Physical characteristics of two moisture tolerant fissure sealants immersed in commercial products with different pH range

Naglaa I. Ezzeldin[a], Mona F. Mohamed[b], Ahmed Abdou[c,d]

Departments of [a]Pediatric Dentistry, [b]Conservative Dentistry, Faculty of Dentistry, October University of Modern Sciences and Arts, [c]Department of Biomaterials, Faculty of Dentistry, Modern University for Information and Technology, Cairo, Egypt, [d]Department of Cardiology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

Correspondence to Naglaa I. Ezzeldin, PhD, Faculty of Dentistry, October University of Modern Sciences and Arts, 26 July Mehwar Road Intersection with Wahat Road, 6th of October, Cairo, Egypt
tel: +20 100 198 4184; fax: +20 238 371 543;
e-mail: niezz@msa.eun.eg

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Objective

This study aimed to evaluate and compare between four physical variables (sorption, solubility, color change and fluoride release) for glass ionomer (GI) sealant against moisture tolerant resin sealant in solutions with different pH in different time intervals.

Materials and methods

Thirty discs were prepared from GI Fuji Triage and Embrace WetBond (EWB) resin sealant. Sorption, solubility and color change were measured after different time intervals (1 day, 1 week, and 1 month) of immersion of the specimens in different solution having different pH. In addition 10 disks were assessed for fluoride release of both sealants after being immersed in distilled water.

Results

EWB showed greater sorption in all media after 1 day and 1 week. GI sealant showed highest water sorption after 1 week and this sorption decreased again after 1 month. GI showed greater initial solubility in Pepsi and water after 1 day ($P = 0.016$). Pepsi showed higher solubility for EWB while no significant effect was found for GI with different media. The color change was greater for EWB when immersed in Pepsi and ginger. After 1-month storage, the color change was the highest specially for Pepsi for both sealants. Fluoride release was greater for GI compared to EWB.

Conclusion

Both sealants showed different physical performance depending on the type of immersion media and time period. This is expected to affect their mechanical properties and hence their clinical performance.

Keywords:
dental caries, Embrace WetBond, glass ionomer, pit and fissure sealants, solubility

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Introduction

Using pit and fissure sealants is a noninvasive procedure recommended for preventing caries development or progression in high caries risk patients [1–3]. Although pits and fissure caries has declined in high risk patients, the percentage of decrease is still less than the decrease noticed in smooth surface caries [4]. Despite the effectiveness of sealants in reducing dental caries, they are still under used [5–7]. The less frequent use of sealants may be related to; the lack of certainty about the effectiveness of the sealants particularly when placed on carious lesion, its retention rates and difficulty in managing young aged patients [8–10]. The effectiveness and retention rate of different sealants may be related to their physical properties such as sorption and solubility that may induce an interaction between dental material and oral environment [11].

Glass ionomer (GI) and resin based are major types of sealants frequently used. Their properties may be determined by; type of resin matrix, chemical composition, polymer matrix density, powder liquid ratio and presence of microvoids [12–16].

One of the main points of weakness for any adhesive restoration is moisture sensitivity. Achieving such requirement may be extremely difficult in some clinical situations [17] for instance; improper accessibility, uncooperative patients or early stages of erupting permanent molars [18,19]. In addition, previous studies showed that food and beverages of different pH may induce volumetric changes such as swelling or solubility with subsequent hydrolysis affecting its survival rate in the oral cavity [20,21].

Therefore different materials that are moisture tolerant have been developed. Embrace WetBond (EWB) is fluoride releasing light cured resin with no Bis-GMA monomer [17,18]. Studies showed that the presence of Bis-GMA affect the degree of polymerization which in turn affect physical and mechanical properties [22,23]. It contains multifunctional hydrophilic monomers that is activated when placed on slightly moist acid etched enamel surface [24,25]. After curing the sealant becomes neutral reducing its solubility in water during its clinical service [26]. However there is lack of studies regarding its physical behavior especially when placed in solutions of different pH. On other hand GI has several merits over resin sealant especially when moisture isolation is difficult [27]. This is beside its fluoride release and adhesiveness to tooth structure. However its poor retention and solubility has questioned its success rate [28].

In the view of this background the present study aimed to evaluate and compare between four physical variables (sorption, solubility, color change and fluoride release) for GI sealant against moisture tolerant resin sealant in solutions with different pH in different time intervals. The tested null hypothesis was that there were no statistically significant differences between both sealants when used in different pH solutions.

Materials and methods

Specimen preparation

The composition of pit and fissure sealants used in this study is presented in Table 1. A total 30 discs 9 mm in diameters and 3 mm in thickness (15 discs from each fissure sealant, five specimens for each solution) were prepared using a Teflon mold. The sample size determination was based on previous studies by Abdelfattah *et al.* [29] and Dinakaran [30], a large effect size (f = 0.4) is expected. Using power 90% and 5% significance level total sample size of 30 discs would be sufficient. The sample size was calculated by G* power software, version 3.1.2, for MS Windows (Franz Faul, Kiel University, Germany). The specimens from both sealants were prepared according to manufacturer's instructions and condensed probably in the mold between microscopic glass slide to squeeze the excess material and obtain smooth surface. The EWB specimen was cured using Eliper LED curing light (3 M ESPE, St Paul, Minnesota, USA) for 20 s. After curing, both sides of specimens were finished using 800 grit silicon carbide abrasive paper. Additionally GI Fuji Triage specimens were coated with GC Fuji Varnish (GC Corporation, Tokyo, Japan) and the varnish cured according to the manufacturer's instructions.

Sorption

The test was determined by the procedures described in the ADA specification no. 27 for resin-based filling materials [31]. The specimens were transferred into a glass desiccator containing dehydrated silica gel (Fischer Scientific, Leicester, UK) maintained at 37 ± 1°C and stored for 22 h then transferred to another desiccator for 2 h at 23 ± 1°C. Then the specimens were weighed using an electronic balance with four digits precision (Sartorius AG, Gottingen, Germany). These steps were repeated until constant weight was achieved ± 0.1 mg to ensure complete dehydration (original weight) in 24 h period. The specimens of each fissure sealant were divided into three groups (n = 5) immersed in 10 ml of three solutions having different pH at 37 ± 1°C. The acidic solution was Pepsi (pH 4.5) (Pepsi Cola, Cairo, Egypt S.A.E), the alkaline solution used was prepared from fresh ginger (pH 8) and the neutral solution was distilled water (pH 7). Fresh ginger was washed then softened in sterile mortar, and the homogenate was filtered by using cotton cloth. Finally the material was milled to powder by using blinder. A 20 mg of powder was added to 100 ml boiled distilled water [32,33], the solution was cooled to 37 ± 1°C then filtered. The pH of each solution was measured by pH meter (JENAWY, Model 3505; Stone, Staffs, UK). Each specimen was immersed in 10 ml solution in separate container. The solutions were changed every day. Sorption was assessed by weight changes, which were measured at the following time intervals of immersion: after 1 day, 1 week and 1 month. Sorption was reported in weight percent. The specimens were removed from the solutions; filter paper was used for blot-drying, and then waved in air for 15 s to evacuate any visible moisture. The final wet weight was obtained after 1 min from the removal of the specimen from the solution. The final weight was measured several times until equilibrium was reached. The sorption percentage was calculated as follows [34]:

Table 1 Tested fissure sealants

Sealant name	Composition	Manufacture
Embrace WetBond, Lot: 160210	Matrix: UEDMA, BMEP, HEMA, TMPTMA, H_2O, catalysts, Fillers: SiO2, NaF (37wt %)	Pulpdent Corporation, Watertown, Massachusetts, USA
GC Fuji Triage Lot: 1504142	Glass ionomer, alimuno fluorosilicate glass, polyacrylic acid, distilled water and poly base carboxylic acid	GC Corporation, Tokyo, Japan

BMEP, bis-methacryloyl ethyl phosphate; HEMA, 2-hydroxyethyl methacrylate; TMPTMA, trimethyloyl propane trimethacrylate; UEDMA, aliphatic diurethane dimethacrylate.

$$\text{Sorption}\,\% = \frac{\text{weight gained - original weight}}{\text{original weight}} \times 100$$

Solubility

Solubility was measured by registration of desorption of the specimens that gained solution. This was done by transferring the specimens in a firmly closed glass desiccator containing silica gel, as the methods described previously to obtain the original weight. Solubility percentage was calculated by the measurement of dehydration weight, which represents the amount of leached material from the specimens [34]:

$$\text{Solubility}\,\% = \frac{\text{original weight -dehaydrated weight}}{\text{orginal weight}} \times 100$$

Color change

The color of specimens was measured using a reflective micro-spectrophotometer (X-Rite, model RM200QC; Neu-Isenburg, Germany). Aperture size was set to 4 mm to be aligned with the device, while using a white background and the measurements were taken according to the CIE L*a*b* color space relative to the CIE standard illuminant D65. The color changes (ΔE) of the specimens were calculated by using the following formula [35]:

$$\Delta E_{CIELAB} = (\Delta L*2 + \Delta a*2 + \Delta b*2)½.$$

where $L*$=lightness (0–100), $a*$=change the color of the axis red/green, and $b*$=color variation axis yellow/blue.

Fluoride release

Five discs from each fissure sealant (5 mm diameter, 2 mm thickness) were prepared in Teflon mold. The specimens were kept to set in relative humidity 100% then immersed in a polyethylene tube containing 2 ml of deionized water. The specimens were stored in an incubator with a constant temperature of 37°C, and transferred to new tubes containing new solution every 2 days for 14 days. The released fluoride concentration was measured every 2 days using an ion meter (F-53, Horiba, Kyoto, Japan) attached to fluoride specific electrodes and reference electrodes (2060A and 8010, respectively; Horiba) that had been calibrated using standard 0.05, 0.1, 0.5, 1, 5, and 50 ppm fluoride solutions. Prior to measurement, Total Ionic Strength Adjustment Buffer II (Orion Research, Cambridge, Massachusetts, USA) was added to each solution (1: 1 ratio) and the fluoride electrode readings for each sample solution was recorded and converted to ppm concentration.

Statistical analysis

Statistical analysis was performed with IBM SPSS (SPSS Inc., IBM Corporation, New York, USA) statistics, version 25 for Windows. Kolmogorov–Smirnov test showed a nonparametric distribution of data. Therefore, Kruskal–Wallis test was used to compare between immersion media and time followed by Mann–Whitney U test for pairwise comparison. Mann–Whitney test was used to compare between tested materials. Spearman was used to correlate between tested parameters ($\alpha = 0.05$).

Fluoride release data showed parametric distribution, so repeated measures analysis of variance was used to compare between the different follow-up periods and tested materials. The significance level was set at P value less than or equal to 0.05.

Results

EWB showed greater sorption in all media after 1 day and 1 week compared to GI but after 1 month this sorption was comparable with GI. Whereas the solubility of EWB was lower compared to GI in Pepsi and water after 1 day. Conversely GI showed greater solubility in Pepsi and water ($P = 0.016$) and lower sorption after 1 day and 1 week. In regard to the effect of pH, EWB showed increased sorption in all media,while GI showed increased sorption only in water. As for solubility, EWB showed increased solubility in Pepsi ($P = 0.006$). In contrast GI showed nonsignificant difference in solubility between the three media (Tables 2, 3).

The color change was greater for EWB compared to GI when immersed in Pepsi and ginger. The color change increased after 1 month for both sealants especially for Pepsi (Table 4, Fig. 1). Sorption showed a negative correlation with solubility for both sealants. Color change showed a positive correlation with solubility and negative correlation with sorption for EWB (Table 5).

The highest fluoride release was detected in GI for all time periods. The fluoride release for both sealants was significantly higher after 2 days followed by 4 and 6 days. This was followed by significant decrease in the subsequent periods (Fig. 2).

Table 2 Sorption data for tested fissure sealants

	Embrace WetBond				GI Fuji Triage				*P*
	Mean	Median	Quartile 25%	Quartile 75%	Mean	Median	Quartile 25%	Quartile 75%	
Sorption									
Pepsi									
1 day	5.09[b]	4.86	4.81	5.48	−0.69[bc]	−0.58	−0.965	−0.515	0.016*
1 week	8.02[ab]	7.04	6.495	10.03	−1.56[c]	−0.97	−2.63	−0.79	0.008*
1 month	−0.48[c]	−2.08	−2.585	2.435	−1.45[c]	−1.72	−2.155	−0.62	0.841 NS
Ginger									
1 day	6.17[b]	6.04	4.855	6.8525	0.76[bc]	0.00	−0.18	2.075	0.004*
1 week	10.20[a]	8.24	7.03	14.36	0.67[bc]	0.00	−0.54	2.218	0.008*
1 month	−0.59[c]	−0.55	−1.595	0.385	1.02[bc]	0.72	−0.98	3.3275	0.413 NS
Water									
1 day	5.76[b]	5.60	5.1225	6.3225	−0.34[bc]	−0.53	−0.59	0	0.004*
1 week	9.48[a]	9.77	6.675	11.4675	3.89[a]	3.86	1.11	6.69	0.048*
1 month	−0.47[c]	−0.63	−1.2275	0.2275	1.23[b]	0.94	−0.82	3.5675	0.352 NS
P			≤0.001*				0.009*		

GI, glass ionomer. Different letters within each column are significant at *P* value less than or equal to 0.05. **P* 0.05.

Table 3 Solubility data for tested fissure sealants

	Embrace WetBond				GI Fuji Triage				*P*
	Mean	Median	Quartile 25%	Quartile 75%	Mean	Median	Quartile 25%	Quartile 75%	
Solubility									
Pepsi									
1 day	−0.37[b]	−0.45	−3.525	2.825	4.84	5.57	3.715	5.6	0.016*
1 week	5.83[a]	5.48	3.885	8.1325	4.34	4.17	3.26	5.59	0.629 NS
1 month	5.22[a]	5.88	2.785	6.98	6.34	4.10	3.45	11.4825	0.889 NS
Ginger									
1 day	−1.14[c]	−3.13	−4.655	2.975	2.74	2.46	0.3925	5.3525	0.257 NS
1 week	0.63[b]	1.45	−3.84	4.68	1.67	1.04	0.845	3.115	1.00 NS
1 month	3.63[ab]	3.29	2.5	4.925	1.90	1.34	1.1925	3.165	0.190 NS
Water									
1 day	−3.09[c]	−2.41	−4.805	−1.725	1.16	0.91	0.6125	1.9425	0.016*
1 week	1.54[ab]	2.34	−2.395	3.9575	2.72	1.11	0.645	5.605	0.931 NS
1 month	4.52[a]	3.80	3.0175	6.38	2.52	1.11	0.645	5.1	0.082 NS
P			0.006*				0.068 NS		

GI, glass ionomer; NS, nonsignificant.Different letters within each column are significant at *P* vale less than or equal to 0.05. **P* 0.05.

Discussion

Despite the documented efficiency of clinical guidelines for the placement of pit and fissure sealants [36–38], several studies [4,39,40] showed that a dispute still exists regarding the use of GI over resin based composite as fissure sealants. In addition Azarpazhooh and Main [36] recommended that sealants should be postponed if isolation cannot be achieved as this would affect the retention rate.

An attempt to solve the problem of field isolation hydrophilic resin sealant has been developed. The data available on EWB is limited particularly physical properties and its comparison to glass ionomer.

According to the present study, the null hypothesis was rejected as EWB showed increased sorption either when placed in solutions with different pH or when compared to GI Fuji Triage. The possible explanation was that water sorption took place through the intermolecular spaces of the polymeric matrix. In addition the composition of EWB may facilitate this sorption. It contains bis-methacryloyl ethyl phosphate; an acidic monomer resembling self-etch adhesives and 2-hydroxyethyl methacrylate (HEMA) which increases the wettability of the material on the tooth surface [41]. Both monomers contain polar groups (phosphate group in bis-methacryloyl ethyl phosphate and hydroxyl group in HEMA) that form hydrogen bonds with water resulting in 'bound water' that cannot be easily eliminated [42,43]. Also it contains TMPTMA; a low viscosity monomer that contains three methacrylate groups in each molecule making it a good cross-linking agent between the polymerized chains [44]. Although it contains many double carbon bonds than monomers containing difunctional groups, such increase in double bonds may enhance the reactivity of the monomer to the surrounding media.

Sorption in polymeric material is usually complicated with solubility. However, sorption is faster than

Table 4 ΔE data for tested fissure sealants

	Embrace WetBond				GI Fuji Triage				*P*
	Mean	Median	Quartile 25%	Quartile 75%	Mean	Median	Quartile 25%	Quartile 75%	
Δ*E*									
Pepsi									
1 day	2.49^{d}	2.82	1.565	3.24	3.93^{c}	4.12	3.07	4.695	0.032*
1 week	9.20^{b}	8.13	5.465	13.48	3.08^{c}	3.20	2.325	3.785	0.008*
1 month	20.69^{a}	19.83	17.4775	25.91	11.68^{a}	9.26	8.015	16.55	0.03*
Ginger									
1 day	3.86^{cd}	3.71	2.985	4.79	4.63^{bc}	3.73	3.2875	6.88	1.00 NS
1 week	6.20^{bc}	6.27	5.665	6.71	3.53^{c}	3.13	2.68	4.765	0.032*
1 month	20.17^{a}	18.82	16.39	24.63	4.25^{bc}	4.04	3.395	5.315	0.016*
Water									
1 day	3.48^{cd}	3.44	3.245	3.685	4.63^{bc}	5.26	2.53	6.0975	0.257 NS
1 week	5.11^{bc}	5.01	3.68	6.495	6.18^{bc}	5.60	5.05	7.6	0.329 NS
1 month	5.91^{bc}	6.03	5.295	6.585	8.29^{ab}	6.73	3.58	13.775	0.662 NS
P			≤0.001*				0.008*		

GI, glass ionomer; NS, nonsignificant. Different letters within each column are significant at *P* value less than or equal to 0.05. **P* 0.05.

Figure 1

Color change for tested fissure sealants.

solubility; there was initial weight gain until full saturation followed by weight loss due to solubility [29]. This might explain the negative correlation found between sorption and solubility. The water sorption permits the hydrolysis of different types of bonds such as ethers, amides or ester bonds present in long polymer chain into smaller chains or individual monomers [45]. This may have led to development of internal stresses and increase the microgaps present in the polymer network allowing the free unreacted monomers and hydrolyzed components to diffuse outward into the surrounding media [46]. This can be expressed as degree of solubility. This was in agreement with Abdelfattah *et al.* [29] and Gavranovi *et al.* [47]. Concerning the effect of pH, EWB showed greater solubility in Pepsi compared to ginger and distilled water, this was in accordance with Giti *et al.* [48]. The acidic solution diffuses into the polymer matrix providing sufficient amount of H^{+} ions which may induce the hydrolysis of ester group in resin matrix namely HEMA cleaving it into methacrylic acid and ethylene glycol. This allows more diffusion of water and consequently increases solubility [49].

On the other hand GI showed increased solubility in Pepsi and water compared to EWB. Regarding the effect of pH, the solubility of GI was nonsignificant in the three media.

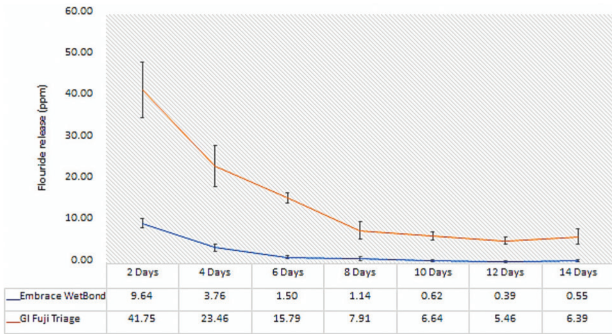
GI Fuji Triage was chosen in this study since it showed higher degree of conversion compared to other resin base materials [50]. It is of low viscosity which may improve its penetration into pit and fissure thus improving its adaptation [51,52]. Moreover, Triage showed high fluoride release [50,53] that may be effective in protecting enamel against demineralization [54].

Table 5 Spearman correlation coefficient for tested parameters

	Variables	By variable	Spearman ρ	P
Embrace WetBond	Solubility	Sorption	−0.3213	0.0178*
	ΔE	Sorption	−0.306	0.0244*
	ΔE	Solubility	0.4799	0.0002*
GI Fuji Triage	Solubility	Sorption	−0.7111	<0.0001*
	ΔE	Sorption	−0.018	0.9065 NS
	ΔE	Solubility	0.1587	0.2978 NS

GI, glass ionomer; NS, nonsignificant. Significant at *P* value less than or equal to 0.05. **P* 0.05.

Figure 2



Fluoride release for tested fissure sealants.

The disintegration of GI in different immersion solutions take place in three steps [55]: superficial surface wash off followed by water diffusion into the GI. This is proceeded by the outward diffusion of the dissolved components into the surrounding media causing surface corrosion [56]. The surface wash off can be credited for the high level of fluoride release. This was in consistent with fluoride release measurement in the present study. GI reported a significant higher fluoride release compared to EWB. This was inconsistent with Neelakantan *et al.* [57] and Markovic *et al.* [58]. In addition the high fluoride release was noticed during the first 6 days in particular at the first 2 days due the phenomena of 'burst effect' followed by slower fluoride release. This finding was in agreement with Poggio *et al.* [53] and Attar and Turgut [59]. This phenomena may have occurred in the three media which might explain the nonsignificant difference in solubility between them. The greater solubility of GI in Pepsi and water when compared to EWB at first day may be related to two factors; the varnish used for coating the GI samples is composed of isopropyl acetate which is moderately soluble in water enhancing the release of different ions [60]. Secondly, to neutralize the acidity of the surrounding media multiple ions such as fluoride and calcium may be released [61].

The sorption in GI was lower than EWB which may be contributed to the difference in the composition. GI showed a large numbers of cross-links between the polymers chains, these cross-links reduced the spaces and water flow inside the materials at first days [62,63]. However the outward diffusion of ions, created pores within the GI allowing inward diffusion of different components from the surrounding media [64]. This was in agreement with the negative correlation between sorption and solubility found in this study and also contribute to the increased water sorption of GI after 1 week compared to Pepsi and ginger. Incorporation of water into GI can occur through several sites such as reaction with Si-O-Si units at the surface of glass particles leading to formation of -Si-OH group [15] or the exchange that may take place between fluoride ion and the hydroxyl group from the surrounding aqueous media [65]. In addition water has low osmotic pressure compared to the ginger solution and high sugar content of Pepsi [63,66], thus increasing water sorption. These results were agreed by Lima *et al.* [63] In contrast Dinakaran [30] revealed that the solubility of the GI increased in acidic media and showed high sorption at all storage media. In Dinakaran [30] study GI was used in powder and liquid form rather than capsules. The procedure of mixing may produce air voides which increase moisture sensitivity. In addition sorption and solubility were measured after 7 days rather than 1 day as in the present study. In the present study, the less sorption noticed in Pepsi may also be related to chelation of phosphoric acid in Pepsi with calcium in GI forming insoluble complexes [67]. In addition the polyacrylic acid polymers in GI may have selective permeability for the diffusion of alkaline media [68].

Color change can be used as indicator for external or internal changes taking place within the restorative materials [69]. When the color change is greater than 3.3 it becomes detectable by human eye [70,71]. In this study the color change for both sealants ranged between 3.3 up to 20. The greater color change detected in EWB in alkaline or acidic media may be related to the increase in H^+ or OH^- ions causing the degradation of different components or silane interface leading to subsurface damage. This was followed by leaching out of hydrolyzed components creating voids allowing diffusion of different stains [69,72]. This may also explain the positive correlation found between solubility and color change. This was in agreement with Gavranovi *et al.* [47]. Moreover, the low filler content and incomplete degree of conversion exhibited in EWB may facilitate the passage of discoloring agents leading to greater color change [41,73]. This was disagreed by Moon *et al.* [74] as they showed lower color change in alkaline and acidic media. The difference may be related to the use of different immersion media and different type of resin composite.

Concerning GI, acidic solution had greater impact on color change compared to ginger and distilled water. This finding was in agreement with Gondim *et al.* [67] and Ahmed and Sajjan [75]. The H^+ ions diffuses into the polymer matrix of GI causing the release of metal ions that are cross linked with carboxyl group [55]. This structural change may create components with different refractive indexes affecting the pattern of light reflection [76]. In conclusion, EWB did not show any superiority over GI at different pH solutions or periods. At the same time the greater fluoride release from GI can be more beneficial to the surrounding environment.

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Conflicts of interest

There are no conflicts of interest.

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