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In vitro comparative study of sorption and solubility of three modified glass ionomer cements

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***Abstract*---**Aim: To evaluate and compare the sorption and solubility of three modified glass ionomer cements – Glass Hybrid Restorative System (Equiaforte), Zirconomer Improved and resin modified glass ionomer cement (RMGIC) in artificial saliva. Materials And Methods: A total of 45cylindrical specimens (4mm diameter and 6mm height) were prepared for each material following the manufacturer's instructions. After subjecting the specimens to thermocycling of 500 cycles between 5 and 55 degree Celsius with the dwell time 15secs,45 specimens evaluated for sorption and solubility, first weighing them by a precision weighing scale (W1), then immersing them in artificial saliva for 28days and weighing them(W2), and finally dehydrating in an oven

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for 24 hours and weighing them(W3). Since the obtained values show normal distribution differences between the groups data were analyzed using the one way analysis of variance (ANOVA). For pairwise evaluation between the groups post hoc dunnet t_3 test was used. Results: The sorption and solubility values in artificial saliva were minimum for zirconomer improved glass ionomer cement with the mean value of sorption 52.91 ± 1.73 μg/mm^3 and solubility 10.47 ± 1.52 μg/mm^3 when compared with other groups.The difference between groups was found to be statistically significant ($p<0.05$) in terms of sorption and solubility. Conclusion: Zirconomer improved glass ionomer cement absorbed less water and is less soluble preceded by glass hybrid restorative system and then the resin modified glass ionomer cement.

***Keywords*---**glass hybrid restorative system, resin modified glass ionomer cement, sorption, solubility, zirconomer improved.

Introduction

Two important physical properties that influence the clinical durability of a restorative material are water sorption and solubility of the material. Since glass ionomer was hydrophilic, the early contamination of glass ionomer resulted in the binding of water molecules by polyacrylic acid (PAA) and ion leachable glass. In this way, the chemical setting was disrupted and the decrease in hardness occurred as a result of the absorption of water as the initial phase of degradation. Moreover, the presence of excess water during the growth of the hydrated silicate phase might have resulted also in a weaker material [(1)].

Cement solubility and water sorption are important in clinical selection, because they show different behaviour when exposed in water and oral fluids for long periods. The water sorption properties and the solubility of the cement change the mechanical characteristics of the material by directly interfering in the half-life of the restorations[(2)]. Water sorption can increase the volume of the material and it can act as a plasticizer and cause deterioration of the matrix structure of the material. There is always an interface between the teeth and restoration margins (about 40 microns) and so the use of cement with low solubility is very more important. Water sorption changes the mechanical properties through two effects: lamination and degradation.

The solubility of the restorative material causes loss of material mass, adversely affecting its mechanical properties, as well as causing tooth/restoration interface failure, increasing the risk of marginal microleakage and leading to restoration failure[(3)]. Resin-Modified Glass-Ionomers were introduced to the dental profession in 1991. They contain the same essential components as conventional glass-ionomers (basic glass powder, water, polyacid), but also include a monomer component and associated initiator system. The monomer is typically 2-hydroxyethyl methacrylate, HEMA, and the initiator is camphorquinone. Resin modified glass-ionomers set by the twin processes of neutralization (acid-base reaction) and addition polymerization, and the resulting material has a complicated structure based on the combined products of these two reactions. Moreover, competition between these two network-forming reactions means that there is a sensitive balance between them. This mixture of setting reactions may jeopardize the reliability of the set material, and as a consequence, close adherence to the manufacturer's recommendations on the duration of the irradiation step is essential in order to produce material optimal properties [4].

A yet new material- Zirconomer improved reinforces structural integrity of restoration and imparts higher mechanical properties befitted for utilization in posterior teeth. This is a high strength restorative material, reinforced with zirconia nano fillers known as zirconomer improved and has been a recent substitute to glass ionomer cement in dentistry. Zirconia (ZrO_2) is a white crystalline oxide of zirconium. It is a polycrystalline ceramic without a glassy phase and exists in several forms. The name "zirconium" comes from the Arabic word "Zargon" which means "golden in color" [5].

The recent development in the glass ionomer cement restoration is the introduction of bulk fill glass hybrid restorative system (Equiaforte) which represents the latest innovation in glass ionomer and resin technologies working in synergy. It is a combination of a self-adhesive, chemically cured, highly filled GIC and a self-adhesive, filled resin surface sealant. Since the comparison of the physical properties of glass ionomer cement would help the clinician to choose the appropriate and best material available for the restoration of weakened tooth structure, the present study has been conducted to comparatively evaluate the physical properties like sorption, solubility of three different types of glass ionomer cement namely resin modified glass ionomer cement, Zirconomer improved and glass hybrid restorative system (Equiaforte) in artificial saliva.

Materials and Methods

Following materials were selected for this study: (fig no: 1)

Group A: Hybrid ionomer restorative system - Equiaforte (GC Corporation, Tokyo, Japan).
Group B: Zirconomer improved(Shofu Inc., Japan).
Group C: Resin modified glass ionomer cement - Fuji II Lc Improved (GC Corporation, Tokyo, Japan).

Fig: no: 1 Restorative materials used in this study

Gupta et al (2011): Composition of artificial saliva

- 0.4gm sodium chloride
- 1.21gm potassium chloride
- 0.78gm sodium dihydrogen phosphate dehydrate
- 0.005gm hydrated sodium sulphide
- 1gm urea and
- 1000ml of distilled water.

To this mixture 10 N sodium hydroxide was added until the pH value was measured to be as 6.75 ± 0.15[6].

Preparation of cylindrical specimen

Metallic split mold (fig no:4) was manipulated with diameter 4mm and height of 6mm according to ISO specification number 9917 – 1: 2007.

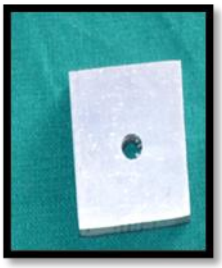


Fig: No: 4 Metallic Split Mold

Total of 45 cylindrical samples measuring 4mm diameter and 6mm height were fabricated in metallic split mold. Prior to the placement of any material in the metallic mold, petroleum jelly was applied on the inner side of the mold. Then the materials were placed in each group according to the manufacturer instructions.

Following the placement material in the mold for each group, back and forth of the mold was covered with mylar strip and pressed against glass slab to remove the excess the material. Finally each cylindrical specimen was smoothed by carbide paper and then it was left undisturbed. After this all the 45 samples were subjected tothermo cycling test comprising 500 cycles between 5°C and55°C with the dwell time 15 secs.

Determination of sorption and solubility:[7]

15 of each material with total of 45 specimens were weighed with precision weighing scale. The initial weight was termed as W_1(µg). Immediately after weighing the samples, they were soaked in artificial saliva (fig no: 16) at pH 7 and placed in incubator at 37°C for 28 days.

Fig No: 16 Samples immersed in artificial saliva

After removing samples from artificial saliva which was stored in incubator with the help of distilled water washed then dried with an absorbent paper, waved in air for 15seconds and weighed. This weight was termed as W_2(µg). Then for determination of solubility specimen were then dehydrated in an hot air oven at 37°C for 24 hours and weighed again; this weight was termed as W_3(µg). By taking the means of two measurements at right angles to each other made to an accuracy of ±0.01 mm using digital vernier caliper, diameter and thickness of each specimen was measured. The volume (V) of each specimen was calculated as follows in cubic millimeters using the mean thickness anddiameter:

$$V = \pi \times r^2 \times h$$

Where r is the mean sample radius(diameter/2) in millimeters and h is the mean sample thickness in millimeters. The loss of material (solubility) was obtained from the difference between the initial and final drying mass of each sample(W_1–W_3). The water sorption was obtained from the difference between initial weighing and the wet weighing(W_2–W_1). The values of water sorption (Wsp) and solubility (Wsol), for each sample were calculated using the following equations:

$$Wsp = (W_2 - W_1)/V$$
$$Wsol = (W_1 - W_3)/V$$

Wsp: sorption of test material (µg/mm^3)
Wsol: solubility of test material (µg/mm^3)
W1: weight prior to immersion in artificial saliva.
W2: weight after immersion in artificial saliva.
W3: dehydrated weight.
V is the volume of test material in mm^3.

Results

The data obtained in this study were calculated using the SPSS version 22 with P value < 0.05 was considered statistically significant. The statistical normal distribution suitability of sorption and solubility values was tested using the

Kolmogorov-Smirnov method. Since the obtained values show normal distribution differences between the groups data were analyzed using the one way analysis of variance (ANOVA). Descriptive statistics were used to determine mean, standard deviation, minimum and maximum values between groups. The difference between groups was found to be significant ($p<0.05$). For pairwise evaluation between the groups post hoc dunnet t_3 test was used.

The mean values of sorption and standard deviation for group A_1, B_1 and C1 were 70 ± 2.00, 52.91 ± 1.73 and 81.52 ± 2.22 μg/mm^3 respectively using ANOVA. By using Dunnett t_3 tests, the mean difference is significant at the 0.05 level. It treat one group as a control, and compare all other groups against it. With highest mean sorption value for RMGIC followed by Equiaforte and Zirconomer Improved. The mean values of solubility and standard deviation for group A, B and C were 20.06 ± 2.24, 10.47 ± 1.52 and 33.46 ± 2.01 μg/mm^3 respectively using ANOVA. With highest mean sorption value for RMGIC followed by Equiaforte and Zirconomer improved.

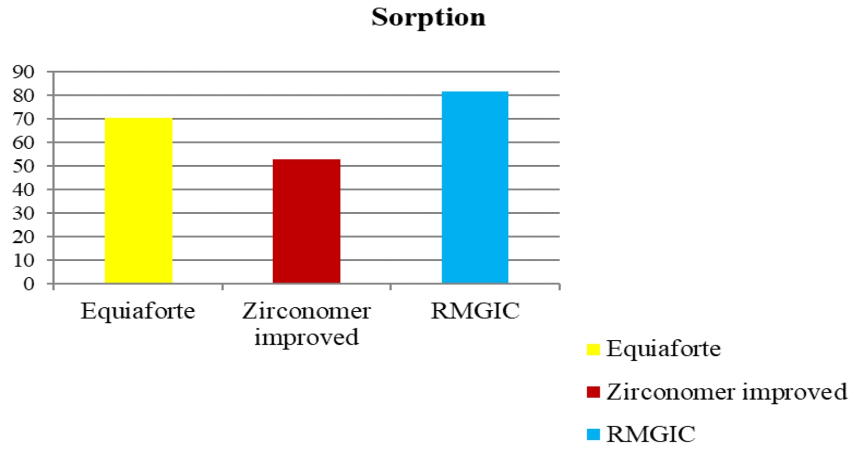
Table 1: Comparison of mean values of Sorption and solubility between three groups- One Way ANOVA test

Variables	Groups	Mean	Std. Dev	95% Confidence Interval for Mean		F value	P value
				Lower Bound	Upper Bound		
Sorption	Group A_1	70.45	2.00	69.34	71.56	35.585	<0.001*
	Group B_1	52.91	1.73	51.95	53.87		
	Group C_1	81.52	2.22	80.29	82.75		
	Total	68.29	12.07	64.66	71.92		
Solubility	Group A_1	20.06	2.24	18.81	21.30	64.832	<0.001*
	Group B_1	10.47	1.52	9.63	11.32		
	Group C_1	33.46	2.01	32.35	34.58		
	Total	21.33	9.72	18.41	24.25		

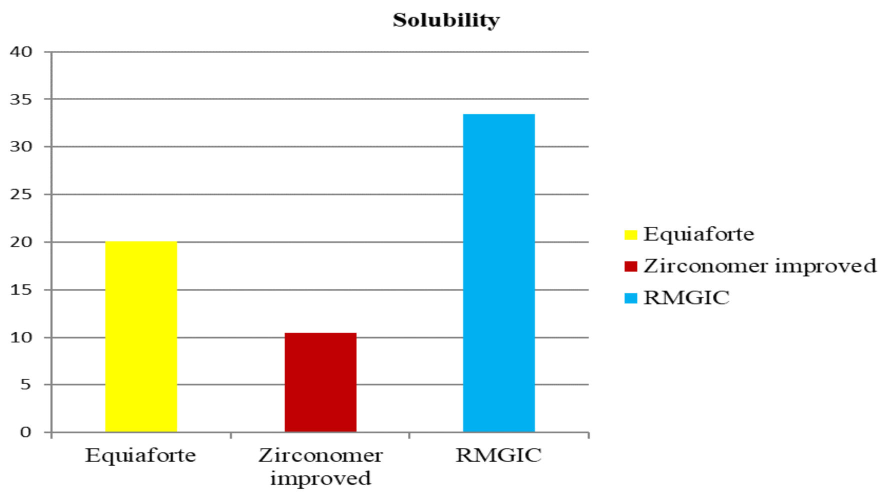
Table 2: Pairwise comparison of sorption and solubility between groups- Dunnett's t3 post hoc test

	(I) material	(J) material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Sorption	Group A_1 (Equiaforte)	Zirconomer improved	17.5400*	.68514	.000	15.80	19.27
		RMGIC	-11.0713*	.77410	.000	-13.03	-9.10
	Group B_1 (Zirconomer	Equiaforte	-17.5400*	.68514	.000	-19.27	-15.80
		RMGIC	-28.6113*	.72899	.000	-30.46	-26.75

	Improved)						
	Group C_1 (RMGIC)	Equiaforte	11.0713*	.77410	.000	9.10	13.03
		Zirconomer improved	28.6113*	.72899	.000	26.75	30.46
Solubility	Group A_1 (Equiaforte)	Zirconomer improved	9.5815*	.70088	.000	7.79	11.37
		RMGIC	-13.4059*	.77852	.000	-15.37	-11.43
	Group B_1 (Zirconomer Improved)	Equiaforte	-9.5815*	.70088	.000	-11.37	-7.79
		RMGIC	-22.9873*	.65246	.000	-24.64	-21.32
	Group C_1(RMGIC)	Equiaforte	13.4059*	.77852	.000	11.43	15.37
		Zirconomer improved	22.9873*	.65246	.000	21.32	24.64



Graph No: 1 Mean values sorption of all the three groups in artificial saliva.



Graph no: 2 Mean values solubility of all the three groups in artificial saliva.

Discussion

Water sorption and solubility is most important in evaluation of adhesive cements clinical durability [8,9,10].Routinely used cements for clinical studies are glass ionomer cements. However, glass ionomer cements have some drawbacks such as early sensitivity to moist or water absorption [11]. Water sorption and dissolution of cement may result in degradation of cement, which leads to debonding of the restoration, recurrent decay, post-operative hypersensitivity, pulpal inflammation and periodontal disease[12]. As a result of water sorption the mechanical properties of the cement, such as flexural strength, hardness and mechanical stability are adversely affected. Added to this, there will be dimensional variation, loss of retention, deterioration of material and discoloration of the esthetic quality of the restoration [13,14,15]. Allegation to dissolution or water sorption during compressive strength reduction was observed.

Adsorption and absorption are the characteristic of sorption. Adsorption is a surface phenomenon while absorption involves penetration of liquid molecules into the structure of the solid material mainly through diffusion [16]. Generally two patterns are known for diffusion of water through materials: one is the pattern following the (free volumetric theory), in which the water diffuses through a micro voids without any mutual relationship to the polar molecules in the material. The other pattern is called (interaction theory), in which water diffuses through material binding successively to the hydrophilic groups. In the case were there was a negative correlation between the diffusion and equilibrium water uptake, the later pattern of diffusion was supposed to occur mainly. Recently it has been assumed that both approaches could be valid, each one for a defined specimen family or both simultaneously [17].

Solubility is the ability of a substance to disperse in another, stated as the concentration of saturated solution of the former in the latter. When solubility is tested, there is no particle in suspension [18]. In the literature it has been reported not all the specimen are equal in size due to variation in dimension of specimen which leads to different periods of time for water to completely infiltrate throughout the polymer matrix[19]. For stabilization, materials which absorbed more water required longer duration and the smaller the specimen, the shorter the period for equilibration with water [20]. Meanwhile water sorption and solubility of materials are mainly based on their relative values and numerical correlations are not always possible[21].

In this study, for sorption and solubility standard test were used involves the storage of cylindrical specimens of materials in artificial saliva for a period of 28 days, the result being quoted as weight gain or weight loss of the specimen respectively. The result revealed water sorption and solubility was higher for resin modified glass ionomer cement (Fuji II LC improved) when compared to glass hybrid restorative system (Equiaforte) and Zirconomer improved. Where, Zirconomer improved exhibited least water sorption and solubility. It may due to variation in filler content between the materials, wear during manipulation process, incorporation of voids during mixing, no protection coating and also hydrophilic nature of RMGIC.

The results are in accordance with the study conducted by Toledano et al (2003), which he attained water sorption and solubility values higher for resin modified glass ionomer (Vitremer and Fuji II LC) when compared with other resin based materials such as Z 100, Prodigy, Compoglass, Compoglass F, Dyract, Dyract AP. It might due to filler content and also hydrophilic constituents such as HEMA increased water sorption values[22]. Nilufer et al (2007) observed solubility of resin modified glass ionomer cements (Vitremer) higher which is similar to present study and the amount of resin solubility could be due to amount of resin affects [23].

Mese et al (2008) studied the amount of sorption and solubility of resin modified glass ionomer cements with other materials and he found that higher water sorption and solubility for RMGIC which is parallel to our study[21]. Significant monomer content of resin modified glass ionomer cements would result in hygroscopic expansion, as observed in there experimental work, and which could lead to adverse forces acting on the tooth structure and the restoration[24]. It is believed hygroscopic expansion may be beneficial by compensating for any polymerization shrinkage, Knobloch et al (2000) and Leevailoj et al(1998) study has shown that resin-modified glass ionomer cements exhibited between 8% and 14% water sorption by weight [20,25,26]. In light of these findings, the use of such cements for the cementation of all-ceramic crowns remains questionable [21]. In correlation with the results of this study, it would seem to indicate that they may not be a good choice of material. Solubility could be due to hydrophilic nature but in comparison with other studies exhibit low solubility than zinc phosphate and polycarboxylate cement [27].

Similar finding was also shown by Dinakaran et al (2014), in his study Fuji II and Fuji II LC improved exhibited higher sorption and solubility values than Dyract in all immersion media [28]. Imbibition of air voids in the material during mixing accelerates the surface to moisture and may lead to inhibition zones with unpolymerized material (Fuji II LC improved) [29]. Matrix dissolution due to loss of the siliceous hydrogel can cause loss of surface hardness of the material [30]. And also, our data were in agreement with previous study conducted by Reshma et al (2016) who compared RMGIC with polyacid modified composite and conventional luting cements and she also gave the same reason as such Dinakaran et al and Mese et al [31].

Glamoc AG et al (2017) reported that resin modified glass ionomer cements (Gc Fuji Plus) has far values of sorption which is in correlating to the present study, it might be mainly influenced by the proportion of hydrophilic nature which holds huge amount of water, differences in the type and composition of the filler, pH value of the solution[32]. Since there are only constricted number of studies reported in the literature respect to Equiaforte and Zirconomer improved with regard to physical and mechanical properties it cannot be compared with other studies. Pertaining to clinical implications properties of resin modified cements such as dimensional changes, hydroscopic expansion leads to fracture of restoration to the tooth root and care must be taken when cementing the post with the root wall which is very thin[26,33].

Consequently the artificial saliva which was used in our study did not contain any enzymes which would present in mouth such as anhydrase, amylase, lysozymes and other esterase [34,35]. Exposure of the material to these enzymes for prolong duration of time might resulted in degradation of surface because of acidic bacteria in oral environment[36]. Therefore higher sorption and solubility values could be reported if future studies done with the use of these enzymes.

Conclusion

In terms of clinical applicability, the values elucidated from this study helps us to determine the quality of employed material. Thematerialsanalyzed inthisstudyinterpreted different levels of sorption and solubility. According to the results, followingconclusions can bedrawn: Zirconomer improved glass ionomer cement absorbedless water and islesssolublethanothe rcements followed by glass hybrid restorative system,andthe highestsorptionandsolubilityvalue inartificialsaliva were seen with resin modified glass ionomer cement. There were significant differences among the tested materials.

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