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ChemFilTM



Advanced Glass Ionomer Restorative

(For Individual Attention Only) 2011-02-14

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For better dentistry

DENTSPLY

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1 Preface by Dr. Joel Berg

February 14, 2011

Dear Colleagues,

Glass ionomer materials are some of the most important materials in dentistry. They are commonly used in all aspects of dentistry for cementation, adhesion, lining cavities, as core buildup materials, as permanent restorations, and as transitional materials. Yet, their versatile inherent properties have not received the attention they deserve. Since the introduction of traditional glass ionomers into the worldwide market in the early 1980s, there has been a slow but consistent growth in their use, but not nearly the growth in development or use relative to their exceptional value in dental practice. This is in part because of the superimposed explosive growth of resin-based polymerizable materials, and in part because of the lack of understanding of the exceptional value of glass ionomer materials.

This lack of awareness about the strengths of glass ionomers is dramatically changing for the better, and now with the launch of ChemFil Rock, I am exceptionally pleased that many more will realize the strengths and advantages of traditional glass ionomer. Additionally, ChemFil Rock offers a new formulation, and as the studies reported in this document indicate, some of the leading researchers and clinicians have demonstrated that glass ionomer can continue to be improved, and clinicians can gain more value for their patients.

Let me review some of the very important clinical properties unique to glass ionomer materials. Because glass ionomer is a salt when it is “set”, it does not undergo polymerization in its setting reaction. It uses a pre-polymerized co-polymeric acid which combines with an aluminum containing glass (base) that forms a stable material in a few minutes, with minimal shrinkage. There is no elution of monomers after the material is set, as the original material contains no monomers. During the setting reaction after the material is placed onto tooth structure, a chemical bond forms between the glass ionomer material and the dentin and/or enamel. There is no other dental filling material which chemically bonds to tooth structure. In fact, although bond strengths as measured by traditional means (such as shear or tensile bond strength destructive measurement) might reveal low bond strength values, these readings are not clinically meaningful, as the material’s bond to dentin and enamel is so strong that glass ionomer material will usually break within the material

itself before its bond to tooth breaks. Clinicians who have seen poorly fitting orthodontic bands or stainless steel crowns be lost will see a protective coping of glass ionomer cement remaining on the tooth. Some of the original clinicians who used the first traditional glass ionomers have many cases in their hands of patients who have received class V facial restorations with glass ionomer restoratives wherein because of wear, abfraction or via other means, although the tooth structure has fractured away circumferentially around the restoration, the glass ionomer restoration itself has remained intact after decades, cantilevered out even in the absence of its cavity walls. This phenomenon has made an indelible image in my mind about the power of glass ionomer to bond to dentin and enamel over long periods of time.

I often tell colleagues that one of the singular most important properties of glass ionomer is that they contain water as core ingredient. Dentin is 25% water and enamel is 4% water. When considering the daily technique challenges we face in trying to create a perfect bond of (the intentionally hydrophobic) resin-based restoratives to tooth structure, it is comforting to note that glass ionomers do not require a “conversion” of the hydrophilic surface of the tooth into a hydrophobic surface prior to placing the material. Glass ionomer welcomes the wet tooth surface (without excess added moisture). When considering the clinical challenges of trying to maintain the exactly appropriate moisture amount during resin-based adhesive techniques, particularly in young children, or in small preparations, or in places with limited access or visibility, one can further appreciate the advantages of a material that is inherently hydrophilic.

We must also consider the importance of the fact that glass ionomer has a coefficient of thermal expansion nearly the same as that of dentin. Over months and years of restorations residing within a cavity preparation which expands and contracts at a significantly different amount than the material, such as with most other materials besides glass ionomer, it is no wonder that even materials with inherently good physical properties in terms of their strength can suddenly fail, or that the tooth structure itself will fail around the material. Because of the compatible expansion coefficient, glass ionomers are deemed to be excellent dentin replacements and are used in the so-called sandwich technique, wherein glass ionomer replaces the dentin lost due to decay or cavity preparation, and composite above replaces the enamel (where the incompatible expansion coefficient is less of a clinical concern exteriorly).

The fluoride release from traditional glass ionomer restorative is important for a variety of clinical reasons. Studies indicate that this fluoride release is adequate to re-mineralize the margins around the restoration after acid attack which might otherwise destroy the integrity of the margins of the restoration. Adjacent teeth can also benefit from the fluoride release. On a daily basis in my practice, I depend upon glass ionomer as a material which can not only help in restoring the tooth in which I place it, but also can “heal” an adjacent incipient lesion, seen or unseen. For example, I frequently see caries lesions on radiographs on the distal surface of a primary second molar, wherein there is no radiographic evidence of decay on the mesial surface of the juxtaposed first permanent molar, even after it has been in contact for a year or more. A glass ionomer restoration placed into the second primary molar can not only effectively restore this tooth until it naturally exfoliates, but can potentially avert progression of a seen or unseen lesion on a permanent tooth. This procedure is of high value to our patients. As we develop improved caries lesion detection methods that are ever more sensitive and specific, so will the value of glass ionomer grow for our practice.

ChemFil Rock has further expanded the developmental of traditional glass ionomers. It incorporates itaconic acid into the polyacrylic acid mixture enhancing physical and handling properties. Additionally, adding zinc into the aluminum fluorosilicate glass mixture provides improvements as described in the many studies contained in this compendium. I am pleased that DENTSPLY has launched this new and improved glass ionomer, and am certain that its introduction will increase the use of the ever more important glass ionomer material by corroborating the value of glass ionomers and by their continued clinical improvements.

Sincerely,



Dr. Joel Berg
Professor and Chair
Lloyd and Kay Chapman Chair for Oral Health
The University of Washington School of Dentistry
Seattle, Washington

Director, Department of Dentistry
Seattle Children's Hospital

2 Introduction

Conventional glass ionomers (GI), used in restorative treatment procedures, still retain an important place in a dentist's armament since this material class features a number of benefits which includes:

- Adhesion to moist tooth substrate. Less invasive preparation techniques vs. amalgam, no need for undercutting
- Chemical cure allows for single-step bulk placement without layering of material increments. Ease of packing and contouring in the cavity
- Ease of repair while maintaining the internal seal with no further loss of tooth structure due to the optical contrast of the GI restorative vs. the surrounding tooth structure.
- Anticariogenic properties and remineralization of surrounding tooth structure due to release of fluoride and calcium ions
- Thermal expansion compatible with tooth substrate
- Low shrinkage and shrinkage stress during setting
- Biocompatibility and low toxicity due to lack of monomers

Due to their forgiving nature, glass ionomers can be used in situations where other materials are not suitable either because of limitations imposed by the patient, the cavity, or the environment. The properties of dental materials are being constantly improved, and those of the glass ionomers are no exception. With ChemFil™Rock important advances have been made which make it more durable and suitable for use in stress bearing situations and in posterior cavities.

2.1 History of glass ionomer development

Glass ionomers were introduced by Wilson and Kent in 1972 (Wilson et al., 1972). The first commercial glass ionomer was introduced in the same year by DENTSPLY DETREY under the brand name ASPA. The product name was derived from the material components applied: Alumino-Silicate-Polyacrylic Acid. In order to control the setting reaction better ASPA II, containing tartaric acid as a setting modifier, was introduced as the next generation product. Later the so called "water – hardening" glass ionomer cements were introduced: In order to overcome the problem of storage stability of the liquid part, the acidic polymer was dried, milled and blended with the

glass filler to yield a GI powder which is mixed with water or a tartaric acid solution. As an example for this GI product class ChemFil™ Superior (DENTSPLY DETREY) can be mentioned. Over the last decades a lot of attempts have been undertaken to improve the performance of glass ionomers, e.g. reinforcement with glass or alumina fibres or the use of metal particles for reinforcement (cermet glass ionomer).

Further development led to high-viscosity glass ionomer restoratives especially designed for the use in the ART technique (Atraumatic Restorative Treatment) supported by the WHO (World Health Organisation).

The objective of the development of ChemFil™ Rock was to provide a high-viscosity, easy to work Glass ionomer restorative, providing higher strength and durability compared to other leading GI brands. It was shown that ChemFil Rock facilitates posterior restorations with at least 2 years of survival under simulated clinical conditions. ChemFil™ Rock comes in an easy to use mixing and dispensing capsule.

2.2 Glass ionomer mode of action

Glass ionomers are self curing materials setting by the acid-base reaction of an acidic polymer (polyacid) with a basic reactive glass filler. In the first step the glass filler powder and the aqueous polyacid liquid are being mixed leading to a hydration of the glass filler particles and an attack of the particle surface by the protons from the polyacid (Figure 1a). During this step, cations (such as Al^{3+} , Ca^{2+} , Sr^{2+}) are being released from the reactive glass filler and hydrated by water which is part of the formulation.

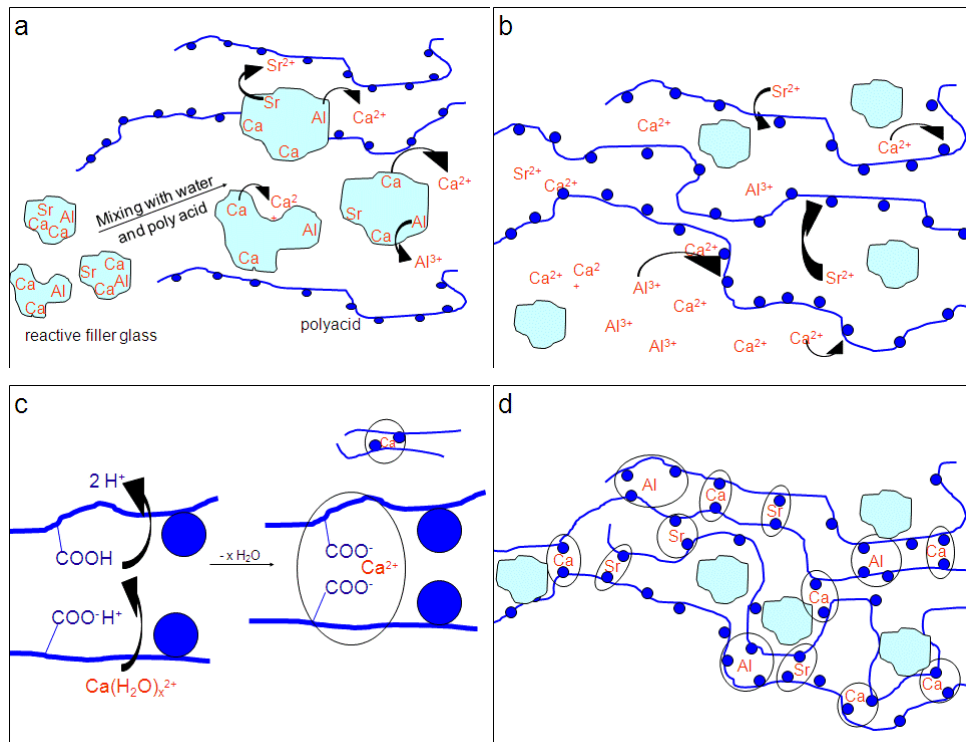


Figure 1 Schematic drawing of the glass ionomer setting reaction

The hydrated cations react with the carboxylic acid groups (COO^-) of the polyacid leading to ionic cross-linking (salt bridges) of the polyacid chains causing an increase in paste viscosity (Figure 1b and c) which finally leads to hardening of the material. Figure 1d shows the situation in the set glass ionomer where the polyacid chains are cross-linked via salt bridges by the cations and the glass filler particles are incorporated in the polyacid matrix via their surface charge.

3 ChemFil™Rock Technology

3.1 Polyacid technology

The polyacid used in a GI formulation has a strong influence on the mechanical properties, shelf life and handling properties of a GI product. From a material point of view, polyacids with high molecular weight tend to maximize mechanical strength. However, handling properties may suffer due to high viscosity of the polyacid liquid associated with its high molecular weight. Additionally, high molecular weight

polyacids are prone to gelation due to hydrogen bond formation (H-bonds), potentially reducing the shelf life of the product (see Figure 2).

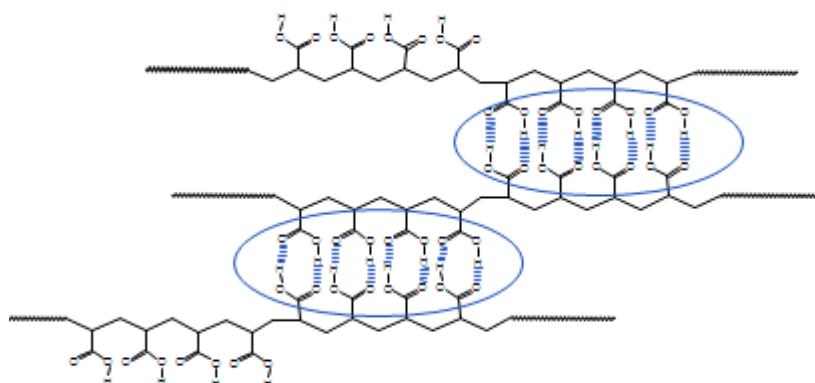


Figure 2 Schematic drawing of polyacrylic acid illustrating the aggregation of the polymer chains via H-bonds

In order to overcome this problem, the polyacrylic acid of ChemFil™ Rock comprises also itaconic acid moieties. By introducing itaconic acid the intermolecular interactions of the polyacid chains are being decreased preventing gel formation over time (see Figure 3).

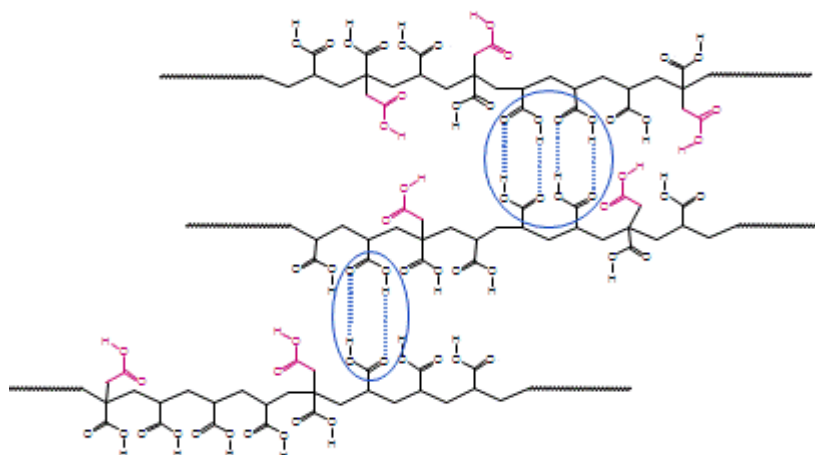


Figure 3 Illustration of itaconic acid effect on the degree of hydrogen bonding of ChemFil™ Rock polyacid chains

Hence, via the introduction of itaconic acid high molecular weight polyacid is being achieved facilitating high mechanical strength and sufficient shelf life without compromising workability of ChemFil™ Rock.

3.2 Filler technology

For ChemFil™Rock a novel reactive zinc-modified fluoro-alumino-silicate glass filler was developed. This novel reactive Zn-glass filler offers a unique ion release pattern leading to high strength of the material due to the immediate release of zinc ions during the setting reaction. The zinc ion release data of the Zn-glass in an acidic environment is depicted in Figure 4. Samples of the glass were extracted with 20% acetic acid reflecting the pH value of a freshly mixed glass ionomer. The zinc ion content of the extracts was determined by ICP-OES (inductively coupled plasma optical emission spectrometry).

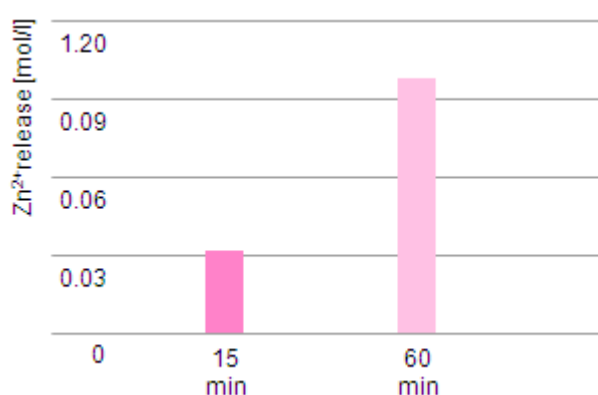


Figure 4 Cumulative zinc ion release from Zn-glass used for ChemFil™Rock

The released zinc ions form zinc-polyacid complexes which are stronger compared to complexes of other bivalent cations such as strontium or calcium ions resulting in an accelerated build-up of flexural strength. Since the strength of zinc ion complexes are comparable to those of aluminium ions, which are also released from zinc-fluoro-alumino-silicate glass filler, the final strength of ChemFil™Rock is superior to glass ionomer restoratives that do not contain zinc.

To demonstrate the effect of the novel Zn-glass filler technology, experimental formulations containing increased amounts of Zn-glass were prepared and biaxial flexural strength was determined.

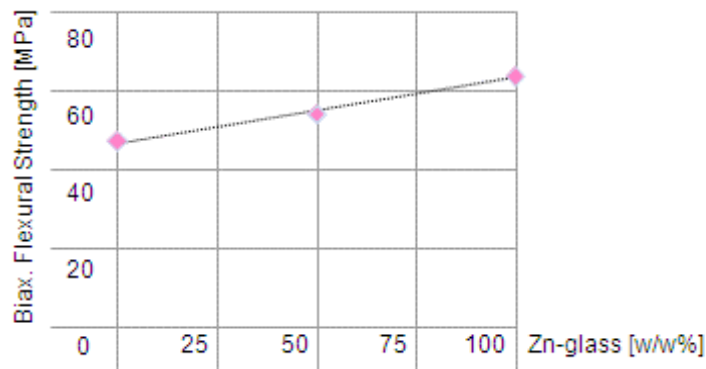


Figure 5 Biaxial flexural strength of experimental GI formulations containing Zn- and/ or Sr-glass with increasing Zn-glass weight percentage.

As depicted in Figure 5 the biaxial flexural strength of a GI formulation containing 100 w/w% of a F-Sr-Al-silicate glass (Sr-glass) was increased by replacing the Sr-glass with increasing Zn-glass weight percentage. In this context, one might refer to ChemFil™Rock as a zinc reinforced GI restorative.

The bimodal particle size distribution of the Zn-glass filler with a mean particle size of about 3.5 µm allows for a relatively high filler loading (~ 70 wt%, ~ 50 vol%) contributing to mechanical strength without compromising product handling properties. (see Figure 6)

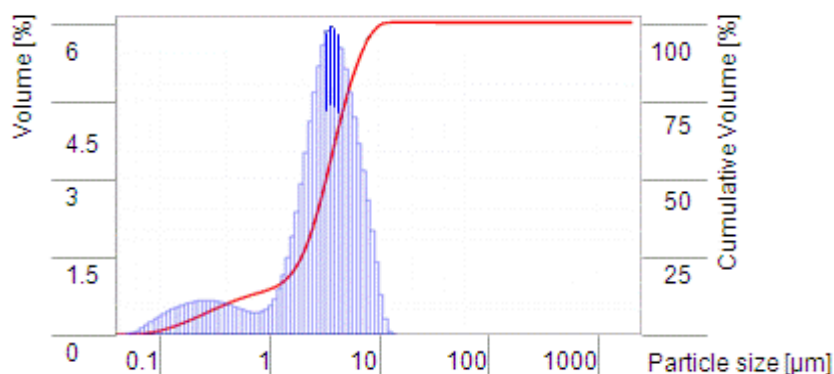


Figure 6 Bimodal particle size distribution.

4 In-vitro Investigations

This chapter describes the in-vitro studies conducted in order to verify that ChemFil™Rock is suitable for its intended use in posterior class I and II cavities.

4.1 Chewing Simulation

Frankenberger R, University of Marburg (D)

Besides wear – either generalized or in the occlusal contact area – fractures at the margins or in the bulk of the restoration are the most relevant failure mode of GI materials especially when used in large class II cavities.

In order to simulate clinical conditions chewing simulation was applied. Wear, marginal quality, and occurrence of fractures were investigated. User evaluations among European and US dentists have shown that roughly 50% of GI restorative users apply neither conditioning, nor coating, nor incremental placement and light application described in the respective instructions for use (IFU). Therefore, these techniques were included. Test parameters are listed in Figure 7.

- Method
 - n = 8 (box in dentin or enamel)
- Storage & Stressing
 - 24 hours at 37°C
 - 2500 TC 5-55°C for 30 seconds
 - 100,000 cycles with 50 N
- Evaluation
 - Margins (SEM)
 - Wear (CLSM)
 - Fractures (3.5x lupes)



Figure 7 Parameter for simulating clinical conditions by chewing simulation (Frankenberger R)

Note: Based on in vitro-in vivo comparison 100,000 chewing cycles in this chewing simulator were reported to correspond to 2 years of clinical service when investigating marginal quality of composite restorations (Frankenberger et al., 2007).

Results: For marginal quality the percentage of gap free margins are shown in Figure 8.

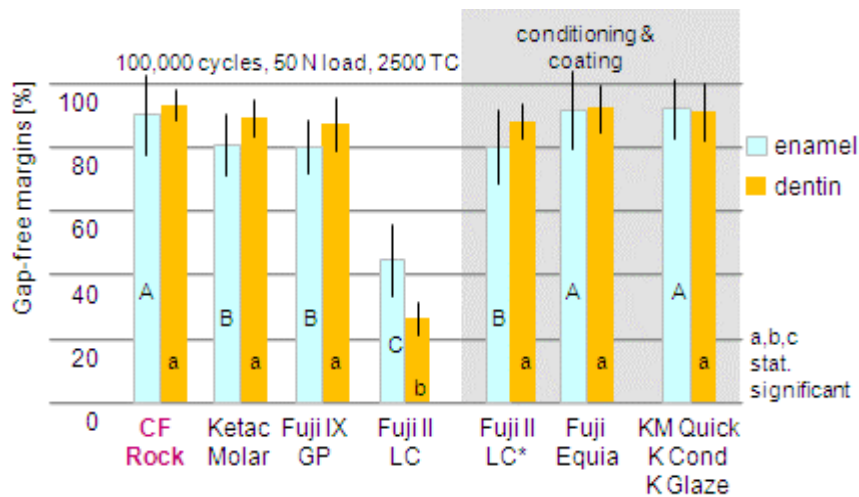


Figure 8 Percentage of gap-free margins in enamel and dentin after thermo-mechanical loading (Frankenberger R, 2010) (* increments of 1.8mm; all other materials bulk fill)

In dentin all tested GI products provided a high percentage of gap free margins except when a resin modified GI was not applied and light cured in increments. In enamel ChemFil™Rock performed better compared to most competitive products tested. Only those competitive GI systems – for which the cavity was conditioned and a coating was applied according to the respective manufacturer’s instruction for use – performed equal to ChemFil™Rock.

Important notice: For ChemFil Rock conditioning or coating is not required.

Wear in the Occlusal Contact Area (OCA) was analyzed using a Confocal Laser Scanning Microscope (CLSM) and is shown in Figure 9.

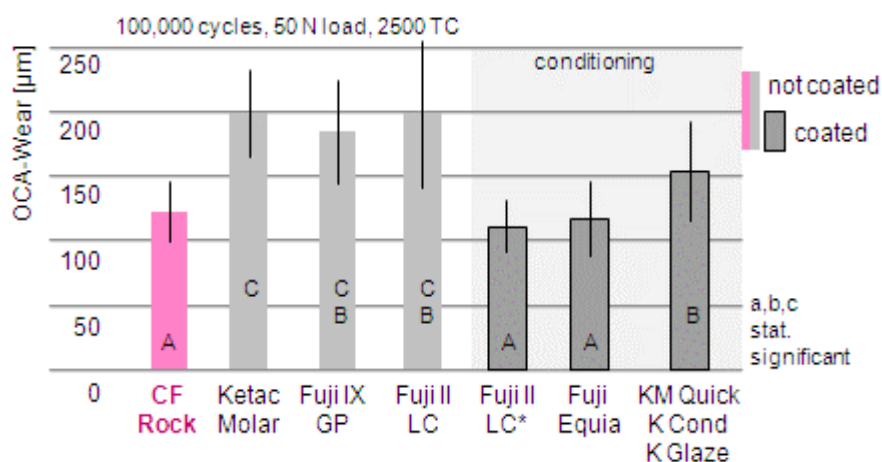


Figure 9 Occlusal wear after chewing simulation (Frankenberger R, 2010) (* increments of 1.8mm; all other materials bulk fill)

ChemFil™Rock applied in the most simplified technique – without conditioning or coating – performed better compared to other GI materials using the same technique. Even those GI restorative systems for which conditioning and coating was applied including a resin modified GI applied in 1.8mm increments did not perform better compared to ChemFil™Rock.

Besides marginal quality and wear the most interesting result obtained from the chewing simulation testing was fracture rate. Two modes of fracture were differentiated: a) marginal fracture and b) bulk fracture when more than 50% of proximal box volume was lost. Figure 10 shows total fracture rates and the contribution of each fracture mode.

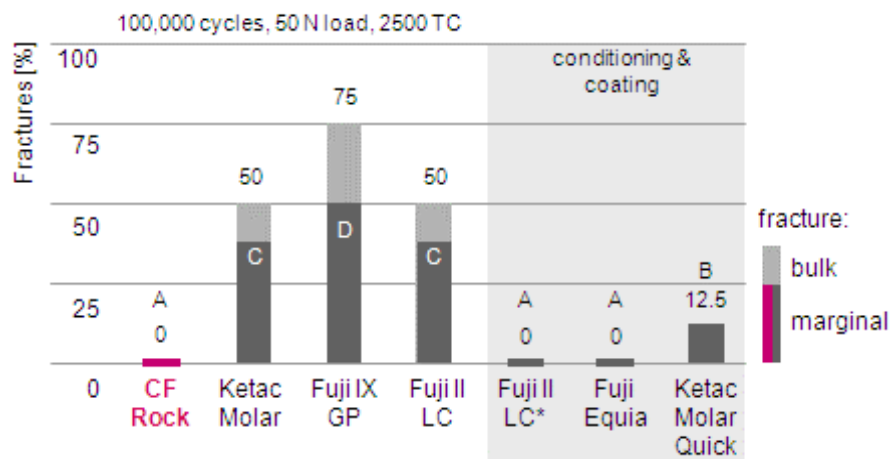


Figure 10 Total fracture rate after chewing simulation (Frankenberger R, 2010) (* increments of 1.8mm; all other materials bulk fill)

After 100,000 chewing cycles, ChemFil™Rock did neither show marginal nor bulk fracture and performed significantly better compared to most GI materials tested.

In a second run additional 100,000 chewing cycles were applied on the surviving restorations from selected materials. Results are depicted in Figures Figure 11 and Figure 12:

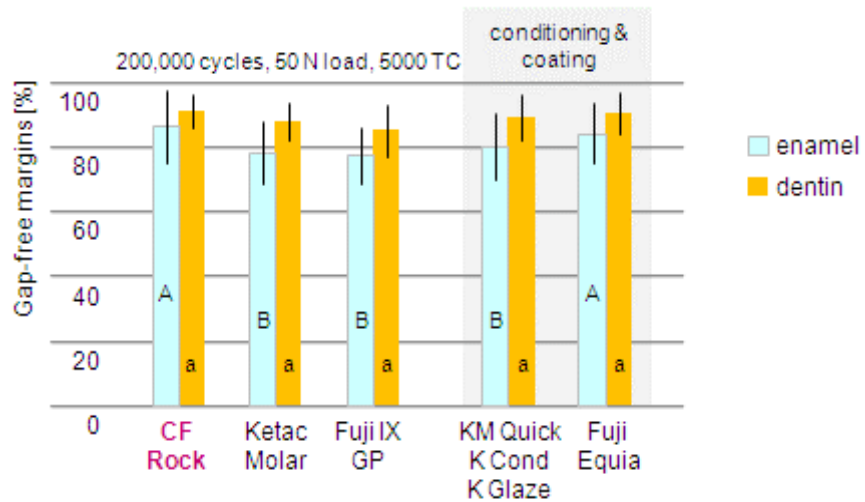


Figure 11 Percentage of gap-free margins in enamel and dentin after thermo-mechanical loading – 2nd run (Frankenberger R, 2010)

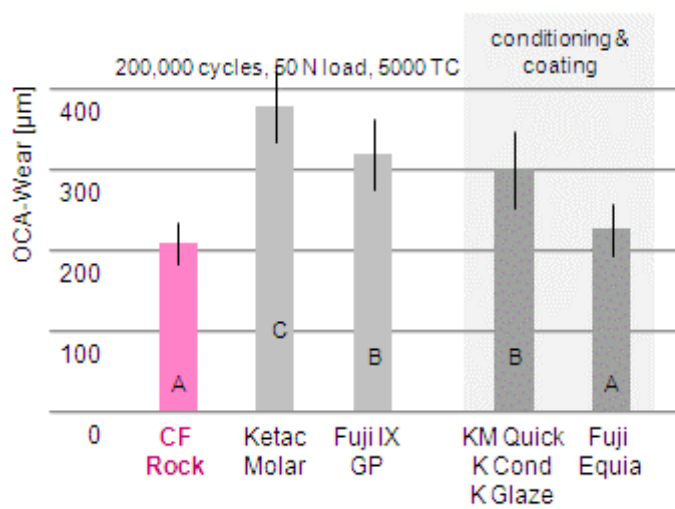


Figure 12 Occlusal wear after chewing simulation – 2nd run (Frankenberger R, 2010)

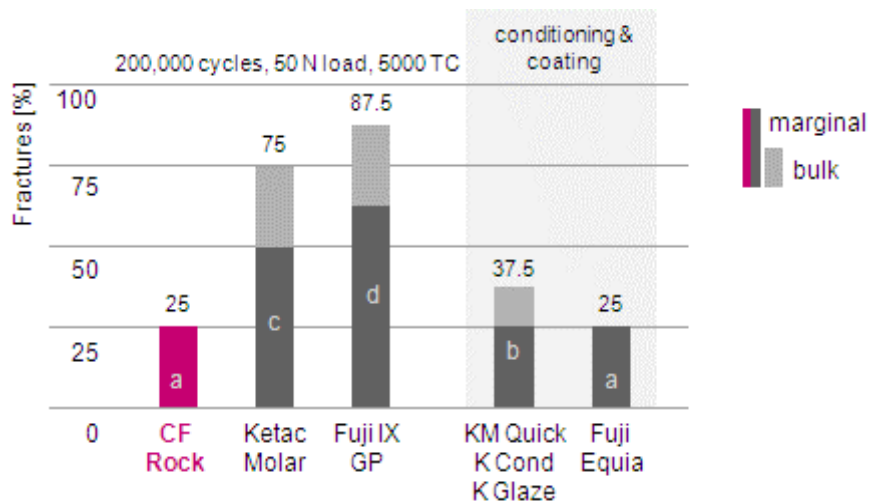


Figure 13 Total fracture rate after chewing simulation – 2nd run (Frankenberger R, 2010)

ChemFil™Rock continued to perform better compared to other GI materials used in a simplified application technique. Only Fuji Equia, for which dentin conditioning, varnishing, and glazing were applied, showed comparable results to ChemFil™Rock.

Conclusion: based on the above results achieved under simulated clinical conditions it is predicted that ChemFil Rock is suitable for and simplifies posterior restorations with at least 2 years of clinical survival.

4.2 Wear

Besides fracture strength wear resistance is the second most important property of GI materials used for posterior restorations in class I and II.

4.2.1 ACTA Wear

Kleverlaan CJ, University of Amsterdam (NL)

The three body wear simulator developed at ACTA (Academic Centre for Dentistry Amsterdam) and described by DeGee et al. in 1994 uses a spring loaded antagonist wheel which abrades the materials to be tested with a slip of 15% in a suspension of rice and millet seeds. This suspension can be buffered to different pH. (See Figure 14)

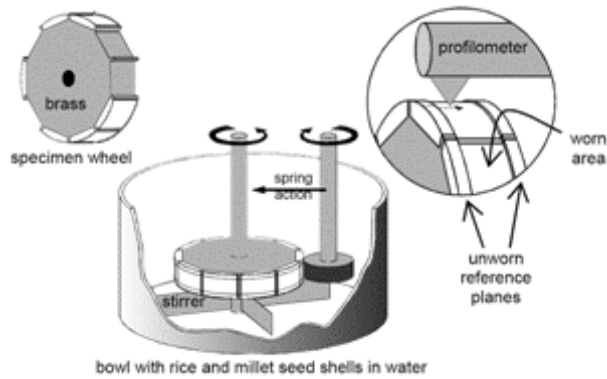


Figure 14 The ACTA wear machine (deGee A, 1994)

As the fillers in GI materials need to be made of reactive glass – being the base in the acid-base-reaction on which these materials harden – it was investigated whether the new Zn-glass used in ChemFil™Rock is resistant to lower pH that may occur when dental plaque is formed on the surface.

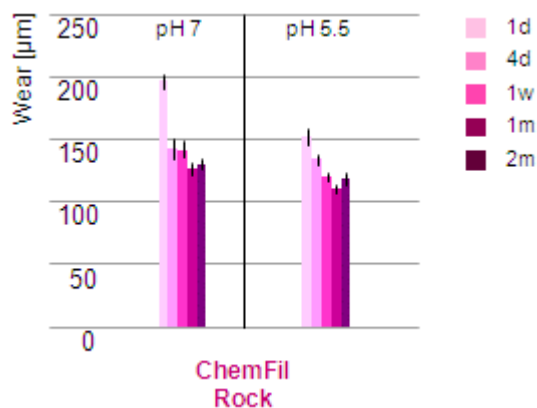


Figure 15 ACTA wear at different pH values (Kleverlaan CJ, 2009)

Though the influence of the buffer solution, which was different for both pH values, could not be tested, ChemFil™Rock showed better wear resistance under lower pH conditions (see Figure 15).

4.2.2 Leinfelder Wear

Latta MA, Omaha, NE (US)

Another three body wear machine known as “Leinfelder Wear Machine” was recently improved (Barkmeier et al., 2008). This device allows testing in two different wear modes.

In the so called generalized wear mode a steel piston is pressed while rotating through a slurry of PMMA beads towards the specimen without touching it (see Figure 16).

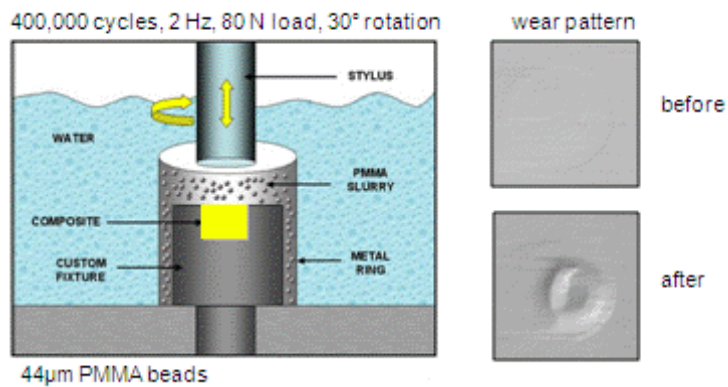


Figure 16 Generalized wear mode and typical wear pattern (Latta MA)

Results from generalized wear are given either as mean depth or volume loss. Results are shown in Figure 17.

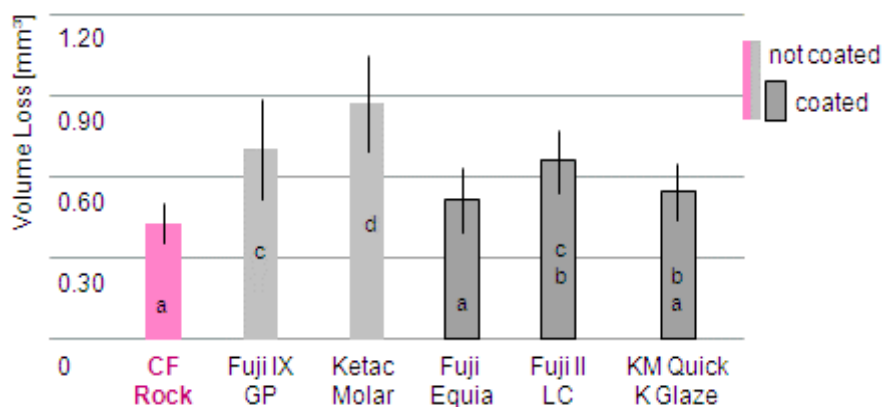


Figure 17 Volume loss in generalized mode (Latta MA, 2011)

ChemFil™Rock showed significantly less volume loss compared to a resin modified GI being coated after finishing and other GI materials when no coating was applied.

In the localized wear mode the piston holds a stainless steel bearing which is pressed against the specimen (see Figure 18).

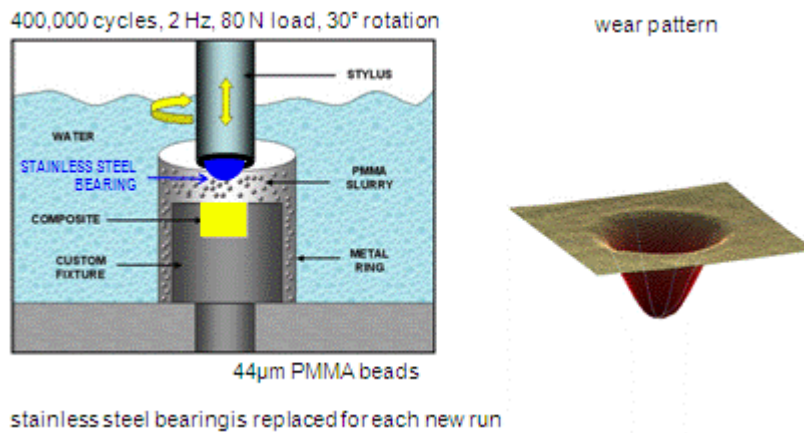


Figure 18 Localized wear mode and typical wear pattern (Latta MA)

Results from localized wear are given either as volume loss or maximum depth. Results are shown in Figure 19.

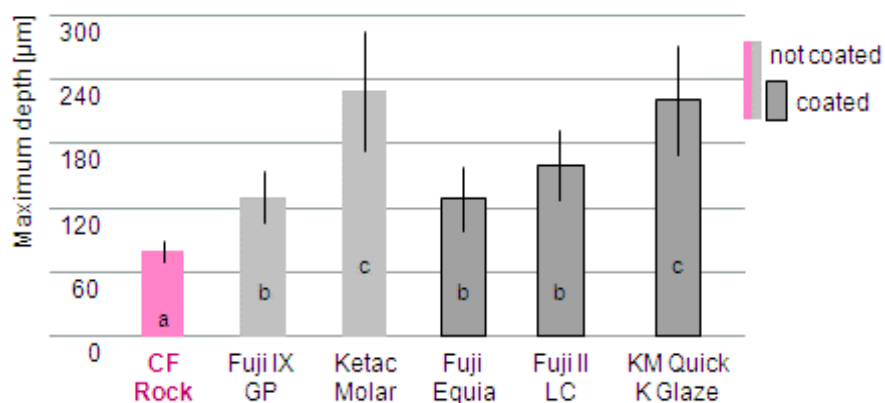


Figure 19 Maximum depth in localized mode (Latta MA, 2011)

In contrast to the generalized wear mode, coating did not have any protective influence in the localized mode. ChemFil™Rock without coating was better compared to all other GI materials tested – including a resin modified GI.

Conclusion: Based on the above results ChemFil Rock (even without coating) showed equal or better wear resistance compared to competitive materials.

4.3 Fracture Toughness

Fracture toughness indicates how much energy it takes to break a material which has already a flaw. Glass Ionomers (GI) are known for their rather brittle nature compared to composites. Once a crack is induced in brittle materials they are prone to catastrophic failure due to crack growth and crack propagation. Hence, in order to improve the durability of GI materials it is important to improve their fracture toughness. Two methods were applied to investigate fracture toughness of ChemFil™ Rock – a) fracture toughness test applying macro indentation and b) single edge notched bending (SENB) test.

4.3.1 Fracture Toughness Measurement Using Indentation

Lach R, Polymer Service Merseburg – University of Halle-Wittenberg (D)

For this test cylindrical specimens having a diameter of 10 mm and a thickness of about 3 mm were prepared. Before testing the specimens were stored in moulds for 1 h at 37 °C and > 90 % r h, and subsequently in demineralised water at 37 °C until the respective storage time was reached.

Cracks were induced by indentation using a Vickers diamond (see Figure 19).

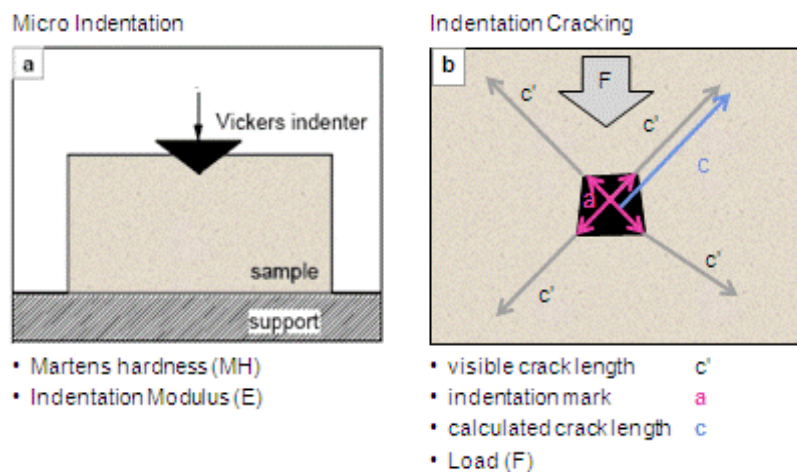


Figure 19 Measurements using micro indentation (a) and crack measurement (b) after indentation cracking (Lach R, 2009)

In order to measure Martens Hardness (MH) and indentation modulus (E) micro indentation was applied using a Vickers indenter (see Figure 19a). In a separate experiment specimens were indented with a Vickers indenter under a load (F) that induced stable cracking (such that the specimen does not fracture). It was observed that for ChemFil™Rock a significant higher load (98.1 N) was necessary to induce

stable cracks compared to Ketac Molar (29.4 N) indicating a higher resistance against crack formation of ChemFil™Rock.

Each crack (c') and the respective part of the indentation mark (a) was measured in order to calculate the true crack length (c) which is the sum of the half length of the impression diagonal a and the visual crack length c' (see Figure 19b). Fracture toughness was calculated according to the following formula:

$$K_I = 0.0015 \cdot \left(\frac{c}{a} - 1\right)^{-1/2} \cdot \left(\frac{E}{MH}\right)^{2/3} \cdot \frac{F}{c^{3/2}}$$

(E = Indentation modulus; MH = Martens Hardness; F = load;
0.015 = empirical factor)

Formula 1 Formula for fracture toughness based on micro indentation measurements and indentation cracking

Using formula (1) fracture toughness for each crack was calculated. From the resulting data median values were calculated. In order monitor fracture toughness development over time the median value of ChemFil™Rock at each time point was set to 100% and the relative fracture toughness was calculated respectively for the control materials.

Figure 20 shows an overview of the relative fracture toughness data obtained up to 6 months after preparation.

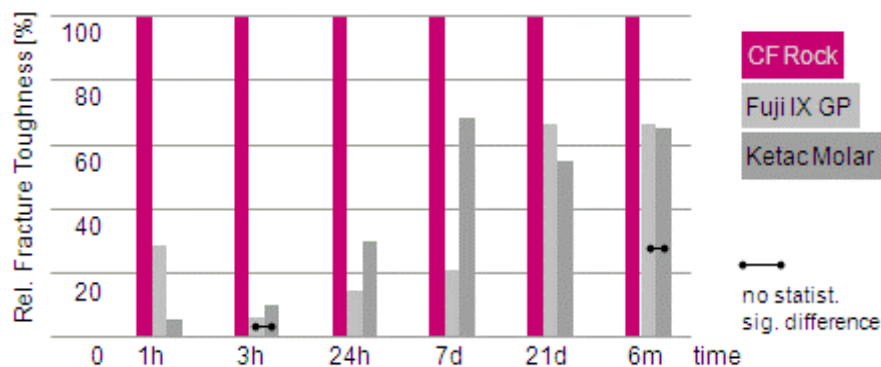


Figure 20 Relative Fracture Toughness over time using indentation cracking (Lach R, 2009)

Conclusion: From the above results it can be concluded that ChemFil™Rock is much more resistant against crack formation induced by indentation like loading (i.e. sharp occlusal contacts). In addition ChemFil™Rock shows much higher fracture toughness especially in the early phase after initial setting.

4.3.2 Fracture Toughness by SENB

Lohbauer U, University of Erlangen (D)

For this method bar shaped specimens (2 x 2 x 25 mm) were prepared. The specimens were removed from the moulds after 5 min of air drying and 10 min storage in distilled water. A notch was cut to almost 1/3 of each specimen thickness and was then sharpened with a razor blade using a custom made device to control load and depth of sharpening (see Figure 21).

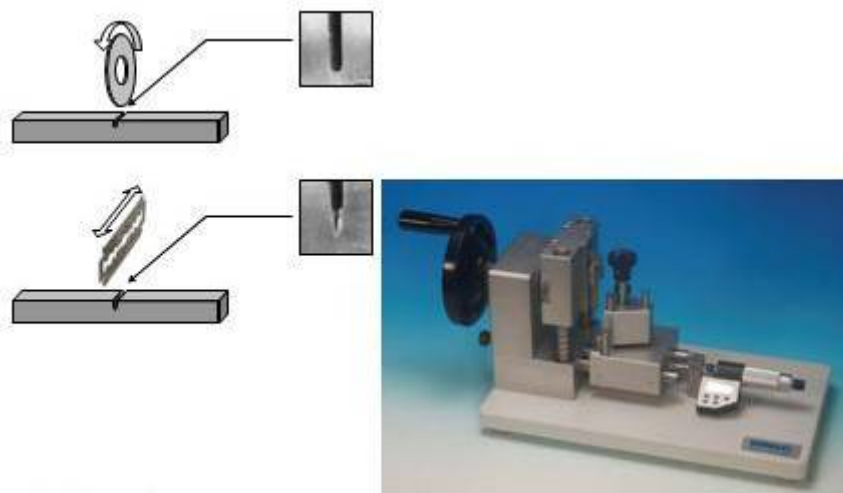


Figure 21 Notch cutting and sharpening using a custom made device (Lohbauer U, 2009)

After breaking the specimen in a four point bending apparatus the crack length was measured under light microscope using 40x magnification (see Figure 22).

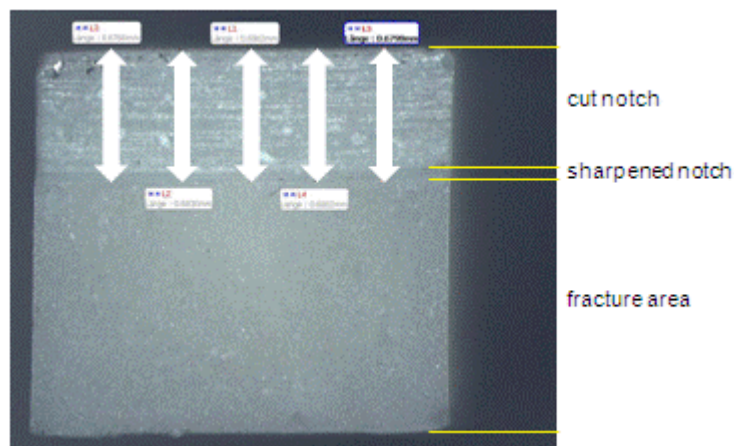


Figure 22 Measurement of crack length (Lohbauer U, 2009)

In this study fracture toughness was measured after 3 hours, 24 hours, 7 days, and 21 days storage in distilled water at 37 °C.

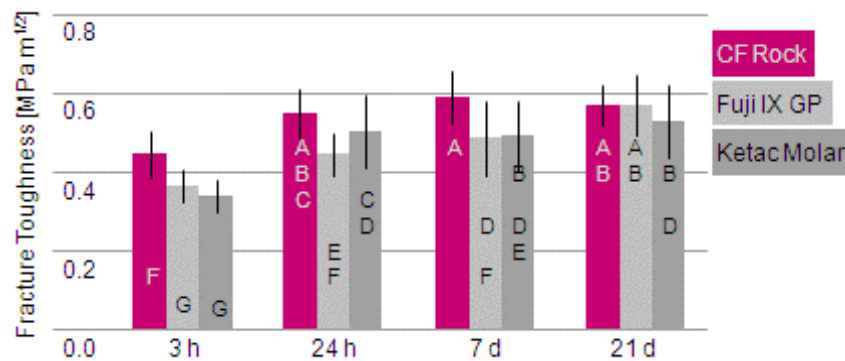


Figure 23 Fracture Toughness over time (Belli R, 2010)

As can be seen from Figure 23 ChemFil™Rock showed significantly higher fracture toughness (K_{Ic}) at 3 hours compared to the control materials and up to 7 days compared to Fuji IX GP.

Conclusion: If flaws were induced in the early phase after setting it would take more energy to break ChemFil™Rock compared to the control materials.

4.4 Other Material Properties

4.4.1 Biaxial flexural strength

The flexural strength test is being considered to produce the most appropriate measure of a GI material strength (Lohbauer 2010). As a test method the biaxial flexural strength described by Ban and Hasegawa (Ban et al., 1992) was chosen as the specimen preparation is easier and more reliable compared to the method described in ISO 4049. This is supported by a publication from Pick et al. (Pick et al., 2010) who reported that the biaxial flexural strength method applied for composites produced more consistent results.

To determine the biaxial flexural strength disc like specimens (20 x 1 mm) were prepared according to the instructions for use. The specimen were applied on a ring-shaped support of 15 mm diameter and loaded centrally with a ball like piston until failure. The force necessary to break the specimen was recorded and the biaxial flexural strength was calculated.

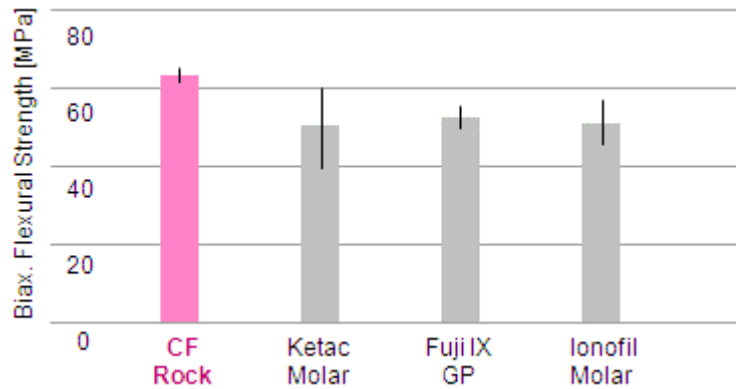


Figure 24 Biaxial flexural strength of ChemFil™Rock and competitor materials

Figure 24 shows the biaxial flexural strength for ChemFil™Rock and competitive materials demonstrating the superior flexural strength of ChemFil™Rock.

4.4.2 Fluoride release

Since fluoride has caries protective effects a continuous fluoride release over a long period is desirable. For ChemFil™Rock and the competitor materials fluoride release was determined according to DIN 38405-4:1985 using an ion selective electrode.

Figure 25 shows the results of the fluoride release investigations conducted with ChemFil™Rock, Ketac Molar Aplicap, Fuji IX GP, Fuji IX GP Extra and Ionofil Molar AC.

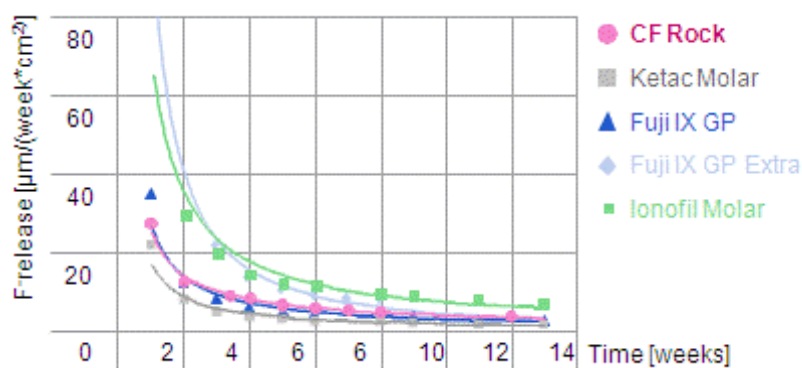


Figure 25 Fluoride release over 3 months for ChemFil™Rock and competitor materials

For Fuji IX GP Extra and Ionofil Molar a large amount of fluoride is released within the first week but after about 1 month when all the materials had reached a steady state the release of fluoride was similar for all tested materials.

4.4.3 Adhesion to enamel and dentin

For adhesion testing human teeth were used without pre-treating the enamel and dentin surface with a cavity conditioner.

In order to determine the adhesion the specimens were supported by a metal ring during shear testing leading to adhesive fractures (Figure 26).



Figure 26 Glass ionomer specimen supported by a metal ring for shear bond strength testing

The results from the adhesion testing to enamel are shown in Figure 27. Pair wise statistical analysis by student's t-test showed that the adhesion of ChemFil™Rock to enamel is comparable to the adhesion of Ketac Molar Aplicap but statistically significant higher than the adhesion of Fuji IX GP.

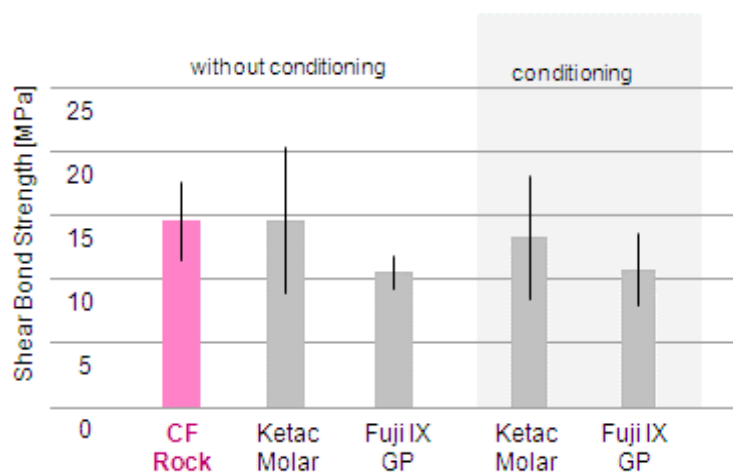


Figure 27 Adhesion to enamel of ChemFil™Rock and the main competitors

Figure 28 shows the results for the adhesion to dentine. Again pair wise statistical analysis by students t-test showed that the adhesion of ChemFil™Rock to dentin is comparable to the adhesion of Ketac Molar Aplicap and Fuji IX GP.

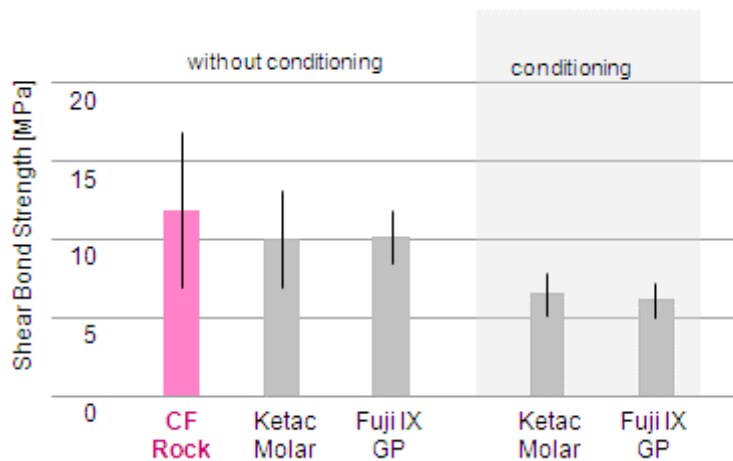


Figure 28 Adhesion to dentin of ChemFil™Rock and competitor materials

4.4.4 Acid erosion according to ISO 9917-1:2007

Acid erosion is a test to simulate the material behaviour when stored under low pH conditions like beneath plaque where the pH-value is in the acidic region due to acid production by bacteria. The acid erosion of ChemFil™Rock, Ketac Molar Aplicap, Fuji IX GP Caps and Ionofil Molar AC was determined according to ISO 9917-1:2007. Specimens are prepared in a PMMA cavity of 5.0 ± 0.5 mm in diameter and 2.0 ± 0.5 mm in depth. After storage for 24 h at 37 °C and > 90 % r. h. the specimens were ground and their height compared to the PMMA plate is determined. Afterwards the specimens were immersed in a 1 molar buffered lactic acid solution of pH = 2.74. The specimens were stored in the lactic acid solution at 37 °C for 24 h and their height relative to the PMMA plate was determined afterwards. The acid erosion was derived from the difference of the measurement before and after storage in the lactic acid solution.

The results of the acid erosion test are shown in Figure 29 demonstrating that for all materials tested the ISO limit of 0.17 mm was easily met.

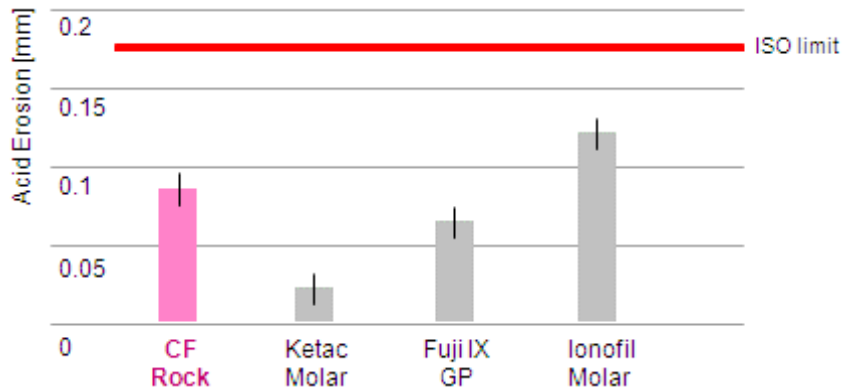


Figure 29 Acid erosion of ChemFil™Rock and competitor materials according to ISO 9917-1:2007

4.4.5 Opacity and color

Opacity and color of the materials were determined using a Luci 100 (Dr. Lange GmbH, Germany) color determination instrument. L a b values are measured using white and black backgrounds. From these L a b values shade and opacity was determined. ChemFil™Rock shades were adjusted to the Vita Classical Shade Guide allowing shade selection comparable to composites. The high opacity of ChemFil™Rock assures ideal contrast to the tooth substance for easy and complete removal of the material when using it as a temporary restorative.

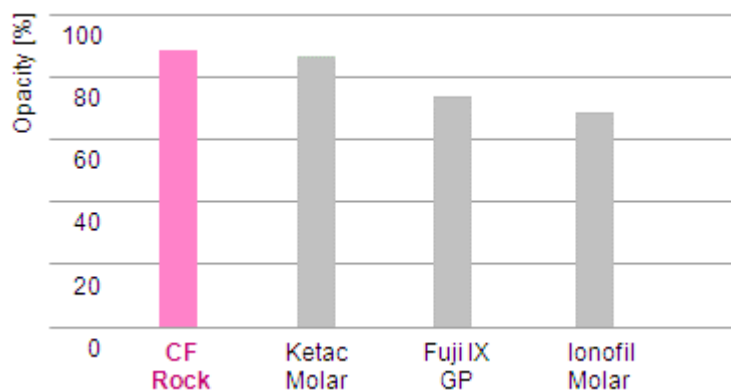


Figure 30 Opacity of ChemFil™Rock and competitor materials

4.5 Summary in vitro studies

Based on the above in-vitro studies the most important properties of ChemFil™ Rock in comparison to other GI materials can be summarized as following:

- ChemFil™Rock has superior biaxial flexural strength
- ChemFil™Rock is more resistant against crack formation induced by indentation. Fracture toughness is significantly higher especially in the early phase after initial setting.
- Based on its higher fracture and wear resistance it is expected that ChemFil™ Rock is suitable for and simplifies posterior restorations with at least 2 years of clinical survival.

4.6 Technical data sheet

Typical technical data of ChemFil™Rock are listed below:

| | | |
|-----------------------------|-------|---------|
| Compressive strength | MPa | 200 |
| Flexural strength (Biaxial) | MPa | 63 |
| Filler content (weight) | % | ≤ 70 |
| Glass filler size (mean) | µm | 3.5 |
| Molecular weight of polymer | Da | 120,000 |
| Acid erosion | mm | 0.08 |
| Expansion in water (linear) | % | 0.02 |
| Radiopacity | mm Al | 2 |
| Opacity | % | 80 – 90 |
| Working time | min | > 1.5 |
| Setting time | min | 3 |
| Minimum time before Finish | min | 6 |
| Extrudable amount | mg | ≥ 280 |

5 Clinical study

ChemFil™Rock is presently evaluated in one clinical multi-center study. A brief information of which can be found below.

5.1 Post-marketing Surveillance Study on the Clinical Performance of ChemFil™Rock in Class II Lesions

In this longitudinal prospective randomized post-marketing surveillance study, posterior glass ionomer restorations (Class II) are placed and evaluated by a group of dental practitioners in their own practice.

The objective of the post-marketing surveillance study is to demonstrate the product's safety and efficacy for restoration of Class II cavities in permanent posterior teeth.

26 dental practitioners have placed 214 ChemFil™Rock ("K130") and 209 Ketac Molar ("KMA") restorations, which were routinely made under conditions of their practices. In order to assess the performance of the materials, the restorations made and included into the study are monitored by the respective dentists for a period of up to 36 months.

A short design overview is provided in Table 1.

Class II Trial

| | |
|---------------------|---------------------------|
| Scientific Advisor: | Prof. Dr. Reinhard HICKEL |
|---------------------|---------------------------|

Design

| | |
|--|--|
| Number of Study Sites: | 26 dental surgeries, previously using Ketac™ Molar Aplicap™ (3M ESPE) which have switched to ChemFil™Rock |
| Teeth: | First or second molars where after cavity preparation at least one of the occlusal contacts is on sound tooth substance. |
| Number of Restorations: | 423 (214 with ChemFil™Rock and 209 with Ketac™ Molar Aplicap™). |
| Number of Patients: | 423 |
| Method of Evaluation: | Clinical Examination |
| Number of Patients per observation period: | ≥ 60 |
| Observation Periods: | Baseline (1 week – 1 month), 2-4 months, 4-8 months, 9-15 months, 16-24 months, 25-36 months. |

Table 1 Design aspects of Class II trial

423 fillings in 423 patients with Glass Ionomer restorative materials were placed and included for reevaluation (209 with Ketac Molar, 3M ESPE, and 214 with ChemFil™Rock, DENTSPLY DETREY).

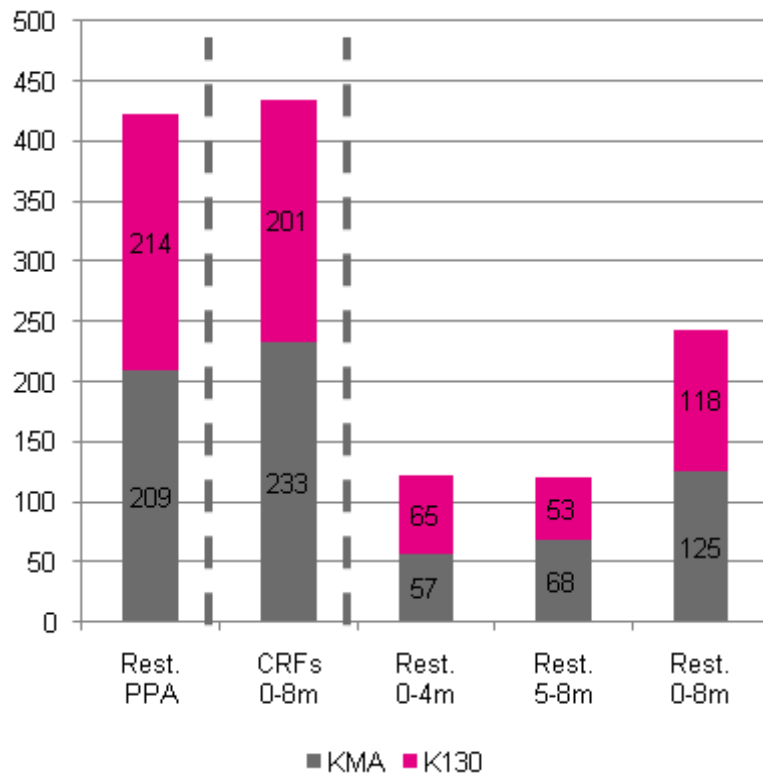


Figure 31 Restorations included; Case Record Forms (CRFs) received; Restorations recalled

122 restorations (KMA: 57; K130: 65) could be reevaluated in the period from 0 to 4 months. Each material showed 3 failures (KMA: 1 “Tooth Integrity” and 2 “Post.-op. Sensitivity and Vitality”; K-130: 1 “Fracture and Retention” and 2 “Post.-op. Sensitivity and Vitality”).

During the second interval, 5 to 8 months, 121 restorations (KMA: 68; K130: 53) have been reevaluated. K130 showed no failures; KMA showed in total 6 failures (1 “Tooth Integrity”, 2 “Post.-op. Sensitivity and Vitality”, 3 “Fracture and Retention”) of which one was a combined failure for “Fracture and Retention” and “Post.-op. Sensitivity and Vitality”.

In total since placement 11 replacements (8 Ketac Molar, 6.4%; 3 ChemFil™Rock, 2.5%) of 243 reevaluated fillings had to be done.

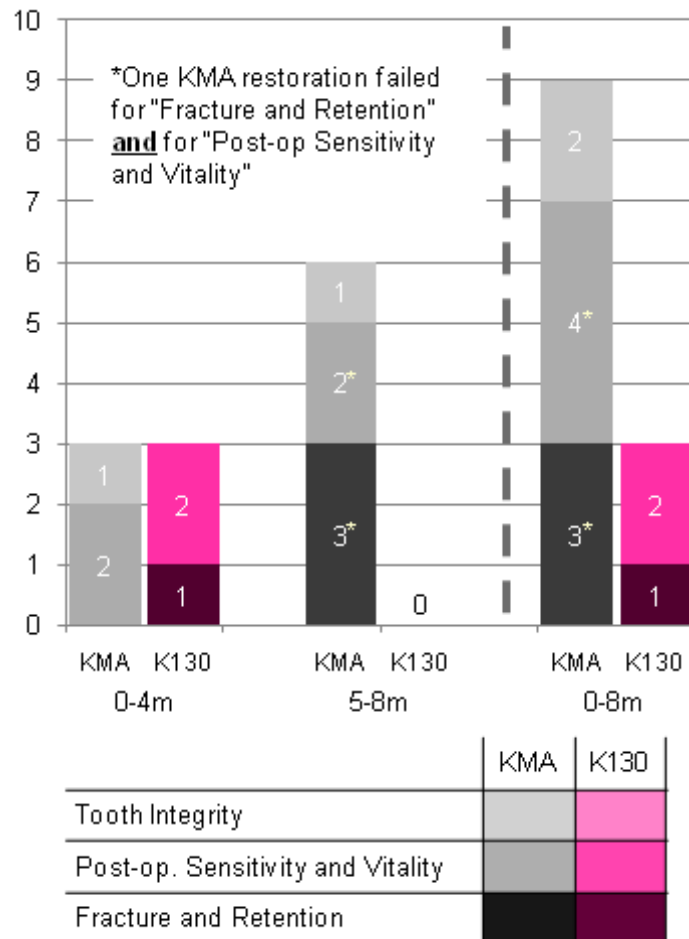


Figure 32 Failures

No adverse events were recorded.

6 Directions for Use

The up-to-date directions for use can be found in all European official languages on the Internet at www.dentsply.eu.

7 References

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8 Glossary and Abbreviations

| | |
|---------|--|
| CLSM | Confocal Laser Scanning Microscope |
| GI | Glass Ionomers |
| ICP-OES | Inductively Coupled Plasma Optical Emission Spectrometry |
| IFU | Instructions for Use |
| OCA | Occlusal Contact Area |
| SENB | single edge notched bending |
| SFR | static flexural strength |
| TC | Thermo Cycling |
| TML | Thermo-Mechanical Loading |

The following materials are not trademarks of DENTSPLY International:

Brand (abbreviation(s); Manufacturer):

Ketac Conditioner (KC, K Cond; 3M ESPE)

Ketac Glaze (KG, K Glaze; 3M ESPE)

Ketac Molar Applicap (KM; KMA, 3M ESPE)

Ketac Molar Quick (KMQ, KM Quick; 3M ESPE)

Fuji II LC (GC)

Fuji IX GP (FIX; GC)

Fuji Equia (GC)

Ionofil Molar (IM; Voco)