Applying glass ionomer cement to MTA flow™ and biodentine™ and its effects on the interface layer

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Abstract. This study compared the interface layer formation between Glass Ionomer Cement (GIC) with Biodentine™ and between GIC with MTA Flow™. There were 10 samples in each group. Biodentine™ and MTA Flow™ were filled with GIC in plastic molds then incubated at a temperature of 37°C with 100% humidity for 24 hours. Samples were sectioned vertically with diamond discs and examined using a scanning electron microscope. The statistical analysis was performed using the Mann-Whitney Test. In group 1, 80% of the samples showed a score of 1 and 20% of the samples showed a score of 2. In group 2, 30% of the samples showed a score of 2 and 70% of the samples showed a score of 3. This clinical trial showed that the formation of interface layers in Biodentine™ and MTA Flow™ were significantly different.

1. Introduction
Bioactive materials are dental materials that are highly developed at this time. They can trigger the biological response of tissues, organisms or cells [1]. Some bioactive materials that are still constantly being developed are calcium silicate based materials such as Mineral Trioxide Aggregate (MTA), Biodentine™ and MTA Flow™. Biodentine™ and MTA Flow™ were developed to overcome the shortcomings of MTA. Both are produced in a smaller particle size and has been improved. Biodentine™ contains tricalcium silicate and dicalcium silicate and acts as a bioactive dentin substitute on the crown or the root of a tooth using Active Biosilicate Technology [2]. Biodentine™ has a short setting time of up to 12 minutes and has biological and physical properties which are claimed to be better than MTA. It has a pH of 11.7 and a 5µm particle size after setting time [2,3]. The mechanical properties of Biodentine™ are more stable and it needs less water than MTA due to its water reducing agent. It is also more resistant to acids [3].

MTA Flow™ is a calcium silicate based material with a smaller particle size and a more pure composition than conventional MTA. Both are available in a powder and a gel. The powder’s particle size is less than 10µm. The other physical characteristics of MTA Flow™ are the same as conventional MTA [4]. MTA Flow™ has a short setting time of up to 15 minutes. Stirring the powder into a liquid produces a mixture with a consistency that is not like sand, making it easier to apply. Biodentine™ and MTA Flow™ require a moist environment to facilitate the setting process properly, so wet cotton would be left in the cavity during a procedure and then covered with a temporary filling. However that technique was not effective because of the uncontrolled moisture which could lead to increased porosity and solubility, reducing the strength of the materials. The moist conditions required can basically be obtained through the structure of the tooth itself since 20% of the composition of
dentin is water. They can also be obtained by diffusion of moisture through cementum and accessories canals [5,6].

Various researches have been done to develop protective materials that will be in direct contact with calcium silicate based materials and do not interfere with the setting reaction or the characteristics of the materials. The contact between both materials can be observed through the interface layer. Evaluating the use of GIC as a protector that will be in direct contact with calcium silicate based materials has been studied through observing the interface layer.

According to Nandini et al., glass ionomer cement can be placed 45 minutes after the application of MTA without affecting the setting reaction and the properties of MTA [7]. From the initial setting through the final setting of GIC, polyacrylic acid would be highly soluble to water (loosely bound water) and cements that set almost perfectly would reabsorb water (tight bound water) [8,9]. According to Ashraf et al., the working time of GIC’s application to MTA did not affect the setting reaction of MTA, and both materials’ interfacial adaptation increased over time leading to a good biological seal [10]. Some studies showed that the interface layer between MTA and GIC had a tendency to not converge, possibly due to the withdrawal of water from MTA to GIC resulting in the disruption of MTA’s hydration and setting time. This also increased the porosity in the interface layer between MTA and GIC [11].

Research on the interface layer of MTA and GIC by Camilleri, said micro cracking occurred in the interface layer but that there was a bond strength between both marked by unreleased materials during the cutting process. High porosity, which is found in MTA, is suspected to have a role in increasing the bond with GIC [12]. The observation of ion migration on the interface layer of MTA and GIC was reported by Camilleri [12]. There was a migration of calcium, silicon and aluminium with an undetermined pattern due to the similar contents of the elements. There was no migration of bismuth but the migration of strontium in MTA was found to be as much as 200 μm. No changes were found on the deeper layers of MTA or GIC [12]. The impact of the ion exchange should be investigated further.

The setting reaction of GIC is known to form an acidic pH that ranges from 1.9–2. An acidic environment could affect calcium silicate based materials, thus reducing the strength and the hardness of MTA, as well as inhibit the setting time and increase the solubility leading to high leakage. Acidic conditions are also known to affect the quality of crystals produced by the hydration process [6]. Biodentine™ and MTA Flow™ are expected to be materials that can overcome the shortcomings of MTA. Both have a shorter setting time and smaller particle sizes than MTA. Research on the application of GIC as a protective material that will be in direct contact with both Biodentine™ and MTA Flow™ still does not exist. The short setting time of both materials can shorten the time of patient visits, but in its clinical application, the impact of GIC placed immediately on Biodentine™ and MTA Flow™ is still questionable. This effect is what will be observed through the interface layer of both materials.

2. Materials and Methods
A group of ten samples of BiodentineTM and a group of ten samples of MTA Flow™ (Ultradent Products Inc., USA) were applied plastic tubes with a diameter of 5mm, a height of 5mm and a thickness of 2mm. Biodentine™ (Septodont, UK) was mixed in accordance with the manufacturer’s instructions. The powder in the capsule was mixed with liquid and whipped using a machine for 30 seconds until it turned into a paste. MTA Flow™ was stirred into a thick consistency with the ratio of powder and gel at 2:2. GIC (GC Corporation, Japan) was also mixed in accordance with the manufacturer’s instructions until it became a solid paste. The BiodentineTM and MTA Flow™ mixtures were then immediately placed in each plastic tube. The materials in the plastic tubes were condensed using a plugger and wet cotton and then GIC was applied to them.

The specimens were stored in an incubator for 24 hours at a temperature of 37 °C with 100% humidity. They were then observed using a scanning electron microscope (SEM) with a magnification of 50x, 100x, 200x and 500x. Prior to observation, samples were vacuumed with high pressure nitrogen for 20 minutes and then coated with gold. The data obtained from the different formations
and pHs of the interface layers was analyzed using the Mann-Whitney Test with significance p < 0.05 in SPSS 20.0 software.

3. Results and Discussion

3.1 Results
The result of this study included an SEM examination on the interface layers which formed between GIC with Biodentine™ and GIC with MTA Flow™. The interface layers were analyzed using a scoring system which referred to the research of Asrianti [13], namely (1) blending, which evaluates the unification of the two materials; (2) no blending, where both materials did not fuse together; and (3) cracking, where there was a separation between the two materials creating empty spaces within them. The data was processed using SPSS 20.0 software. The interface layers in this study represented a categorical group so a normality test was not performed. The Chi Square was performed first because the expected score count of less than 5 exceeded 20% of the total samples. Then, the Mann-Whitney Test was performed.

Table 1. The distribution of the interface layer scores in each group

| Group   | N  | Score 1 | Score 2 | Score 3 | p-value |
|---------|----|---------|---------|---------|---------|
| Group 1 | 10 | 8(80%)  | 2(20%)  | 0(0%)   | 0.000   |
| Group 2 | 10 | 0(0%)   | 3(30%)  | 7(70%)  |

Group 1 : MTA Flow™
Group 2 : Biodentine™

In Table 1, group 1 showed that 80% of the samples have the score of 1 and 20% of the samples were scored 2. In group 2, 30% of samples showed a score of 2 and 70% of samples showed a score of 3. The result of the interface layer observation using an SEM in each group is showed in Figure 1.

![Figure 1](image1.png)

Figure 1. The result of the SEM with magnification of 200x based on scoring 1) blending, 2) no blending and 3) cracking

3.2 Discussion
Contact between GIC and Biodentine™ or MTA Flow™ could be seen through observation of the interface layer where, with a good marginal adaptation, it was expected to create a tight seal and close cracks that can cause inflammatory reactions and tissue degeneration. Additionally, the merging of both materials was not expected to cause an adverse reaction in either material.

Calcium silicate based materials are known to contain high pH. In an alkaline pH of 12.5, these materials have potential to be antibacterial and antifungal agents as well as create an alkaline environment to support the healing process. The pH values are known to perpetuate up to 28 days. That alkaline condition was expected to remain despite contact with other materials. Some studies reported the acidic environment could increase solubility and porosity, affect setting time, and reduce the strength and quality of the crystals produced from the hydration process of calcium silicate based materials.
Biodentine\textsuperscript{TM} and MTA Flow\textsuperscript{TM} are calcium silicate based bioactive materials with smaller particle sizes and short setting times of 12 minutes for Biodentine\textsuperscript{TM} and 15 minutes for MTA Flow\textsuperscript{TM}. In this research, the interface layer between MTA Flow\textsuperscript{TM} and GIC showed 80\% blending and 20\% no blending, while the interface layer between Biodentine\textsuperscript{TM} and GIC showed 30\% no blending and 70\% cracking. Based on the statistical analysis, there is a significant difference between the interface layers in both groups. Thus, the hypothesis of this research, which stated the interface layer of MTA Flow\textsuperscript{TM} and GIC would be equal to the interface layer of Biodentine\textsuperscript{TM} and GIC, was rejected. The research proved that the application of GIC immediately above Biodentine\textsuperscript{TM} and MTA Flow\textsuperscript{TM} produced significantly different interface layers with $p < 0.05$.

The occurrence of no blending and cracking on the interface layer between Biodentine\textsuperscript{TM} and GIC is most likely due to the setting process and low porosity of Biodentine\textsuperscript{TM}. In the setting process, Biodentine\textsuperscript{TM} forms an impermeable barrier to protect the materials. The porosity in Biodentine\textsuperscript{TM} is low because of its water reducing agent. Thus, the reduced water needs increase the flow of materials and reduce porosity. These conditions did not allow deposition and crystal adhesion [3,14]. The occurrence of blending between MTA Flow\textsuperscript{TM} and GIC is related to the hydration process where, according to Damamaschke \textit{et al.}, the mechanics of MTA Flow\textsuperscript{TM} ensure that in 24 hours, the hydration of tricalcium alumina forms a gel hydrate colloidal as described in the reaction below [15].

$$3\text{CaO}.\text{Al}_2\text{O}_3 + 6\text{H}_2\text{O} \rightarrow 3\text{CaO}.\text{Al}_2\text{O}_3 + 6\text{H}_2\text{O}$$

GIC had three stages in the setting reaction: dissolution, the precipitation of salt hardening or gelatin stage, and the rehydration of salts. In the dissolution stage, H$^+$ ions from polyacrylic acid were released to attack the surface of the glass. Metal ions were released from the glass followed by H$^+$ ions, the metal reacted to the polyacrylic acid, and then the surface of the glass particles formed the layer of silica gel. The initial setting reaction occurred rapidly in 4-10 minutes from the start of stirring the cements due to the crossing movement of Ca. Maturation occurred after for more than 24 hours when the F and PO$_4$ ions formed salt. Sodium contributed in the formation of orthosilicic acid on the surface particles as the pH increased. In the final setting, the progressive hydration of the salt matrix initiated the physical form of the material's refinement [8].

There were two reactions on the interface layer of MTA Flow\textsuperscript{TM} and GIC. COO$^-$ chains on polyacrylic acid interacted with Ca on MTA Flow\textsuperscript{TM} to form calcium salt, and silicate hydrate gel on MTA Flow\textsuperscript{TM} condensed with silicate hydrate gel on GIC to form by-product. The existence of no blending between MTA Flow\textsuperscript{TM} and GIC could have occurred because of a 30 minute period nearing the initial setting of MTA Flow\textsuperscript{TM} (45 minutes). At this stage, according to Lee [16], MTA Flow\textsuperscript{TM} starts to leave a solvent and the anhydrous materials that are still in the initial stages of crystallization remained bound to each other (interlocking mass). Some studies showed that MTA Flow\textsuperscript{TM} and GIC have a tendency not to converge, possibly due to the withdrawal of water from MTA Flow\textsuperscript{TM} to GIC resulting in the disruption of MTA Flow\textsuperscript{TM}’s hydration and setting time and also increasing porosity in the interface area between MTA Flow\textsuperscript{TM} and GIC.

\textbf{4. Conclusion}

In conclusion, this research showed the difference between the interface layers of MTA Flow\textsuperscript{TM} with GIC and Biodentine\textsuperscript{TM} with GIC in which MTA application created a better seal for restoration. Nonetheless, it is necessary to do further studies on the physical properties of Biodentine\textsuperscript{TM} and MTA Flow\textsuperscript{TM}. In addition, there should be advanced research done with variations in the materials used and the time range of GIC application.

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