CHAPTER 3

PRELIMINARY TESTING

3.1. Introduction & Literature review

There are four types of fluoride releasing restorative materials as described in Chapter 2. The first group is conventional glass ionomer cement. The most advantages of this type are fluoride release over a long period and adhesion to tooth structure due to chemical bonding with enamel and dentin. The mechanism of bonding is suggested by many investigators (Wilson and Mesley, 1974; McLean and Wilson, 1974a; Wilson, Prosser and Powis, 1983) that there is intermediate interfacing layer between cement and tooth surface. Polyacrylate enters the molecular surface of hydroxyapatite displacing and replacing surface phosphate. Calcium and phosphate ions are part of complex. Series of ionic exchanges and the intermediate layer of calcium, aluminum phosphate and polyacrylates would form at the interface between cement and apatite. There are also some disadvantages of this material for instance, the cement still requires mixing procedure. This type of cement is also sensitive to the balance of water during placement and setting because water contamination can cause dissolution of the matrix forming and deteriorate its strength. When compared to composite resin, it has lower strength and less esthetics (Akpata, 1996; Shen , 1996).

The second group of fluoride releasing restorative materials is called resin-modified glass ionomer cement which was developed by Mitra in 1991. The resin-modified glass ionomer cement combines the fluoride releasing property of a conventional glass ionomer cement and some favorable physical properties of resin composite. The metal ions released from glass particles react with polyacrylic acid while HEMA which is part of resin cures concurrently. This reaction forms a hardened mixture and at the same time solves the disadvantage of conventional glass ionomer cement especially water balance. Water sensitivity could be reduced by incorporation

of photopolymerization reaction and rapid setting also provides better color stability. However, a mixing procedure of the cement is required.

The third group is called polyacid modified resin composite or compomer which offers easy usage. The polyacid modified resin composite is combinations of glass ionomer glass powder or prereacted glass ionomer and a polymerizable acidified monomer (Barnes et.al, 1995; Swift and Vann 1995; Mcclean and Wilson 1994). This material was developed to improve the physical properties and clinical handling of glass ionomer cement. It is similar to resin composite in its chemical structure and reaction, ion-leachable glass particles and polymerizable acidic monomer. The importance of mixable polyacid modified resin composite has significantly decreased. The major advantages of this material are excellent handling characteristics, moderate mechanical properties and ability to release fluoride (Christensen, 1997). The polyacid modified resin composite releases fluoride less than resin modified glass ionomer cement because initially there is no water in the compression. Water must diffuse from environment through limited hydrophilic matrix to liberate ionic components from glass filler (Meyer et al.1998).

The fourth group is resin composite that can release fluoride by adding the polymer matrix-forming material with the soluble fluoride. The poor stability and discoloration properties still prevent their popular uses as a restorative material. This group requires more development (Combe, Burke and Douglas 1999).

Many investigators compared the fluoride release from these types of material. For instance, Bertacchini et al. (1999) used three groups of fluoride releasing materials to evaluate fluoride release at 1 h, 24 h, 7 d, 15 d, 30 d, 60 d and 90 d. They found that time factor and type of material had significant influence in the result. Fluoride release was significantly less with polyacid modified resin composite than glass ionomer cement. These materials have bonding and margin-sealing abilities equivalent to resin composite (Yap, Lim and Neo,1995). Then the thorough investigators examined the caries inhibitive of these kind of materials. Dionysopoulos et al. (1998a and b) examined

artificial caries after filled with tooth colored restorative materials. They found that the use of glass ionomer cement and/or polyacid modified resin composite may prevent both primary and secondary caries around restoration in the surface enamel adjacent to the restoration. Forss and Seppa (1990), Hicks (1986a) and Tantjbirojn et al.(1997) also found some inhibitive effect against enamel caries formation.

As many investigators convinced of having fluoride on glass ionomer cement and polyacid modified resin composite depending on type and company. Fluoride released into the environment was also found to have inhibitive effect on secondary caries in vivo and in vitro. Elusion of free fluoride has demonstrated over 15 years ago (Forsten et al., 1976). However, it is believed that the progression of the demineralization was attacked in the lower pH environment. The setting reaction of glass ionomer cement was complex and involved with many ions. When the outer layer of glass particles are decomposed, Ca^{2+}, Al^{3+} and F^{-} ions are released. The cations migrate into the aqueous phase, and cross-link the polyalkenoate chains, causing hardening of the material. Even the resin modified glass ionomer cement which contains HEMA or Bis-GMA together with a photoinitiator, the material still has the acid-base glass ionomer reaction (Combe, Burke and Douglas 1999). The reaction of the ions could possibly make the pH in the artificial caries change. The polyacid modified resin composite which is formulated as anhydrous one component material produces ionic bond to the inorganic part of the tooth by acid-base reaction. The acid-base reaction takes place between strontium fluorosilicate glass filler and the polycarboxylate groups. When the setting process of these materials starts, the loose ions could possibly alter pH adjacent to the surface and inhibitive effect occurred.

The objective of this preliminary test was carried out to quantify amount of fluoride released from the studied materials as to confirm the fluoride releasing properties of the materials.

3.2. Amount of fluoride released from tooth-colored restorative materials

3.2.1 Materials and Methods

Equipment

1. Plastic tube (size 10x10x40 mm)

2. Silicone mold (size 3x3x1mm)

3. Ion analyzer model EA 940 (ORION Research Incorporated, Boston, USA) and combination fluoride electrode (Select Bioscience Ltd, Sudbury, UK)

4. Incubator (Memmert, Schwabuch,Germany)

Materials

1. Resin composite (Clearfil liner bond 2, Kuraray, Osaka, Japan)

2. Resin modified glass ionomer (Fuji II LC, GC company, Tokyo, Japan)

3. Polyacid modified resin composite or compomer (F2000, 3M company, Minnesota, USA)

4. Sterile deionized water

Methods

Ten specimens of each materials were prepared following the manufacture's instruction into a size of 3x3x1 mm (Figure 3.1). Then all the specimens were put separately into tube contained deionized water 10 cc. for 24 hours, 7 days and 30 days at 37° C. The fluoride release was measured by a specific fluoride electrode (Orion ion analyzer) (Figure 3.2) at each duration. The amount of fluoride release from those materials

versus time was analyzed using one way ANOVA and Paired-Samples T test was compared between materials ($p<0.05$).

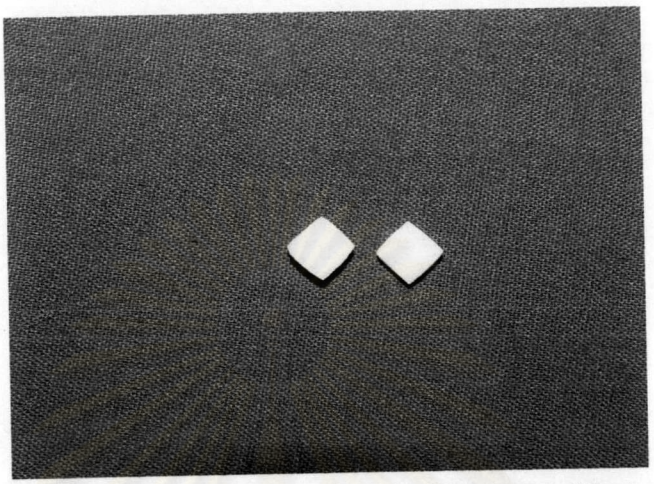

Figure 3.1. Prepared specimens (size 3x3x1 mm) of Resin modified glass ionomer cement (Fuji II LC) and Polyacid modified resin composite (F 2000)

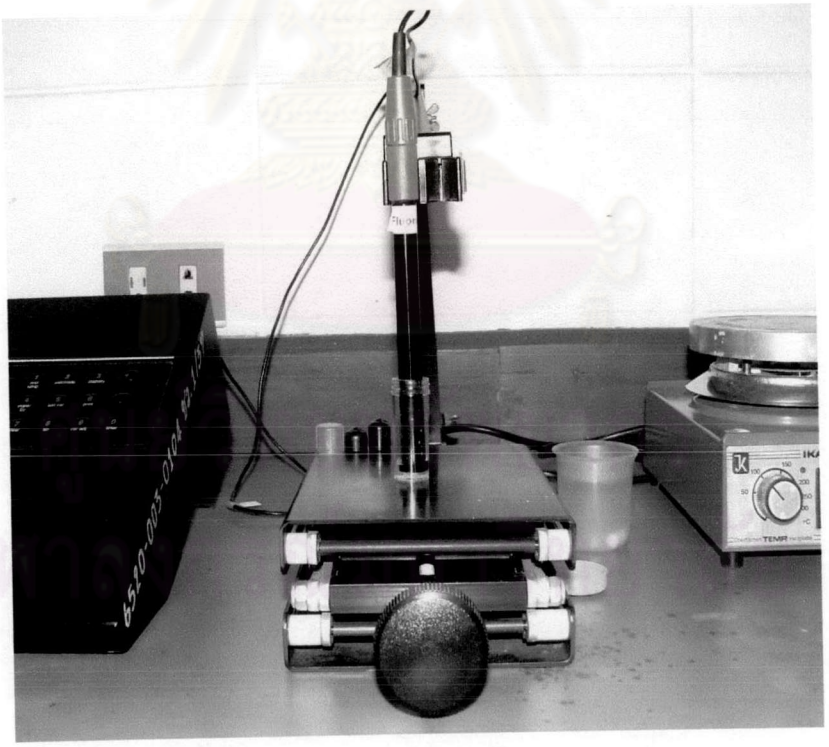

Figure 3.2. Fluoride electrode was positioned vertically to the specimen which was placed at the bottom of the tube

3.2.2. Results

Resin modified glass ionomer cement (Fuji II LC) showed substantial amount of fluoride release in 24 hours, 7 days and 30 days (6.74 ± 1.15, 10.74 ± 1.98, 21.45 ± 3.68 ppm respectively) (Figure 3.3).

Polyacid modified resin composite (F2000) also released fluoride in 24 hours, 7 days and 30 days (4.99 ± 0.90, 10.49 ± 1.7, 20.18 ± 2.87 ppm respectively). Resin composite (Clearfil APX) as negative control material did not show any fluoride release as shown in Figure 3.3.

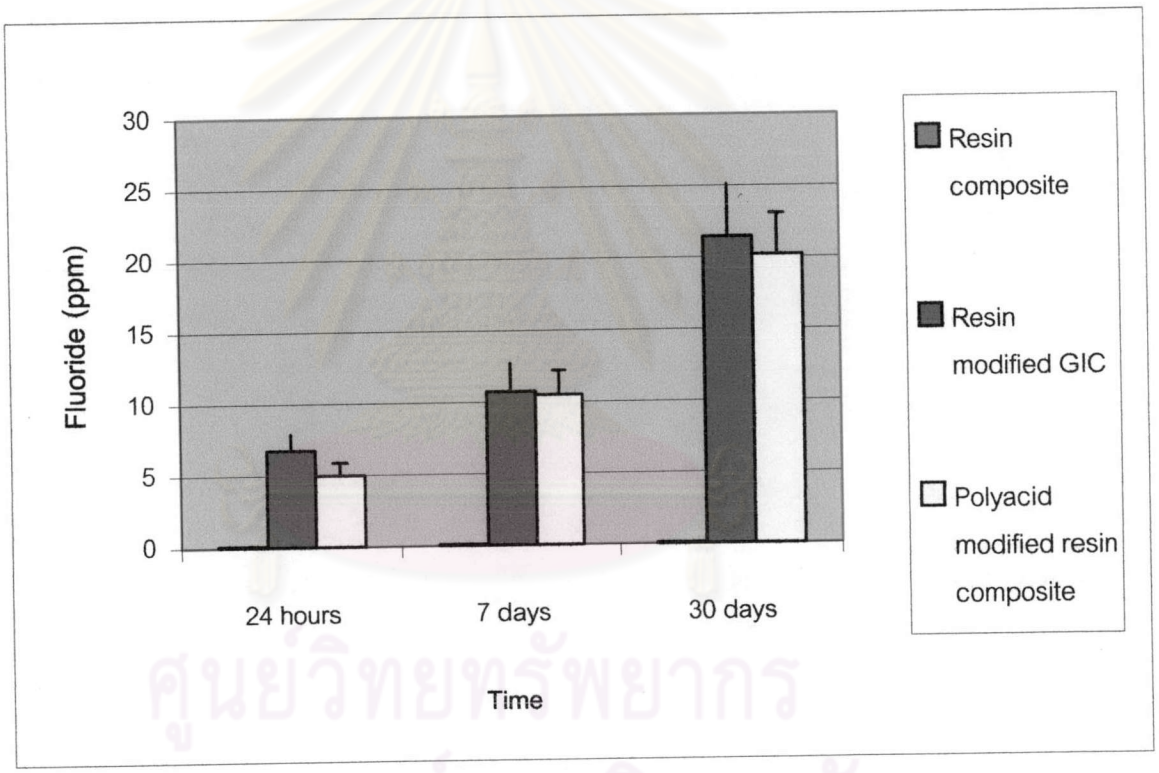

Figure 3.3. Fluoride release of the test materials versus time

The data of fluoride release from the resin modified glass ionomer cement and polyacid modified resin composite versus time was analyzed by Paired-Samples T test. It was found that there was significant difference between amount of fluoride versus time in both materials ($p < 0.05$). And one way ANOVA was used to compare each group versus time. There was significant difference of fluoride between the two groups in 24

hours at $p<0.05$. But there was not any significance difference among both groups for 7 days and 30 days.

3.2.3. Discussion

There have been many attempts to incorporate fluoride-releasing agents to the restorative materials in order to reduce caries. Since silicate cements dissolved in oral fluids and released fluoride, glass ionomer cement was developed and proved to provide cariostatic properties (Combe, Burke and Douglas 1999). A lot of studies explained the fluoride ions released from filling materials play the important role of inhibiting secondary caries (Brown, Gregory and Chow, 1977; Attin et al., 1999; Millar,Abiden and Nicholson, 1998; Zimmerman,Rawls and Querens, 1984. Release of fluoride from restorative materials may be deemed to get a beneficial in cariostatic effect, especially fluoride is in the solution around and adjacent to the restoration. This experiment was designed to investigate the fluoride release from the studied restorative materials. In general fluoride can be found releasing in some solution such as deionized water and acid challenge solution (Featherstone et al.,1990). Many investigators reported the amount of fluoride released from glass ionomer cement and polyacid modified resin composite (Attin et.al. 1999; Bala et.al,1997; Bertacchini et.al.,1999; de Araujo et.al.,1996; Dionysopouls et.al.,1998 a and b; Eichmiller and Marjenhoff,1998; Forsten, 1991). The amount of fluoride release varied depending on many factors such as types of material, solution and soaking period (Arends et.al.1988).

The present study confirmed the fluoride release from the experimental fluoride releasing materials when soaking in the deionized water. It was found that the amounts of fluoride released from glass ionomer cement were 6.74 ± 1.15, 10.74 ± 1.98, and 21.45 ± 3.68 ppm for 24 hr, 7 days and 30 days respectively. And polyacid modified resin composite were 4.99 ± 0.90, 10.49 ± 1.70 and 20.18 ± 2.87 ppm for 24 hr, 7 days and 30 days respectively. As a control material, the resin composite specimen did not release any fluoride. The amount of fluoride release from resin modified glass ionomer cement and polyacid modified resin composite investigated by other studies was widely

recognized, with the fluoride release being dependent on factors such as time of mixing, powder to liquid ratio, method of mixing and the material itself (Combe, Burke and Douglas 1999). Although there were great differences in amount of fluoride release among many studies but the pattern was similar. The greatest amount of fluoride release occurred in 24 hours especially for the glass ionomer cement (Karentakis et al. 2000). The cumulative of fluoride release of resin modified glass ionomer cement in 30 days was in the wide range from 15 up to 200 ppm in 7 days (Itota et al. 1999; Karentakis et al. 2000; Combe, Burke and Douglas 1999). However, the amount of fluoride release must be carefully interpreted since each experiment used various sizes of specimens.

The specimen size of 3x3x1 mm was designed so as to simulate the cavity size prepared for the next experiment. The amount of fluoride released from the similar area of exposed specimen surface could be estimated. Normally, the resin modified glass ionomer cement released less fluoride than glass ionomer cement. A thin mix (low viscosity) of resin modified glass ionomer cement also resulted in higher fluoride release than a thick mix (Forsten, 1995). Polyacid modified resin composites which has an advantage on ease of uses showed less amount of fluoride release compared to glass ionomer cement. This was also confirmed in the present study (Figure 3.3). However, Grobler et. al. (1998) reported that even relatively low amount of fluoride was released, it may result in significant concentration of fluoride at the gap between restored material and tooth margin.