

Comparison Of The Solubility Of Luting Cements Immersed In Artificial Saliva - An Invitro Study

Abstract

Background and Objectives: Luting cements are susceptible to attack by moisture during the initial setting period that can result in an increased solubility. The purpose of this study is to observe the effect of early water contact on solubility of zinc phosphate cement, glass-ionomer cement, resin cement and resin-modified glass-ionomer cement when immersed in artificial saliva.

Material and Methods: Four commercial luting cements were used (Fuji I, De Trey Zinc, Rely X ARC, Fuji CEM). For each material, 5 resin cement holders were made with 2 circular cavities, (diameter of 5mm and depth of 2mm). One minute after start of mixing, the specimens are placed in a humidifier at 37°C and 100% relative humidity. After 2 minutes, 5 minutes and 8 minutes of storage time, they were immersed in 50 ml of artificial saliva in a glass weighing bottle where they were stored for 3 hours at 37° C. Next, the specimens are removed and water was evaporated from the weighing bottle at 130° C for 2 hours. The difference between the final and initial weight of the bottle was taken as amount of solubility. Data were analysed by analysis of variance and Duncan's test.

Results: The lowest cement loss was for Rely X ARC at 9 minutes immersion time (1.475 mg/cm²). The greatest loss was for Fuji CEM after 3 minutes immersion time (14.758 mg/cm²). The solubility of cements decreased by 33% for Fuji I, 33% for De Trey Zinc, 50% for Rely X ARC, and 17% for Fuji CEM at various time intervals between the various groups. The percentage reduction in solubility at 6 minutes immersion time was 45% for Fuji I, 40% for De Trey Zinc, 41.7% for Rely X ARC, and 28% for Fuji CEM cement. The degree of solubility of these cements from 3 minutes to 9 minutes from start of mixing was 71.2% for Fuji I, followed by 71.1% Rely X ARC, 59.8% for De Trey Zinc, and 33.3% for Fuji CEM.

Interpretation and Conclusion: Increasing the time from start of mixing until immersion in artificial saliva from 3 to 9 minutes resulted in a marked decrease in loss of substance from the surface of all 4 cements. Resin cements were less sensitive to early water contamination.

Key Words

Solubility, Luting cements, artificial saliva.

Introduction

'Luting' is a word that is often used to describe the use of a moldable substance to seal a space or to cement two components together. They can also be described as the use of a flowable substance to seal joints and cement two surfaces together. Traditionally, the term 'Cements' in dentistry has been applied to a powder liquid material which when mixed to a creamy consistency sets to a hard mass which is used clinically to join the restoration to the teeth. Cements have been used in restorative dentistry for luting purposes for a long period of time. However, since the properties of various cements differ from each other the choice of cement depends on a larger degree to the functional and biological demands of the particular clinical situation. If optimal performance is to be attained, the physical and biological properties along with working characteristics like manipulation time, working time and ease of removal of excess

flash must be considered when selecting cement for a specific purpose. Solubility is one of the most important essential factors in assessing the quality of luting agents in restorative dentistry. Generally the luting cements require an extended setting time and a working time which makes them more susceptible to intraoral saliva contamination^[1]. Early cement exposure to saliva during setting of cement not only alters most of its properties, especially solubility and resulting in microleakage and affecting durability of the restoration. Luting cements have been known to undergo early dissolution when exposed to moisture immediately after initial hardening. Prevention of moisture contamination is therefore necessary^[2].

However, even with utmost care, sometimes it is not possible to have a moisture free environment when rubber dam is not applied. During the recent years, several

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thickness also play an important role^[4, 5]. Third, the dimensional changes occurring in the cement during setting should be minimized. Sources include, gain or loss of water and differences in the coefficients of thermal expansion among the tooth, the prosthesis, and the cement^[6, 7]. It is, therefore, important to isolate the cement immediately after removal of the excess^[4].

Fixed prosthesis can debond because of biologic or physical reasons or a combination of the two. Recurrent caries results from a biologic origin^[8]. Disintegration of the cements can result from fracture or erosion of the cement. In the oral environment luting cements are immersed in an aqueous solution^[6]. In this environment, the cement layer near the margin can dissolve and erode leaving a space. This space can be susceptible to plaque accumulation and recurrent caries; therefore the margin should be protected with a coating to allow continuous setting of the cement^[9].

A study was therefore undertaken to compare the solubility of some of the conventional luting cements during their initial contact with water or moisture. In this study, solubility of four types of luting cements during initial moisture contamination and effect of time lapse between mixing and moisture contamination on solubility of these four luting cements is being compared.

Material And Methods

The conventional specification test for assessing the solubility and disintegration of dental cements was developed by Paffenbarger et al^[18, 19]. It consists of immersing 2 thin flat disks of cement in water 1 hour after preparation. Twenty-four hours later, the disks are removed from the water and the water evaporated to dryness. The amount of material lost is determined from the weight of the residue and is usually expressed as a percentage of the original weight of the cement. This method consists of the basis of solubility evaluations because the organic or polymeric materials that might be collected would not have been degraded or volatilized during the drying steps. The conventional method was originally intended for the quality control of commercial products. However, the method has often been altered by research workers to evaluate new materials and to compare different types of dental cements.^[15, 16, 17] Um and Øilo^[15] altered the conventional method to evaluate the solubility of luting cements at

the early stages after mixing. Instead of thin flat disks, they used resin molds as cement holders and immersed cements in distilled water at the early stages of mixing before the final set of the cement. The same method, except immersion times and immersion in artificial saliva^[14] was used in this study to record the early solubility of luting cements.

To quantify loss of substance from 4 different luting cements (**Table 1**), 2 circular cavities (diameter of 5 mm, depth of 2mm) were made in resin block (**fig 2**). The cements were all mixed on a glass slab at room temperature by the stiff blade. The powder/liquid ratios used were in accordance with the manufacturers' recommendations. If the powder/liquid ratios were not given in the instructions, they were calculated from the proportioning system given by the manufacturer by weighing scoops of powder or drops of liquid. The powder/liquid ratios of each cement are given in **Table 1**. In each case, the powder was weighed on a digital balance (**fig 3**) that had a measuring accuracy of 0.1 mg and the liquid was dispensed from a 1 mm tuberculin syringe calibrated to the nearest 0.1mL. All cements were mixed within 30 seconds and placed in small resin cups in 30 seconds. The surface was flattened all the excess cement was removed with a spatula and a dry cotton pellet. One minute after the start of mixing, the specimens were placed in a humidifier (**fig 4**) at 37°C and 100% relative humidity. After 2, 5, or 8 minutes of storage time, specimens were removed from the humidifier and immersed in 50 ml of artificial saliva in a glass weighing bottle with the exposed surface area pointed upward.

The mass of the empty bottle had been previously established by using a digital balance that had a measuring accuracy of 0.1mg. Before weighing, the bottle was carefully rinsed, dried at 130°C for 2 hours

in hot air oven (**fig 5**), and cooled in a desiccation for 20 minutes. Bottles were stored for 3 hours at 37° C and thereafter, the cement holders were removed from the water and the bottles were stored at 130° C for 2 hours to allow evaporation of water. Bottles were then cooled in a desiccators (**fig 6**) and weighed as previously described. The amount of substance dissolved was determined by subtracting the first established weight of the bottle from the second. The time schedule for the total procedure is illustrated in Flow chart. Five specimen of each type of cement were used at each immersion stage. The mean weight loss per square centimeter was calculated. The exposed surface area of each specimen was calculated ($r = 2.5 \text{ mm}$) and the exposed surface area was doubled as each cement holder had 2 cavities. Data were analyzed by 2-way analysis of variance ($p < 0.01$). Duncan's multiple range test analysis was also used to distinguish statistically significant groups.

Results

The recorded mean values and the standard deviations for each luting cements at each immersion time are presented in **table 2**. The lowest cement loss was recorded for resin cement after 9 minutes immersion time (1.47 mg/cm²). The greatest loss was for resin modified glass ionomer cement after 3 minutes immersion time (14.75 mg/cm²). For all of the observed luting cements, resin cement showed the lowest mean loss of substance at all immersion times. However, the loss of resin cement was significantly different from all cement at 3 minutes immersion time. Increasing the time from the start of mixing until immersion in artificial saliva from 3 to 9 minutes resulted in marked decrease in loss of substance from the surface of all 4 cements (**Graph 1**.) This inter-comparison result was obtained from the "BONFERRONI". The solubility of the cement decreased by 33% for Fuji I, 33% for

Flow chart 1. Time schedule for experimental procedure.

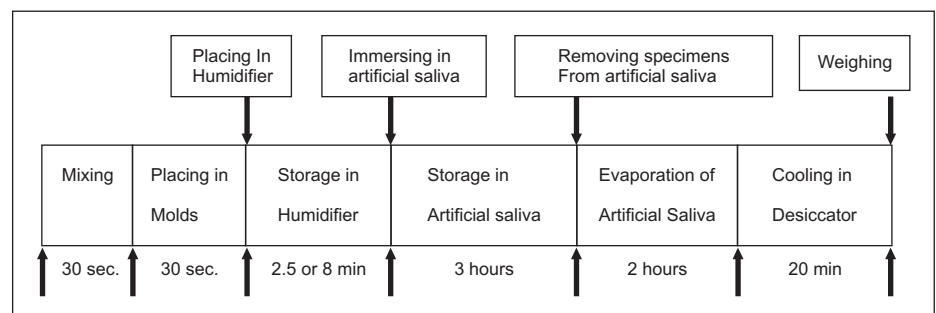


Table 1.

Product Name	Manufacturer	Composition	Scoops
			P:l/two paste system
FUJI I	GC	Powder:- Calcium Fluoroalumino Silicate Glass Liquid:- Conc.aqueous Solution Of Polyacrylic Acid	1.8:1 g
DeTreyZinc Crown & Bridge Fixodont Plus	DENTSPLY	Powder:- 10% Mgo, 90% Zno Liquid:- 67% Phosphoric Acid 33% Water With Aluminium And	2.8:1 g
Rely X ARC	3M ESPE	Base:- Strotium Alumino Fluoro Silicate Glass Aerosol Initiator Catalyst:- Macromonomer Aminopenta	1:01
FUJI CEM	GC	Base:- Fluoroalumino Silicate Glass, Poly Hema (Resin) Catalyst:- Aqueous Solution Of Modified Polyalknoic Acid.	

De Trey Zinc, 50% for Rely X ARC, and 17% for Fuji CEM at various timer intervals between the various groups. The pair wise comparison was done using the "ANALYSIS TEST". For all the four groups there is a significant reduction in solubility at 3 minutes versus 6 minutes and 3 minutes versus 9 minutes and 6 minutes versus 9 minutes. When the specimens were immersed in artificial saliva 6 minutes after

mixing, the difference in solubility between 3 and 6 minutes was most marked for RC cement, which was around 50%.The percentage reduction in solubility at 5 minutes immersion time to be 45% for GIC, 40% for ZnPO4, 41.7% for RC, and 28% for RMGIC cement. The degree of solubility of these cements from 2 minutes to 8 minutes from start of mixing was 71.2% for GIC followed by RC by 71.1%, 59.8% for

Graph 1. graphic representation of solubility of luting cements in relation to immersion time.

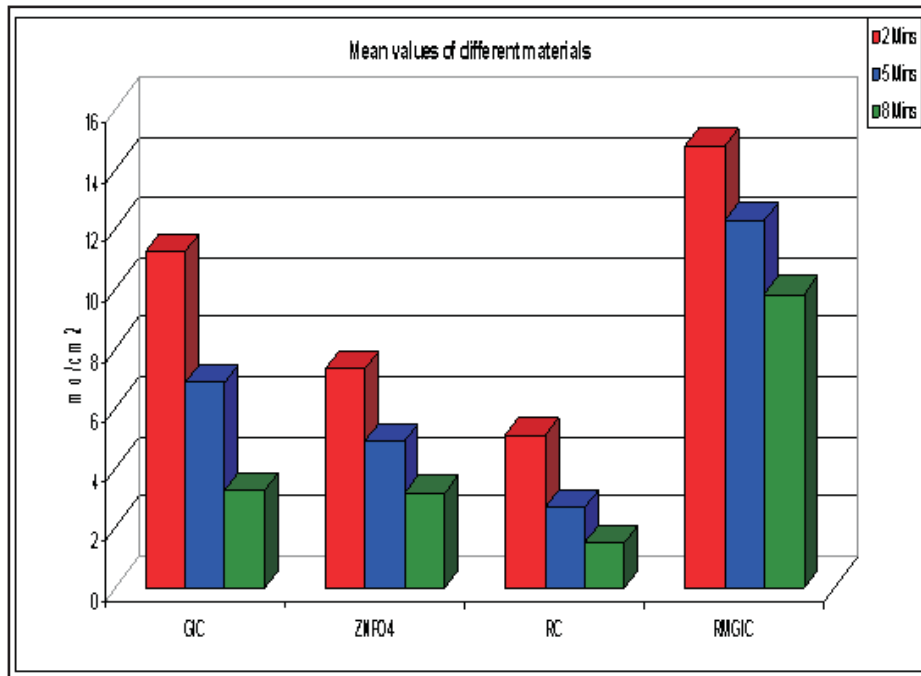


Table 2. Mean and standard deviations of loss of substance (mg/cm²) for 4 luting cements immersed in artificial saliva at different intervals.

Cement	3min		6min		9min	
	Mean	SD	Mean	SD	Mean	SD
Fuji I	11.2	0.3	6.8	0.6	3.2	0.3
De Trey Zinc	7.3	0.7	4.8	0.4	3.1	0.2
Rely X ARC	5	0.6	2.6	0.5	1.4	0.3
Fuji CEM1	14.7	0.4	12.2	0.3	9.8	0.2

ZnPO4, and 33.3% for RMGIC.

Discussion

From the results obtained, it can be seen that the resin cement, gave the least solubility at all periods of time. This can be explained by the fact that principle cement is more of resin cement and less of glass ionomer cement. Also it has a dual cure capability and the margins can be light cured. The next ideal cement in terms of reduced solubility was the century old zinc phosphate cement. As far as its solubility was concerned, it was found less soluble than glass ionomer cement in our vitro study. Studies [2] have shown the importance of protecting the glass ionomer cements immediately after cementation, as they have been shown to disintegrate rapidly in the presence of moisture. Zinc phosphate showed a lower solubility than the glass ionomer cement, therefore it is necessary that adequate protection is given against water after cementation. The reason for the dissolution of glass ionomer cement in water has been attributed to two factors: firstly [2], they contain sodium that forms water soluble salts with the matrix forming anions. Secondly [2], free calcium and aluminum ions that are present in the fresh cement can be removed by chemical reactions. In addition, aluminum ions react rather slowly with the matrix forming anions and before they are bound, is vulnerable to early water leaching. As the setting progresses, the cement becomes more and more solid, preventing water penetration and reducing the outward transportation of cations. Finally⁴, the resin modified glass ionomer cement showed the highest solubility which was more than glass ionomer cements. Because they contain a resin HEMA (hydroxy ethylmethacrylate) which is hydrophilic in nature, there is an increased water sorption and subsequent plasticity and hygroscopic expansion. This behavior is

analogous to a synthetic hydrogel. All the four luting agents show a decreased amount of solubility when extending the time between the start of mixing and immersion in artificial saliva. A significant decrease was observed during a 2-8 minute period after commencement of mixing and the loss of substance was greatly reduced at 8 minutes after the commencement of mixing. As regard, the percentage reduction of solubility from 2-8 minutes, we find that the resin modified glass ionomer cement showed the least reduction in solubility which can be explained as mentioned earlier due to the presence of HEMA resin. Although invitro tests have limited clinical significance because it does not give an indication of stability of the set cement in oral environment, they are important for screening the quality of different cement types.

Conclusion

From the results of this study the following conclusions were obtained. The resin cement showed the least solubility to water at various time intervals of immersion. Zinc phosphate and glass ionomer cement followed this to various time intervals of immersion. The resin modified glass ionomer cement showed the highest solubility. The glass ionomer cement showed a greater percentage of reduction in solubility as the time interval of immersion in water increased when compared to other luting cements. This study shows that the resin modified glass ionomer cement and glass ionomer cements requires protection from moisture in the early period after mixing (8 minutes).

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