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ORIGINAL ARTICLE

Effect of PH and Powder to Liquid Ratio on the Solubility of a Conventional Glass Ionomer Luting Cement

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ABSTRACT

Introduction: Properties of GI luting cements are influenced by complex oral environment. This in-vitro study is designed to simulate oral conditions for better understanding behavior of GI luting cement in oral cavity and to evaluate its solubility. Fluctuating oral environment from acidic to alkaline is thought to cause solubility of luting agent, which insults the integrity of luting agents leading to micro leakage and ultimately luting failure of restorations. An attempt via this study was made to funnel powder liquid ratio for dental practice by exposing different P/L ratio to varying pHs of artificial saliva solution.

Materials and Methods: Four groups of specimens were prepared using different powder liquid ratios (Group A 1:2, group B 1:3, group C 1.5:3 and group D 2:3S). Specimens were desiccated for 2 hours in hot air oven and weighted with digital analytical balance. Specimens were immersed in respective artificial saliva for a week, desiccated for 2 hours, weighed and solubility was calculated. Micrographs of specimens were taken with scanning electron microscope. Descriptive statistics such as mean, range and standard deviation values were presented as tables and charts. variation of means amongst groups were analysed using One-way analysis of variance (ANOVA). P value of ≤ 0.05 was considered significant

Results: Solubility of specimens immersed in acidic artificial saliva solution showed elevated solubility. One-way ANOVA showed statistically significant results, Tukey's HSD showed that specimen of group A2 showed elevated solubility of 0.000246790 $\mu g/mm^3$ and specimens of group D1 showed less solubility of 0.000003466$\mu g/mm^3$.

Conclusion: Unlike manufacturer recommended P/L ratio, this study concluded that P/L ratio of 2:3 is a better GI luting cement as it exhibited least solubility in both acidic and basic environments.

Keywords: Luting, GI luting cement, solubility, salivary pH

INTRODUCTION

Demand for fixed prosthesis is increasing with increase in number of partially edentulous individuals, glass ionomer cement (GIC) is a lute cement used commonly for cementation of fixed prosthesis (Saran et al., 2020). GI luting cement was reported in early 1970s (Nicholson et al., 2020). Glass ionomer is a group of materials, tailored to react the powdered silicate-glass and polyacrylic acid-based liquid which was originally intended for aesthetic restoration of anterior teeth. But due to its adhesive chemical bonding with tooth tissues & metal alloys, biocompatibility with pulp and its caries prevention properties by fluoride release expanded its use as a luting agent, orthodontic bands adhesives, pits and fissure sealants, liners, bases, core buildups and intermediate restorations (Park & Kang, 2020; Saran et al., 2020).

Prognosis & longevity of fixed prosthesis rely on mechanical and chemical properties of lute cements. Clinically, solubility insults marginal integrity which causes marginal leakage in materials bulk, secondary caries in the prepared tooth, dentinal hypersensitivity as the tubules are exposed in underlying prepared tooth, accumulating and releasing toxins to the neighboring tissues which causes periodontal disease and ultimately fractures the material bulk and debonds the restoration. Pertinent to disintegration of luting agent; most important characteristic is its stability and degradation (MS et al., 2017).

Previous studies observed different solubility values for conventional GIC as a luting agent. Bharali et al. in 2017 evaluated solubility of different luting agents in artificial saliva of pH 5 and pH 7, they concluded elevated solubility in conventional GIC compared to resin cement, resin-modified GIC and zinc phosphate lute cement (Bharali et al., 2017a). Al-Razooki et al concluded from their study that GI lute cement showed least resistance to solubility in distilled water as compared to resin cement, polycarboxylate, zinc phosphate (Al-Shekhli, 2010). Cattani-lorente et al found impaired physical and mechanical properties in different lute-cements. They evaluated the specimens for water sorption after storing them for extending time in an aqueous environment. (Cattani-Lorente et al., 1999). Saleem and Haq evaluated solubility of GI lute in artificial saliva solution with different pH and concluded that GI lute presented with highest solubility at a low pH of 2.46 but was less soluble at high pH of 6.57 (Saleem & Haq, 2011).

Manufacturers of GC Company, (GC Corp, and Tokyo, Japan) have recommended powder to liquid ratio of 1: 2 for commonly used luting cement in our region, this is seldom followed by dental care providers which may cause detrimental effects on its physical properties. This study was hence aimed to provide information regarding a better choice of powder/ liquid ratio to clinicians for luting indirect restorations.

MATERIALS AND METHODS

A total of 120 test samples were prepared for current study using conventional GI lute (GC I, GC Corp, Tokyo, Japan) using a stainless-steel mold of 10 x 2mm (ISO specification 4049).

Calculated amount of powder liquid ratio was poured on to glass slab. They were mixed thoroughly and poured into the mold and slightly over filled. Both sides of mold were cover by cellulose acetate strips and a glass slab was used to press material to prepared mold cavity. After initial setting was achieved in approximately 6 to 8 minutes sample was retrieved from the mold and excess material if any was cut with dry-slow speed finishing bur. This procedure was applied to prepare samples of all sub groups of group A, B, C and D (Table 1).

Table 1: Distribution of specimen in various groups and sub groups

Groups	Sub-groups	Samples	Total Specimens	P/L ratio	pH
Control A	A1	10	30	1/2	7
	A2	10		1/2	5.4
	A3	10		1/2	7.4
Experimental B	B1	10	30	1/3	7
	B2	10		1/3	5.4
	B3	10		1/3	7.4
Experimental C	C1	10	30	1.5/3	7
	C2	10		1.5/3	5.4
	C3	10		1.5/3	7.4
Experimental	D1	10	30	2/3	7

D	D2	10		2/3	5.4
	D3	10		2/3	7.4

Artificial Saliva solution (A.S.S) was used as immersion medium and was prepared according to Saleem & Haq in 2011, a stock of NaCl (0.400g/dm^3), $CaCl_2$ (0.795g/dm^3), NaS (0.005 g/dm^3), NaH_2 PO_4 (0.780 g/dm^3), $(NH_2)CO$ (10.000 g/dm^3) and KCL (0.400 g/dm^3). This stock was dissolved into 1 liter of de-ionized water. Hydrochloric acid and sodium hydroxide were used to adjust pH of artificial saliva solution to pH 7, 7.4 and 5.4 with the help of digital pH meter (Saleem & Haq, 2011). Samples were desiccated in dry-air oven (S-0703140, M-D50 300D) for 2 hours at 37^{o}C in presence of silica gel as a moister absorbent. In order to calculate volume, masses (m_o & m_1) were recorded using analytical balance and diameters (d) were recorded using digital calipers. Cycles were repeated to get a constant mass (m^o).

Samples were kept in their respected artificial saliva solution for a period of seven days. samples were then removed from artificial saliva solution, damped in tissue paper and moved again into hot air oven for 2 hours at 37 C^o to obtain m^1.

The value of solubility was computed using equation 1 (ISO 4049:2000)

$$S = \frac{M^{\circ} - M^{1}}{V} \ (\mu g/mm^3) \text{---- 1}$$

In the given equation m^0 signifies mass done prior immersion (mg), m^1 indicates mass post immersion (mg) and V indicates specimen volume before immersion (mm^3). (Al-Shekhli, 2010).

Scanning Electron Microscopy: Micrographs of specimens were taken using scanning electron microscope [(JSM- IT 100) in National Centre of Excellence, Department of Geology, University of Peshawar. Four Specimens (one from each ratio) were subjected to SEM analysis before immersing them into artificial saliva solution. One specimen from each subgroup was subjected to SEM analysis after immersing them into their respective artificial saliva solution. Each specimen was desiccated for 2 hours before gold sputtering. Gold sputter (DII-29030SCTR Smart Coater) was used to coat each specimen with 24 carat gold. (Lad et al., 2014)

Descriptive statistics such as mean, range and standard deviation values for solubility for specimens in each of sub-groups were computed and presented as tables and charts. For statistical analysis of variation from means within groups and between groups were computed using ANOVA. P value of ≤ 0.05 was kept constant to be significant.

RESULTS

Amongst experimental groups specimens of sub group A2 having P/L ratio 1:2 and treated with pH 5.4 showed elevated solubility (mean 0.0001354μg/mm^3) and sub group D1 having P/L ratio 2:3 and treated with pH 7 showed decreased solubility (mean 0.0000034656μg/mm^3) among all other groups. These results are plotted in a bar chart having sub groups plotted on X' axis and solubility values plotted on Y' axis (Figure 1).

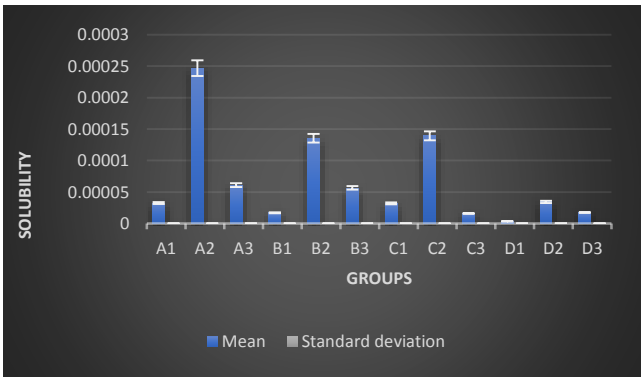

Figure 1: Box plot shows Mean values of Solubility (μg/mm^3) and Whiskers Showing Standard Error of GIC specimens solubility of various groups

One-way analysis of variance showed, the collected data are significant and their values as a result of statistical analysis are less than 0.05. (Table 2).

Table 2: Results of one-way ANOVA

ANOVA					
Solubility					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.000	11	.000	92.654	.000
Within Groups	.000	108	.000		
Total	.000	119			

Means in homogenous subsets computed via Tukey HSD revealed that sub group D1 having P/L ratio 2:3 treated with pH 7 artificial saliva, sub group C3 having P/L ratio 1.5:3 treated with pH 7.4, sub group B1 having P/L ratio 1:3 treated with pH 7 and subgroup D3 having P/L ratio 2:3 with pH 7.4 showed decreased values of solubility. Among these homogenous subsets, sub group A2 having P/L ratio 1:2 treated with pH 5.4 showed highest values of solubility. (Table 3).

Table 3: Means for solubility values (μg/mm^3) for groups and subgroups in homogenous subsets are displayed

Solubility					
Tukey HSD[a]					
Group	N	1	2	3	4
Group D1	10	.000003466			
Group C3	10	.000015817			
Group B1	10	.000016847			
Group D3	10	.000017422			
Group C1	10	.000032072	.000032072		
Group A1	10	.000032533	.000032533		
Group D2	10	.000034466	.000034466		
Group B3	10		.000056603		
Group A3	10		.000060997		
Group B2	10			.000135400	
Group C2	10			.000139290	
Group A2	10				.000246790
Sig.		.149	.229	1.000	1.000

Scanning Electron Microscopy results: Specimens were subjected to SEM analysis after desiccating them for 2 hours. All specimens showed micro cracks on their surfaces which because of desiccation. (Lad et al., 2014)

Group A: Amongst micrographs of group, A specimens, group A2 showed cracks which are propagated to material bulk. It may be because of its treatment with acid artificial saliva (Figure 2).

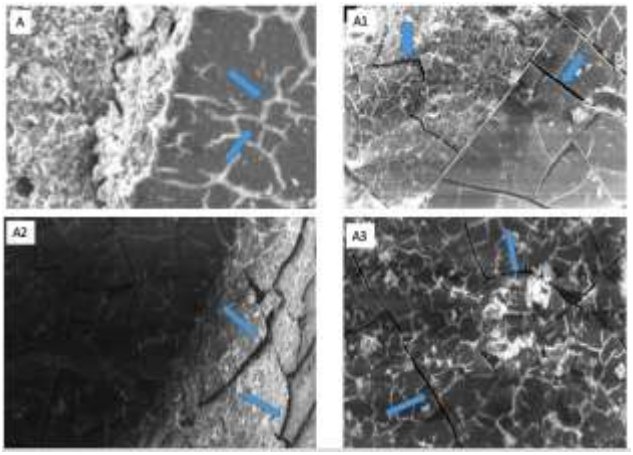

Figure 2: Showing micrographs of group A specimen. (*A without treatment, *A1- pH 7, *A2- pH 5.4 & A3- pH 7.4).

Group B: All the specimen of group B, showed wider cracks on surface propagated to material bulk, this may be because of artifact from desiccation or its treatment with artificial saliva solution (Figures 4).

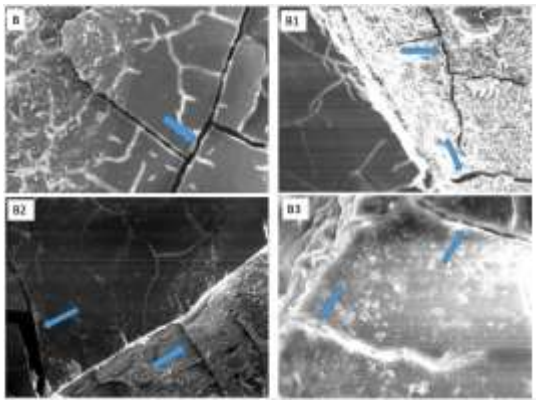

Figure 4: Showing micrographs of group B specimen. (*B without treatment, *B1- pH 7, *B2- pH 5.4 & B3- pH 7.4).

Group C: Scanning electron micrographs of specimen of group C, without treatment with artificial saliva solution showed minimal surface cracks. However, cracks and also small pits were observed on surface of specimen which were treated with artificial saliva solution (Figures 5).

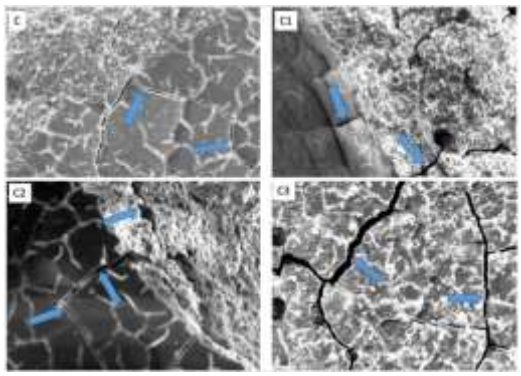

Figure 5: Showing micrographs of group C specimen. (*C without treatment, *C1- pH 7, *C2- pH 5.4 & C3- pH 7.4).

Group D: Numerous small craze lines were seen on surface of group D specimens, treated with artificial saliva, this may be because of artifact from desiccation or due to stresses during sample preparation and removal of set specimen from the mold (Figures 6).

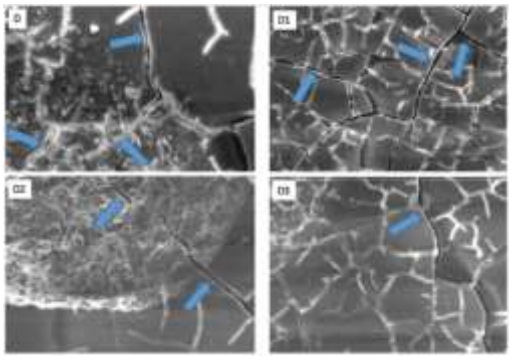

Figure 6: Showing micrographs of group D specimen. (*D without treatment, *D1- pH 7, *D2- pH 5.4 & D3- pH 7.4).

DISCUSSION

Results of our study were accordance to Nazirkar et al. They explained that the marginal gap should be kept 50 µm clinically and any increase in the said marginal gap will expose the lute cements to the challenging oral environment and integrity of the lute cement will descends to increase solubility (Nazirkar et al., 2014).

There existed significant differences in the solubility values among the tested GI lute and artificial saliva solution. Which may be due to the differences in powder liquid ratio or the final set composition and the surrounding media in which they were immersed. The detected high sorption for the specimens of group B2 was in accordance with the findings of previous investigations. Giti et al. they showed that GI lute had highest sorption in both water and ethanol in comparison with other cements. On the contrary, the sorption and solubility of specimens of D1 were less than all other cements, which could be attributed to its higher filler loading (powder content) than other tested groups. Variation in acidic content of artificial saliva solution influences and is likely to be the cause of detected difference between these groups. (Giti et al., 2016)

At the end of the study all specimens presented water sorption

Specimens of all tested groups and sub groups presented with some degree of water gain after the immersion phase. Hydrophobicity and hydrophilicity of a specific materials is determined by its chemical nature, cross linking in its polymers and the presence of reactive carboxyle, hydroxyle or phosphat groups which makes a material more susceptible to water gain and thus more hydrophilic. Cracks on the surface of the specimens tested may also contribute to gaining water during the immersion phase. Zankuli et al conducted a study on micro leakage of GI lute cement and funneled their observations that crack zones and craze lines in tested samples are because of the increased proportions of dye infiltration, which most probably be the justification for deviations in solubility values mentioned in studies conducted earlier. (Zankuli et al., 2014)

Oral environment is moisture laden, which was simulated in the present study to check the solubility of set cement by immersing the specimens of all sub groups in artificial saliva solution. The result documented from our study is in accordance with a study done by Yan et al, they incorporated chlorohexidine in GIC and concluded from their study that a group of restorative dental materials might captivate chemicals, water and fluid from fluctuating oral environment which disintegrated the complex structure of the cited material and in turn might liberate their components or dissolves to surroundings in environment. Fact that GI lute cement is water loving, this hydrophilic nature makes this luting material perceptive to oral fluids which give rise to hydrolytic degradation that ultimately results in their impaired mechanical properties and reduced longevity in terms of function and service.(Yan et al., 2017)

At all levels of pH, specimens of groups that were submersed in A.S.S of pH 7 & 7.4 showed smoothest surface compared with groups that were submersed in A.S.S of pH 5.4 which is in accordance to a study conducted by Reddy et al. They concluded that with each degree decrease in pH value, roughness of the immersed specimen increases. All samples in their study immersed in pH 2 presented higher roughness and samples that were immersed pH 7 presented lowest values in terms of roughness. They added that, H+ ions of citric acid infiltrates into set GIC disintegrates their components and substitutes metal cations in their matrix. Afterwards these free-cations are dispersed to the surface and released. Following this phenomenon metal cations declines in material matrix as more and more cations are disintegrated and released from adjoining glass particles and consequently causing its dissolution. Ultimately, set GIC material surface will be roughened with undissolved and projected glass particles (Reddy et al., 2014).

In present study specimens of groups that were immersed in artificial saliva solution of pH 5.4 showed elevated amount of solubility which may be because of presence of more H+ ions, readily available for replacement of metal cations from glass ionomer matrix.

Due to the unique properties of GI luting cement, as a result of immersion into artificial saliva solution detected in SEM analysis identified the topographical aspect interaction of GIC. It is necessary to investigate the chemical interaction between GIC composition and chemical interaction with that of artificial saliva solution by specific analyses, such as transmission electron microscopy and sophisticated spectroscopies. Findings of SEM analysis showed that the artificial saliva solution of lowered pH (5.4) left the specimens cracked till the depth of the material and not only the surfaces whereas the pH 7 and 7.4 caused only surface roughness that may be because of the desiccation. (Garcia-Contreras et al., 2015)

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