Evaluation of the Solubility of Dental Cements in Artificial Saliva of Different pH Values

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The aim of this study was to determine the solubility of dental cements in artificial saliva of different pH values. A total of 180 specimens—20 specimens (20 mm diameter × 1.5 mm thickness) for each dental cement—were prepared. After the specimens were weighed, they were set in the media with different pH values. After 24 hours, 72 hours, seven days, and 28 days, the specimens were taken out, dried in a desiccator, and re-weighed. Percentage of solubility at each time period was calculated as 100 per cent times weight loss divided by the initial weight of the specimen.

Analysis of variance and LSD multiple comparison test were applied. It was found that cement type, storage time, and medium pH exerted statistically significant (p<0.001) effects on solubility. Among the tested cement types, zinc phosphate cement (Adhesor Carboline) presented the highest solubility. In terms of medium pH, the cements seemed to be most stable in the medium with pH 7.

Keywords : Solubility, Permanent cements, Temporary cements

INTRODUCTION

Presently, various types of adhesive cement are used for permanent and temporary cementation of indirect restorations. These cements have different mechanical and biological characteristics. Among which, the most important characteristic is stability in the oral cavity—that is, resistance against decomposition and degradation. Decomposition of cements results in deterioration of restorations, and may even cause secondary caries.

Zinc phosphate cement is one of the oldest materials used for the cementation of restorations. But of late, many new adhesive cements have been developed. These cements give better results than zinc phosphate cement. They adhere to the surfaces of both dentin and metal prostheses more firmly, and thus reduce microleakage to a greater extent. These newly developed adhesive cements include glass ionomer cement, polycarboxylate cement, zinc oxide eugenol cement, and resin cement. Besides, composite resins with dual-cure system for bonding inlays and laminate veneers have also been introduced.

Provisional luting cements are of two main types: calcium hydroxide and zinc oxide cements (with eugenol or alternative substances). Zinc oxide-eugenol cements have a good sealing capacity and antibacterial properties. However, eugenol occasionally might cause allergic reactions. Therefore, eugenol-free cements are advised for patients who are sensitive to eugenol.

In terms of the solubility of conventional luting agents, many related investigations have been performed by many researchers. However, information is scarce regarding the solubility of temporary cements in artificial saliva with different pH values. The aim of this study, therefore, was to compare the solubility of permanent and temporary cements in artificial saliva with different pH values.

MATERIALS AND METHODS

Dental cement specimens
Table 1 lists the brand names and contents of five permanent and four temporary cements used in this study. A total of 180 samples—20 specimens for each cement type—were prepared. A standard mix of cement of 0.5 ml was placed in a split stainless steel ring (inside diameter 20 mm and 1.5 mm thick) on a thin polyethylene sheet backed by a flat glass plate. Another flat plate lined with a thin polyethylene sheet was used to press the cement into the ring. One end of a piece of preweighed corrosion-resistant wire was placed in the soft cement as the specimens were formed to provide a convenient means of holding the specimens.

At three minutes after start of mix, the plates and cement were placed in an oven with a relative humidity of 95-100% at 37±1°C. After one hour, the specimens were withdrawn from the oven and removed from the rings, and any excess or loose material was carefully removed. Specimens of each cement type were separated into four groups for evaluation and comparison in distilled water and artificial saliva with different pH values.

Artificial saliva
The artificial saliva was of the following composition: NaCl, 0.400 g; KCl, 0.400 g; CaCl\(_2\), 0.795 g; Na\(_2\)PO\(_4\), 0.69 g; NaS • 9H\(_2\)O, 0.005 g; urea 1.0 g; distilled water, 1000 ml. The pH was then adjusted to...
3, 7, or 9 with NaOH or HCl, and the volume made up to 1 L.

**Experimental procedure**
Dental cement specimens were weighed and submerged by pouring 50.1 ml of distilled water and artificial saliva (pH 3, 7, or 9), and stored at 37°C for 24 hours, 72 hours, seven days, and 28 days. At the end of each time period, specimens were removed from the saliva, blotted with clean absorbent paper, and stored in a desiccator containing thoroughly dry anhydrous calcium sulfate until constant weight was attained. Amount of weight loss was calculated as the difference between the initial weight of the specimen and its final constant weight after storage in the desiccator. Percentage of solubility was calculated as 100 per cent times weight loss divided by the initial weight of the specimen.

**Statistical analysis**
For each type of cement, five specimens of each medium (i.e., distilled water, artificial saliva of pH 3, 7, and 9) were analyzed and their average values taken into consideration.

For statistical evaluation, analysis of variance and LSD (Least Significant Difference) multiple comparison test were used. Means and standard deviations were calculated.

**RESULTS**
According to variance analysis, cement type, pH value (pH 3, pH 7, and pH 9), and storage time were found to be very significant factors to solubility (p<0.001). Figure 1 shows that the percentage of solubility values were dependent on storage time at different pH values (pH 3, pH 7, and pH 9).

With the permanent cements, Meron (glass polyalkenoate (ionomer) cement) presented the lowest percentage of solubility value (0.531%) at pH 7 after one day, while Adhesor Carbofine (zinc phosphate cement) presented the highest percentage of solubility value (5.081%) at pH 3 after 28 days.

With the temporary cements, Temp Bond (zinc oxide-eugenol cement) presented the highest percentage of solubility value (5.331%) at pH 7 after 28 days, while Sinogol (zinc oxide without eugenol cement) presented the lowest value (3.536%) at pH 7 after 28 days. In other words, solubility of zinc oxide without eugenol cement was more stable than the zinc oxide-eugenol cement.

**DISCUSSION**
The most important characteristic required for cements used in dentistry is their resistance to dissolution in oral fluids. Possible factors that influence solubility include immersion time, concentration of solute in dissolution medium, medium pH, specimen shape and thickness, and powder-liquid ratio of cement. In the present study, the powder-liquid proportions used for cement preparation complied with the manufacturers’ specifications.

In many studies concerning glass ionomer cement, it was reported that this material was more resistant to solubility than zinc phosphate cement. In a review and comparison of presently used dental cements, Oilo further found that glass ionomer cement had the highest strength and retentive properties and a low solubility. But for all dental cements in general, it was emphasized that they must be isolated from humidity and saliva, especially within the first few hours of cementation. In this study, Meron (a glass polyalkenoate
(ionomer) cement) - which included polyacrylic acid - showed the significantly lowest solubility value than the other permanent cements. In other words, the solubility result of glass ionomer cement in this study corroborated those of previous studies.

In an in vitro study, it was found that glass ionomer cement was more resistant to acidic erosion than polycarboxylate cement. On the same issue about erosion, it was found in the present study that the percentage of solubility of polycarboxylate cement (3.989%) was higher than that of glass ionomer cement (1.867%) at pH 7 after 28 days, which corroborated reported findings that the solubility of polycarboxylate cement in water was relatively weaker.

With zinc phosphate cement, Mesu and Reedijk stated that its biggest disadvantage was its greater solubility in the presence of organic acids of oral secretions. When compared against zinc phosphate cement, Mortimer et al. and Yoshida et al. found that the solubility values of polycarboxylate cement were higher. However, this was not so with Phillips et al., whereby it was suggested that there were no differences between zinc phosphate and polycarboxylate cements in terms of solubility.

In this study, the percentage rates of solubility of Adhesor Carbofine (zinc phosphate cement) and PolyCarb (polycarboxylate cement) were found proximate to each other. This finding was in accordance with that of Phillips et al. Further, Adhesor Carbofine (zinc phosphate cement) presented the highest percentage solubility (5.081%) at pH 3 after 28 days, while Yoshida et al. obtained a value of 8.0% after 30 days for zinc phosphate cement.

In the mouth, luting cements constantly come into contact with oral flow causing dissolution. Luting cement around the margins of a restoration is in an area of plaque stagnation, and is therefore subjected to a pH value lower than 7 because plaque bacteria ferment sugars to produce lactic acid. Apart from plaque build-up, other situations such as intake of acidic foods may also play a role in solubilizing luting cements. While variations and peculiarities in clinical situations lead to differences in solubility of luting cements, there is increasing evidence of a lack of correlation between standard test results and in vivo phenomena observed in the mouth.

On this note, great care and discernment must be exercised when interpreting reported results.

In studies pertaining to dental cements, water, acids, and other solvents have been used to act as food-simulating liquids. On this note, Levine et al. suggested the usage of artificial saliva to produce a setting similar to the oral medium. In general, it is known that the solubility of cements in organic acids is much higher than in water. However, it is also very important to have a good knowledge of the solubility of various types of cement when exposed to different pH values. Lower pH values increase the solubility of cements. In this connection, Walls et al. obtained the highest solubility at pH 4, whereas no dissolution was observed in a buffer solution at pH 10 even after 24 hours. In light of this finding by Walls et al., artificial salivas of different pH values were prepared in this study. Indeed, it was found that the tested cements demonstrated maximal solubility at 28 days in artificial saliva of pH 3 - a finding compatible with the result of Wall et al. It should be highlighted that erosions understandably occurred most frequently within the first 5-10 minutes of cement preparation. Therefore, it was found that the longer it remained in a desiccated medium, the higher its stability would be.

It was suggested that in vitro studies per se were not adequate to reflect the true and exact nature of
water absorption and solubility of cements. In other words, in vivo studies are required. In an in vivo study conducted on this issue\(^6\), it was realized at the end of 12 months that glass ionomer and silicophosphate cements demonstrated minimal solubility, while zinc phosphate cement underwent maximal dissolution. In addition, it was proposed that not only in vivo conditions, but that powder-liquid ratio of the tested cements also affected the results obtained\(^7\). To supplement the list of factors that influence solubility, an in vitro study has further indicated of a statistically significant positive relationship between solubility and immersion period\(^8\). In in vivo researches performed by Mitchem and Gronas\(^9\) and Hersek and Canay\(^10\), it was also detected that the solubilities of glass ionomer and silicophosphate cements in water were similar but lower than those of zinc phosphate and polycarboxylate cements. Likewise, in the present study, the percentage rates of solubility of silicophosphate and glass ionomer cements were lower than those of zinc phosphate and polycarboxylate cements.

Currently, three main types of conventional cement are commonly used: zinc phosphate, polycarboxylate, and glass ionomer cements\(^11\). In an effort to provide improved clinical success, new classes of luting agent with improved physical properties and new therapeutic capabilities have been developed\(^12\). Of late, two additional types of luting agent have gained considerable popularity. These include the resin-modified glass ionomer cements and resin cements. The resin cement category includes light-cured, dual-cured, and chemically cured agents\(^13\).

Twenty years ago, the use of resin composite luting agents to cement dental restorations was not common. Now, resin cements account for a growing proportion of cement use, the reasons for which include their better mechanical properties compared with conventional luting agents. One of the most important properties determining the durability of luting cements in the mouth is resistance against dissolution and disintegration\(^14,15\). Hence, although resin luting cements are more difficult to use, they provide greatly increased bonding capabilities\(^16\) and exhibit low solubility values\(^17\). Yoshida et al.\(^18\) indicated that resin luting cements were markedly less soluble than conventional luting agents when placed at pH 4 over a 30-day period.

Further, recently introduced resin-modified glass ionomer luting materials offer increased resistance to dissolution and improved physical and biological attributes\(^19\). Due to their hydrophilic nature, resin-modified glass ionomer cements showed significantly higher water sorption compared to composite cements\(^20\). At this juncture, it must be highlighted that the solubility of resin-modified glass-ionomer and resin luting cements given in various studies\(^21-23\) were higher than those obtained in the present study. Notwithstanding, the cements used in this study were the ones routinely used in our clinics.

For temporary restorations, the required retention duration might span from a few weeks to a month. As such, a temporary cement must be resistant to solubility, has good retentive properties, and must also facilitate easy removal by clinician when needed\(^24\). Time is another important factor which needs to be considered when evaluating the retentive properties of temporary cements. Temporary cements, which have low solubility and compressive strength from medium to high level, are advised for acrylic or metal temporary restorations. However, cements with high solubility and low retention strength are recommended for temporary luting of permanent restorations\(^25\).

Olin et al.\(^26\) stated that cements without eugenol are used not only in patients who are sensitive to eugenol, but also in restorations which require high retention. In this study, it was determined that temporary cements without eugenol showed lower solubility than those with eugenol. Thus, if it is considered that low solubility has positive effect on retention, then our results were compatible with those of Olin et al.\(^26\).

In this study, Temp Bond (zinc oxide-eugenol cement) showed high solubility (5.331%) compared to other temporary cements. However, while some studies indicated that high solubility was a disadvantage of zinc oxide-eugenol cements\(^27,28\) Lewinstein et al.\(^29\) stated that there were no significant differences in the solubility of temporary cements. Presently, there is no existing literature concerning the solubility of temporary cements. Hence, there is no basis nor platform upon which discussion of this subject can leverage nor proceed further at this point.

**CONCLUSIONS**

Statistically significant differences in solubility were found among the specimens stored in acidic, basic, and neutral artificial salivas. It was observed that the cements were more soluble in the acidic medium but were stable in pH 7 medium.

With the permanent cements, the highest solubility was presented by Adhesor Carbofine (zinc phosphate cement), followed by PolyCarb, Satellit, and Harvardid. Least solubility was presented by Meron. With the temporary cements, the highest solubility was presented by Temp Bond (zinc oxide-eugenol cement).

In conclusion, the findings of this study indicated that cement type, storage time, and medium pH exerted statistically significant (p<0.001) influences on cement solubility.
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