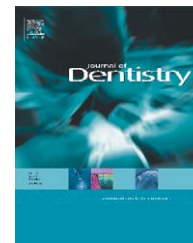


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# Chemical exchange between glass-ionomer restorations and residual carious dentine in permanent molars: An in vivo study

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## ABSTRACT

**Objective:** To evaluate the remineralization of carious dentine following the restoration of an extensive lesion in a permanent molar with a high strength glass-ionomer cement (GIC).

**Materials and methods:** Thirteen first permanent molars, which were scheduled for extraction because of the presence of extensive caries lesions, were selected for this study. They were first restored, according to the ART technique, using encapsulated Fuji IX<sub>GP</sub>, which contains a strontium glass rather than the traditional calcium glass. The cavities were prepared with a clean enamel margin and minimal removal of the carious dentine around the walls. After a period of 1–3 months they were harvested and subsequently sectioned and examined using an electron probe microanalysis (EPMA) and scanning electron microscopy (SEM).

**Results:** EPMA demonstrated that both fluorine and strontium ions had penetrated deep into the underlying demineralized dentine. The only possible source of these ions was the GIC restoration.

**Conclusion:** The pattern of penetration of the fluorine and strontium ions into the dentine was consistent with a remineralization process.

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## 1. Introduction

Since the time of Dr. G.V. Black the profession has been taught to completely remove softened and discoloured dentine to eliminate infected tissue and create a hard foundation to support a proposed restoration. The suggested routine has been to remove all demineralized dentine, using aggressive hand instrumentation or a round bur, until sound, normal dentine formed the entire pulpal floor. The objective was to ensure the elimination of all remaining microorganisms thus eliminating a possible recurrence of caries. However, Lager et al. showed that this is not always successful and some microorganisms may remain even after

all softened dentine has been removed and the cavity treated with sodium hypochlorite.<sup>1</sup> The main risk with this traditional approach is the possible accidental exposure of the pulp, particularly in young patients, where the rate of pulp exposure following excavation of large carious lesions in permanent molars has been rated at 40%.<sup>2</sup>

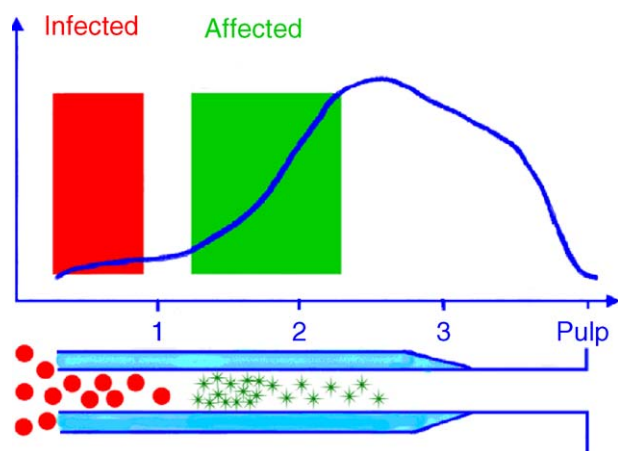
A step-wise excavation technique was introduced by Bodecker<sup>3</sup> designed to decrease the risk of mechanical pulp exposure. Bodecker recommended partial removal of the soft demineralized dentine on the cavity floor followed by immediate restoration with a temporary material such as zinc oxide/eugenol. The transitional material was expected to remain for a brief period of weeks and then replaced with

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**Fig. 1 – Schematic drawing to illustrate the relationship between the hardness curve corresponding to the infected and affected zones found in carious dentine. The diagram below the chart shows the relationship between the bacterial invasion front and the mineralization of the dentinal tubule (Adapted from<sup>5</sup>).**

a permanent restoration. When the transitional restoration was due for replacement, further excavation was to be carried out to completely remove any remaining demineralized dentine. Compared with the traditional G.V. Black approach, it was found in a randomized clinical trial that the incidence of direct pulp exposure was 17.5% for the step-wise excavation method compared with 40% for the traditional method.<sup>2</sup>

Massler and Fusayama both suggested that there were two layers in carious dentine. The outer layer is heavily infected by microorganisms and is broken down to the extent that it cannot be remineralized at all. However, the inner layer, immediately adjacent to sound dentine on the floor of the lesion, will be partially demineralized, will contain some bacterial flora, but will still have some of the original dentine tubule structure present. This was identified as the affected layer and it has been shown that this can be remineralized, at least to a degree (Fig. 1).<sup>4,5</sup> These authors suggested that only the outer infected layer should be removed during cavity preparation and the inner affected layer be retained and treated with a therapeutic lining or base of calcium hydroxide, glass-ionomer or a similar material. However, it has always been acknowledged that clinical differentiation between the infected and affected layer poses problems and there is still no reliable technique available to identify them separately. Recently it has been suggested that there is no need to differentiate between the two layers and therefore unnecessary to re-enter a properly sealed lesion. Mertz-Fairhurst et al. showed that 10 years after completely sealing a lesion the soft, wet, demineralized dentine left on the floor of a lesion, did not progress or jeopardize the restoration placed above it. The clinical result was sound providing a complete seal had been established and maintained.<sup>6</sup> This was later confirmed in another study.<sup>7</sup>

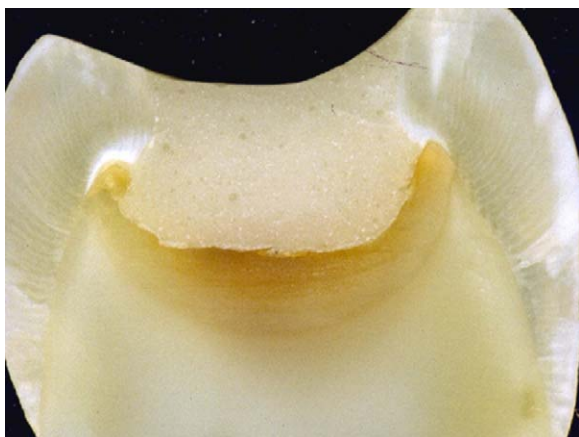
The advent of the glass-ionomers provided the profession with a biocompatible material, with low technique sensitivity, which allows for the development of an ion exchange adhesion with both enamel and dentine. One of the more

interesting uses of glass-ionomer has been the atraumatic restorative technique (ART)<sup>9</sup> for stabilisation of caries lesions in countries where routine dental care is not available. This “low tech-low cost” approach enables the delivery of restorative care without the need for electricity and high-tech equipment. The concept means it is possible to remove the infected layer on the floor of the cavity using dental hand instruments only leaving the affected layer or partly demineralized dentine underneath. A glass-ionomer can then be placed to restore and seal the lesion. It has been shown in both laboratory and clinical studies that fluoride ions are released from glass-ionomer and taken up by the adjacent sound enamel as well as the demineralized dentine.<sup>10-16</sup> There is also some in vitro evidence to suggest that demineralized dentine can be remineralized by the placement of glass-ionomer adjacent to an artificial lesion and that glass-ionomer can impart a certain degree of protection in tooth surfaces adjacent to it.<sup>17-19</sup> However, to date there has been little clinical evidence that such direct internal remineralization of a caries lesion will occur underneath glass-ionomer restorations.<sup>20,21</sup> It is essential to gain a more thorough understanding of the way in which glass-ionomer interacts with partially demineralized dentine in order to ensure, not only that it is used to greatest advantage, but also to permit further material developments to be investigated. The purpose of this clinical trial was to study the interaction, at a chemical level, between glass-ionomer and demineralized dentine under in vivo conditions.

## 2. Materials and methods

As this was designed as a clinical trial, it was necessary to find a source of carious teeth that needed to be extracted for the benefit of the patient. At the time, one of the authors had been seconded to the Dental School of the Department of Medicine, Colonial War Memorial Hospital, Suva, Fiji and was assisting in the introduction of a new curriculum and the development of a full BDS course of training for the University of the Pacific. He was aware of a series of patients in the age range from 12 to 16 years who had already had two or more of their first molars extracted due to rampant caries and it was apparent that the loss of the remaining carious first molars would, in fact, offer some level of balance in their occlusion. A proposal was placed before the Department of Health, Government of Fiji, requesting permission to approach young people, and their parents, who were due to have such serial extractions, to ask if they would be willing to have the carious teeth due for extraction, restored initially using the ART technique. Permission was granted by the Government of Fiji, and Ethical approval was also granted by the Committee for the Ethics of Human Experimentation, The University of Adelaide, South Australia.

A clear description of the proposed action was provided in writing to each patient and their parents, and their consent was also obtained in writing. One of the authors carried out the ART restorations, abiding strictly by the methods outlined by WHO for this technique to be most effective.<sup>9</sup> A total of 13 subjects volunteered, and the restorations were placed using Fuji IX<sub>CP</sub> (GC Corp., Tokyo, Japan) as the restorative material.



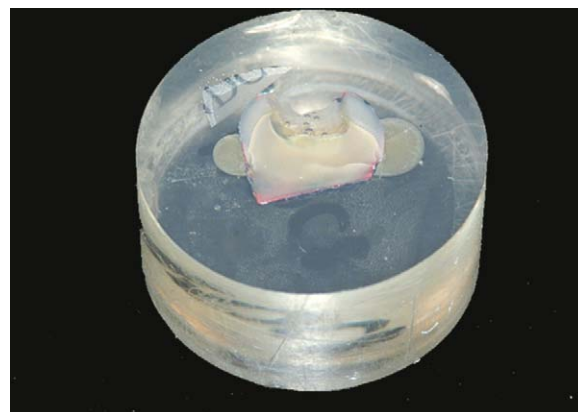
**Fig. 2 – A sample immediately after sectioning, showing the extent of demineralized dentine left under the glass-ionomer restoration.**

Fuji IX<sub>GP</sub> is an encapsulated glass-ionomer, which contains a strontium glass to enhance radiopacity rather than the conventional calcium glass. An incidental advantage of the presence of strontium is that it has been shown to be universally present in whole enamel.<sup>22</sup> As an element, it has close similarity to Ca in both chemical and physical properties, it has a similar ionic radius and valence to that of calcium ( $\text{Sr}^{2+} = 1.13 \text{ \AA}$ ,  $\text{Ca}^{2+} = 0.99 \text{ \AA}$ ), so it can replace calcium without disrupting the structure of hydroxyapatite.<sup>23</sup> This suggests that calcium lost from dentine during progress of the caries lesion should be able to be replaced with strontium from glass-ionomer in the healing process and that this replacement should be able to be traced using EPMA techniques.

The protocol for the experiment involved extraction of the series of teeth over a period between 1 and 3 months following restoration placement. In all 13 teeth were harvested over this period. Immediately following extraction the teeth were preserved in 2% glutaraldehyde until they could be prepared for EPMA analysis.

Each tooth was sectioned mesio-distally through the centre of the glass-ionomer restoration (Fig. 2) To minimize shrinkage the specimens were then dehydrated using a series of solutions with decreasing concentrations of alcohol as previously described.<sup>24</sup> The specimens were embedded in epoxy resin (Adelaide Epoxy Supplies, Adelaide, Australia), plano-parallel specimens were prepared then polished progressively down to a 0.3  $\mu\text{m}$  diamond grit (Struers, Copenhagen, Denmark), before coating with carbon (Fig. 3).

The electron probe microanalysis (EPMA) was carried out using a Cameca SX51 microanalyser (Cameca, Corbevoie, France) set at 15 kV and 20 mA, with a beam diameter of 1000  $\text{\AA}$ , running in wavelength dispersive spectrometry (WDS) mode and a collection time of 10 s at each point. Calibration for the EPMA used a minerals mount MINM25-53 (Astimex Scientific, Toronto, Canada), with apatite, fluorite and celestite used as standards to calibrate Ca and P, F and Sr, respectively. The calculated minimum detection limits (MDL) are listed in Table 1. A programmed spot analysis for calcium, phosphorus, fluorine and strontium was performed along a line perpendicular to the GIC/dentine interface at 5  $\mu\text{m}$



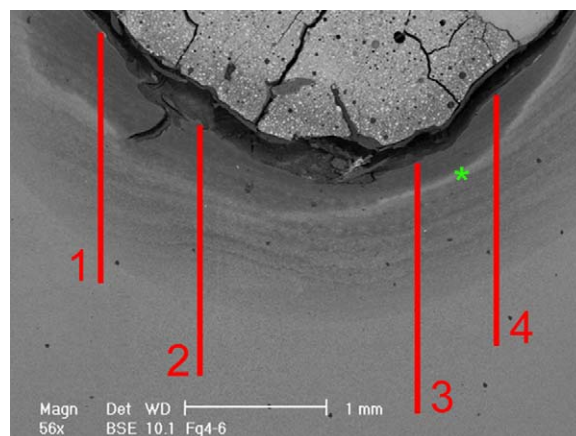
**Fig. 3 – Sample ready for coating with carbon before EPMA and SEM.**

**Table 1 – Concentrations found in the standard used for calibration and minimum detection limits for the elements of interest**

	Calibration levels (wt.%)	Minimum detection limit (wt.%)
Ca	39.85	0.05
P	18.50	0.12
F	48.67	0.05
Sr	47.52	0.05

intervals, reaching into sound dentine. Data was collected from several regions in each sample and a set of three lines was performed for each region<sup>25</sup> (Fig. 4). An average was calculated from the three lines to represent the mineral profile for that region of analysis.

The scanning electron microscopy (SEM) utilized a Philips XL30 field emission scanning electron microscope (Philips, Eindhoven, The Netherlands) set at 10 kV with a spot size of



**Fig. 4 – For this sample, EPMA was undertaken in four separate regions, marked by the four red lines. The green star shows a region with high mineral content, Ca, P and Sr. This is the same sample shown in Fig. 2. For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.**

**Table 2 – Average depth of carious dentine and average depth of penetration of Sr and F**

Sample ID	Average depth of carious dentine in $\mu\text{m}$ (S.D.)	Average depth of penetration of Sr in $\mu\text{m}$ (S.D.)	Average depth of penetration of F in $\mu\text{m}$ (S.D.)
1	1178 (101)	1160 (104)	1056 (164)
2	900 (218)	940 (298)	577 (125)
3	350 (71)	350 (71)	350 (71)
4	300 (141)	400 (141)	350 (212)
5	100 (0)	150 (0)	50 (0)
6	1735 (78)	1645 (120)	1515 (191)
7	1210 (241)	1380 (85)	1105 (148)
8	845 (92)	875 (35)	830 (56)
9	707 (192)	700 (201)	577 (172)
10	790 (497)	917 (578)	723 (441)
11	550 (14)	540 (28)	375 (70)
12	497 (199)	467 (179)	487 (179)
13	905 (205)	1105 (233)	1120 (212)
Mean	774 (121)	818 (121)	701 (113)

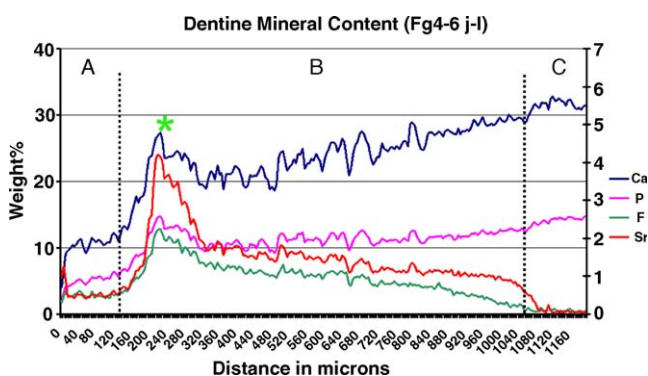
3  $\mu\text{m}$  and using in back scattered mode to highlight the overall mineral distribution in the samples. The SEM micrographs were used together with the mineral profiles derived from EPMA to determine the average depth of the lesions and the depth of penetration for Sr and F as shown in Table 2.

### 3. Results

The results from the EPMA are presented as mineral profiles (Fig. 5). This is the analysis of the sample previously shown in Figs. 2 and 4. The horizontal axis represents distance from the interface into dentine expressed in  $\mu\text{m}$ , with the interface being on the left side of the chart. The concentrations of the elements are shown on the vertical axes, where the weight percentages for calcium and phosphorous are shown on the left vertical axis (0–40%) and those for fluorine and strontium are shown on the right vertical axis (0–7%).

Figs. 5 and 6 show typical profiles of Sr and F, found in a large and a small lesion in carious dentine. These profiles can be roughly divided in to three distinct zones, namely:

(A) Adjacent to the glass-ionomer restoration. This area is heavily demineralized with the levels of Ca and P being



**Fig. 5 – Typical mineral profiles found in carious dentine, in a large lesion, adjacent to the glass-ionomer restoration. Ca and P are charted on the left hand scale. F and Sr are charted on the right hand scale.**

close to or below 10 wt.% in comparison to normal dentine which is approximately 30 wt.%.

(B) The transition zone between zone A and zone C, which is sound dentine.

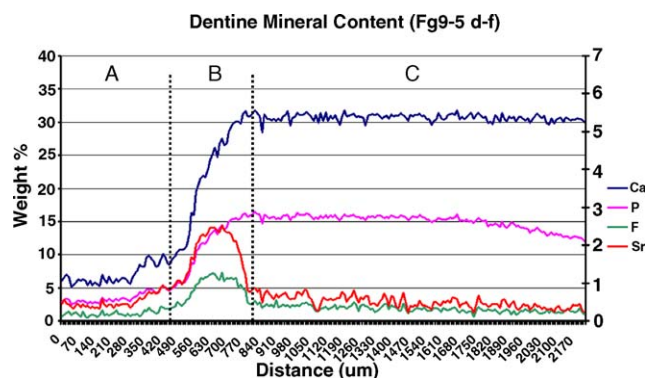
(C) Sound dentine, with level of calcium approximately above 30 wt.%.

Table 2 shows the average depth of carious dentine found in each of the 13 samples and the depth of penetration of both Sr and F into both zones A and B. It is evident that the penetration of these two elements finishes where zone C starts. This observation is confirmed by the strong correlation coefficient between the depth of carious dentine and the depth of penetration of Sr ( $r = 0.98$ , significant at 0.01 level). When the depth of penetration of fluorine is compared with the depth of carious dentine, the correlation is again significant ( $r = 0.95$ , significant at 0.01 level) (Fig. 7).

Table 3 shows that there is a strong correlation between the level of strontium and fluorine, especially in zone B.

#### 3.1. Scanning electron microscopy

All specimens were examined using a SEM in back scattered electron (BSE) mode to highlight the overall mineral distribution of the sample and also to measure the depth of the demineralized area, zones A and B.



**Fig. 6 – Typical mineral profiles found in a small carious lesion in dentine, adjacent to a glass-ionomer restoration.**



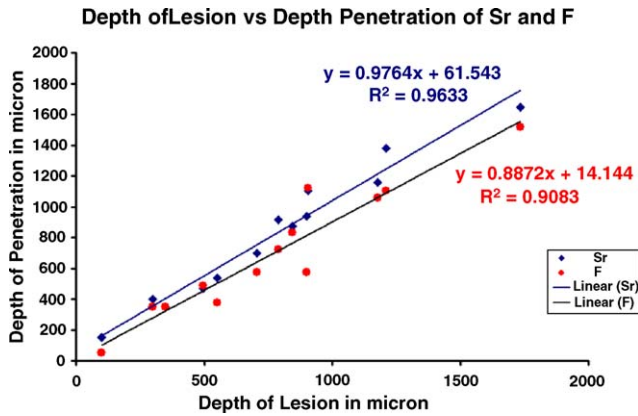


Fig. 7 – Depth of lesion and depth of penetration of Sr and F found in the 13 samples.

Table 3 – Correlation coefficient for the levels of strontium and fluorine in the three zones A, B and C

	A	B	C
Large lesion (Fig. 5)	-0.13	0.93*	0.28
Small lesion (Fig. 6)	0.82*	0.97*	0.84*

\* Significant at 0.01 level.

The green star in Fig. 4 corresponds to a thin bright band, which is due to a higher level of mineral as well as the presence of Sr, which has a higher atomic number. This zone of elevated mineral content was quantitatively confirmed in Fig. 5.

#### 4. Discussion

The objective in these analyses was to determine the level of ion exchange between glass-ionomer and partially demineralized dentine. Remineralization in enamel has previously been defined as the deposition of mineral in demineralized defects at a molecular level.<sup>26</sup> It has been suggested that the mineral deposited should be apatitic and it should not be very different from the mineral of sound enamel and dentine. It has also been suggested that the term remineralization should only be used if the mineral is “brought in from outside” the enamel. It is suggested that the above definition and observations should also be applicable to the remineralization of carious dentine.

Fuji IX<sub>GP</sub> was selected as the restorative material because, like all glass-ionomers, it contains fluoride ions. However, it is different from the original glass-ionomers in as much as it is manufactured with a strontium glass rather than the conventional calcium glass. This means that there will be strontium ions available rather than calcium so, for the purposes of this experiment, any possible remineralization will occur through deposition of strontium. In fact, this study showed that a substantial amount of both strontium and fluorine crossed the interface into the partially demineralized dentine adjacent to the restorative material. Any strontium and fluorine above background levels, which were

found in these samples, must come from the restorative glass-ionomer because these elements are not normally found at high levels in dental tissues. This observation was confirmed by the low levels of strontium and fluorine recorded by EPMA in sound dentine (Figs. 5 and 6).

The presence of strontium and fluorine in the remineralized dentine is most likely controlled by both diffusion and remineralization. However, observation of the profiles of strontium and fluorine distribution in Figs. 5 and 6 allows the following general observations to be made:

- (1) The depth of penetration of the remineralizing ions is related to the physical state of the demineralized dentine. The accumulation is greatest in zone B where the concentrations of the two major elements—calcium and phosphorus—vary between 10 and 30 wt.% for calcium and 5 and 15 wt.% for phosphorus.
- (2) The accumulation of strontium and fluorine ceases in the region of the junction between demineralized (zone B) and sound dentine (zone C). The correlation coefficient between the depth of penetration and the depth of the lesion are highly significant, at 0.98 for strontium and 0.95 for fluorine.
- (3) Both strontium and fluorine accumulated deep into the lesion. For strontium, the mean depth was 818 μm and the maximum depth was 1645 μm. The equivalent figures for fluorine were 701 and 1515 μm.
- (4) There is a clear relationship between the concentration of strontium and fluorine in zone B, for both large (r = 0.93) and small lesions (r = 0.97).

It is known that the pH of the liquid component of a glass-ionomer is close to pH 1.0 and that, as the powder is added to the liquid, there will be an ion release from the surface of the powder particles that will include both fluorine and strontium. As the freshly mixed material is placed against a cavity wall there will be a release of ions from the enamel and dentine as well leading to the exchange of ions, which is recognised as the ion exchange adhesion. It is suggested that the same ion exchange can occur in the presence of partially demineralized carious dentine. The ions released from both the cement and the tooth structure will combine to buffer the low pH until such time as it rises to a level where ion activity ceases. During this period of activity there will be both fluorine and strontium ions available to undertake apatitic activity in relation to areas in dentine where calcium ion levels are low with strontium ions replacing the missing calcium ions.

It is suggested that this occurs through a diffusion process driven partly by the concentration gradient, which exists between the glass-ionomer and the dentine with respect to these two elements. As both strontium and fluorine are apatite-forming elements, they react with the demineralized dentine. If the process is purely controlled by diffusion then one would expect to see the level of strontium and fluorine to be highest at the interface and lowest deep into the sound dentine.

These observations offer support to the original hypothesis. There remains a need to design an in vitro model to study the effects of other possible variables such as time of exposure and prior levels of demineralization.

Remineralization of demineralized dentine has been demonstrated by various glass-ionomers in vitro<sup>18,19,27</sup> and hypermineralized dentine has been reported using an in situ model.<sup>17</sup> The results from this study demonstrate that at least partial remineralization is possible in an in vivo model.

## 5. Conclusions

Findings from this clinical study support the laboratory evidence that glass-ionomer can contribute directly in the remineralization of carious dentine. However, there are two important requirements for this to happen; firstly the restoration has to provide a total seal to the external environment and secondly there is intimate contact between the glass-ionomer and the partly demineralized dentine.

The extent to which the remineralization from glass-ionomer re-establishes the physical property of carious dentine that had undergone remineralization could not be determined in this study.

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