

Evaluating the Solubility of Various Dental Cements In Artificial Saliva in Different Mediums at different Time Intervals: In-Vitro Study

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Abstract

Background: Different varieties of cements have been used in dentistry though many years to retain the restoration in a fixed position within the mouth. Cement solubility is considered to be a primary cause for the failure of cast restoration. The rate of luting cement dissolution is directly related to the degree of marginal opening. Larger the marginal gap, more the exposure of dental cement to oral environment and more rapid is the rate of cement dissolution Solubility contributes to loss of marginal integrity, surface properties and decreased aesthetics, resulting in restoration failure.

Objective: To evaluate the solubility of various commercially available dental cements in artificial saliva at different pH and time.

Material and methods: One forty-four samples of zinc polycarboxylate, zinc phosphate, glass ionomer cements and resin cement were prepared according to manufacturer's instructions. After setting, they were weighed and each specimen were immersed in artificial saliva at three different pH (3.3, 6.7 and 12.3) for 24 hour, 48 hours and 7days. After specific time interval, samples were placed in desiccator and again weighed. Solubility of the cements were calculated by weight analyses.

Results and Observation: The results of the study showed that the zinc phosphate cement has the highest solubility in acidic, basic as well as neutral environment followed by Zinc Polycarboxylate and glass ionomer cement. The least soluble cement is resin cement.

Conclusion: Within the limit of this study, Zinc Phosphate showed highest solubility in acidic medium followed by basic medium and then in neutral environment. Resin cement is the least soluble. Further studies are warranted to better explore the results of this study.

Keyword: Dental Cements, Solubility, Marginal Integrity, Restoration Failure.

Introduction

Dental cements are an integral part of restorative dentistry. Although these are used in very small quantity but these are important materials which are used as luting agents to retain restorations or appliances in a fixed position within the mouth. The term luting is derived from the Latin word Lutum which means clay or mud. Cement is a generic term for a joining medium provided adhesion and/or micromechanical locking between the two surfaces to be

connected (Simon JF& de Rijk WG 2006). It flows and locks the restoration in place between the irregularities of tooth and crown or bridge.

Solubility is an important factor that determines the longevity of crowns, bridges and posts. Once the dissolution of cement occurs, loosening of the fixed prostheses will follow. Because of the property of solubility, luting cement in general, have been described as the "weak link" when restoring teeth with cast restorations. Different methods are used to calculate the solubility such as weight analysis, chemical analysis of the ion concentration in the erosion liquid, profilometry, stereoscopic measurements.

In most of the the historical studies, the solubility of different provisional and permanent dental cements such as Zinc Phosphate, Zinc polycarboxylate, Glass ionomer, Zinc oxide eugenol, and Calcium hydroxide were evaluated. But there is a paradigm shift from Zinc phosphate to Resin cement. This paucity of information has currently provoked a need to make the comparison between resin cement with conventional dental cements. Moreover, in the previous studies the solubility was assessed, either in an acidic environment or in distilled water, studies have not considered the effects of basic environment on the solubility of the cements, so in the present study it was planned to evaluate the solubility of different dental cements in acidic, neutral and basic mediums.

Materials and Method

Materials

Dental cements: Zinc Phosphate cement (De Trey Zinc, Dentsply, Germany), Zinc Polycarboxylate cement (Poly F, Dentsply, Germany) , Glass ionomer cement (Gold Label, GC Dental Product Corp, Japan) , Resin cement (Maxcem Elite, Kerr)

Medium: Distilled water (Sankalp distilled water) and Artificial saliva (freshly prepared), Sodium Hydroxide and Hydrochloric acid.

Armamentarium

Customized stainless-steel die, Desiccator and Oven, Digital pH meter (Figure 1), Digital weighing Balance (Figure 2),

Study Method

A total of 144 specimens were prepared. Further two types of storage mediums were used for conditioning of the specimens before testing. The specimens were tested in three-time intervals (after 24 hours, after 48 hours and after 7 days) and at each time interval specimen were tested at three different pH values (3.3, 6.7, and 12.3). Grouping and sub categories of the groups have been shown in the table below.

Table 1: Categorization of Groups and Subgroups

	GROUP A (Artificial Saliva)			GROUP B (Distilled water)
SUBGROUP I	3.3	6.7	12.3	
(ZINC PHOSPHATE)	I A	II A	IIIA	IV A
(Hours)	24 I A₁	24 II A₁	24 III A₁	24 IV A₁
(Hours)	48 I A₂	48 II A₂	48 III A₂	48 IV A₂
(Days)	7 I A₃	7 II A₃	7 III A₃	7 IV A₃
SUBGROUP II				
(Zinc Polycarboxylate)	I B	II B	IIIB	IV B
(Hours)	24 I B₁	24 II B₁	24 IIIB₁	24 IV B₁

(Hours)	48 I B ₂	48 II B ₂	48 IIIB ₂	48 IV B ₂
(Days)	7 I B ₃	7 II B ₃	7 III B ₃	7 IV B ₃
SUBGROUP III				
(Glass ionomer)	IC	IIC	IIIC	IV C
(Hours)	24 I C ₁	24 II C ₁	24 III C ₁	24 IV C ₁
(Hours)	48 I C ₂	48 II C ₂	48 III C ₂	48 IV C ₂
(Days)	7 I C ₃	7 II C ₃	7 III C ₃	7 IV C ₃
SUBGROUP IV				
(Resin)	ID	IID	IIID	IV D
(Hours)	24 I D ₁	24 II D ₁	24 III D ₁	24 IV D ₁
(Hours)	48 I D ₂	48 II D ₂	48 III D ₂	48 IV D ₂
(Days)	7 I D ₃	7 II D ₃	7 III D ₃	7 IV D ₃

Specimen Preparation

A customized stainless-steel die (8 cavities with dimensions of 20mm diameter and 1.5mm thickness) was prepared for fabrication of the specimens. Die was lubricated with a layer of petroleum jelly for ease of separation of the specimens from it.

The Zinc Phosphate Cement Subgroup I, Zinc Polycarboxylate Subgroup II and Glass Ionomer cement as Subgroup III are supplied in powder and liquid form. The powder and liquid were mixed according to the manufacturer's instructions and placed on the mould concavities of die. They are covered using glass slab until flash came out. Similarly, Resin cement -Subgroup IV was manipulated using auto-mixing tips with a mixing ratio of 1:1. The mould cavities were approximated with glass slab and then light cure for 30 seconds, the samples were retrieved. After recommended setting time, the specimens were carefully separated from the mould and examined for any porosity or defect.

Artificial Saliva

The freshly prepared artificial saliva was used by immersing prefabricated granules into 500ml of distilled water. The pH was then adjusted to 3.3 and 12.3 with hydrochloric acid and sodium hydroxide. The other solution selected was distilled water which acts as control.

First 36 specimens of each cement were stored in these mediums for 24 hours, out of which one set of 12 specimens were stored at 3.3 pH, other set of 12 specimens were stored at 6.7 pH and remaining 12 specimens were stored at 12.3 pH. Second set of 36 specimens of each cement were stored in above mentioned mediums for 48 hours. Third set of 36 specimens were stored for 7 days. The weighing of the specimen was done prior to its placement in the storage medium by digital weighing balance.

Solubility Evaluation

The specimens were subjected for evaluation of solubility by measuring the weight loss. Amount of weight loss will be calculated as the difference between initial weight of specimen before placing in the artificial saliva and its final weight after its storage in the desiccator. Percentage of solubility will be calculated.

Weight loss= Initial Weight-Final weight

$$\text{Percentage of solubility} = \frac{\text{weight loss} \times 100}{\text{Initial weight}}$$



FIGURE 1: Digital pH Meter



FIGURE 2: Digital Weighing Balance

Data thus collected was put to Statistical Analysis

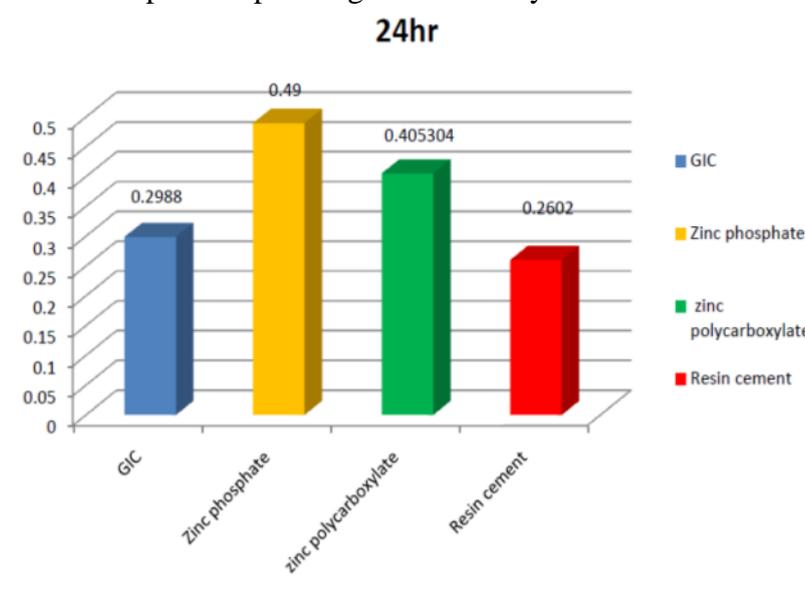
Statistical Analysis

Statistical analysis was performed using Statistical Package for Social Sciences(SPSS Inc, Chicago, IL, version 20.0 for Windows).The results of the measurements were analyzed using one –way analysis of variance (ANOVA) test.

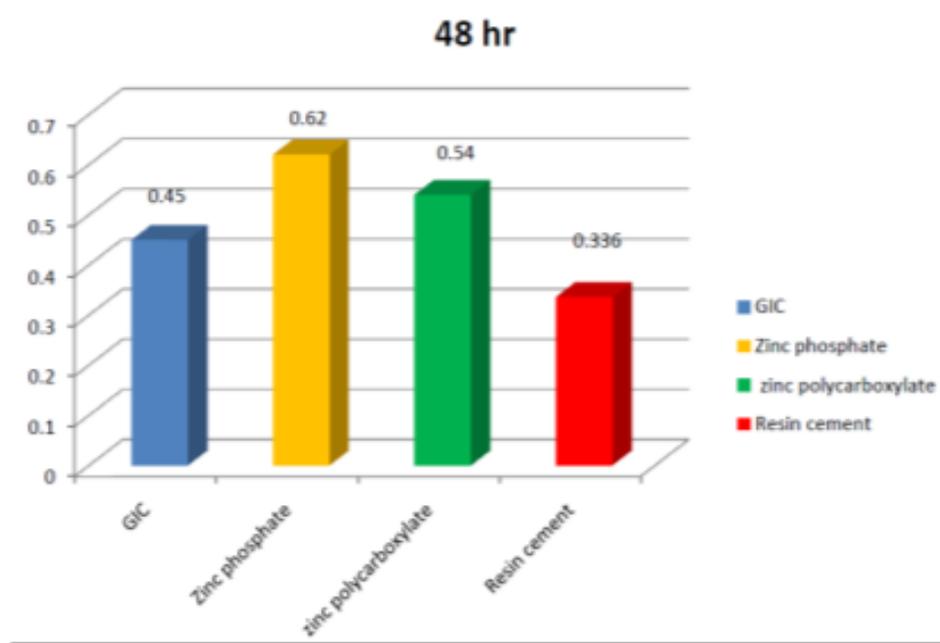
Results and Observations

Intragroup and Intergroup comparison of Group I

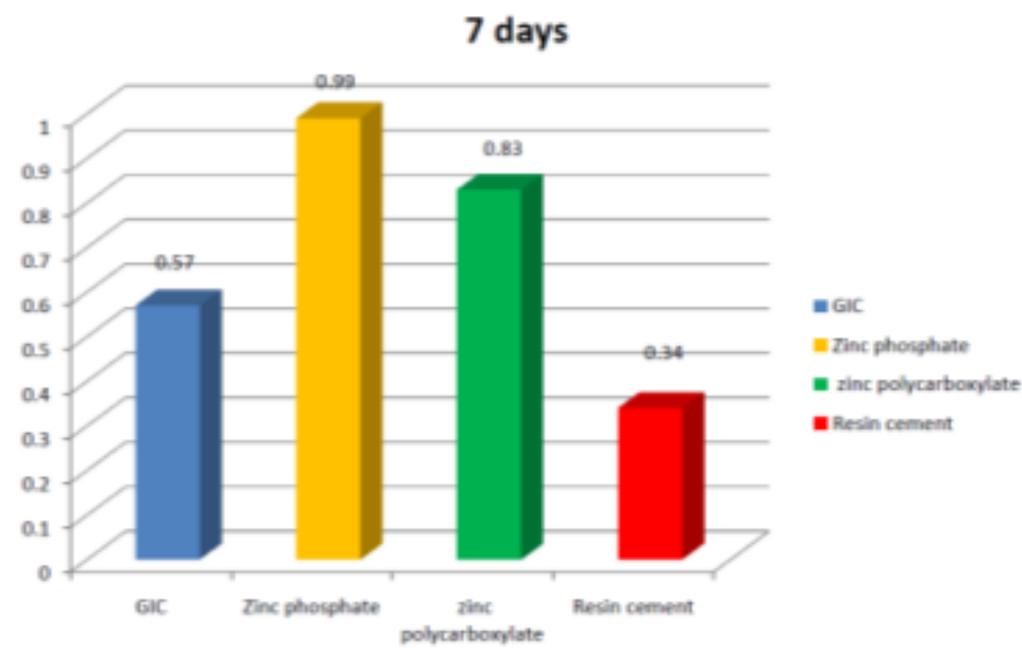
Graph 1, 2 and 3 depicts comparison of different cements in artificial saliva at different time interval and pH. Zinc Phosphate depicts highest solubility with time.



GRAPH 1: Solubility of different dental cement at 24 hours



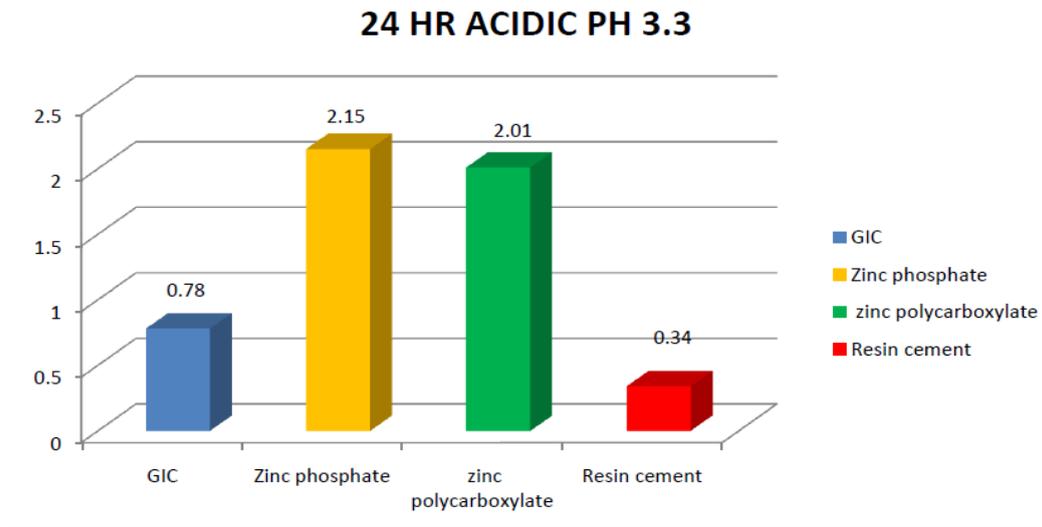
GRAPH 2: Solubility of different dental cement at 48 hours



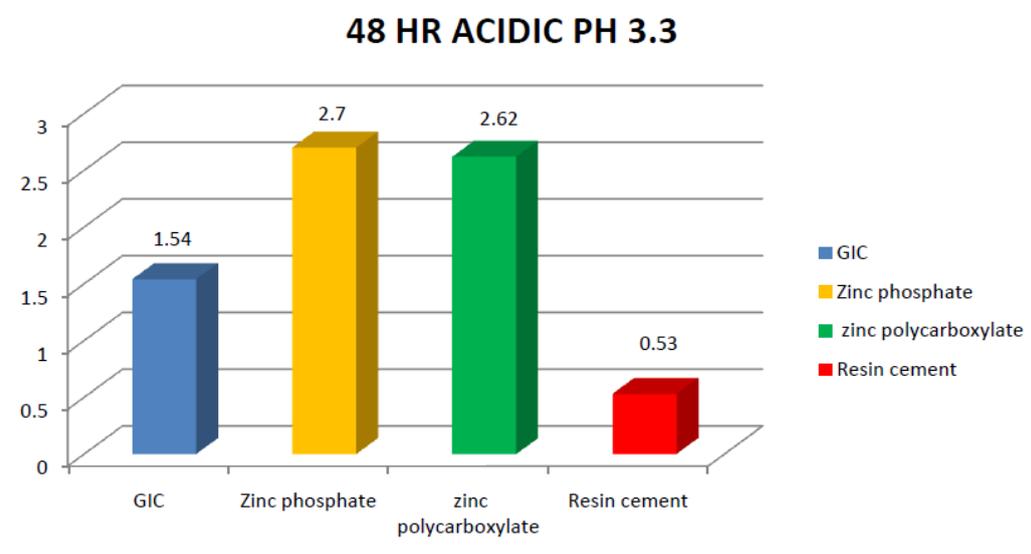
GRAPH 3: Solubility of different dental cement at 7 days

Intragroup and Intergroup comparison of Group I at acidic pH

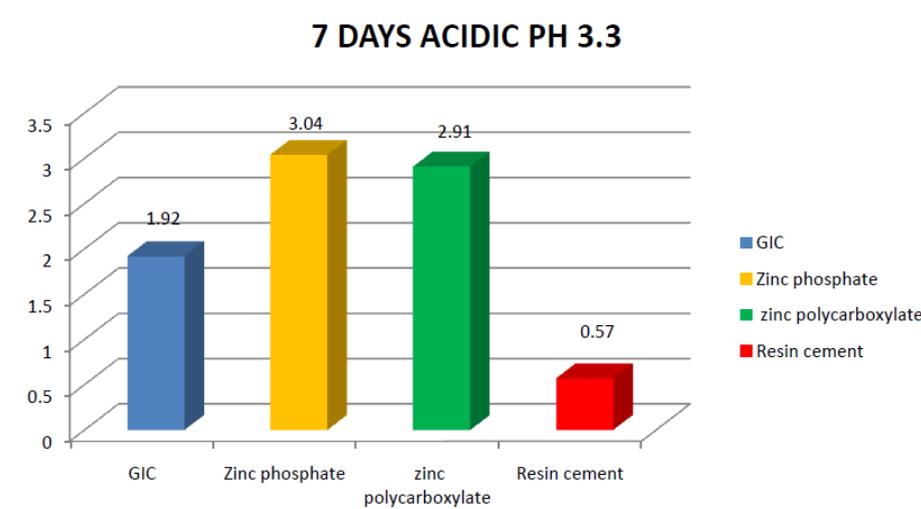
Graph 4, 5 and 6 depicts comparison of different dental cement in acidic pH (3.3) at 24 hours, 48 hours and 7 days. Subgroup yielded higher solubility rate at 7 days than 24 hours. A highly significant difference was seen between dental cements with the P value of <0.05. Zinc Phosphate depicts highest solubility with time and resin cement depicts lowest solubility.



GRAPH 4: Solubility of different dental cement at 24 hours at 3.3ph



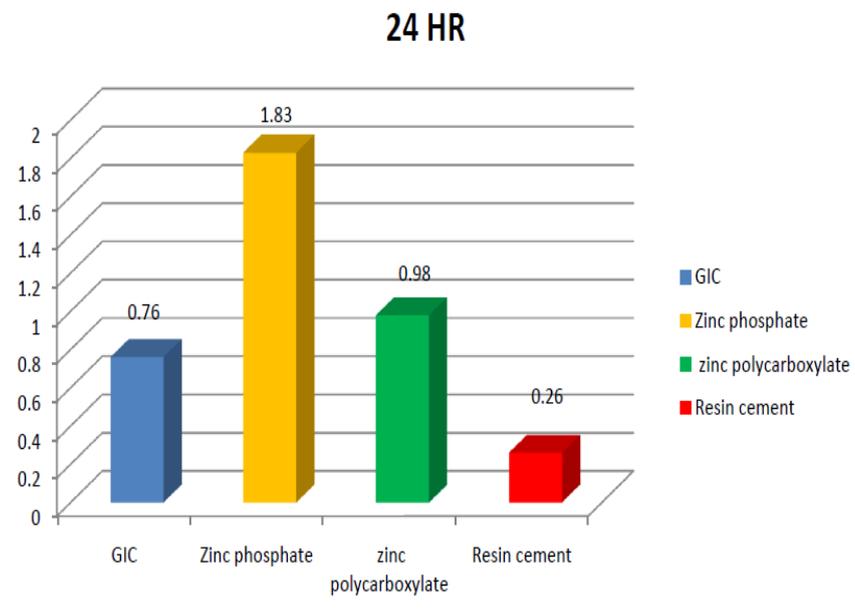
GRAPH 5: Solubility of different dental cement at 48 hours at 3.3 pH



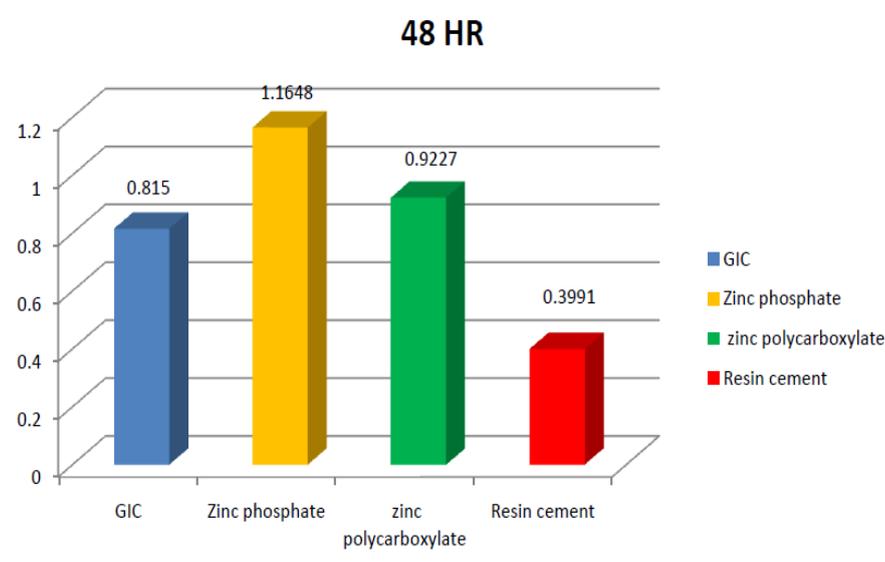
GRAPH 6: Solubility of different dental cement at 7 days at 3.3 pH

Intragroup and Intergroup comparison of Group I at basic pH

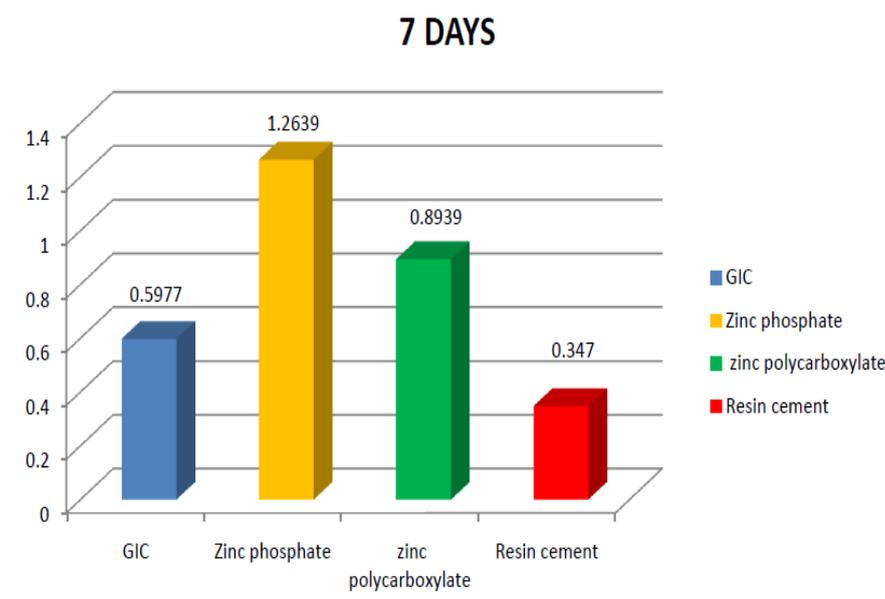
Graph 7, 8 and 9 depicts comparison of different dental cement in basic pH (12.3) at 24 hours, 48 hours and 7 days. Subgroup yielded higher solubility rate at 7 days than 24 hours. A highly significant difference was seen between dental cements with the P value of <0.05.



GRAPH 7: Solubility of different dental cement at 24 hours at 12.3 pH



GRAPH 8: Solubility of different dental cement at 48 hours at 12.3 pH



GRAPH 9: Solubility of different dental cement at 7 days at 12.3 Ph

The **results** of this study showed: Zinc Phosphate is most soluble followed by Zinc Polycarboxylate then Glass ionomer and least soluble is resin cement. All the cements showed **more solubility in acidic** environment (3.3) followed by basic medium (12.3) and least soluble neutral environment (6.7).

Discussion

Dental cements play an essential part in fixed prosthodontic treatment. Their most important characteristic property is the resistance to dissolution in oral fluids. The oral cavity has a dynamic environment and dental luting cements are subjected to multiple sources of fluid flow. According to **Tae Hyung Kim 2006**, low solubility and high strength are the most desirable properties for any luting material.

Three main types of conventional cements are included in this study. Zinc phosphate cement is included as it is considered as the gold standard against which other cements are compared due to its long history in clinical application among other luting agents.. Zinc Polycarboxylate cement is the first chemically adhesive cement introduced by **Dr. Dennis Smith in 1968**. This cement also sets by acid-base reaction. Glass ionomer cement is widely used for luting the prosthesis. Advantage of using this cement is fluoride content of the powder that ranges from 10 to 23% which is responsible for its anticariogenic property. It is sensitive to water erosion, due to hydrolysis of the cement components. **Deniz et al(1998)** stated that higher levels of solubility are related to the exposure of mixed cement with water.

These days resin cements are among the commonly used dental cements. Resin cement can be light cured, chemically cured and dual cured. This luting cement has better mechanical properties are compared to other conventional luting cements. The water absorption capacity of resin cement depends on resin polarity as dictated by the concentration of polar sites available to form hydrogen bonds with water. The choice of these cements used for evaluation is based on the fact that these are the most commonly used luting materials in the clinical practice.

Due to change in socio-economic lifestyle of society there is shift in eating habits from healthy to packed foods. Food components, beverages and different oral fluids from the oral environment have adverse effects on the solubility of the luting cements. During consumption, various food or drinks comes in contact with the restoration surface before it is washed away by saliva. Luting cement around the margins of the restoration is an area of plaque stagnation and hence is subjected to lower pH as plaque bacteria ferments sugars to produce acid. So,

evaluation of solubility of dental cements at acidic and basic pH is carried out.

Initial weight of each sample was measured before placing it in the medium by digital weight balance. After specific time interval each sample was removed, and then placed in a desiccator. Longer the samples are placed in the desiccator, the higher is its stability as stated by **Levine (1987)**. Initially there is a gain in weight as a result of the ingress of water molecules and egress of monomers and other small molecules. Generally, this diffusion of water molecules through polymeric materials can occur in two patterns, free volumetric theory where without any mutual relationship diffusion occur and interaction theory in which water diffuses through material binding successively to the hydrophilic groups. Individual samples weights are again measured by digital weight balance and the solubility of cements were measured. In present study weight analysis method was selected to evaluate solubility as it is simple, easy to carry and no expensive instrumentation is required. Overall, each cement specimens showed loss in final weight due to the loss of unpolymerized and soluble components into the storage medium that was replaced by water.

The results of this study showed highest solubility for Zinc Phosphate followed by Zinc polycarboxylate then Glass ionomer and least values for Resin cement. Zinc phosphate and Zinc polycarboxylate cements form a weaker bond matrix than the calcium and aluminium ions in the glass ionomer cements. Resin cements showed least solubility as they are composed of resin matrix of bis-GMA and filler of inorganic particles. Solubility depends upon the filler content of the resin cement. More the filler content less will be the solubility. Bis-GMA is also responsible for the low solubility due to its cross-linking density. Mixing of resin cement was different from mixing of conventional cement. Later was mixed on paper pad and former was auto mixed. These leads to formation of number of voids which gets incorporated within the cement thus forming oxygen-inhibition zones of unpolymerized materials, which affect the solubility of the set cement.

The results obtained in the present study are in accordance with the studies done by **Osborne et al. (1978)**, **Yoshida (1998)** and **Eisenburger, Addy, and Robbach (2002)** for acidic and neutral mediums.. In all the previous studies, solubility of conventional and resin cements was checked in acidic environment only, but in the present study solubility was compared in basic medium as well.

However, two studies conducted by **Hajmiragha et al (2008)** and **Saleem and HAQ (2011)** **contradicted** results to the present study. Hajmiragha reported that Zinc polycarboxylate showed greater solubility than Zinc phosphate. The difference in the result obtained can be due to the difference in the exposure medium. As the exposure medium in that study was circulated around the specimens with the help of magnetic field. Study by **Saleem and HAQ (2011)** stated that Glass ionomer cement is more soluble than Zinc phosphate cement. This may be attributed to use of different methodology to check the solubility of various cements. In their study, after preparation and immersion period, samples were washed with copious water and openly dried before measuring final weight. However, in our study, in order to stimulate the intra-oral conditions, freshly prepared artificial saliva was used. Though every effort was made to simulate oral cavity condition, the factors which were not considered might affect the clinical performance of the dental cements. These include 1) microbial flora; which is present in oral cavity and is responsible for plaque or activity was not taken into consideration; 2) constant salivary flow; samples were immersed in the same medium no change in the medium was done whereas as in oral cavity salivary flow is constant and keep on changing; 3) effects of plaque metabolism and tooth brushing habits-these effects the cement solubility as plaque accumulation around the restoration alters the pH and can be responsible for restoration failure.

Conclusion

Within the limits of this study, Zinc Phosphate showed highest in all types of mediums and at

all study intervals. Resin cement was found to be least soluble. Future studies with larger sample sizes are warranted to better explore the solubility and other properties of dental cements taking other aspects of the oral conditions in consideration.

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