerise. Protecting light curing materials from ambient light during application is impractical and with dental loupes becoming more popular, very light-sensitive composites have a clear disadvantage.

Tetric N-Ceram Bulk Fill therefore features a patented light sensitivity filter. The inhibitor delays polymerisation when low level blue light is present, but does not impair the polymerisation process under the intensive blue light of a properly functioning curing light.

A material's sensitivity to ambient light is usually determined according to conditions defined in the standard ISO 4049. The longer the period of time before the material polymerises, the less sensitive to light it is. Tetric N-Ceram Bulk Fill features a modelling-time of more than three minutes (200 s) under defined light conditions of 8000 lux. This was the longest period of the materials tested below.

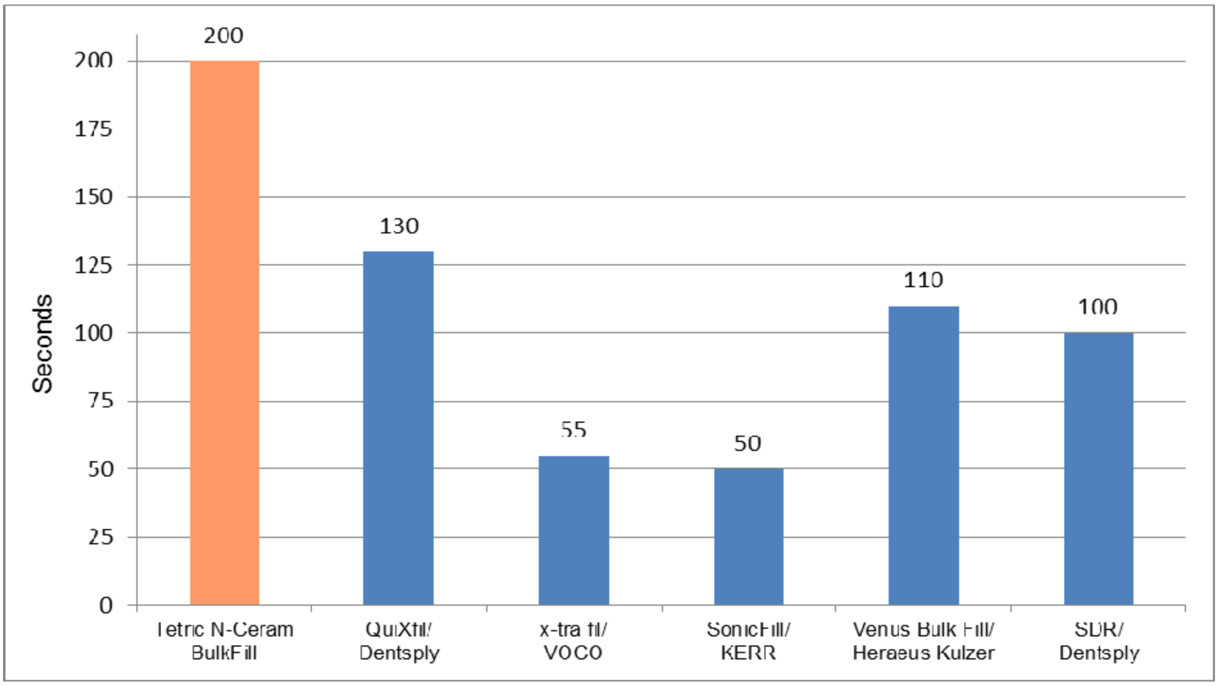


Fig. 19: Sensitivity to ambient light (modelling-time) of various bulk fill composites determined according to ISO 4049. (*R&D Ivoclar Vivadent, July 2011*)

4.3 Polymerisation Shrinkage

Minimising the shrinkage stress is particularly important in a material that is applied in increments of up to 4 mm. Tetric N-Ceram Bulk Fill therefore contains a shrinkage stress reliever with a low modulus of elasticity. It acts like a microscopic spring, attenuating the forces generated during shrinkage. Reduced polymerisation shrinkage should translate as lower volumetric shrinkage, improved marginal integrity and reduced shrinkage stress force over the composite surface/on the adhesive bond.

4.3.1 Volumetric shrinkage

The polymerization shrinkage (% vol) of various composites was tested. The volumetric shrinkage of the scuptable bulk fill products Tetric N-Ceram Bulk Fill, Sonic Fill/Kerr, QuiXfil/Dentsply and x-tra fil/Voco are shown below. The percentage of volumetric shrinkage after 1 hour was measured with a mercury dilatometer. The graph below shows that in this test the volumetric shrinkage of Tetric N-Ceram Bulk Fill is similar to that of other sculptable bulk fill materials.

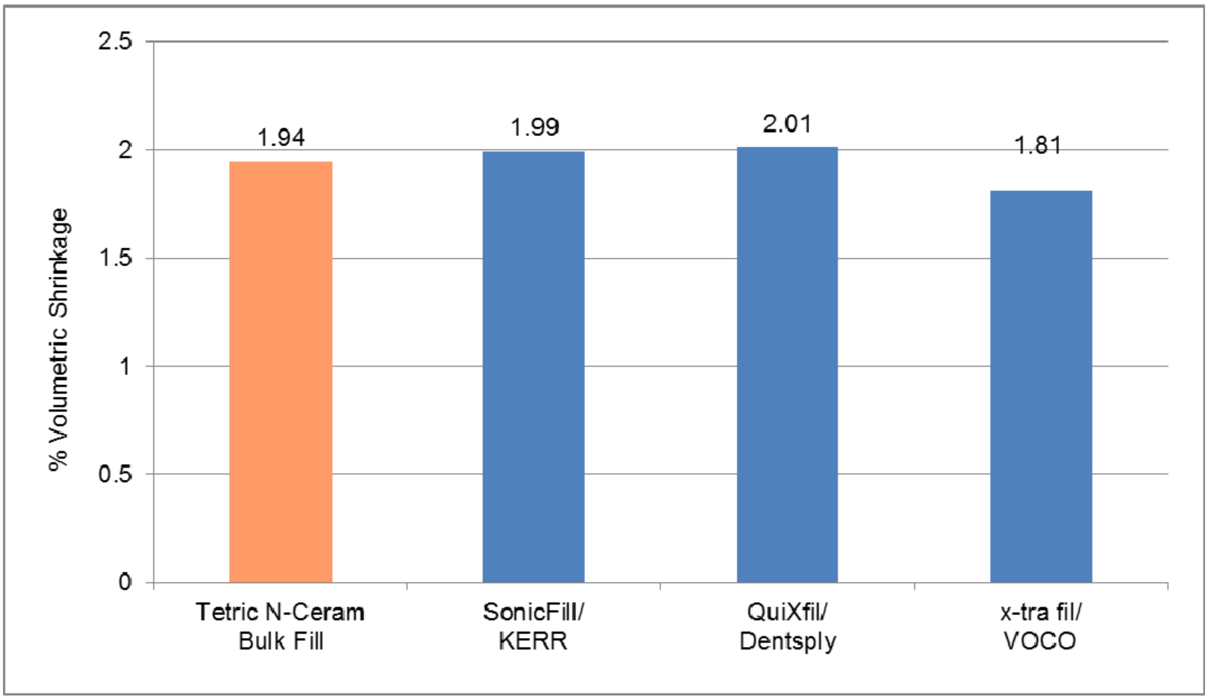


Fig. 20: Comparison of the polymerisation shrinkage of various scultpable bulk fill composites *(R&D Ivoclar Vivadent, December 2011)*

4.3.2 Shrinkage force and stress

Composites are fixed to the tooth structure by means of an adhesive and cannot shrink freely during the shrinkage process. The shrinkage force that builds up in the course of the shrinkage process puts a strain on the adhesive bond. The shrinkage force of various materials was examined in different layer thicknesses. The measurements were performed by means of a Bioman shrinkage stress measuring device (light exposure with Bluephase G2 - HIP, for 10 seconds, shrinkage force measurement over a period of 30 min). The results show that Tetric N-Ceram Bulk Fill exhibits less shrinkage stress in both 2 mm and 4mm than the universal bulk fill materials SonicFill/KERR and x-tra Fil/Voco, when applied in comparable thicknesses. The test also revealed that the shrinkage stress measured in the 4 mm layers were not substantially higher than that in the 2 mm layers.

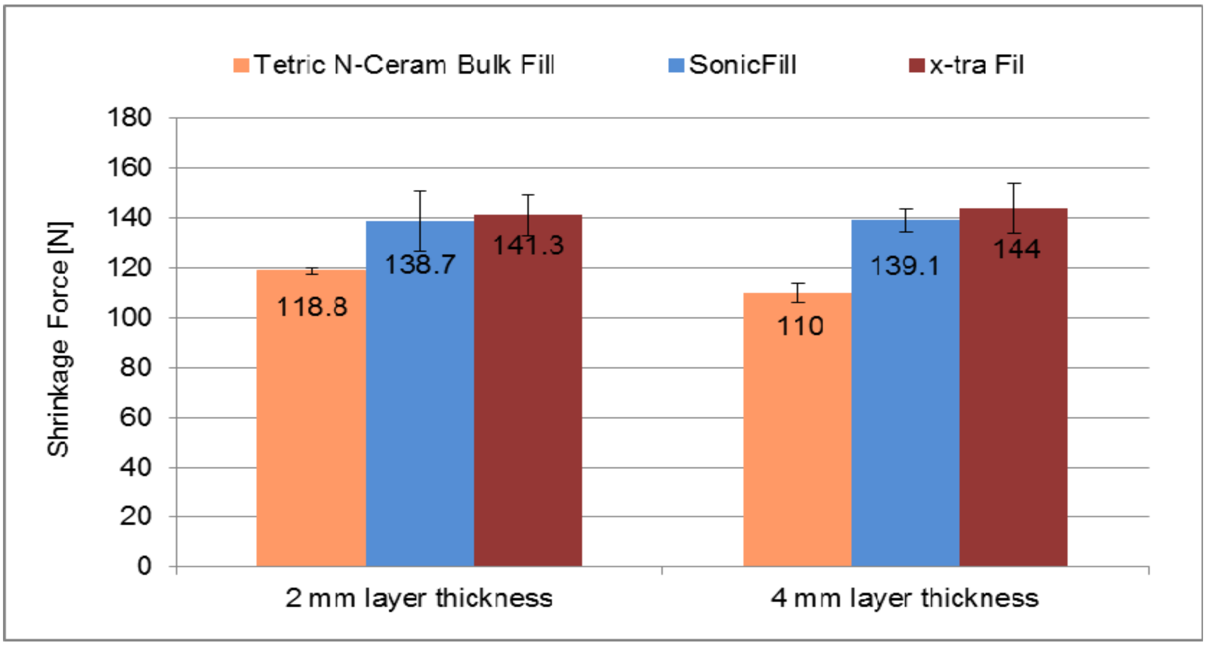


Fig. 21: Shrinkage force of Tetric N-Ceram Bulk Fill compared to other bulk fill composites *(R&D Ivoclar Vivadent, 2013)*

Shrinkage stress is determined by means of the shrinkage force measured on the surface of the test specimens, i.e. the shrinkage stress is the shrinkage force per unit area (MPa = N/mm^2). The following graph shows that the shrinkage stress exhibited by a 4 mm increment of Tetric N-Ceram Bulk Fill is lower than that of other bulk fill products when applied in the same layer thickness and is also lower than conventional composite products when applied in just 2 mm increments.

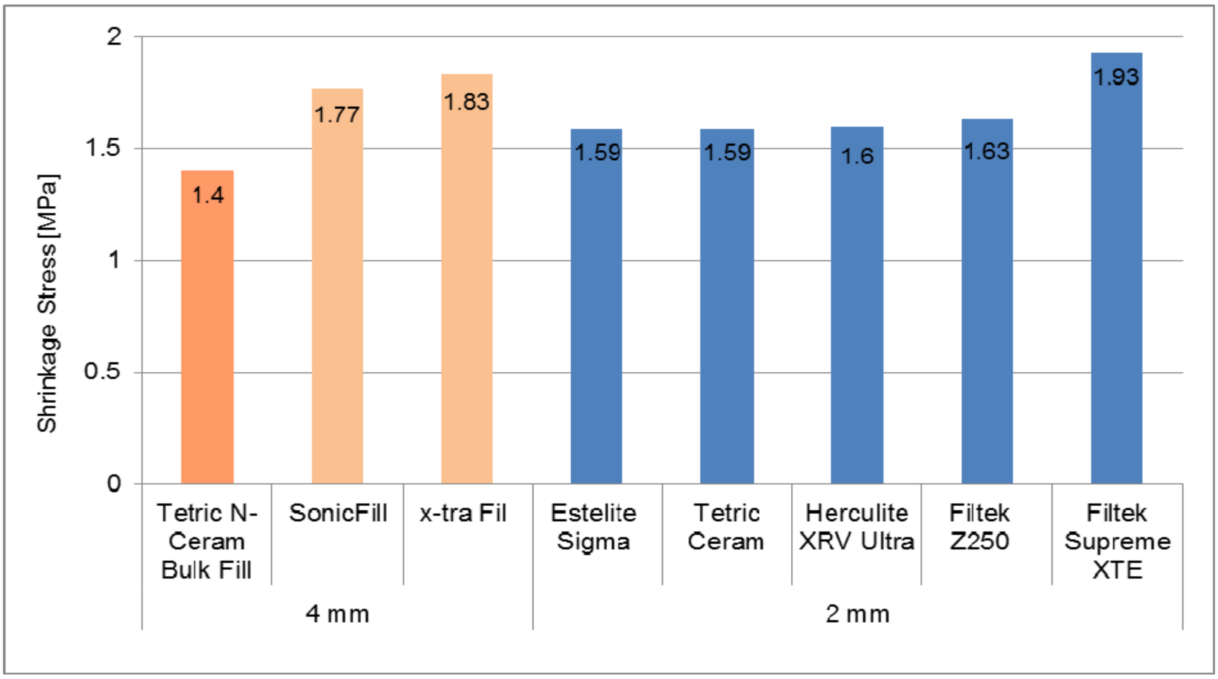


Fig. 22: Shrinkage stress occurring in Tetric N-Ceram Bulk Fill in a layer thickness of 4 mm compared to the shrinkage stresses of other bulk fill composites (4 mm) and conventional composites (2 mm). Measurement according to Watts. *(R&D Ivoclar Vivadent, 2013)*

The results showed that the average shrinkage force (Fig. 22) and shrinkage stress (Fig. 23) of Tetric N-Ceram Bulk Fill is lower than that of other composites.

4.3.3 Marginal integrity

In order to investigate marginal integrity in Tetric N-Ceram Bulk Fill restorations, 24 class II cavities were prepared in 12 lower jaw molars (mesial and distal) with an approximal depth of 4mm, an occlusal depth of 3 mm and an approximal width of 5 mm. The entire cavity margin lay in the enamel. Four groups were defined using 2 different Ivoclar Vivadent composites (conventional and bulk fill) and 2 different Ivoclar Vivadent adhesives (etch and rinse and self-etch). The two cavities of each tooth were filled with Tetric N-Ceram (mesial) and Tetric N-Ceram Bulk Fill (distal) using the same adhesive.

Group	Adhesive	Molars	Composite	Cavities
Group 1	Tetric N-Bond Self-Etch	6	Tetric N-Ceram	6
Group 2			Tetric N-Ceram Bulk Fill	6
Group 3	Tetric N-Bond	6	Tetric N-Ceram	6
Group 4			Tetric N-Ceram Bulk Fill	6

Table 3: Composite and adhesive groups for the evaluation of marginal integrity.

Tetric N-Ceram was applied in three layers with each layer cured for 10 seconds using Bluephase Style. Tetric N-Ceram Bulk Fill was applied in one layer and cured for 10 seconds using Bluephase Style. Teeth were stored in water for 1 day at 37°C and thermocycled x10,000 at 5°C/55°C.

Cavities were evaluated as to whether the fillings could be manually dislodged from the cavity and marginal integrity was analysed using a stereomicroscope and SQUACE (semi quantitative clinical evaluation) criteria. A distinction was made between marginal gaps and irregularities. Margins were assessed occlusally, axio-approximally and cervically – for both marginal gaps and marginal irregularities

All 24 fillings remained in the cavities and could not be dislodged. The fillings bonded with Tetric N-Bond showed somewhat higher marginal adaptation than those bonded with Tetric N-Bond Self-Etch but the difference was not significant. There was also no statistically significant difference in marginal integrity of Tetric N-Ceram Bulk Fill fillings compared to Tetric N-Ceram restorations within the same adhesive group. Only for the criteria marginal gaps (axio-approximally) in the Tetric N-Bond Self-Etch group were slightly more (significantly) gaps found in the Tetric N-Ceram Group. When the data is pooled, independent of adhesive there was no statistically significant difference between Tetric N-Ceram (3 layers) or Tetric N-Ceram Bulk Fill (1 layer) regarding either marginal gaps or marginal irregularities.

4.4 Wear

In order to gauge the clinical wear-behaviour of dental materials, laboratory-based chewing simulation tests are often used. Ivoclar Vivadent uses a Willytec chewing simulator to measure the wear resistance of restorative materials. The aim is to emulate mastication processes using a standardised procedure in order to obtain results that can be compared with each other. To achieve this, standardised ceramic antagonists (IPS Empress) are employed and plane test samples are subjected to 120,000 masticatory cycles, with a force of 50N and a sliding movement of 0.7 mm. The vertical substance loss is measured by means of a 3D laser scanner. A vertical loss of less than 200 µm is considered low and a loss ranging between 200 – 300 µm is considered medium. The graph below compares the wear exhibited by Tetric N-Ceram Bulk Fill and other bulk fill composites.

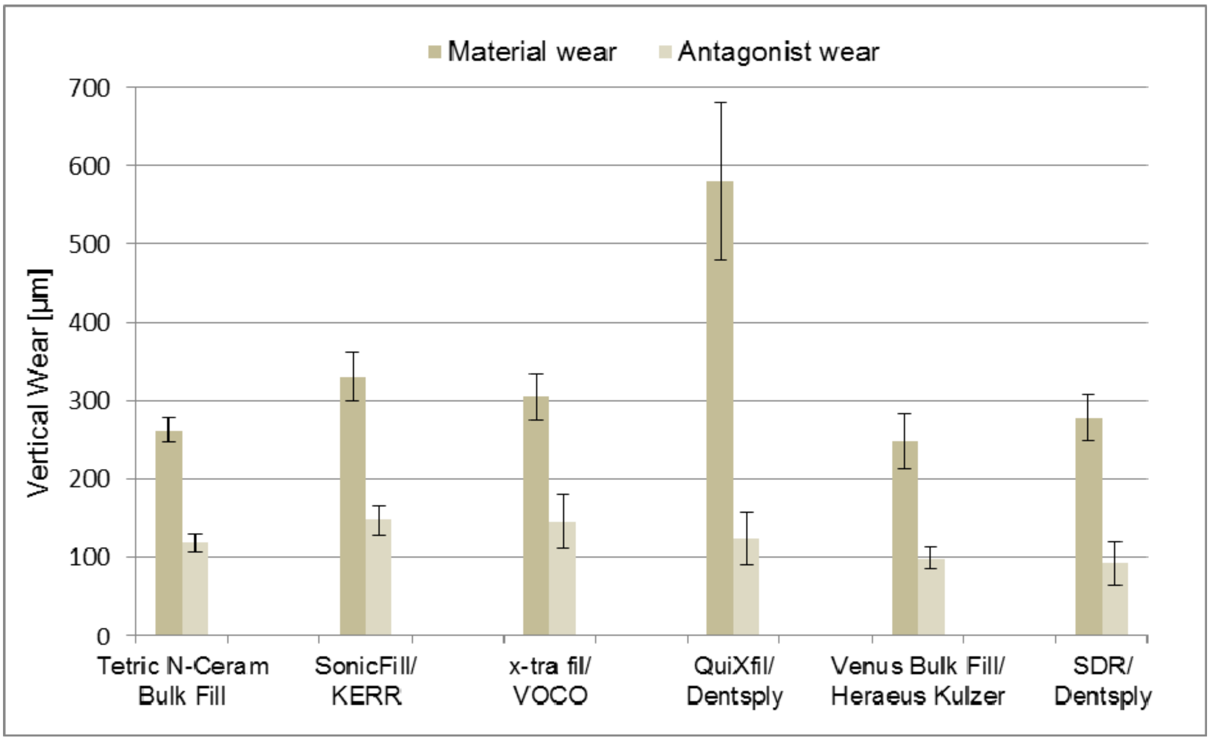


Fig. 23: Mean vertical wear of restorative materials and their antagonists *(R&D Ivoclar Vivadent, 2011)*

The material QuiXfil exhibited significantly higher wear than the other bulk fill materials. SDR, Venus Bulk Fill and Tetric N-Ceram Bulk exhibited low and comparable wear, whereas Quixfil and SonicFill showed significantly higher wear. With regard to antagonist wear, there was less variation but significantly higher wear was recorded for SonicFill and X-tra fil test samples.

4.5 Polishability and Colour Stability

Polishing represents a critical step in direct restorative treatment . A pleasing surface gloss is decisive for the clinical success and esthetic appearance of a composite restoration.

Restoration surfaces that are too matte in relation to the surrounding tooth structure are unesthetic and rough surfaces are conducive to staining and plaque accretion. Special attention was therefore given to achieving advantageous polishing properties in the development of Tetric N-Ceram Bulk Fill.

For the experiment below, eight specimens were prepared for each material according to the manufacturer's directions. The specimens were roughened with sand paper (320 grit) to achieve a defined initial surface roughness. The specimens were then stored in a dry-storage area at 37 °C for 24 hours, whereupon their gloss was measured with a novo-curve gloss meter and surface roughness was determined with an FRT MicroProf measuring device.

The specimens were polished using a single-step OptraPol polisher at a pressure of 2N at 10,000 rpm under water cooling. Specimens were polished for 30 seconds in total, with the surface roughness measured at intervals of 10 seconds.

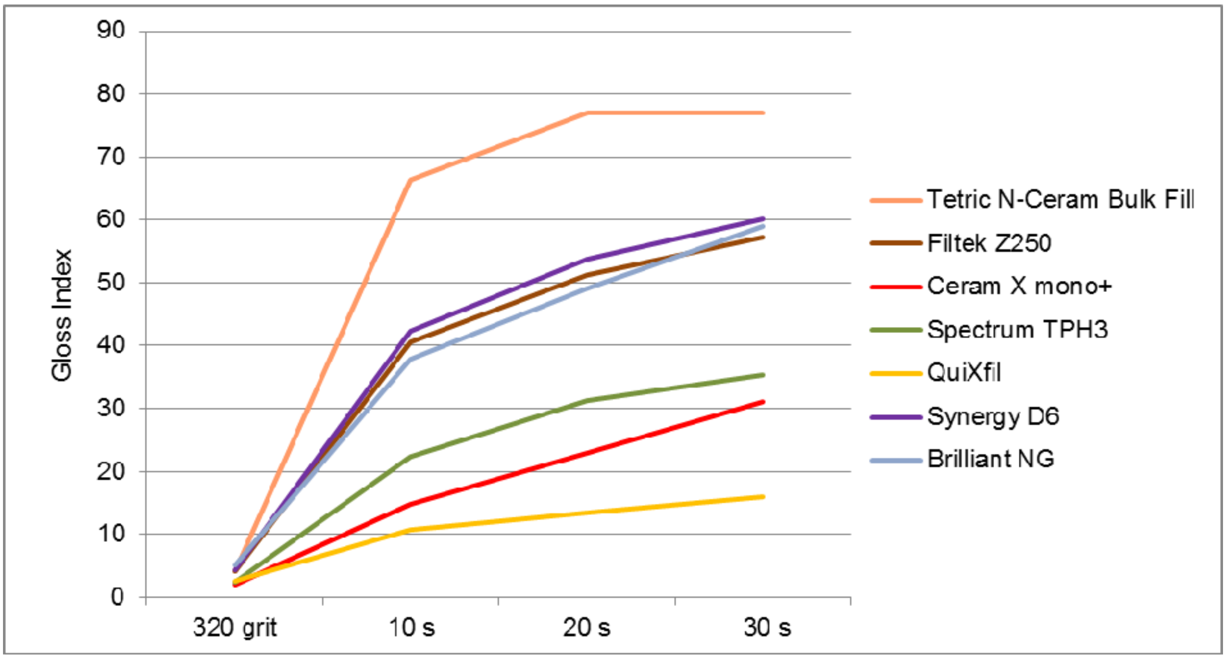


Fig. 24: Mean surface gloss of various composite materials compared to Tetric N-Ceram Bulk Fill after polishing with OptraPol in relation to polishing time. Reference material: black glass = 92.6 *(R&D Ivoclar Vivadent, 2012)*

Tetric N-Ceram Bulk Fill showed a significantly higher surface gloss than all the other materials investigated. The materials investigated produced the following surface gloss results after 30 seconds (in descending order; according to ANOVA post hoc Tukey B with $p<0.05$):

Tetric N-Ceram Bulk Fill > Synergy D6 = Brilliant NG = Filtek Z250 > Spectrum TPH3 = Ceram.X mono+ > QuiXfil

In a further test, surface roughness was also determined after 10, 20 and 30 seconds of polishing. The lower the surface roughness value, the better the polishability of the material. A mean surface roughness of <0.1 µm indicates excellent polishability, <0.2 µm suggests good polishability, a value between 0.2 -0.4 µm corresponds to medium polishability and >0.4 µm means poor polishability. Tetric N-Ceram Bulk Fill was found to show excellent polishability in the above test.

This investigation did not show any significant differences in the surface roughness of Tetric N-Ceram Bulk Fill, Synergy D6, Brilliant NG and Filtek Z250.

The following surface roughness values were measured for the materials after 30 seconds (in ascending order, low roughness → high roughness; according to ANOVA post hoc Tukey B with p<0,05):

Tetric N-Ceram Bulk Fill = Synergy D6 = Brilliant NG = Filtek Z250 < Spectrum TPH3 = Ceram.X mono+ < QuiXfil

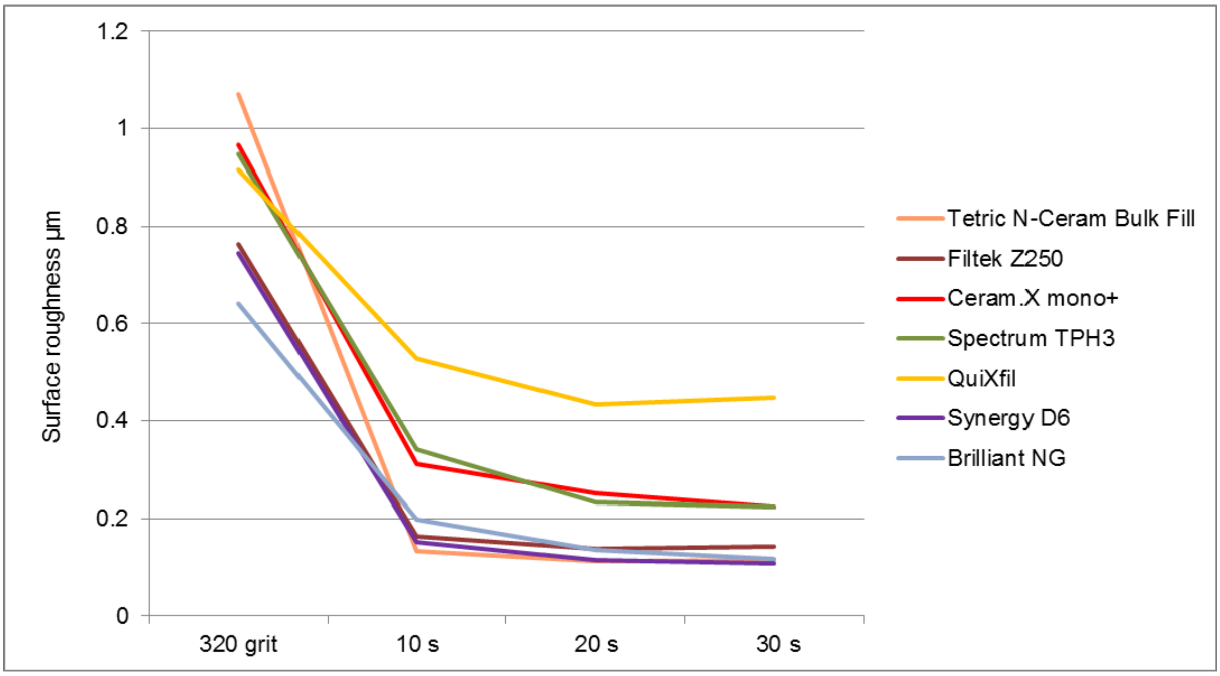


Fig. 25: Mean surface roughness (µm) of various composite materials compared to Tetric N-Ceram Bulk Fill after polishing with OptraPol in relation to polishing time. *(R&D Ivoclar Vivadent, 2012)*

The mix and size of the fillers are responsible for the excellent polishability and high gloss of Tetric N-Ceram Bulk Fill. Large fillers do not produce the same smooth and glossy surface as small fillers. Compared to other materials, Tetric N-Ceram Bulk Fill comprises fillers of a comparatively small size. The differences in filler size are clearly visible in the scanning electron microscope (SEM) pictures shown in section 2.

Colour stability and polishability of bulk fill composites. S. Mehta[1], S, Singhal[1], D. Antonson[2], S. Antonson[1]. [1]R&D Ivoclar Vivadent, Amherst, New York, USA. [2]State University of New York, Buffalo, USA. Poster 516, AADR, Charlotte USA, 2014.

The objective of this study was to compare the colour stability and gloss values of bulk composite resins versus conventional composites.

Method
Six different composites were tested. Two conventional composites: Tetric N-Ceram and Filtek Z350 XT/3M ESPE and four bulk fill composites: Tetric N-Ceram Bulk Fill, QuiXX/Dentsply, x-traFil/Voco and SureFil HDP/Dentsply. Five specimens of each product were tested regarding colour stability and gloss. Specimens were prepared and light cured (using Bluephase Style) according to the manufacturer's instructions.

Colour stability: specimens were surface finished (320-1200 SiC paper + alumina (50 µm) slurry. Baseline L*a*b* values were measured against white and black backgrounds (UltraScan VIS spectrophotometer). Specimens were stored in caffeinated solution for 48 hours and any change in colour (ΔE) was measured.

Gloss: specimens were finished using 320 SiC paper (Baseline/T1), followed by polishing with OptraPol for 20, 40 and 60 seconds (T2, T3 and T4 respectively). Three gloss values per specimen were measured (Novo-Curve Gloss meter) after each polishing (T) event and the average was considered the final value.

Results:

Data was analysed using ANOVA and Tukey's post-hoc test:

	Change in Colour		Change in Gloss			
	ΔE White Background (SD)	**ΔE Black Background (SD)**	**T1 Baseline (SD)**	**T2 20 s (SD)**	**T3 40 s (SD)**	**T4 60 s (SD)**
Tetric N-Ceram	7.2 (0.4)	7.6 (0.6)	11.4 (1.9)	64.9 (1.5)	71.8 (1.1)	74.2 (0.7)
Filtek Z350 XT	12.3 (2.1)	13.3 (1.7)	13.6 (0.6)	71.5 (1.5)	82 (1.3)	88.03 (0.7)
Tetric N-Ceram Bulk Fill	7.0 (0.8)	7.6 (0.3)	13.1 (1.0)	52.0 (4.9)	62.8 (4.7)	76.652.4)
QuiXX	11.2 (0.5)	10.4 (0.5)	3.0 (0.2)	3.7 (0.2)	4.8 (0.4)	6.23 (0.3)
x-traFil	9.2 (1.4)	8.1 (0.1)	3.7 (0.3)	5.9 (0.3)	6 (0.8)	6.58 (0.3)
SureFil HDP	11.8 (1.3)	12.4 (0.8)	12.6 (0.8)	37.1 (1.6)	53.4 (2.5)	63.07 (1.5)

Table 4: Comparison of change in colour and gloss of various bulk fill and conventional composites. (*Poster AADR 2014)*

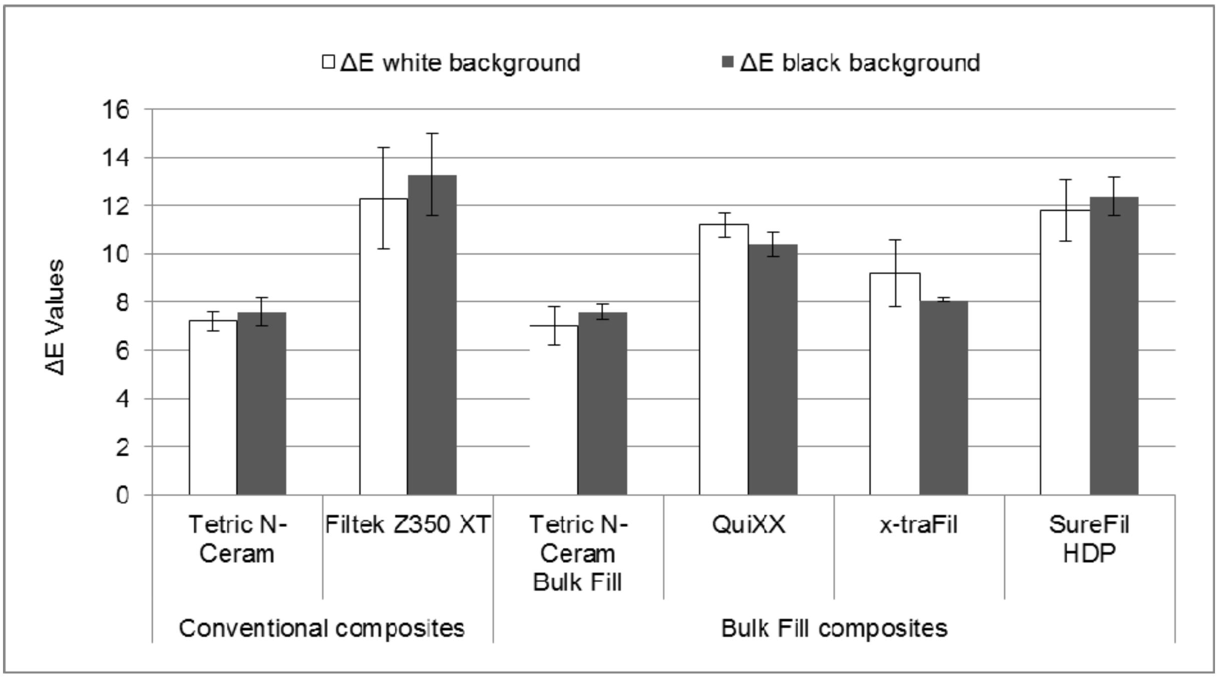


Fig. 26: Colour stability on a white and black background of various conventional and bulk fill composites. *(S. Mehta, poster AADR 2014)*

Tetric N-Ceram Bulk Fill showed statistically significant low ΔE values after 48 hours compared to other bulk fill composites tested. Filtek Z350 XT showed significantly higher ΔE values after 48 hours compared to the conventional composite Tetric-N Ceram on which Tetric N-Ceram Bulk Fill is based. Tetric N-Ceram Bulk Fill and Tetric N-Ceram exhibited similarly low levels of colour change on both the white and black background.

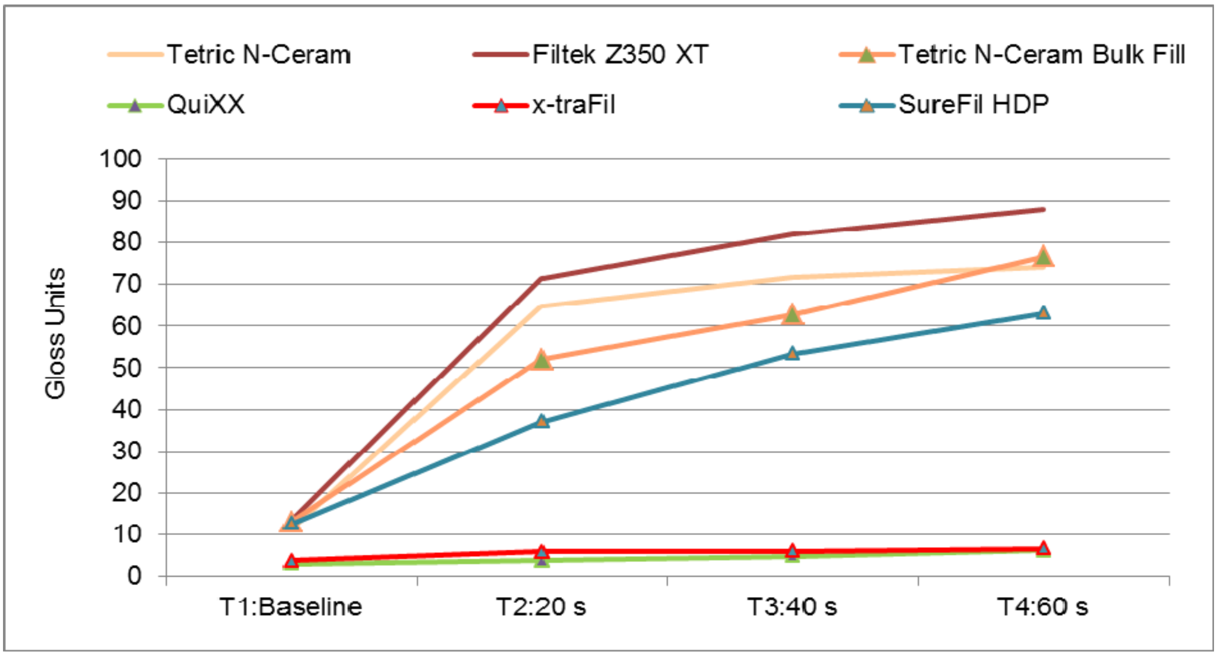


Fig. 27: Change in gloss of various conventional and bulk fill composites after polishing for different amounts of time. *(S. Mehta, poster AADR 2014)*

Tetric N-Ceram Bulk Fill showed a statistically significantly high surface gloss values compared to the other bulk fill resin composites tested.

Conclusion: Tetric N-Ceram Bulk Fill showed the lowest ΔE and highest gloss values amongst the bulk fill composites tested.

5. Clinical Case with Tetric N-Ceram Bulk Fill

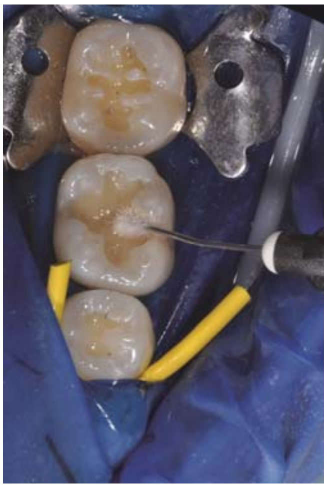
Class I restorations

Pre-operative situation

Placement of Optradam

Removal of amalgam

Enamel was etched for 30s and dentin for 10s



Tetric N-Bond adhesive was applied

Adhesive was light cured for 10s with Bluephase Style

Both cavities filled with one increment of Tetric N-Ceram Bulk Fill and light cured

Polishing with OptraPol

Final result at recall after 1 week

Source: Figures: 28a-h: (*Dr Eduardo Mahn, Santiago, Chile)*

6. Biocompatibility

To minimise the risks related to biocompatibility as far as possible from the outset, care is taken to ensure that mainly raw materials that have been used in dental composite materials for many years and have been proven *in vivo* to be safe, are used in the development of new materials. The toxicological properties of Tetric N-Ceram Bulk Fill can therefore be evaluated using data from well-established dental composites and their ingredients.

Cytotoxicity
Samples of Tetric N-Ceram Bulk Fill were extracted in RPMI 1640 medium according to ISO 10993-12. Subsequently, L929 cells were brought into contact with this extract for 24 hours. The vitality of these cells was measured after 24 hours with the help of tetrazolium dye (XTT). Extracts of Tetric N-Ceram Bulk Fill did not show any relevant effects on the cell cultures. Tetric N-Ceram Bulk Fill was found to be non-cytotoxic.

Mutagenicity
Extracts of material samples were examined in a reverse mutation test (Ames test). None of these tests indicated any mutagenic activity.
Ivocerin was also subjected to extensive testing, and showed no signs of mutagenic activity.

Irritation and sensitization
Like virtually all light-curing dental materials, Tetric N-Ceram Bulk Fill contains methacrylates and dimethyacrylates. These materials (notably in their uncured state), may have an irritating effect and may cause sensitization. This can lead to allergic reactions, such as contact dermatitis. Allergic reactions are very rare in patients but occur more frequently among dental staff, who handle uncured composite material on a daily basis. Such reactions can be minimized/avoided by clean working conditions and avoiding skin contact with uncured material. It should be noted that commercially available medical gloves do not provide effective protection against the sensitizing effects of methacrylates.

Tetric N-Ceram Bulk Fill must not be used in patients who are known to be allergic to any of its constituents.

Conclusion
On the basis of the data available, it can be concluded that Tetric N-Ceram Bulk Fill poses no health hazard if used correctly. To ensure correct use, the notes and directions in the Instructions for Use must be observed and followed.

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