# The effect of resin based coatings on fluoride release of glass ionomer cement, an *in vitro* study.

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**A mini-thesis submitted in partial fulfilment of the requirements for the degree of MSc in Paediatric Dentistry**

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**April 2018**

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***MSc Mini thesis, Faculty of Dentistry, Department of Paediatric Dentistry, University of the Western Cape***

## ABSTRACT

The use of glass ionomer cement (GIC) restorative materials assists in the prevention of dental caries due to its long-term fluoride release. However, poor physical strength is one of the main drawbacks of GIC. A surface coating is recommended to improve the physical strength and is considered necessary during the overlapping stages of setting reactions of GIC restorative materials. The development of resin based coatings has improved the properties of the material but the effect on fluoride release needs investigation.

**Aim** To study the effect of three resin-based coatings on the fluoride release property of a conventional glass ionomer restorative material, namely Ketac Molar Aplicap ®.

**Methods**

Twenty eight disc-shaped specimens were fabricated from the conventional GIC Ketac Molar Aplicap (3M ESPE, Europe) and divided into four groups (Group I, II, III and IV) of seven each. Group I specimens were uncoated (control), group II coated with G-coat plus, group III coated with Scotchbond Universal and group IV coated with Riva coat. After polymerization, the discs were immersed in individual plastic bottles containing deionized double distilled water and the bottles were sealed. The fluoride concentration of the water in each container was changed at 24, 48, and 72 hours using an Orion fluoride ion selective electrode (Orion Research Inc, Boston, USA) using a standard technique after calibration with 2.5, 5 and 10 part per million of standard fluoride solution. After each fluoride measurement, the deionized distilled water was replaced with new deionized distilled water. The fluoride concentration in the water was used as an indication of fluoride released from the glass ionomer cement. The 3 different 'coating' materials were strategically chosen to represent different formulations.

**Result**

This study found that fluoride release during the entire period of the experiment was the greatest from the uncoated group, followed in ranking by Riva coat, Scotchbond coated and G coat plus coated samples.

## Conclusion

Resin based coatings reduced the fluoride release from GIC samples, particularly during the first seven days. Unfilled resin based coating (Riva coat) behaved differently by allowing more fluoride release compared to microfilled (Scotchbond Universal and nanofilled (G coat plus) coatings. However, microfilled coatings and nanofilled coatings allowed for a slow and steady release of fluoride. Therefore, it is recommended that these protective coatings be placed over GIC restorative materials.

# KEYWORDS

Glass ionomer cement

Resin-based coatings

Nanofilled coatings

Microfilled coatings

Acrylic coatings

Fluoride Release

Physical Properties

# LIST OF ABBREVIATIONS

| | |
|---|---|
| **GIC** | Glass ionomer cement |
| **F** | Fluoride |
| **Ca** | Calcium |
| **Al** | Aluminum |
| **ISE** | Ion selective electrode |
| **IC** | Ion chromatography |
| **pH** | potential Hydrogen |
| **TISAB** | Total Ionic Strength Adjustment Buffer |
| **GC** | G coat plus |
| **SB** | Scotchbond Universal |
| **RC** | Riva coat |

# DECLARATION

I declare that the thesis entitled "*The effect of resin based coatings on the fluoride release of glass ionomer restorative material*" is my own work, that it has not been submitted before for any degree or examination in any other university, and that all the sources I have used or quoted have been indicated and acknowledged as complete references.

Fayez N Shatat

April 2018

Signature.

# ACKNOWLEDGEMENTS

I would like to first say a very big thank you to my supervisor, Dr. Peerbhay and my co-supervisor, Dr. Adam for the patient guidance and assistance during my research work.

I thank profusely Prof Mohamed, the head of department, for her endless support during my study. The door to Prof. Mohamed's office was always open whenever I ran into a trouble spot.

I would also like to show my gratitude to Prof. Grobler for his assistance and guidance during the lab work.

I am gratefully indebted to Ms. Basson for her very valuable comments on this thesis.

Finally, I must express my very profound gratitude to the staff of the medical library and the staff of the Paediatric Dentistry Department who directly or indirectly assisted and motivated me to complete this study.

# DEDICATION

*This thesis is dedicated to:*

*My parents*
*My Brothers and Sisters*
*My close friends Hisham, Ibrahim, Abdul Rahman & Jasim*

*Thank you for your endless love, encouragement and support.*

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# CHAPTER 1:

## INTRODUCTION AND LITERATURE REVIEW

### 1.1 Introduction:

Currently there are a variety of dental materials to restore primary teeth. Such a range of dental restorative materials may cause confusion for clinicians (Berg, 1998). Most of these materials are tooth colored and have been in high demand for the past decade. Some of the available tooth colored materials used in dental practice is composite resins, conventional glass ionomer cements (GIC), resin modified glass ionomer cements (RMGIC) and polyacrylic acid modified composites (Compomers) (Burke *et al*., 2002).

Direct restorative materials in dentistry are used mainly for replacing missing tooth structure. Ideal properties of a restorative material include good adhesion to tooth structure, high physical strength, high aesthetic and bacteriostatic properties, biocompatibility and similar wear rate and thermal expansion to tooth structure (Rodríguez-Farre *et al*., 2016). In general, dental restorative materials are usually categorized in three main groups: amalgam, composites and glass ionomers (Lohbauer, 2009).

Glass ionomers exhibit specific advantages as restorative materials in the primary dentition (Hickel and Voss, 1990; Cho and Cheng, 1999). These advantages are mainly due to the ability to release fluoride and the ability to chemically adhere to mineralized tooth structure (Cho and Cheng, 1999). The ease of use and the short application time is another advantage when treating paediatric patients (Hickel and Voss, 1990; Cho and Cheng, 1999).

## 1.2 Literature review

### 1.2.1 Glass Ionomer development

Glass ionomer cements (GICs) consist of a powder and liquid that is mixed to set as a solid. It was originally invented by Wilson and Kent in 1969. The newly developed material was first used in Europe in 1972 and in North America in 1977 (Valanezhad *et al.*, 2016). It was seen as a replacement for the zinc poly-carboxylate cements which had been around for many years. Zinc poly-carboxylate cements could not be used as restorative materials because of the high solubility of unsatisfactory mechanical properties and unacceptable aesthetics. Glass ionomer cements exhibited outstanding properties such as specific adhesion to tooth structure and fluoride release. The use of these materials in pediatric dentistry makes it an interesting tool in the prevention of dental caries in children.

### 1.2.2 Glass ionomer classification and clinical properties

Glass ionomer restorative materials are generally classified according to their function namely: luting, restorative and lining/base cements (Croll and Nicholson, 2002). The chemistry for the three categories is similar, however, they exhibit variations in powder /liquid ratio and in the size of the powder particles (Nagaraja Upadhya and Kishore, 2005; Lohbauer, 2009). These variations are present to accommodate the clinical situation and the desired function (Nagaraja Upadhya and Kishore, 2005). Restorative cements may be further categorized into conventional, metal-reinforced and resin-modified cements (Cho and Cheng, 1999).

The use of GIC is highly versatile in restorative dentistry. Besides the reasonable aesthetic properties of GIC compared to amalgam, it has great clinical potential due to its unique adhesion. Glass ionomers chemically bond to the dental structures (Marquezan and Raggio, 2009). The adhesion of the cement is due to the ionic bonds formed between the tooth and the cement (Berg, 1998). This chemical adhesion of GICs to tooth structure and its ability to release fluoride renders it a valuable material in preventing microleakage and recurrent caries (Marquezan *et al.*, 2010).

These materials are highly biocompatible because of the weak and high molecular acid used, namely polyacrylic acid, which cannot diffuse through the dentinal tubules (Rizzante *et al.*, 2015). However, GIC is characterized by weak mechanical strength and sensitivity to moisture during the first setting stages (Bonifácio *et al.*, 2012).

### 1.2.3 Glass ionomer composition and setting reaction

The glass component of the GIC consists mainly of silica ($SiO_2$), alumina ($Al_2O_3$) and calcium fluoride ($CaF_2$), while the liquid can be of a wide range of polyacrylic acid analogues (Khoroushi and Keshani, 2013) with a concentration of forty five percent (Lohbauer, 2009). Both components: the acidic liquid and the basic powder may be substituted by polyalkenoic acid dried into the powder and water as a liquid (Tyas and Burrow, 2004).

The initial setting reaction of GIC occurs a few minutes after mixing. The mixing procedure can be done manually using a spatula on a pad or glass block (Sidhu and Nicholson, 2016). The material can also be mixed in a measured ratio inside a bespoke capsule with a membrane separating the two parts. Once the membrane has been broken, the capsule is immediately placed and vibrated rapidly in a specially designed auto-mixer (Sidhu and Nicholson, 2016).

The setting reaction process is based on an acid-base reaction that includes dissolution, gelation, hardening and maturation (Khoroushi and Keshani, 2013). As seen in Figure 1, the acid destroys the glass network and releases metal cations such as $Al_3$+ and $Ca_2$+ to form a polyacrylate matrix (Sidhu and Nicholson, 2016). This process starts within 10 minutes after mixing. The material at this stage is sensitive to water uptake for up to one hour (Bonifácio *et al.*, 2012). Fluoride and sodium ions are then released but they do not play any role in the setting process (Bonifácio *et al.*, 2012). As the reaction continues, more metal cations are slowly released within the matrix lasting for the first 24 hours (Francisconi *et al.*, 2009; Bonifácio *et al.*, 2012). At this stage of the reaction, the material is sensitive to dehydration (Bonifácio *et al.*, 2012).

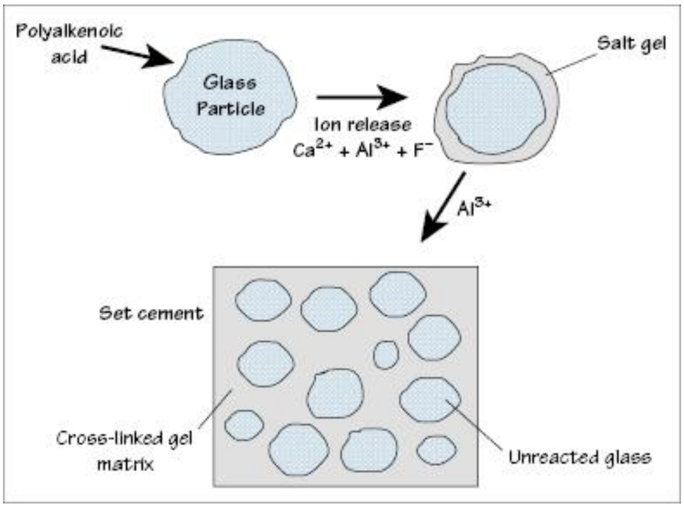


**Figure 1: Glass ionomer cement setting reaction (Von Fraunhofer, 2013)**

### 1.2.4 Glass ionomer clinical uses:

Glass ionomers have a wide range of clinical uses because their physical properties can be modified (Nicholson, 1998; Lohbauer, 2009). However, their poor physical properties limit their use as permanent restorations in stress bearing areas (Xie and Brantley, 2000). Glass ionomers may be used in primary dentition for class I preparations (Curzon *et al.*, 1996). They are the best choice to cement stainless steel crowns in primary teeth (Croll and Nicholson, 2002). Moreover, they are used to cement space maintainers and single orthodontic bands (Francisconi *et al*., 2009). GIC has been successfully used as a sealant to seal fissured primary molars and partially erupted permanent molars (Manhart *et al*. 2000). Resin modified glass ionomer is used for small to medium sized Class II, small Class III and Class V restorations (Rutaret *et al.*, 2002).

### 1.2.5 Fluoride release and recharge

#### 1.2.5.1 Fluoride release

GIC has the ability to release fluoride in the short and long term (Mousavinasab and Meyers, 2009). The initial fluoride release is considered crucial in preventive dentistry for those who are at high risk for caries (Zafar and Ahmed, 2015). The high release of fluoride in the first few days is rapid, followed by a gradual reduction and then a constant release (De Moor *et al.*, 1996; Mousavinasab and Meyers, 2009).

Fluoride is released through three possible mechanisms: superficial rinsing, diffusion through micro pores and mass diffusion (De Moor *et al.*, 1996). The first mechanism is more rapid and includes the dissolution and flush of fluoride from the superficial surface of GIC, which is in contact with the oral environment (Dionysopoulosa, 2014). This mechanism continues up to four weeks once GIC is exposed to an aqueous medium (Nicholson, 1998). The other two mechanisms are slower and more gradual. It includes sustained diffusion of fluoride from the inner core bulk of GIC through pores and cracks (Dionysopoulosa, 2014).

Many studies have been conducted both *in vivo* and *in vitro* to predict the long-term fluoride release from GIC (Hatibovic-Kofman and Koch, 1991; Forsten, 1998; Mousavinasab and Meyers, 2009; Nicholson, 2015). Karantakis *et al*. (2000) established that the highest fluoride release of GIC was recorded in the first 4 hours after mixing, *in vitro*. The initial fluoride release may vary with the different commercial brands of GIC restorative materials (Attar and Turgut, 2003; Bell *et al*., 1999; Creanor *et al*., 1994). It was found that GIC restorations release fluoride over a period lasting several months to over three years (Wiegand *et al*., 2007).

Fluoride release is considered to be a complex process and is influenced by many factors such as the composition of saliva, the pH value of the oral environment and biofilm formation (Dionysopoulos, 2014). Procedural factors which include the powder - liquid ratio, mixing procedure and the curing time can also affect the release of fluoride (Wiegand *et al.*, 2007).

Studies showed that the pH of saliva and salivary enzymes may have a direct effect on GIC and its fluoride release (Bell *et al.*, 1999; Yan *et al.*, 2007; Carvalho *et al.*, 1999; Rošin-Grget and Linčir, 2001). The decrease in the pH of saliva increases the dissolution of the material, which in turn increases the amount of fluoride in the acidic environment (Yan *et al.*, 2007; Carvalho *et al.*, 1999). Moreover, it was concluded by Bell *et al.* (1999) that saliva may retard the effect of fluoride release. Nevertheless, the activity of salivary enzymes may increase the fluoride rate (Rošin-Grget and Linčir, 2001).

According to Komatsu and Shimokobi (1993), the release of fluoride results in decreasing the solubility of tooth structure, suppressing the growth of bacteria, specifically *Streptococcus mutans* and changing the adhesive properties of bacteria to teeth. It was believed that dental biofilm surrounding restorations changes the properties and deteriorates the surface integrity of these materials (Fúcio *et al.*, 2008). As a result, more biofilm formation will be observed on the roughened surface (Fúcio *et al.*, 2008; Busscher *et al.*, 2010). Moreover, according to their findings, Fúcio *et al.* (2008) and Busscher *et al.* (2010) concluded that glass ionomer materials alter the adherence of bacterial biofilm.

A recent study showed the correlation of cariostatic activity of GIC with its fluoride release rate during plaque formation (Chau *el al.*, 2015). GIC are well known by its cariostatic property that makes enamel less soluble to acids produced by bacteria. The release of fluoride from those materials enhances the hardness of hydroxyapatite and restrains the growth of *streptococcus mutans* (Rošin-Grget and Linčir, 2001; Chau *et al.*, 2015). This cariostatic action is essential to heighten enamel acid resistance, which eventually changes the nature of plaque surrounding the restorations. The benefit of fluoride release from GIC is seen not only in enamel adjacent to the restoration, but also has been reported in areas up to three millimeters away from the restoration's margin and may even offer protection for the entire tooth (Hotta *et al.*, 1992 ; Miyazaki *et al.*, 1996).

### 1.2.5.2 Fluoride recharge

GIC restorations have the ability to reabsorb fluoride from the oral cavity as they act as reservoirs (Zafar and Ahmed, 2015). The ability to recharge is dependent on how permeable the material is, the concentration of the fluoridating agent and the frequency of the fluoride exposure (Han *et al.*, 2002; Preston *et al.*, 1999).

Dental restorations in the mouth are frequently exposed to many sources of fluoride. These exogenous sources include fluoridated toothpastes, mouthwashes and fluoride gels. Daily exposure of these restorations to fluoridated toothpastes has shown a high recharge capability of GIC (Freedman *et al.*, 2003). The fluoride ions are released from the outer surface of the restoration once it is treated with fluoridated agents. This release is attributed to the acidic attack of these agents resulting in detachment of the surface bound fluoride (Gao and Smales, 2001). However, some studies confirmed that fluoride recharge declines with maturation. They suggested that fluoride uptake may be more of a surface rather than a bulk diffusion effect and that re-exposure of fluoride will enhance fluoride release (Arbabzadeh-Zavareh *et al.*, 2009; Sidhu and Nicholson, 2016).

Re-fluoridation is effective when fluoridated agents are used frequently and in higher concentrations (Freedman *et al.*, 2003 and Han *et al.*, 2002). Recharging restorations with fluoride will also ensure their antimicrobial effect against bacteria over the long term. This antimicrobial action is mainly against *Streptococcus sanguis* and *Streptococcus mutans* (Barkhordar *et al.*, 1989).

### 1.2.6 Protective coatings

The maturation process of glass ionomers may be compromised by dehydration and water contamination (Bonifácio *et al*., 2012). Water is the liquid component of the GIC system that acts as a medium in which calcium and aluminum cations are formed and then transported into the reaction with the polyacid powder to form the final matrix (Leirskar *et al.*, 2003).

It was found that moisture may affect the integrity of the GIC surface when it is in contact with the restoration before the end of its maturation phase (Sangappa *et al.*, 2005). This is due to the 'wash-out' effect resulting in a weakened surface that is prone to erosive and abrasive forces (Sukumaran and Mensudar, 2015; Kamatham and Reddy, 2013; Gorseta *et al.*, 2016).

On the contrary, if the water component has been dehydrated or lost from the cement during the setting reaction, it will lead to discontinuation of this reaction. As a result the restoration will be immature and show signs of crazing and poor physio-mechanical properties (Sukumaran and Mensudar, 2015; Miyazaki *et al.*, 1996). Both of these negative outcomes may disturb the setting reaction of the restoration and reduce the mechanical strength of the material which eventually leads to poor clinical performance (Kamatham and Reddy, 2013). Glass ionomers have short working times and rather long setting times which make their handling properties, technique sensitive, clinically due to their susceptibility to water dehydration or contamination (Bonifácio *et al.*, 2012).

To minimize the possibility of any drawbacks, many authors recommended protecting the outer surface of GIC restorations by using surface coating agents immediately, to maintain the water balance during the maturation process (Sukumaran and Mensudar, 2015 ; Bonifácio *et al.*, 2012). Small *et al.*, (1998) concluded that surface protection is essential during overlapping stages of setting reactions of GIC to maintain the ideal physical strength of those restorations. *In vitro* studies revealed optimum compressive strengths when sealing the immature GIC for at least one hour (Leirskar *et al.*, 2003). The advantages obtained from this protection are mainly to improve the strengthening properties of GIC, while sacrificing the benefit of the initial release of fluoride by occluding the mechanism of fluoride release (Hotta *et al.*, 1992).

GIC has been coated with varnishes, cocoa butter, petroleum jelly and light cured bonding resins (Lohbauer, 2009; Bonifácio *et al.*, 2012). In a study conducted by Kamatham and Reddy (2013), using two coatings (varnish and the petroleum jelly), they concluded that surface coatings can impede the fluoride releasing property of glass ionomer restorations. However, these coatings have also been shown to peel from the surface shortly after placement (Hattab and Amin, 2001). Bonifácio *et al.* (2012) stressed the fact that the longer a protective coating is in contact with a GIC surface, the less chance exists that the GIC will have its physical properties reduced.

The advantage of light activated resin based coatings has been highlighted by Castro *et al.* (1994) and Gorseta *et al.* (2016) who reported on their effectiveness in protecting the GIC from dissolution compared to other conventional coatings (Castro *et al.*, 1994; Gorseta *et al.*, 2016). Light activated resin based coatings are sticky and have low water permeability over GIC compared with other conventional coatings like varnishes and petroleum jelly which are lost by masticatory wear (Sukumaran and Mensudar, 2015). Gorseta *et al* (2016) has revealed the advantage of using a light curing unit after the application of resin- based coatings. The heat produced by these units was found to improve the surface characteristics of GICs (Kuter *et al*., 2010; Gorseta *et al*., 2016).

Resin based surface coatings contain enhanced formulations, including resin, low molecular weight monomers, photo-initiators and other modifiers (Faraji *et al*., 2017 ). To improve their mechanical properties, light activated resin based protective coatings may contain fillers. Accordingly it can be in two forms: with fillers (filled resins) and without fillers (unfilled resins) (Faraji *et al.*, 2017).

Recently, a new generation of light activated resin based coatings with nanotechnology has been introduced (Bonifácio *et al.*, 2012; Tiwari and Nandlal, 2013). The incorporation of nano-sized fillers increases the overall performance of these coatings (Bonifácio *et al.*, 2012; Tiwari and Nandlal, 2013).

Previous research has studied the compressive and flexural strength of GIC after protecting it with resin-based coating (Bonifácio *et al.*, 2012; Sukumaran and Mensudar, 2015; Gorseta *et al.*, 2016). These studies concluded that GIC coated with light activated resin based coatings exhibited an increase in both compressive and flexural strengths, compared to the uncoated GIC. However, insufficient data is available in the literature on testing the effect of these coatings on fluoride release.

### 1.2.7 Fluoride measurement

Fluoride is present in two different forms when released from dental materials: ionic and complex forms (Okte *et al.*, 2012). Fluoride in free ionic form is effective in increasing the tooth structure resistance to secondary caries around restorations (Itota *et al.*, 2004).

The determination of the fluoride concentration is a sensitive method (Tokaliglu *et al.*, 2004). The most commonly used techniques to measure fluoride released from dental materials are ion selective electrodes (ISE) and ion chromatography (IC) (Itota *et al.*, 2004).

Ion selective electrodes have been used extensively in research to replace the costly and time consuming ion chromatography methods (Tokaliglu *et al.*, 2004). The ion selective electrode method has the ability to detect both free ionic and complex forms of fluoride (Okte *et al.*, 2012). In contrast, the ion chromatography method can only measure fluoride concentrations in ionic forms and in low concentrations that are undetectable by electrodes (Okte *et al.*, 2012; Itota *et al.*, 2004).

This study was done by using an ion selective electrode (ISE) which is among the most frequently used methods to measure the fluoride ions present in biological environments (Itota *et al.*, 2004 ; Torabzadeh *et al.*, 2015). This electrode can respond to changes in a wide range. The only important interference of this electrode is with the hydrogen ions (Harhash *et al.*, 2017). In order to prevent this particular interference and other possible ionic interferences, and also for standardization of pH and ionic strength, a total ionic strength adjustment buffer (TISAB) was used. Generally speaking, fluoride measurement using ISE is easier, faster, more accurate, allows continuous monitoring and more cost-effective than other methods (Tokaliglu *et al.*, 2004; Torabzadeh *et al.*, 2015; Harhash *et al.*, 2017).

# CHAPTER 2:

# AIMS AND OBJECTIVES

## 2.1 Aim:

To study the effect of resin-based coatings on the fluoride release of a conventional glass ionomer restorative material (GIC).

## 2.2 Objectives:

1. To measure the fluoride concentration in water containing uncoated GIC specimens at 1, 2, 3,7,14 and 28 days
2. To measure the fluoride concentration in water containing GIC specimens coated with a micro-filled resin at 1, 2, 3,7,14 and 28 days
3. To measure the fluoride concentration in water containing GIC specimens coated with a nano-filled resin at 1, 2,3,7,14 and 28 days
4. To measure the fluoride concentration in water containing GIC specimens coated with acrylic resin at 1, 2, 3 ,7,14, and 28 days
5. To compare the water fluoride concentrations among different groups at different time intervals.

## 2.3 Null Hypotheses

Resin-based surface protective coatings do not affect the fluoride release from a GIC restorative material.

# CHAPTER 3:

## MATERIALS AND METHODS

### 3.1 Study design:

An *in-vitro* controlled comparative study

### 3.2 Materials

A conventional glass ionomer restorative material, Ketac Fil Aplicap®, was used in this laboratory study. Ketac Molar Aplicap is a conventional glass ionomer filling material with enriched ultra-fine radiopaque aluminium-calcium-lanthanum-fluorosilicate glass designed to control active carious lesions (3M ESPE, 2016). The powder component of the capsule also contains 5% dried polycarbonate acid. This acidic addition will increase the cross linkage and improve overall mechanical properties without any remarkable increase in the viscosity (Katsuyama *et al*., 1993).

This material was sampled, grouped and then coated with three different protective coatings as shown in Table 1. G coat plus is a light-cured protective clear coating formulated with adhesive monomer and uniformly dispersed nanofillers, while Scotchbond Universal is a light-cured dental adhesive that is widely used in restorative dentistry. The material itself is viscous and can be used on dry or moist surfaces. Riva coat is a pure light acrylic monomer introduced into the market along with Riva light cure GIC (Sukumaran and Mensudar, 2015).

**Table 1: Materials used in the study**

| Material | Product type | Manufacturer | Composition |
|---|---|---|---|
| Ketac-Molar Aplicap® | Capsulated self-cured glass ionomer | 3M ESPE | Powder: radiopaque aluminum–calcium–lanthanum–fluorosilicate glass, polycarbonic acid; Liquid: polycarbonic acid aqueous solution, tartaric acid. |
| G coat Plus® | Low viscosity, self-adhesive light cured Nano filled resin | GC Corporation | Methyl methacrylate, colloidal silica, camphorquinone, urethane methacrylate, phosphoric ester monomer. |
| Scotchbond Universal® | Self-adhesive light cured filled resin | 3M ESPE | DP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators, Silane. |
| Riva coat® | Acrylic monomer | SDI | 100% Acrylic Monomer. |

## 3.3 Laboratory procedures:

### 3.3.1 Specimen preparation:

Twenty eight specimens of the material were created using disposable cylindrical polytetrafluoroethylene moulds in the form of round disc-shaped samples (5 mm diameter x 1.5 mm height). The restorative material was prepared according to the manufacturers' instructions (Appendix C ) at room temperature (23 ± 1ºC) and controlled relative humidity (50 ± 5%), according to ISO specification #7489 (Paschoal *et al*, 2011).

After placement of the material in the mould, the surface of the restorative material was covered with a Mylar strip and a glass slab to create pressure to expel excess material from the mould. A length of dental floss was incorporated into the discs during fabrication to allow for suspension into the test medium.

These discs were randomly assigned to 4 groups as shown in Table 2. Three test groups of 7 each (*n*=21) were coated with the three different protective coatings by applying one layer using a micro brush, then light cured with a Light Emitting Diode (LED) curing light with a wavelength range of 440– 480 nm at an output of 1500mW/cm2. The control group (*n*=7) remained uncoated. To control for bias, the investigator and laboratory assistant were blinded.

**Table 2. Study samples grouping**

| | | |
|---|---|---|
| **Ketac Fil**<br>**Aplicap®**<br>***n*=28** | Uncoated | Control Group |
| | Coated with G-coat plus | Experimental Group |
| | Coated with Scotchbond Universal | Experimental Group |
| | Coated with Riva coat | Experimental Group |

### 3.3.2 Determination of fluoride ion release:

The specimens were suspended in 5 ml distilled water for different time periods. The bottles were sealed. Fluoride ion measurement was performed at the end of the first, second, third, seventh, fourteenth, and twenty-eighth day under normal atmospheric conditions and at room temperature (23 ± 1ºC) using a combination fluoride ion electrode (Orion research electrode, Orion Research Inc, Boston, USA). The electrode was connected to an ion analyzer (Ion 85 Radiometer, Copenhagen, Denmark). The electrode was calibrated before each series of measurements with fluoride concentrations of 0.10, 0.30, 1.00 and 3.00 parts per million, prepared from a 100 ppm fluoride standard (Orion research electrode, Orion Research Inc, Boston, USA). These standards were prepared in a 1:1 buffer/water solution.

The TISAB buffer was prepared by adding 4 grams of CDTA (cyclohexylenedinitrilo-tetra-acetate) to 57 ml of glacial acetic acid and 58 grams of sodium chloride and dissolved in 500 ml of double distilled water. The pH of this solution was then adjusted between 5.3 and 5.5 by slowly adding 6N sodium hydroxide. This strong buffer solution was always used in a ratio of 1:1 with any solution to neutralize any small change in the ionic strength which may influence the reading and to maintain the pH of all solutions between 5.3 and 5.5 to cancel out the possible effect of any variation of the ionic strength on the electrode.

At the end of Day 1, each disc was lifted by the floss above the water level; rinsed with an equal amount of the buffer, blot dried using absorbent paper and then transferred to a new plastic container with fresh double distilled water. Similar steps were followed for Day 2, Day 3, Day 7, Day 14 and Day 28. The containers were stored for measurement. When the fluoride concentrations of the samples were too high and out of the range of the electrode, it was diluted as necessary and the fluoride concentration recorded in ppm F.

### 3.4 Data analysis

The data was categorized and coded and then entered into a data capture sheet using a Microsoft Excel sheet (Microsoft Corporation®, Redmond, Washington). The database was imported into the Social Package of Statistical Analysis (SPSS-IBM Corporation®, Armonk, New York) to perform statistical analysis. A statistician was consulted and the results were presented appropriately in the form of frequency tables and graphs. Fluoride release was measured over six fixed time periods: Day 1, Day 2, Day 3, Day 7, Day 14 and Day 28. The analysis was done using the Mann Whitney test at a significance level of 0.001. Firstly, the effect of the coating was evaluated by comparing the coated samples with the uncoated samples over time. Thereafter, the differences in fluoride blocking effect of the three coatings were evaluated over time again, using a General Linear model test.

### 3.5 Declaration of interest:

The researcher reports no conflicts of interest. Project registration and Ethics approval was granted by the Senate Research Committee of the University of the Western Cape (15/7/34) (Appendix G). The researcher did not receive any funding, materials or equipment from any dental company to conduct the research. The researcher alone is responsible for the content and writing of the paper.

# CHAPTER 4:

## RESULTS

The aim of this study was to investigate the effect of three different protective coatings on the fluoride concentration of water containing GIC specimens.

### 4.1 Comparison of fluoride concentration in water containing coated and uncoated GIC samples

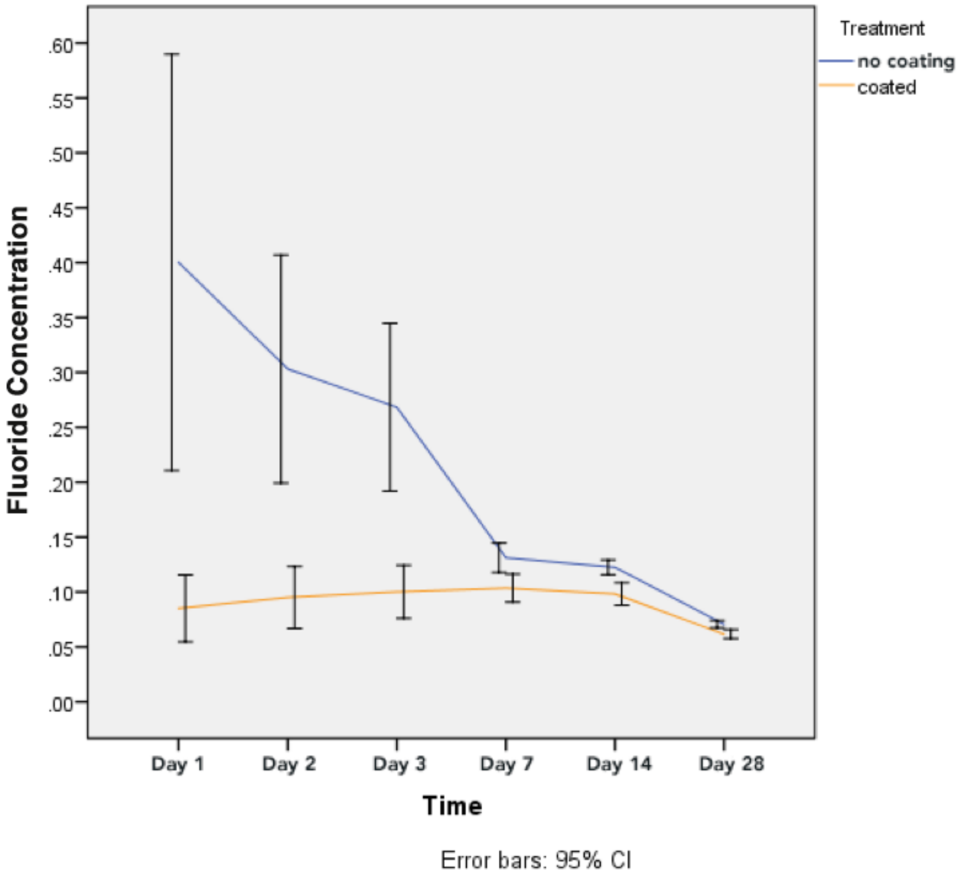


**Figure 1: Fluoride concentration in water containing coated and uncoated specimens.**

As shown in Figure 1, the difference is prominent between the coated and uncoated samples, particularly at early stages (time) of release. Figure 1 also shows large differences in variance. The Mann Whitney test indicated that the difference in concentration is significantly lower when discs were coated compared to discs not coated ($p$<0.001*). The difference between the coated and uncoated samples remained present and highly significant ($p$<0.01*) over the period of 28 days. The fluoride concentration in water of the coated samples increased from day 1 up to about the 7th day from when it decreased, while the water concentration with uncoated samples showed a drop from day 1. After 7 days, the fluoride concentration in water with coated and uncoated samples became similar (Figure 1) and after 28 days the fluoride concentration in water was small (Figure 1).

## 4.2 Comparison of fluoride concentrations among the groups with the coated GIC specimens

The difference in the concentration of fluoride in the water between the three types of coating was statistically analyzed using a General Linear model with repeated measurements.

**Table 3: Pairwise comparisons of fluoride concentrations among the groups with the coated GIC specimens**

| Coat | Day | Mean | Std. Error | 95% Confidence Interval | |
|---|---|---|---|---|---|
| | | | | Lower Bound | Upper Bound |
| Nano-filled resin (G coat Plus®) | 1 | .042 | .011 | .019 | .065 |
| | 2 | .053 | .009 | .034 | .072 |
| | 3 | .063 | .009 | .045 | .082 |
| | 7 | .083 | .008 | .067 | .099 |
| | 14 | .075 | .004 | .066 | .084 |
| | 28 | .054 | .002 | .049 | .059 |
| Micro-filled resin (Scotch Bond Universal®) | 1 | .044 | .011 | .021 | .067 |
| | 2 | .057 | .009 | .038 | .076 |
| | 3 | .070 | .009 | .052 | .088 |
| | 7 | .097 | .008 | .081 | .113 |
| | 14 | .098 | .004 | .089 | .107 |
| | 28 | .061 | .002 | .056 | .066 |
| Acrylic resin (Riva coat®) | 1 | .169 | .011 | .146 | .192 |
| | 2 | .175 | .009 | .156 | .194 |
| | 3 | .167 | .009 | .149 | .185 |
| | 7 | .131 | .008 | .115 | .147 |
| | 14 | .122 | .004 | .112 | .131 |
| | 28 | .070 | .002 | .065 | .075 |

Table 3 presents mean, standard error and 95% confidence intervals for the fluoride concentrations in milligram/liter for the three groups with the coated GIC specimens for each time interval.

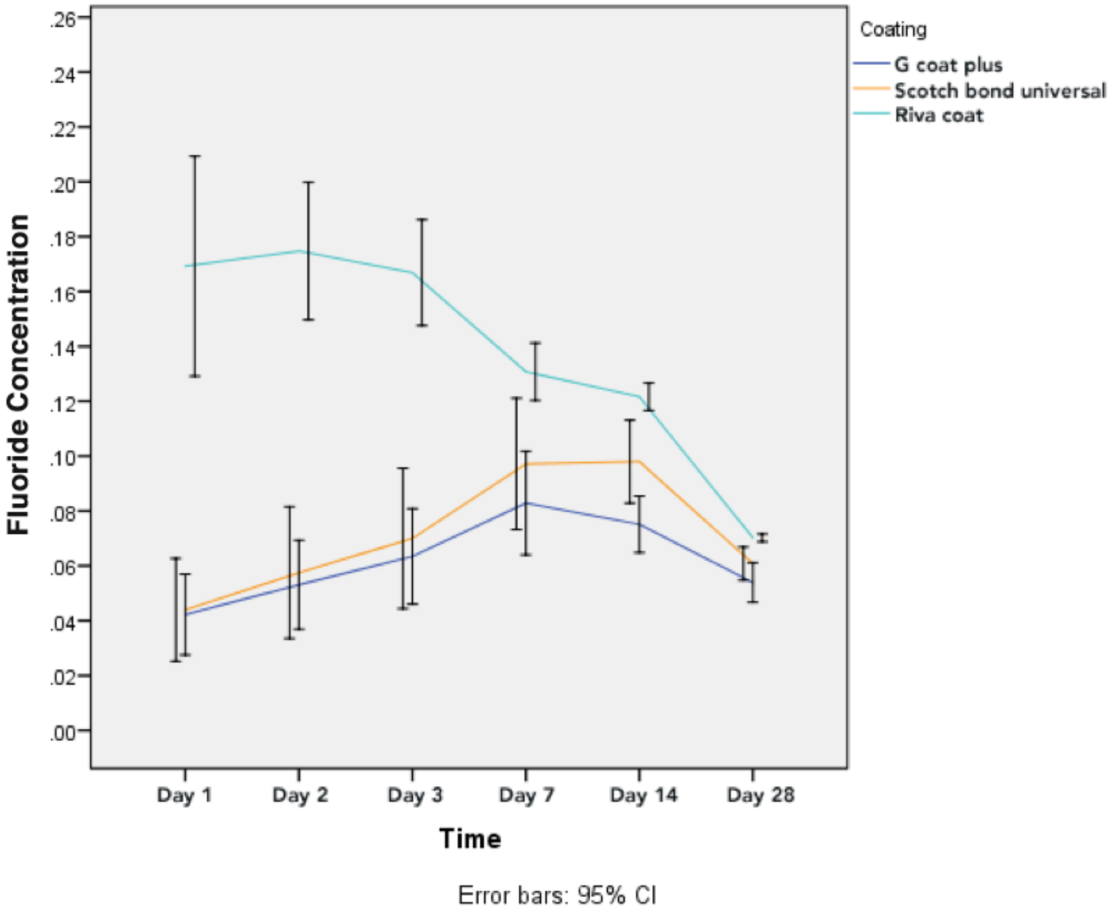


**Figure 2: Fluoride concentrations for the groups containing the coated GIC specimens for the different time intervals.**

From Figure 2, it is clear that the fluoride concentration in water that was released through the GIC was the highest for the Riva coated samples and stayed as such for 28 days. This was significant as seen in Table 1. The fluoride concentration in water between Scotchbond and G coated samples did not differ significantly over time (Table 1), although the concentration of fluoride in water from Scotchbond was always a little more as illustrated in Figure 2. Furthermore, the concentration of fluoride in water in Scotchbond and G coat specimens groups increased up to about 14 days and then decreased (Figure 2, Table 3). Riva coat was decreasing from the beginning.

### 4.3 Summary

This study found that fluoride released during the entire period of the experiment was the greatest from the uncoated group, followed in ranking by Riva coated, Scotchbond coated and G coat plus coated samples.

# CHAPTER 5:

## DISCUSSION

This study aimed to determine the effect of resin-based coatings on the fluoride released by a conventional glass ionomer restorative material. Our findings suggest that uncoated conventional glass ionomer released more fluoride than the coated conventional glass ionomer. Significant differences were recorded for fluoride release when different coatings were used over a period of seven days.

In order to analyze and assess the fluoride released from glass ionomers, we have to consider the factors that influence this property. The release of fluoride from GIC material is governed by various intrinsic and extrinsic factors (Upadhyay *et al*., 2013; DeSchepper *et al.*, 1991 and Hattab and Amin 2001). The intrinsic factors include the material composition, powder/liquid ratio, mixing time, temperature, specimen geometry, permeability, surface treatment and finishing (Upadhyay *et al.*,2013 ; Hattab and Amin 2001). However, the composition, powder/liquid ratio and mixing time vary according to the materials studied (Hattab and Amin 2001). Extrinsic factors that may affect the fluoride release include pH and temperature of the medium (Upadhyay *et al.*, 2013; Yan *et al.*, 2007).

In order to control the influence of some of these factors in this study, materials were prepared similar to previous studies and by following the manufacturer's instructions (Kamatham and Reddy, 2013; Sukumaran and Mensudar, 2015; Appendices C, D, E and F). In addition, Ketac Molar Aplicap in its encapsulated form was used in this study. The encapsulated form of GIC restorative material is popular and widely available among dentists (Nomoto *et al*., 2004). Encapsulation allows standardized powder/liquid ratios and mixing which will eliminate any susceptibility to clinician induced variability (3M ESPE, 2016). Manual mixing may incorporate air porosity resulting in weakening of the material (3M ESPE, 2016).

## 5.1 Fluoride concentrations of water from groups with coated and uncoated specimens:

In this study, all the coated and uncoated discs showed different concentrations of fluoride in water which suggests different patterns of fluoride release. This research confirmed studies conducted by De Moor *et al*., (1996); Yip and Smales (2000); Yap *et al*., (2002); Kamatham and Reddy (2013), where uncoated glass ionomer displayed higher fluoride release for the first three days and then a gradual decrease to nearly a constant level over a period of three weeks. Similar fluoride release patterns were also confirmed by Castro *et al*., (1994), Mazzaoui *et al*., (2000) and Tiwari and Nandlal, (2013).

These results suggested that despite the variation in the amount of fluoride released, the pattern of fluoride release was changed when a surface coating was used. Surface protection reduces the burst effect of fluoride release but does not completely prevent fluoride passage (Castro *et al.*, 1994). In this study, the control group, which had no surface coating, released the most fluoride. Without coatings, these immature GIC discs are in direct contact with the distilled water and are quite soluble (Wilson and McLean, 1988).

The clinical significance of using surface protection over GIC restorations is mainly to avoid early exposure to water. The correlation between early exposure to water and poor clinical performance of GIC has been reported by Kamatham and Reddy (2013). Both water uptake and water loss in the early stages of setting reactions can compromise the physical properties of GIC restorations (Karaoglanoglu *et al.*, 2009).

In clinical practice, in order to achieve the maximum utilization of fluoride release and at the same time maintain the optimum physical strength, it is desirable to use a surface coating that seals the surface for the first 24 hours and allows slow and steady release of fluoride over an extended period to prevent secondary caries (Karaoglanoglu *et al.*, 2009; Bonifácio *et al.*, 2012). In contrast, Kamatham and Reddy (2013) believe that the use of protective coating to improve the mechanical strength of primary teeth is of less importance; this is due to the short life span of these teeth. Moreover, the mastication forces of primary teeth are comparatively less in children (Kamatham and Reddy, 2013).

## 5.2 Comparison of fluoride concentration of among groups with the coated GIC specimens:

In this study, both nano-filled and micro-filled coatings provided good sealing ability in contrast to acrylic resin coatings which eluted more fluoride in the first week. It was expected that the filled resin surface coatings (G coat plus and scotch-bond Universal) would show less fluoride release compared to the unfilled resin surface coating namely Riva coat. Generally, the resin-based coatings have the ability to properly seal the GIC surface porosities and cracks (Karaoglanoglu *et al.*, 2009; Faraji *et al.*, 2017). The coating does not improve the wear resistance of the underlying restoration, but the protection mechanism against water contamination allows for the improvement of the physical properties and full maturation of the GIC (Karaoglanoglu *et al.*, 2009). This protection mechanism works by retaining the forming ions including Ca, Al, silicate and phosphate within the matrix which eventually contribute to its high physical properties (Czarnecka *et al.*, 2002). Without a coating, these ions will be lost by dissolution into the aqueous medium (Gorseta *et al.*, 2016).

Castro *et al*., (1994) considered the amount of fluoride released from the coated samples as a measurement of the sealing ability of the tested coatings. The greater the amount of fluoride released, the poorer the sealing capability of the coating (Castro *et al.*, 1994). In spite of the good sealing property of resin based protective coatings, permeability of the coatings has been reported after light cure polymerization and these have been largely attributed to differences in the formulations (Wang *et al*., 2004).

Furthermore, factors such as the thickness and viscosity of the coating can also play a role in blocking fluoride release (Mazzaoui *et al.*, 2000; Wang *et al*., 2004). Similar to Mazzaoui *et al*., (2000), the protective coatings were applied using a brush. Therefore it was not possible to control the thickness of the applied coating layer. The thicker the coating, the greater the barrier against fluoride release (Mazzaoui *et al.*, 2000). These factors may give some variability in the amount of fluoride released by glass ionomer specimens.

Ribeiro *et al*. (1999) related the high performance of surface coatings by its low viscosity and resistance to disintegration. The low viscosity coating will provide a low contact angle between the coating and the surface of GIC, allowing for good wetting on the surface and thus provide optimum protection (Ribeiro *et al.*, 1999). The incorporation of filler particles within the coating resin matrix also improves the sealing capability of protective coatings

(Faraji *et al.*, 2017). However, the addition of fillers to any dental material may increase their viscosity. The enhanced formulation in the current coatings, with the use of a low molecular monomer background, maintained the low viscosity (Faraji *et al*., 2017).

Riva coat has a low viscosity and does not contain any filler particles. This may explain the high fluoride release recorded which may result in poor sealing capability. Methacrylate-based resins are hydrophilic and fillers are added to decrease the water sorption ability (Faraji *et al.*, 2017). The filler content within the resin matrix formula can affect the coating performance, specifically wear resistance, hardness and strength (Faraji *et al.*, 2017). Unlike the filled resin coatings (G coat plus and Scotchbond Universal) unfilled resin coatings (Riva coat) can undergo hydrolysis and degradation due to high water sorption (Drummond, 2008; Faraji *et al.*, 2017).

Fluoride release from the nano-filled resin coatings was slightly less than from micro-filled coated GIC in the first seven days. Due to the composition, it might have been expected that the nanofilled coating (G-coat plus) has a better sealing ability compared to the microfilled coating (Scotch-bond Universal). Hepdinz *et al.* (2016) concluded that the nano sized fillers within the resin matrix formula in G coat plus had a better efficacy in sealing the micro pores and gaps in the outer surface, while Tiwari and Nandlal (2013) reported a decrease of passage of fluoride. However, according to ESPE 3M study results, it is noteworthy to mention that the micofilled Scotchbond Universal performance was found to be very close to that of nano-filled G coat plus. The manufacturer, ESPE 3M, related the enhancement of coating/restoration adhesion to the replacement of traditional methacrylate monomers with phosphorylated methacrylate monomers (ESPE 3M, 2016). Our study results showed close fluoride concentration in water for the 28 days for specimen groups coated by G coat plus and Scotch bond Universal.

Although the longevity of surface coating agents was not addressed in this study, it may be assumed that after several weeks the surface coatings are lost and the pattern and amount of subsequent fluoride release would be similar to that of an uncoated specimen. It should also be noted that mastication forces and brushing habits may influence the retention of these coatings (Karaoglanoglu *et al*., 2009). Therefore, the results obtained from these laboratory studies may differ from those obtained clinically (Karaoglanoglu *et al*., 2009).

A significant difference has also been observed in the compressive and flexural strengths of GIC when a protective coating has been applied. This could mean that the protective coatings act as mechanical barriers as well (Wang *et al.*, 2004; Sukumaran and Mensudar, 2015).

It is worth mentioning that heat produced by a dental cure lamp, used to cure resin based coatings, improves surface characteristics of GIC. Gorseta *et al.*, (2016) stated that heat accelerated the setting reaction in the surface layers of the specimens. However, the heating effect still needs further investigation.

To date, there is no conclusive clinical evidence whether the reduction of fluoride release caused by these coatings impairs the cariogenic properties of glass ionomers (Nicholson and Czarnecka, 2007), that makes the overall effects of these protective coatings appear beneficial.

# CHAPTER 6:

## CONCLUSIONS AND RECOMMENDATIONS

### 6.1 CONCLUSIONS

The null hypothesis, that resin-based surface protective coatings do not affect the fluoride release from a GIC restorative material, was rejected.

This study revealed that fluoride release is altered at the first stage. The greatest amount of fluoride was released from GIC coated with Riva coat, followed by Scotchbond universal and then G coat plus.

This study proved that microfilled (Scotchbond Universal), nanofilled (G coat plus) resin based and acrylic coatings (Riva) have the ability to allow the passage of fluoride from GIC to the outer environment during the initial fluoride release phase.

The three clinical implications of this study are as follows: Firstly, proof is provided that coating GIC makes a significant difference to the release of fluoride when compared to no coating.  Secondly, proof is provided on the behavior of the different coating materials over time, whether there is a difference and whether this difference is significant. Thirdly, it is evident that from day 7 approximately, the effect of coatings was reduced.

**The limitations of the study include:**

- The use of one glass ionomer material (Ketac Molar Aplicap)
- The sample size was relatively small but a statistician was consulted prior to the laboratory work...
- This is an *in vitro* study and does not mimic the oral cavity conditions that can be influenced by many factors such as forces of mastication, dietary habits and brushing habits.
- The study was limited to 28 days.

## 6.2 RECOMMENDATIONS

The clinical use of GIC in children with high caries risk is relevant. It can be concluded from this study and Kamatham and Reddy (2013) that if the need for fluoride release from a GIC is more important than the mechanical properties, it is recommended to coat it with the least fluoride blocking coating.

More studies should be done to evaluate the sealing of Riva coat and its ability to protect GIC. The chemical formula of Riva coat is still ambiguous as the manufacturers did not mention the specific chemical composition of the coat. They stated that the exact elements and concentrations have been withheld as a trade secret.

# REFERENCES

Attar, N. & Turgut, M. D. (2003). Fluoride release and uptake capacities of fluoride-releasing restorative materials. *Operative Dentistry*, 28: 395-402

Barkhordar, R.A., Kempler, D., Pelzner, R.R.B. & Stark, M.M. (1989). Antimicrobial action of glass-ionomer lining cement on S. sanguis and S. mutans. *Dental Materials*, 5(4): 281-282.

Bell, A., Creanor, S.L., Foye, R.H. & Saunders, W.P. (1999). The effect of saliva on fluoride release by a glass-ionomer filling material. *Journal of Oral Rehabilitation*, 26: 407- 412.

Berg, J.H. (1998). The continuum of restorative materials in pediatric dentistry-a review for the clinician. *Pediatric Dentistry*, 20: 93-100.

Bonifácio, C.C., Werner, A. & Johanes, C. (2012). Coating glass-ionomer cements with a nanofilled resin. *Acta Odontologica Scandinavica*, 70(6): 471-477.

Burke, F.J.T., Fleming, G.J.P., Owen, F.J. & Watson, D.J. (2002). Materials for restoration of primary teeth: 2. Glass ionomer derivatives and compomers. *Dental Update*, 29(1): 10-17.

Busscher, H.J., Rinastiti, M., Siswomihardjo, W. & Van der Mei, H.C. (2010). Biofilm formation on dental restorative and implant materials. *Journal of Dental Research*, 89(7): 657-665.

Carvalho, A.S. & Cury, J. A. (1999). Fluoride release from some dental materials in different solutions. *Operative Dentistry*, 24: 14-19.

Castro, G.W., Gray, S.E., Buikema, D.J. & Reagan, S.E. (1994). The effect of various surface coatings on fluoride release from glass-ionomer cement. *Operative Dentistry*, 19: 194-194.

Chau, P. T., Pandit, S., Cai, J., Lee, M. & Jeon, M. (2015). Relationship between fluoride release rate and anti-cariogenic biofilm activity of glass ionomer cements. *Dental Materials*, 31: 100-108.

Cho, S.Y. & Cheng, A.C. (1999). A review of glass ionomer restorations in the primary dentition. *Journal of Canadian Dental Association*, 65: 491-495.

Creanor, S.L., Carruthers, L.M., Saunders, W.P., Strang, R. & Foye, R. H. (1994). Fluoride uptake and release characteristics of glass ionomer cements. *Caries Research*, 28: 322-328.

Croll, T.P. & Nicholson, J.W. (2002). Glass ionomer cements in pediatric dentistry: review of the literature. *Pediatric Dentistry*, 24(5): 423-429.

Curzon, M.E., Pollard, M.A. & Duggal, M.S. (1996). Restoration of primary molars. *British Dental Journal*, 180(7): 246.

Czarnecka, B., Limanowska-Shaw, H. & Nicholson, J.W. (2002). Buffering and ion-release by a glass-ionomer cement under near-neutral and acidic conditions. *Biomaterials*, 23(13): 2783-2788.

De Moor, R.J., Verbeeck, R.M. & De Maeyer, E.A. (1996). Fluoride release profiles of restorative glass ionomer formulations. *Dental Materials*, 12(2): 88-95.

DeSchepper, E.J., Berry, E.A., Cailleteau, J.G. & Tate, W.H. (1991). A comparative study of fluoride release from glass-ionomer cements. *Quintessence International*, 22(3): 215-220.

Dionysopoulos, D. (2014). The effect of fluoride-releasing restorative materials on inhibition of secondary caries formation. *Fluoride*, 47(3): 258-265.

Drummond, J.L. (2008). Degradation, fatigue, and failure of resin dental composite materials. *Journal of Dental Research*, 87(8): 710-719.

Faraji, F., Heshmat, H. & Banava, S. (2017). Effect of protective coating on microhardness of a new glass ionomer cement: Nanofilled coating versus unfilled resin. *Journal of Conservative Dentistry*, 20(4): 260-263.

Francisconi, L.F., Scaffa, P.M.C., Barros, V.R.D.S.P., Coutinho, M. & Francisconi, P.A.S. (2009). Glass ionomer cements and their role in the restoration of non-carious cervical lesions. *Journal of Applied Oral Science*, 17(5): 364-369.

Freedman, R. & Diefenderfer, K.E. (2003). Effects of daily fluoride exposures on fluoride release by glass ionomer-based restoratives. *Operative Dentistry*, 28(2): 178-185.

Forsten, L. (1998). Fluoride release and uptake by glass-ionomers and related materials and its clinical effect. *Biomaterials*, 19(6): 503-508.

Fúcio, S.B., Carvalho, F.G., Sobrinho, L.C., Sinhoreti, M.A. & Puppin-Rontani, R.M. (2008). The influence of 30-day-old Streptococcus mutans biofilm on the surface of esthetic restorative materials—an in vitro study. *Journal of Dentistry*, 36(10): 833-839.

Gao, W. & Smales, R.J. (2001). Fluoride release/uptake of conventional and resin-modified glass ionomers, and compomers. *Journal of Dentistry*, 29(4): 301-306.

Gorseta, K., Glavina, D., Skrinjaric, T., Czarnecka, B. & Nicholson, J.W. (2016). The effect of petroleum jelly, light-cured varnish and different storage media on the flexural strength of glass ionomer dental cements. *Acta Biomaterialia Odontologica Scandinavica*, 2(1): 55-59.

Han, L., Li, M., Niwango, K., Okamoto, A., Honda, N. & Iwaku, M. (2002). Effect of fluoride mouth rinse on fluoride releasing and recharging from aesthetic dental materials. *Dental Materials Journal*, 21(4): 285-295.

Harhash, A.Y., ElSayad, I.I. & Zaghloul, A.G. (2017). A comparative in vitro study on fluoride release and water sorption of different flowable esthetic restorative materials. *European Journal of Dentistry*, 11(2): 174-179.

Hatibovic-Kofman, S.A. & Koch, G.O. (1991). Fluoride release from glass ionomer cement in vivo and in vitro. *Swedish Dental Journal*, 15(6): 253-258.

Hattab, F.N. & Amin, W.M. (2001). Fluoride release from glass ionomer restorative materials and the effects of surface coating. *Biomaterials*, 22(12): 1449-1458.

Hickel, R. & Voss, A. (1990). A comparison of glass cermet cement and amalgam restorations in primary molars. *ASDC Journal of Dentistry for Children*, 57(3): 184-188.

Hotta, M., Hirukawa, H. & Yamamoto, K. (1992). Effect of coating materials on restorative glass-ionomer cement surface. *Operative Dentistry*, 17(2): 57-61.

Itota, T., Carrick, T.E., Rusby, S., Al-Naimi, O.T., Yoshiyama, M. & McCabe, J.F. (2004). Determination of fluoride ions released from resin-based dental materials using ion-selective electrode and ion chromatograph. *Journal of Dentistry*, 32(2): 117-122.

Kamatham, R. & Reddy, J. S. (2013). Surface coatings on glass ionomer restorations in Pediatric dentistry-Worthy or not?. *Journal of Indian Society of Pedodontics and Preventive Dentistry*, 31(4): 229-233.

Karantakis, P., Antoniades, H.M., Pahini, T.S. and Papadogiannis, Y. (2000). Fluoride release from three glass ionomers, a compomer, and a composite resin in water, artificial saliva, and lactic acid. *Operative Dentistry*, 25: 20-25.

Karaoglanoglu, S., Akgül, N., Özdabak, H.N. and Akgül, H.M. (2009). Effectiveness of surface protection for glass-ionomer, resin-modified glass-ionomer and polyacid-modified composite resins. *Dental Materials Journal*, 28(1): 96-101.

Katsuyama, S., Ishikawa, T. & Fujii, B. (1993). *Glass ionomer dental cement: the materials and their clinical use*. Medico Dental Media International.

Khoroushi, M. & Keshani, F. (2013). A review of glass-ionomers: From conventional glass-ionomer to bioactive glass-ionomer. *Dental Research Journal*, 10(4): 411-420.

Komatsu, H. and Shimokobe, H. (1993). Section 5: Fluoride release and the strengthening of tooth structure *In*: S. Katsymaya, *Glass Ionomer Dental Cement – The Materials and Their Clinical Use*. Tokyo*: Ishiyaku EuroAmerica, Inc Publishers*: 53-59.

Kuter, B., Eden, E. & Şen, H.B. (2010). The Effect of Heat on the Surface Properties of Conventional Glass Ionomer Cements: SEM Evaluation. *Balkan Journal of Stomatology*, 10(14): 65-71.

Leirskar, J., Nordbø, H., Mount, G.J. & Ngo, H. (2003). The influence of resin coating on the shear punch strength of a high strength auto-cure glass ionomer. *Dental Materials*, 19(2): 87-91.

Lohbauer, U. (2009). Dental glass ionomer cements as permanent filling materials?– Properties, limitations and future trends. *Materials*, 3(1): 76-96.

Manhart, J., Kunzelmann, K.H., Chen, H.Y. & Hickel, R. (2000). Mechanical properties and wear behavior of light-cured packable composite resins. *Dental Materials*, 16(1): 33-40.

Marquezan, M., Osorio, R., Ciamponi, A., L. & Toledano, M. (2010). Resistance to degradation of bonded restorations to simulate caries-effected primary dentin. *Dental Material Research*, 23: 47-52.

Marquezan, M. & Raggio, D.P. (2009). Dental materials in daily pedodontics clinical practice. *Dental Materials Research*: 71-88.

Mazzaoui, S.A., Burrow, M.F. & Tyas, M.J. (2000). Fluoride release from glass ionomer cements and resin composites coated with a dentin adhesive. *Dental Materials*, 16(3): 166-171.

Miyazaki, M., Moore, B.K. & Onose, H. (1996). Effect of surface coatings on flexural properties of glass ionomers. *European Journal of Oral Sciences*, 104(5-6): 600-604.

Mousavinasab, S.M. & Meyers, I. (2009). Fluoride release by glass ionomer cements, compomer and giomer. *Dental Research Journal*, 6(2): 75-81.

Nagaraja Upadhya, P. & Kishore, G. (2005). Glass ionomer cement: The different generations. *Trends Biomater Artificial Organs*, 18(2): 158-165.

Nicholson, J.W. (1998). Chemistry of glass-ionomer cements: A review. *Biomaterials,* 19: 485–494.

Nicholson, J. W. & Czarnecka, B. (2007). Kinetic studies of the effect of varnish on water loss by glass–ionomer cements. *Dental Materials*, 23(12): 1549-1552.

Nomoto, R., Komoriyama, M., McCabe, J.F. & Hirano, S. (2004). Effect of mixing method on the porosity of encapsulated glass ionomer cement. *Dental Materials*, 20(10): 972-978.

Okte, Z., Bayrak, S., Fidanci, U.R. and Sel, T. (2012). Fluoride and aluminum release from restorative materials using ion chromatography. *Journal of Applied Oral Science*, 20(1): 27-31.

Paschoal, M.A.B., Gurgel, C.V., Rios, D., Magalhães, A.C., Buzalaf, M.A.R. and Machado, M.A.D.A.M. (2011). Fluoride release profile of a nanofilled resin-modified glass ionomer cement. *Brazilian Dental Journal*, 22(4): 275-279.

Preston, A.J., Higham, S.M., Agalamanyi, E.A. & Mair, L.H. (1999). Fluoride recharge of aesthetic dental materials. *Journal of Oral Rehabilitation*, 26: 936–940.

Ribeiro, A.P.G., Serra, M.C., Paulillo, L.A. & Rodrigues, A.L. (1999). Effectiveness of surface protection for resin-modified glass-ionomer materials. *Quintessence International*, 30(6): 427-431.

Rizzante, F.A.P., Cunali, R.S., Bombonatti, J.F.S., Correr, G.M., Gonzaga, C.C. & Furusel, A.Y. (2015). Indications and restorative techniques for glass ionomer cement. *The South Brazilian Dentistry Journal* (*Online)*, 12(1): 79-87.

Rodríguez-Farre, E., Testai, E., Bruzell, E., De, W.J., Schmalz, G., Thomsen, M. & Hensten, A. (2016). The safety of dental amalgam and alternative dental restoration materials for patients and users. *Regulatory Toxicology and Pharmacology: RTP*, 79: 108-109.

Rošin-Grget, K. & Linčir, I. (2001). Current concept on the anticaries fluoride mechanism of the action. *Collegium Antropologicum*, 25(2): 703-712.

Rutar, J., McAllan, L. & Tyas, M.J. (2002). Three-year clinical performance of glass ionomer cement in primary molars. *International Journal of Paediatric Dentistry*, 12(2): 146-147.

Sangappa, V.M., Kumar, N.D. & Shivanna, V. (2005). A spectrophotometric evaluation of effectiveness of surface protection for resin modified glass ionomer cement an in vitro study. *Journal of Conservative Dentistry*, 8(2): 15-23.

Sidhu, S.K. & Nicholson, J.W. (2016). A review of glass-ionomer cements for clinical dentistry. *Journal of Functional Biomaterials,* 7(3): 16-30.

Small, I.C.B., Watson, T.F., Chadwick, A.V. & Sidhu, S. K. (1998). Water sorption in resin-modified glass-ionomer cements: An in vitro comparison with other materials. *Biomaterials,* 19 (6): 545-550.

Sukumaran, V.G. & Mensudar, R. (2015). To Evaluate the Effect of Surface Coating on Three Different Types Glass Ionomer Restorations. *Biomedical and Pharmacology Journal*, 8: 445-449.

Tiwari, S. & Nandlal, B. (2013). Effect of nano-filled surface coating agent on fluoride release from conventional glass ionomer cement: an in vitro trial. *Journal of Indian Society of Pedodontics and Preventive Dentistry*, 31(2): 91-95.

Tokalioğlu, Ş., Kartal, Ş. & Şahin, U. (2004). Determination of fluoride in various samples and some infusions using a fluoride selective electrode. *Turkish Journal of Chemistry*, 28(2): 203-212.

Torabzadeh, H., Ghassemi, A., Janani, A., Raoofinejad, F., Naderi, H. & Akbarzadeh B., A. (2015). Effect of Powder/Liquid Ratio on Fluoride Release of Glass Ionomers. *Beheshti University Dental Journal*, 33(1): 98-105.

Tyas, M., J. & Burrow, M., F. (2004). Adhesive restorative materials: A review. *Australian Dental Journal*, 49(3): 112-121.

Upadhyay, S., Rao, A. & Shenoy, R. (2013). Comparison of the amount of fluoride release from nanofilled resin modified glass ionomer, conventional and resin modified glass ionomer cements. *Journal of Dentistry (Tehran, Iran)*, 10(2): 134-140.

Valanezhad, A., Odatsu, T., Udoh, K., Shiraishi, T., Sawase, T. & Watanabe, I. (2016). Modification of resin modified glass ionomer cement by addition of bioactive glass nanoparticles. *Journal of Materials Science: Materials in Medicine*, 27(1): 1-9.

Von Fraunhofer, J.A. (2013). Inorganic cements. *Dental Materials at a Glance*. John Wiley & Sons, 60

Wang, L., Buzalaf, M.A.R. & Atta, M.T. (2004). Effect of one-bottle adhesive systems on the fluoride release of a resin-modified glass ionomer. *Journal of Applied Oral Science*, 12(1): 12-17.

Wiegand, A., Buchalla, W. & Attin, T. (2007). Review on fluoride releasing restorative materials – Fluoride release and uptake characteristics, antibacterial activity and influence on caries formation. *Dental Materials*, 23: 343-362.

Wilson, A.D. & McLean, J.W. (1988). *Glass-Ionomer Cement*. Quintessence Publishing Company.

Xie, D., Brantley, W.A., Culbertson, B.M. & Wang, G. (2000). Mechanical properties and microstructures of glass-ionomer cements. *Dental Materials*, 16(2): 129-138.

Yan, Z., Sidhu, S.K., Mahmoud, G.A., Carrick, T.E. & McCabe, J.F. (2007). Effects of temperature on the fluoride release and recharging ability of glass ionomers. *Operative Dentistry*, 32(2): 138-143.

Yip, H.K. & Smales, R.J. (2000). Fluoride release from a polyacid-modified resin composite and 3 resin-modified glass-lonomer materials. *Quintessence International*, 31(4): 261-266.

Zafar, M.S. & Ahmed, N. (2015). Therapeutic roles of fluoride released from restorative dental materials. *Fluoride*, 48(3): 184-194.

3M ESPE. (2016). 3M™ Scotchbond™ Universal Adhesive: Technical Product Profile. Available from: https://multimedia.3m.com/mws/media/1279638O/3m-scotchbond-universal-adhesive-technical-product-profile.pdf [Accessed 05 April 2018].

## 1. Appendix A:

### Data Sheet

| Discs | Day 1 | Day 2 | Day 3 | Day 3 till Day 7 | Per day | Day 7 till Day 14 | Per day | Day 14 till Day 28 | Per day |
|---|---|---|---|---|---|---|---|---|---|
| **Uncoated** | | | | | | | | | |
| 1 | 0.36 | 0.244 | 0.2 | 0.45 | 0.1125 | 0.76 | 0.10857 | 0.96 | 0.0685 |
| 2 | 0.176 | 0.194 | 0.198 | 0.45 | 0.1125 | 0.84 | 0.12 | 0.96 | 0.0685 |
| 3 | 0.284 | 0.234 | 0.22 | 0.51 | 0.1275 | 0.88 | 0.12571 | 0.96 | 0.0685 |
| 4 | 0.256 | 0.226 | 0.24 | 0.54 | 0.135 | 0.84 | 0.12 | 0.96 | 0.0685 |
| 5 | 0.362 | 0.324 | 0.26 | 0.57 | 0.1425 | 0.88 | 0.1257 | 1.08 | 0.0771 |
| 6 | 0.736 | 0.396 | 0.34 | 0.6 | 0.15 | 0.92 | 0.1314 | 0.96 | 0.0685 |
| 7 | 0.628 | 0.504 | 0.42 | 0.555 | 0.13875 | 0.88 | 0.1257 | 1.04 | 0.0742 |
| **G-Coat** | | | | | | | | | |
| 8 | 0.054 | 0.0628 | 0.068 | 0.34 | 0.085 | 0.52 | 0.0742 | 0.76 | 0.0542 |
| 9 | 0.0368 | 0.0412 | 0.052 | 0.3 | 0.075 | 0.44 | 0.0628 | 0.76 | 0.0542 |
| 10 | 0.0492 | 0.0612 | 0.076 | 0.38 | 0.095 | 0.56 | 0.08 | 0.76 | 0.0542 |
| 11 | 0.048 | 0.0596 | 0.072 | 0.37 | 0.0925 | 0.6 | 0.0857 | 0.76 | 0.0542 |
| 12 | 0.0632 | 0.08 | 0.092 | 0.46 | 0.115 | 0.64 | 0.0914 | 0.96 | 0.0685 |
| 13 | 0.0256 | 0.0336 | 0.044 | 0.24 | 0.06 | 0.48 | 0.0685 | 0.68 | 0.0485 |
| 14 | 0.0188 | 0.0332 | 0.04 | 0.23 | 0.0575 | 0.44 | 0.06285 | 0.6 | 0.0428 |
| **SB-Coat** | | | | | | | | | |
| 15 | 0.0496 | 0.0668 | 0.072 | 0.42 | 0.105 | 0.76 | 0.1085 | 0.8 | 0.0571 |
| 16 | 0.0748 | 0.0928 | 0.112 | 0.51 | 0.1275 | 0.76 | 0.1085 | 0.92 | 0.0657 |
| 17 | 0.0636 | 0.0884 | 0.1 | 0.5 | 0.125 | 0.76 | 0.1085 | 0.96 | 0.0685 |
| 18 | 0.0164 | 0.0232 | 0.034 | 0.26 | 0.065 | 0.52 | 0.0742 | 0.76 | 0.0542 |
| 19 | 0.0408 | 0.0448 | 0.06 | 0.38 | 0.095 | 0.72 | 0.1028 | 0.88 | 0.0628 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 20 | 0.0316 | 0.0456 | 0.064 | 0.4 | 0.1 | 0.76 | 0.1085 | 0.92 | 0.0657 |
| 21 | 0.0308 | 0.0408 | 0.048 | 0.25 | 0.0625 | 0.52 | 0.0742 | 0.72 | 0.0514 |
| **Riva-Coat** | | | | | | | | | |
| 22 | 0.0976 | 0.1168 | 0.124 | 0.465 | 0.11625 | 0.8 | 0.1142 | 1 | 0.0714 |
| 23 | 0.2012 | 0.1956 | 0.18 | 0.51 | 0.1275 | 0.84 | 0.12 | 0.96 | 0.0685 |
| 24 | 0.1956 | 0.1888 | 0.176 | 0.525 | 0.13125 | 0.88 | 0.1257 | 1 | 0.0714 |
| 25 | 0.1532 | 0.1788 | 0.168 | 0.51 | 0.1275 | 0.8 | 0.1142 | 0.96 | 0.0685 |
| 26 | 0.2036 | 0.1912 | 0.188 | 0.525 | 0.13125 | 0.88 | 0.1257 | 1 | 0.0714 |
| 27 | 0.128 | 0.168 | 0.172 | 0.51 | 0.1275 | 0.88 | 0.1257 | 0.96 | 0.0685 |
| 28 | 0.2052 | 0.184 | 0.16 | 0.615 | 0.15375 | 0.88 | 0.1257 | 1 | 0.0714 |

## 2. Appendix B:

### Statistical Analysis

Mean measurements of fluoride concentrations in milligram/liter of the four different groups in double distilled water.

| | *Uncoated* | *G coat plus* | *Scotchbond Universal* | *Riva coat* | *p-value** |
|---|---|---|---|---|---|
| ***Day 1*** | 0.4003 | 0.0422 | 0.0439 | 0.1692 | 0.0001* |
| ***Day 2*** | 0.3031 | 0.0531 | 0.0575 | 0.1747 | 0.0001* |
| ***Day 3*** | 0.2682 | 0.0634 | 0.0700 | 0.1669 | 0.0001* |
| ***Day 7*** | 0.1313 | 0.0829 | 0.0971 | 0.1307 | 0.0006* |
| ***Day 14*** | 0.1224 | 0.0751 | 0.0980 | 0.1216 | 0.0001* |
| ***Day 28*** | 0.0706 | 0.0539 | 0.0608 | 0.0706 | 0.0003* |

## 3. Appendix C:

### Ketac Molar Aplicap Use Instructions

3M ESPE

**Ketac™ Molar Aplicap™**

- (en) **Glass Ionomer Restorative Material**
- (de) **Glasionomer Füllungsmaterial**
- (fr) **Matériau d'obturation en verre ionomère**
- (it) **Materiale per otturazione vetro-ionomero**
- (es) **Material de obturación de vidrio ionómero**
- (pt) **Material de obturação de vidro ionômero**
- (nl) **Glasionomeer vulmateriaal**
- (el) **Υαλοϊονομερής κονία για εμφράξεις**
- (sv) **Glasjonomer fyllningsmaterial**
- (fi) **Lasi-ionomeeritäyteaine**
- (da) **Glasionomer-fyldningsmateriale**
- (no) **Glassionomer fyllingsmateriale**

| | |
|---|---|
| Instructions for Use | Gebruiksinformatie |
| Gebrauchsinformation | Οδηγίες χρήσης |
| Mode d'emploi | Bruksanvisning |
| Informazioni per l'uso | Käyttöinfo |
| Información de uso | Brugsanvisning |
| Instruções de uso | Bruksinformasjon |

**3M Deutschland GmbH**
Dental Products
Carl-Schurz-Str. 1
41453 Neuss – Germany

CE 0123

3M ESPE Customer Care/MSDS Information:
U.S.A. 1-800-634-2249 and Canada 1-888-363-3685.

44000768158/01

## (en) ENGLISH

**Product Description**

Ketac™ Molar Aplicap™ is a radiopaque bulk fill glass ionomer cement in capsules. On account of its chemical adhesion to the dental enamel and dentin, a gentle preparation and an especially tight seal at the margins is possible. The product is available in various shades; it can be applied without lining and releases fluoride ions. The dispensable quantity of a capsule is at least 0.14 ml.

☞ These Instructions for Use should be kept for the duration of product use. For details on all mentioned products, please refer to the respective Instructions for Use. Ketac™ Glaze, Scotchbond™ Universal Adhesive, or Single Bond Universal Adhesive are not available in all countries.

**Indications**

- Linings for single-surface and multiple-surface composite fillings
- Core build-up prior to crown placement
- Primary tooth fillings
- Single-surface fillings in non-occlusal load-bearing areas
- Stress bearing Class II restorations when the isthmus is less than half of the intercuspal distance and with at least one occlusal contact on enamel
- Cervical fillings, if aesthetics is not the prime consideration
- Uniplanar and multi-planar temporary fillings

**Precautionary Measures**

3M MSDSs can be obtained from www.mmm.com, or contact your local subsidiary.

**Cavity Preparation**

- ▶ Remove carious tooth substance only; undercuts are not required.
- ▶ Do not prepare thin beveling margins. A wall thickness of 0.5 mm has to be provided for the material.

**Pulp Protection**

- ▶ To prevent pulpal irritation, cover areas in close proximity to the pulp with local application of a calcium hydroxide material if necessary.

**Conditioning**

- ▶ For optimal chemical bonding to the teeth, the smear layer produced in the preparatory steps must be carefully removed by applying Ketac™ Conditioner to the prepared surfaces and allowing the substance to react for 10 sec.
- ▶ Then rinse with copious amounts of water.
- ▶ Blow the cavity dry in only 2–3 short intervals with water- and oil-free air or dab dry with cotton pellets. Do not overdry! The cavity should have a matt shiny appearance. Excessive drying can result in post-operative sensitivity after filling.
- ▶ Avoid further contamination.

**Times**

The following times apply for an ambient temperature of 23° C/73° F:

| | min:sec |
|---|---|
| Activation | 0:02 |
| Mixing in RotoMix™ | 0:10 |
| Mixing in high-frequency mixer, e.g., CapMix™ | 0:15 |
| Working after start of mixing | 2:00 |
| Setting after start of mixing | 4:30 |

Setting is accelerated at room temperatures over 23° C/73° F. Exceeding the given working time will cause the loss of adhesion to the dental enamel and the dentin.

**Capsule Activation**

- ▶ Place the Aplicap™ Activator on a sturdy surface and insert the Aplicap capsule into the activator. In doing so, keep the application nozzle shut.
  - – Both the Aplicap Activator and the Applier are color-coded in orange.
- ▶ Using the ball of your hands, depress the activator lever firmly and completely to the stop and hold it down for 2 to 4 seconds.
  - – Firmly and completely depressing the lever to the stop and holding it in this position is the only way to ensure that all of the liquid is pressed into the powder. If inadequate pressure is applied or if the lever is not depressed completely, it is possible that too little liquid will be mixed with the powder, increasing the viscosity and changing the product characteristics.

**Mixing**

- ▶ Mix the capsule at approx. 4,300 rpm in a high-frequency mixing device such as CapMix or the RotoMix rotating mixer. In doing so, keep the application nozzle shut.

**Application**

- ▶ Insert the capsule into the Aplicap™ Applier and open the application nozzle.
- ▶ Apply the glass ionomer cement in the cavity and shape the filling.
  - – Ensure that there is no contamination from water, saliva, etc., for the entire duration of application and shaping; the working environment must be kept dry.

**Attention**

- By all means prevent delay between activation, mixing, and application because the beginning of setting of the material in the capsule will render difficult or prevent the extrusion.
- Ketac Molar Aplicap clings to metal instruments and for this reason should be washed off with cold water before setting.

**Filling Protection**

Applying a protective coat to the filling is not mandatory. If a protective coat is desired, Ketac Glaze or Scotchbond Universal Adhesive or Single Bond Universal Adhesive can be used as follows:

- ▶ Immediately after shaping, apply Ketac Glaze or Scotchbond Universal Adhesive or Single Bond Universal Adhesive to all free surfaces of the glass ionomer cement and light-cure according to the respective Instructions for Use.
- ▶ The inhibition layer remaining after the setting of Ketac Glaze or Scotchbond Universal Adhesive or Single Bond Universal Adhesive may obstruct the setting of the addition-type silicone impression materials. For this reason, remove this layer or use a polyether impression material if needed.

**Finishing**

- ▶ Further finishing with Arkansas stones, fine-grain diamonds, abrasive discs with successively decreasing grain size, or silicone polishers should be started 4:30 minutes after start of mixing at the earliest.
- ▶ Optionally apply Ketac Glaze or Scotchbond Universal Adhesive or Single Bond Universal Adhesive again for surface sealing and light-cure according to the respective Instructions for Use.

**Storage and Shelflife**

Store the capsules in the blister packs.
Use within 1 month maximum once the blister pack has been opened!
Store the product at 15–25° C/59–77° F.
Do not use after the expiry date.

**Customer Information**

No person is authorized to provide any information which deviates from the information provided in this instruction sheet.

**Warranty**

3M Deutschland GmbH warrants this product will be free from defects in material and manufacture. 3M Deutschland GmbH MAKES NO OTHER WARRANTIES INCLUDING ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. User is responsible for determining the suitability of the product for user's application. If this product is defective within the warranty period, your exclusive remedy and 3M Deutschland GmbH's sole obligation shall be repair or replacement of the 3M Deutschland GmbH product.

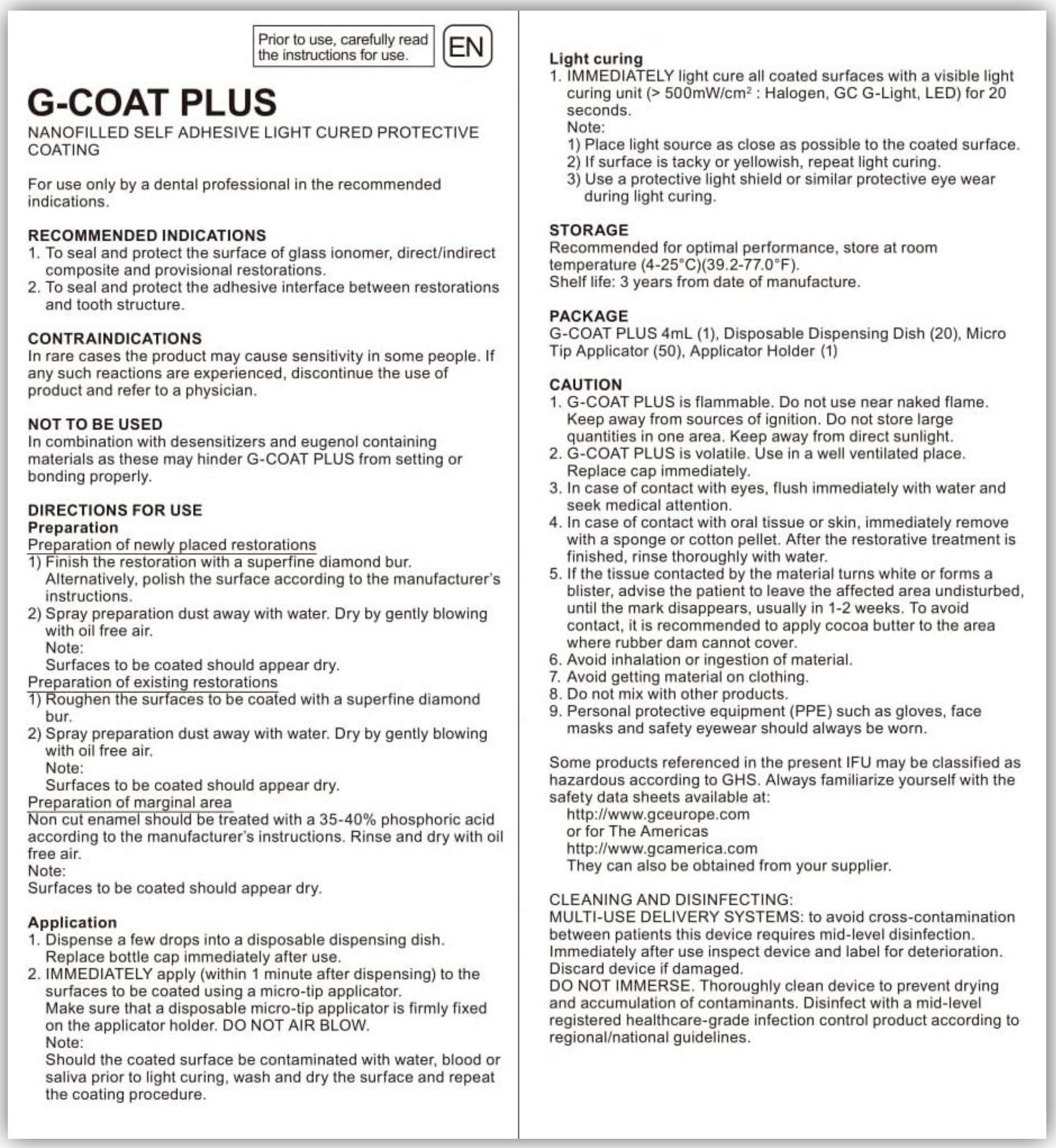
**Limitation of Liability**

Except where prohibited by law, 3M Deutschland GmbH will not be liable for any loss or damage arising from this product, whether direct, indirect, special, incidental or consequential, regardless of the theory asserted, including warranty, contract, negligence, or strict liability.

Information valid as of February 2014

## 4. Appendix D:

## G coat plus Use Instructions



Prior to use, carefully read the instructions for use. EN

# G-COAT PLUS

NANOFILLED SELF ADHESIVE LIGHT CURED PROTECTIVE COATING

For use only by a dental professional in the recommended indications.

**RECOMMENDED INDICATIONS**

1. To seal and protect the surface of glass ionomer, direct/indirect composite and provisional restorations.
2. To seal and protect the adhesive interface between restorations and tooth structure.

**CONTRAINDICATIONS**

In rare cases the product may cause sensitivity in some people. If any such reactions are experienced, discontinue the use of product and refer to a physician.

**NOT TO BE USED**

In combination with desensitizers and eugenol containing materials as these may hinder G-COAT PLUS from setting or bonding properly.

**DIRECTIONS FOR USE**

**Preparation**

Preparation of newly placed restorations

1) Finish the restoration with a superfine diamond bur. Alternatively, polish the surface according to the manufacturer's instructions.
2) Spray preparation dust away with water. Dry by gently blowing with oil free air.
   Note:
   Surfaces to be coated should appear dry.

Preparation of existing restorations

1) Roughen the surfaces to be coated with a superfine diamond bur.
2) Spray preparation dust away with water. Dry by gently blowing with oil free air.
   Note:
   Surfaces to be coated should appear dry.

Preparation of marginal area

Non cut enamel should be treated with a 35-40% phosphoric acid according to the manufacturer's instructions. Rinse and dry with oil free air.
Note:
Surfaces to be coated should appear dry.

**Application**

1. Dispense a few drops into a disposable dispensing dish. Replace bottle cap immediately after use.
2. IMMEDIATELY apply (within 1 minute after dispensing) to the surfaces to be coated using a micro-tip applicator. Make sure that a disposable micro-tip applicator is firmly fixed on the applicator holder. DO NOT AIR BLOW.
   Note:
   Should the coated surface be contaminated with water, blood or saliva prior to light curing, wash and dry the surface and repeat the coating procedure.

**Light curing**

1. IMMEDIATELY light cure all coated surfaces with a visible light curing unit (> 500mW/cm$^2$ : Halogen, GC G-Light, LED) for 20 seconds.
   Note:
   1) Place light source as close as possible to the coated surface.
   2) If surface is tacky or yellowish, repeat light curing.
   3) Use a protective light shield or similar protective eye wear during light curing.

**STORAGE**

Recommended for optimal performance, store at room temperature (4-25°C)(39.2-77.0°F).
Shelf life: 3 years from date of manufacture.

**PACKAGE**

G-COAT PLUS 4mL (1), Disposable Dispensing Dish (20), Micro Tip Applicator (50), Applicator Holder (1)

**CAUTION**

1. G-COAT PLUS is flammable. Do not use near naked flame. Keep away from sources of ignition. Do not store large quantities in one area. Keep away from direct sunlight.
2. G-COAT PLUS is volatile. Use in a well ventilated place. Replace cap immediately.
3. In case of contact with eyes, flush immediately with water and seek medical attention.
4. In case of contact with oral tissue or skin, immediately remove with a sponge or cotton pellet. After the restorative treatment is finished, rinse thoroughly with water.
5. If the tissue contacted by the material turns white or forms a blister, advise the patient to leave the affected area undisturbed, until the mark disappears, usually in 1-2 weeks. To avoid contact, it is recommended to apply cocoa butter to the area where rubber dam cannot cover.
6. Avoid inhalation or ingestion of material.
7. Avoid getting material on clothing.
8. Do not mix with other products.
9. Personal protective equipment (PPE) such as gloves, face masks and safety eyewear should always be worn.

Some products referenced in the present IFU may be classified as hazardous according to GHS. Always familiarize yourself with the safety data sheets available at:
http://www.gceurope.com
or for The Americas
http://www.gcamerica.com
They can also be obtained from your supplier.

CLEANING AND DISINFECTING:
MULTI-USE DELIVERY SYSTEMS: to avoid cross-contamination between patients this device requires mid-level disinfection. Immediately after use inspect device and label for deterioration. Discard device if damaged.
DO NOT IMMERSE. Thoroughly clean device to prevent drying and accumulation of contaminants. Disinfect with a mid-level registered healthcare-grade infection control product according to regional/national guidelines.

## 5. Appendix E:

## Scotchbond Universal Use Instructions

**3M ESPE**

# Scotchbond™ Universal
# Scotchbond™ Universal DCA

ⓔⓝ **Adhesive / Dual Cure Activator**
ⓓⓔ **Adhäsiv / Aktivator für Dualhärtung**
ⓕⓡ **Adhésif / Activateur pour la prise duale**
ⓘⓣ **Adesivo / Attivatore per polimerizzazione duale**
ⓔⓢ **Adhesivo / Activador para polimerización dual**
ⓟⓣ **Adesivo / Activador para dupla polimerização**

Instructions for Use
Gebrauchsinformation
Mode d'emploi

Informazioni per l'uso
Instrucciones de uso
Instruções de Utilização

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Dental Products
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CE 0123

3M ESPE Customer Care/MSDS Information:
U.S.A. 1-800-634-2249 and Canada 1-888-363-3685.

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44000188290/03

### ⓔⓝ ENGLISH

**Product Description**
Scotchbond™ Universal is a single-component, light-curing adhesive which is available in L-Pop blisters for single dosing or in bottles for multiple doses.

Depending on the indication, the adhesive is used:
- In a "self-etching" procedure to enable the shortest possible treatment time and to minimize post-operative sensitivities.
- With selective enamel etching to maximize the adhesion to the tooth enamel and to minimize post-operative sensitivities.
- In a "total etching" procedure with a prior phosphoric acid etching step, e.g., if the etching cannot be restricted to the tooth enamel.

Scotchbond™ Universal DCA activates the dual curing mechanism of Scotchbond Universal and is mixed with the adhesive whenever dual-cure or self-cure composite filling materials, cements, or core build-up materials are being used.
The addition of the activator to the adhesive is not required when using RelyX™ Ultimate Adhesive Resin Cement because the activator is already integrated into the cement.

☞ These Instructions for Use must be kept for reference for the duration of product use. Please see the pertinent information for use for details on all of the products mentioned below.

**Indications**
- All classes of fillings (according to Black) with light-curing composite or compomer filling materials
- Cementation of indirect restorations when combined with RelyX Ultimate Adhesive Resin Cement
- Cementation of veneers when combined with RelyX Veneer Cement
- Bonding of core build-ups made of light-curing composite or core build-up materials
- Bonding of dual-cure cements and core build-up materials and self-cure composites when combined with Scotchbond Universal DCA
- Repair of composite or compomer fillings
- Intraoral repair of composite restorations, porcelain fused to metal, and all-ceramic restorations without extra primer
- Root surface desensitization
- Sealing of cavities prior to cementation of amalgam restorations
- Sealing of cavities and preparation of tooth stumps prior to temporary cementation of indirect restorations
- Bonding of fissure sealants
- Protective varnish for glass ionomer fillings

**Precautionary Measures**

**For Patients and Dental Personnel**
- Scotchbond Universal: contact with eyes may cause severe eye damage. Wear eye protection to prevent injury. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

**For Patients**
- Scotchbond Universal contains substances that may cause an allergic reaction by skin contact in certain individuals. Avoid use of this product in patients with known acrylate allergies.
- If prolonged contact with oral tissue occurs, flush with large amounts of water. If allergic reaction occurs, seek medical attention as needed, remove the product if necessary, and discontinue future use.

**For Dental Personnel**
- Scotchbond Universal contains substances that may cause an allergic reaction by skin contact in certain individuals. To reduce the risk of allergic response, minimize exposure to these materials. In particular, avoid exposure to uncured adhesive. If skin contact occurs, wash skin with soap and water.
- The use of protective gloves and a no-touch technique is recommended. Acrylates may penetrate commonly used gloves. If Scotchbond Universal contacts the glove, remove and discard the gloves. Wash your hands immediately with soap and water and re-glove.
- If allergic reaction occurs, seek medical attention as needed.
- Scotchbond Universal and Scotchbond Universal DCA are highly flammable. Avoid static electricity build-up. Keep away from heat, sparks, open flame, pilot lights, and other ignition sources.

3M MSDSs can be obtained from www.3m.com, or contact your local subsidiary.

**Precautions during Processing**
- Seat temporary restorations using a eugenol-free product (e.g., with RelyX™ Temp NE). The use of temporary cements containing eugenol can inhibit the polymerization process of Scotchbond Universal during the final cementing.
- For the best possible bond, do not treat the surface of Zirconia, aluminum oxide, or metal restorations with phosphoric acid.

**Pulp Protection**
To avoid pulpal irritation, cover areas in close proximity to the pulp by applying small amounts of hard-setting calcium hydroxide material. To avoid bite increases during the cementing of the later restoration, the pulp protection should be applied prior to taking an impression for the final restoration.

**Tooth Preparation**

**Preparation of the Cavity/Tooth Stump**
- ► Remove loose preparation debris by spraying with water, and lightly air dry the stump or the cavity in only 2–3 bursts of water-free and oil-free air, or use cotton pellets to dry it off. Do not overdry.
  - The cavity should be just dry enough that the surface has a slightly glossy appearance. Overdrying can lead to post-operative sensitivity.

We advise against the use of substances such as desensitizers, disinfectants, astringents, dentin sealants, rinsing solutions containing EDTA, hydrogen peroxide, etc. (with the exception of chlorhexidine solutions) after the final cleaning. The residues of these substances can be detrimental to the bonding strength and setting reaction of the adhesive, especially of dual- and self-cure materials.

**Unprepared Tooth Enamel**
- ► Clean the tooth enamel as usual (e.g., with polishing paste or powder blasting), and thoroughly rinse by spraying with water.
- ► Then etch and rinse, see section "Selective Enamel Etching".
- ► Continue with the application of Scotchbond Universal Adhesive.

**Etching of the Tooth Structure (optional)**
Clinically adequate adhesive bonds are achieved by the application of the adhesive. Even higher adhesive values can be achieved by selectively etching the enamel or using the "total etching procedure".

**Selective Enamel Etching**
Accidental etching of the dentin does not have any detrimental effect on the adhesion.
- ► Apply a commonly used phosphoric acid etching gel (about 35%), e.g., Scotchbond Universal Etchant to the prepared and unprepared (if present) tooth enamel and allow to react for 15 sec.
- ► Rinse thoroughly with water and dry with water-free and oil-free air or with cotton pellets; do not overdry.

**Total Etching Procedure**
- ► Apply a commonly used phosphoric acid etching gel (about 35%), e.g., Scotchbond Universal Etchant, to the prepared and unprepared (if present) tooth structure (enamel and dentin) and allow to react for 15 sec.
- ► Rinse thoroughly with water and dry with water-free and oil-free air or with cotton pellets; do not overdry.

**Root Surface Desensitization**
- ► Clean the root surface as usual (e.g., with polishing paste or powder blasting).
- ► Clean thoroughly by spraying with water, and dry with water-free and oil-free air or with cotton pellets.
- ► Continue with the application of Scotchbond Universal Adhesive.

**Pre-treatment of Fillings or Restorations During Repair**
(made of composite, glass ceramic, Zirconia ceramic, aluminum oxide ceramic, metal (incl. amalgam))
- ► Thoroughly roughen the surface of the restoration which is to be luted, preferably by blasting with aluminum oxide ≤ 40 μm; if ceramic, remove the glaze up to 1 mm over the margin.
- ► Clean thoroughly by spraying with water, and dry with water-free and oil-free air or with cotton pellets.
  - Do not etch with phosphoric acid because this can weaken the adhesion to certain materials (e.g., Zirconia, aluminum oxide, metal).
- ► Continue immediately with the application of Scotchbond Universal Adhesive. The use of an additional primer is unnecessary.

**Dosing Scotchbond™ Universal Adhesive**

**Dosing from the Bottle**
The dosing bottle cap has a thumb depression so that it can be opened to one side and closed again with one hand. The cap will remain open if the opening angle is > 90°.
- ► To open, take the bottle in your hand, place your thumb in the thumb depression, and push the cap to the side until it remains in the open position.
- ► Hold the bottle upside down in a vertical position and pour the required quantity of Scotchbond Universal in a mixing well. Protect the adhesive in the mixing well from light.
- ► After use, carefully close the bottle again by applying pressure to the lever from above.
  - The cap tangibly snaps into place when pressed closed and a small, even gap between the cap and bottle can be seen all the way around.

**Dosing from the L-Pop Blister**

**Attention:** Do not squeeze the L-Pop blister without the disposable applicator.
- ► Hold the shaft of the disposable applicator with one hand, covering the blister opening with your thumb. This will prevent the etching liquid from spraying out.
- ► With the thumb and index finger of the other hand, squeeze the reservoir of the L-Pop blister, starting with the outer end, in the direction of the disposable applicator.
- ► Turn the disposable applicator back and forth in the liquid to cover it completely.
- ► For easy application in the cavity, the disposable applicator can be bent while it is still in the L-Pop blister. In order to do this, only pull the disposable applicator out of the blister far enough so that the narrow section of the applicator shaft can be seen. Fold the applicator shaft at this location over your thumbnail.
- ► During the treatment, press all of the liquid into the open depression end with your thumb and index finger to rewet the applicator.

**Dosing Scotchbond™ Universal DCA Activator**
- ► Unscrew the lid of the bottle.
- ► Mix the required quantity of Scotchbond Universal DCA in a ratio of 1-to-1 (drops) with Scotchbond Universal in a mixing well and mix for 5 sec immediately before application. Protect the liquid in the mixing well from light.
- ► Carefully close the bottle tightly after dosing. If necessary, disinfect the bottle in accordance with local hygiene regulations.

**Application**
- ► Avoid any contamination with blood, saliva, or sulcus fluid during application. We recommend the use of a rubber dam to ensure that the treated area is kept sufficiently dry and to prevent exposure of any soft tissue.

**Procedure for direct restorations with light-curing composites and compomers, intraoral repair using light-curing composites, root surface desensitization, sealing of cavities before filling with amalgam, sealing of cavities and stump preparations before temporary attachment of indirect restorations**
- ► Use the disposable applicator to apply the adhesive to the entire tooth structure and **rub it in** for 20 sec. Avoid contact between the adhesive and the oral mucosa.
- ► If necessary, rewet the disposable applicator during treatment.
- ► Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely.
- ► Harden the adhesive with a commonly used curing light for 10 sec.
- ► As appropriate for the indication, continue with the desired material in accordance with the pertinent instructions for use.

**Procedure for dual-cure and self-cure composite and core build-up materials and cements**
When using RelyX Ultimate, the activator is not required because it has been integrated into the cement. Please follow the RelyX Ultimate instructions for use.
- ► Place one drop each of Scotchbond Universal and Scotchbond Universal DCA in a mixing well and mix for 5 sec.
- ► Use the disposable applicator to apply the mixture to the entire tooth structure and **rub it in** for 20 sec. Avoid contact between the adhesive and the oral mucosa.
- ► If necessary, rewet the disposable applicator during treatment.
- ► Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely.
- ► Harden the adhesive with a commonly used curing light for 10 sec.
- ► As appropriate for the indication, continue with the desired material in accordance with the pertinent instructions for use.

**Procedure when cementing indirect restorations and veneers using RelyX Ultimate Adhesive Resin Cement**
- ► When using RelyX Ultimate, Scotchbond Universal DCA is not required. Please follow the RelyX Ultimate instructions for use.

**Use of Scotchbond Universal as a primer for indirect restorations**
When using RelyX Ultimate, Scotchbond Universal DCA is not required. Please follow the RelyX Ultimate instructions for use.
- ► Prepare the surface of the restoration:
  - Etch glass ceramic surfaces with hydrofluoric acid in accordance with manufacturer's instructions.
  - Roughen composite, Zirconia ceramic, and metal surfaces, preferably by sandblasting.
  - Do not etch with phosphoric acid because this can weaken the bonding to certain materials (e.g., Zirconia, aluminum oxide, metals).
- ► Thoroughly rinse the surface with water and dry with water-free and oil-free air or with cotton pellets.
- ► In combination with other composite cements:
  - Place one drop each of Scotchbond Universal and Scotchbond Universal DCA in a mixing well and mix for 5 sec.
  - Immediately after mixing, use the disposable applicator to apply the adhesive to the entire surface of the restoration to be cemented and allow it to **react** for 20 sec. Do not light-cure.
  - Follow the instructions for use from the manufacturer to apply the cement.

**Using RelyX Ultimate to cement posts**
- ► When using RelyX Ultimate, Scotchbond Universal DCA is not required. Please follow the RelyX Ultimate instructions for use.

**Using other composite cements to cement posts**
- ► Prepare the endodontically treated root canal for the post cementing.
- ► Trial fit the post; it should fill 2/3 of the root canal.
- ► Place one drop each of Scotchbond Universal and Scotchbond Universal DCA in a mixing well and mix for 5 sec.
- ► Use the disposable applicator to apply the mixture to the entire tooth structure and **rub it in** for 20 sec.
- ► Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely. Soak up excess liquid with a paper tip.
- ► Harden the adhesive with a commonly used curing light for 10 sec.
- ► Follow the instructions for use for the relevant product to cement the post.

**Procedure when cementing veneers using 3M ESPE RelyX™ Veneer Cement**
- ► The surface of the glass ceramic veneer which is to be cemented should be etched using hydrofluoric acid in accordance with manufacturer's instructions. An additional silane primer is not required when using Scotchbond Universal.
- ► Before applying the adhesive, clean the prepared tooth structure with pumice paste, thoroughly rinse with water, and dry with water- and oil-free air or with cotton pellets.
- ► Trial fit the veneer using RelyX™ Try-In Paste. Use water to rinse off the paste from the tooth and veneer and dry all of the surfaces with water- and oil-free air or with cotton pellets. Isolate the adjacent teeth from the veneer preparation using transparent template strips.
- ► Apply a commonly used phosphoric acid etching gel (about 35%), e.g., Scotchbond Universal Etchant, to the enamel and dentin and allow to react for 15 sec. Then rinse thoroughly with water for 10 seconds and dry with cotton pellets. The surface should be shining and not have any puddles on it.
- ► Use the disposable applicator to apply Scotchbond Universal immediately to the entire surface of the enamel and dentin and **rub it in** for 20 sec. Avoid contact between the adhesive and the oral mucosa. If necessary, rewet the disposable applicator during treatment. Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely. **Do not light-cure.**
- ► Use the disposable applicator to apply Scotchbond Universal to the etched veneer. Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely. **Do not light-cure.**
- ► Carefully cement the veneer with RelyX™ Veneer Cement following the pertinent instructions for use. Remove any excess from the veneer edges and light-cure in accordance with manufacturer's instructions. We recommend light-curing the gingival edges first, then the middle and the incisal edges. Avoid eye contact with the light source.

**Procedure for light-cure sealing of fissures**
In contrast to a composite filling, light-cure fissure sealants can be bonded to untreated tooth enamel using Scotchbond Universal without prior etching with phosphoric acid.
- ► Clean the surface to be sealed with polishing paste or by powder blasting (e.g., using Clinpro Prophy Powder).
- ► Use the disposable applicator to apply Scotchbond Universal to the surface to be sealed and **rub it in** for 20 sec, applying pressure. Continue to rub in for a longer period of time when sealing larger surfaces. **Do not reduce this time.** Applying and distributing the adhesive is not adequate.
- ► Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely.
- ► Apply a light-cure fissure sealant, e.g., Clinpro Sealant.
- ► Polymerize the fissure sealant and the adhesive together with a commonly available light-curing device; the time is dependent on the fissure sealant used.

**Use as protective varnish for glass ionomer fillings**
- ► If a template was used for the shaping, first remove the template.
- ► Use the disposable applicator to apply Scotchbond Universal to the fully modeled filling and **rub it in** for 20 sec. Avoid contact between the adhesive and the oral mucosa.
- ► If necessary, rewet the disposable applicator during treatment.
- ► Subsequently direct a gentle stream of air over the liquid for about 5 sec until it no longer moves and the solvent has evaporated completely.
- ► Use a commonly available light-curing device to polymerize the adhesive for 10 sec on every surface of the filling.
- ► When the filling has been completely cured, grind down the occlusion and reapply the adhesive to the ground places to protect the filling from drying out.
- ► Use a damp cloth or cotton pellet to remove the oxygen inhibition layer.

**Hygiene and Disinfection**
- ► Clean any contaminated adhesive and activator bottles with commonly used agents and disinfect them in accordance with the commonly used hygienic regulations.

**Notes**
- Scotchbond Universal can prematurely polymerize when exposed to natural or artificial lighting. Avoid intensive light exposure during application.

**Storage and Stability**
Store Scotchbond Universal and Scotchbond Universal DCA at 02–25° C/ 36–77° F.
Do not use after the expiration date.

**Customer Information**
No person is authorized to provide any information which deviates from the information provided in this instruction sheet.

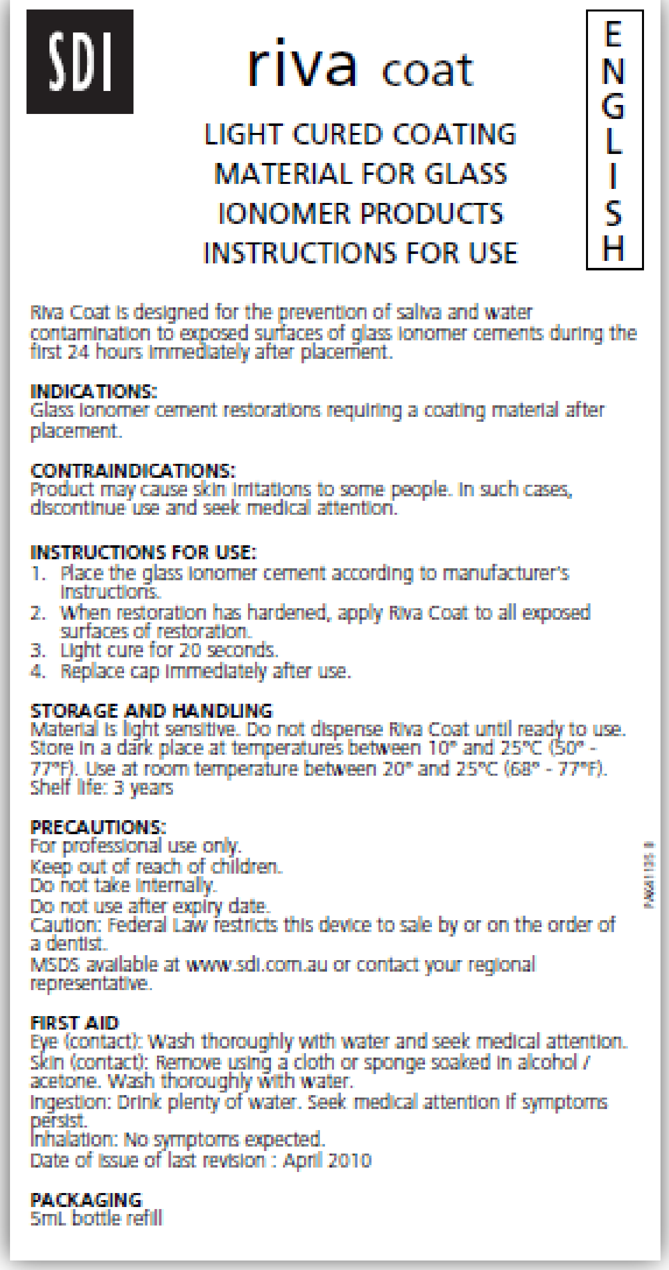
**Warranty**
3M Deutschland GmbH warrants this product will be free from defects in material and manufacture. 3M Deutschland GmbH MAKES NO OTHER WARRANTIES, INCLUDING ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. User is responsible for determining the suitability of the product for user's application. If this product is defective within the warranty period, your exclusive remedy and 3M Deutschland GmbH's sole obligation shall be repair or replacement of the 3M Deutschland GmbH product.

**Limitation of Liability**
Except where prohibited by law, 3M Deutschland GmbH will not be liable for any loss or damage arising from this product, whether direct, indirect, special, incidental or consequential, regardless of the theory asserted, including warranty, contract, negligence, or strict liability.

Information valid as of July 2013

## 6. Appendix F:

### Riva coat Use Instructions



SDI

riva coat

LIGHT CURED COATING
MATERIAL FOR GLASS
IONOMER PRODUCTS
INSTRUCTIONS FOR USE

ENGLISH

Riva Coat is designed for the prevention of saliva and water contamination to exposed surfaces of glass ionomer cements during the first 24 hours immediately after placement.

**INDICATIONS:**
Glass ionomer cement restorations requiring a coating material after placement.

**CONTRAINDICATIONS:**
Product may cause skin irritations to some people. In such cases, discontinue use and seek medical attention.

**INSTRUCTIONS FOR USE:**

1. Place the glass ionomer cement according to manufacturer's instructions.
2. When restoration has hardened, apply Riva Coat to all exposed surfaces of restoration.
3. Light cure for 20 seconds.
4. Replace cap immediately after use.

**STORAGE AND HANDLING**
Material is light sensitive. Do not dispense Riva Coat until ready to use. Store in a dark place at temperatures between 10° and 25°C (50° - 77°F). Use at room temperature between 20° and 25°C (68° - 77°F).
Shelf life: 3 years

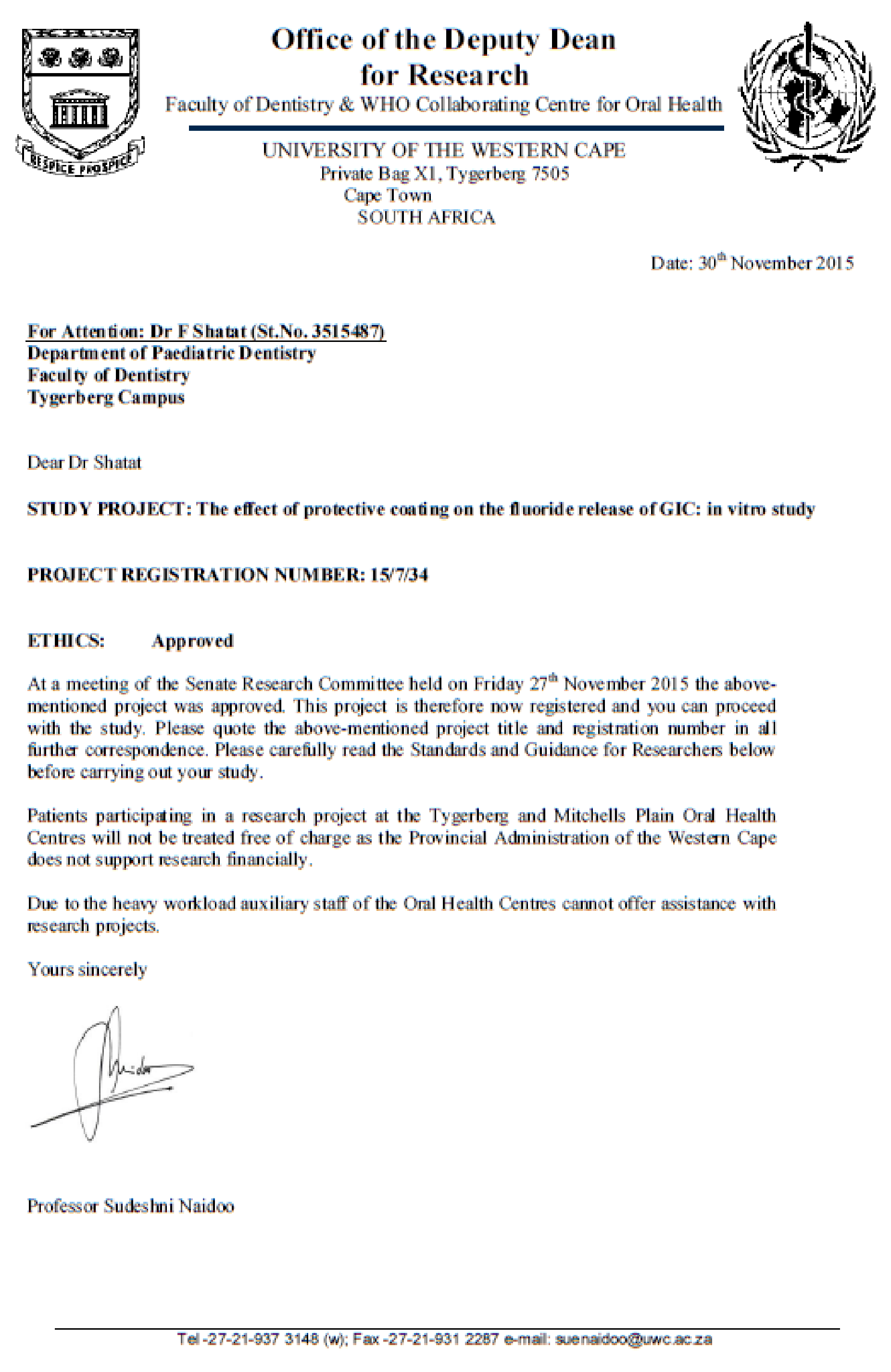
**PRECAUTIONS:**
For professional use only.
Keep out of reach of children.
Do not take internally.
Do not use after expiry date.
Caution: Federal Law restricts this device to sale by or on the order of a dentist.
MSDS available at www.sdi.com.au or contact your regional representative.

**FIRST AID**
Eye (contact): Wash thoroughly with water and seek medical attention.
Skin (contact): Remove using a cloth or sponge soaked in alcohol / acetone. Wash thoroughly with water.
Ingestion: Drink plenty of water. Seek medical attention if symptoms persist.
Inhalation: No symptoms expected.
Date of issue of last revision : April 2010

**PACKAGING**
5mL bottle refill

## 7. Appendix G:

### Project registration and ethics approval letter



Office of the Deputy Dean
for Research
Faculty of Dentistry & WHO Collaborating Centre for Oral Health

UNIVERSITY OF THE WESTERN CAPE
Private Bag X1, Tygerberg 7505
Cape Town
SOUTH AFRICA

Date: 30th November 2015

**For Attention: Dr F Shatat (St.No. 3515487)**
**Department of Paediatric Dentistry**
**Faculty of Dentistry**
**Tygerberg Campus**

Dear Dr Shatat

**STUDY PROJECT: The effect of protective coating on the fluoride release of GIC: in vitro study**

**PROJECT REGISTRATION NUMBER: 15/7/34**

**ETHICS: Approved**

At a meeting of the Senate Research Committee held on Friday 27th November 2015 the above-mentioned project was approved. This project is therefore now registered and you can proceed with the study. Please quote the above-mentioned project title and registration number in all further correspondence. Please carefully read the Standards and Guidance for Researchers below before carrying out your study.

Patients participating in a research project at the Tygerberg and Mitchells Plain Oral Health Centres will not be treated free of charge as the Provincial Administration of the Western Cape does not support research financially.

Due to the heavy workload auxiliary staff of the Oral Health Centres cannot offer assistance with research projects.

Yours sincerely

Professor Sudeshni Naidoo

Tel -27-21-937 3148 (w); Fax -27-21-931 2287 e-mail: suenaidoo@uwc.ac.za