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HYGROSCOPIC/HYDROLYTIC KINETICS OF TWO BULK-FILL RESIN COMPOSITE RESTORATIVE MATERIALS STORED IN DIFFERENT MEDIA: AN INVITRO COMPARATIVE STUDY

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ABSTRACT

Objectives: The aim of this study was to determine the effect of different storage media on Sorption/solubility kinetics of two bulk-fill resin composites, Venus bulk fill flowable resin composite (VF) and Sonic Fill bulk fill resin composite (SF), stored in different media (alkaline water Aw, 75% ethyl alcohol EA and lemon juice LJ)**.**

Materials and Methods: One hundred and twenty disc specimens were fabricated and divided into two main groups (n=60) involve two bulk-fill resin composites, depending on their low viscosity. The specimens in each group were divided into three subgroups according to the storage media (n=20) i.e. alkaline drinking water, 75% ethyl alcohol and lemon juice. All specimens were desiccated before storage to obtain a constant mass (m_1) and volume (V) in mm^3. Specimens were subjected to 4 weeks immersion, then dried and reweighed again to obtain (m_2). The specimens were desiccated again to obtain constant mass (m_3)**.** Sorption and solubility's properties' in water were calculated according to proposed formula by ISO 4049- 2009.

Results: Two-way ANOVA indicated significant difference in sorption and solubility kinetics for both factors, resin composites and immersion media ($p<0.05$). Tukey's test showed that VF significantly absorb fluids than SF, regardless the storage media. The mean weight changes in the VF resin composite showed increasing fluid sorption after storage in AW, EA and LJ. There were significant differences between either AW or EA and LJ. For SF, fluid sorption is increased from AW, followed by EA and LJ. Solubility of VF was significantly higher than SF, when stored in each immersion medium. The mean weight loss in the VF resin composite showed insignificant increasing solubility after immersion in AW, LJ and EA. The mean weight loss for SF revealed increasing solubility after immersion in AW, LJ and EA. VF revealed higher significant fluid solubility when stored in water, than in EA or LJ. EA cause significantly higher solubility of SF resin composite, than AW or LJ storage media,

Conclusions: Within the conditions of this *in-vitro* study, the following conclusions were drawn: The sorption and solubility behaviour of the resin composites is material dependent, as Venus flow bulk-fill resin composite tested in this study showed higher fluids sorption/solubility tendency than that of Sonicfill bulk-fill resin composite. Regarding to the storage media, pH of the solutions seems to have an influence on the sorption/solubility behavior of composite resin materials. Both acidic and alkaline media increase sorption/solubility behaviour of the resin composites.

KEYWORDS: Bulk-Fill resin composite, storage media, Sorption/solubility behavior.

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INTRODUCTION

In recent decades, the face esthetics, including teeth, became of a great interest. In this context, tooth colored restorations have been widely used and encouraged by both the patients and dentists, not only in the anterior teeth, but also in the posterior region. The outspread of dental resin composites is for their esthetic properties, as well for their ability to be bonded to tooth structures.[1] Thence; great evolutions were accomplished in the resin composite restorative materials, since they were introduced to dental markets, to improve their performance, with simplifying their application, avoiding technique sensitivity. One of these developments is the introduction of bulk-fill resin composites.

The bulk fill resin composites have been intended to improve restoration properties, such as reducing polymerization contraction with its associated stresses [2,3], and speed-up their application by reducing number of increments.[4] These products enabled a depth of cure approximately 4-6 mm, without compromising the degree of conversion.[5] This increase in depth of cure may be achieved by improving viscosity of the bulk-fill resin composites. Two methods have been used by the manufacturers to decrease the material viscosity during insertion. The first one was through introduction of flowable bulk-fill resin composites, containing lower filler content and enlarged filler size.[6] An example for this method is Venus bulk fill flowable resin composite. The second was achieved by the development of SonicFill bulk-fill resin composite with its sonic activation handpiece. The material is sonically activated to be rendered like a flowable composite during application, but retain its viscosity after that.[7] The bulk fill resin composites are somewhat recent materials, with controversies about their physical and mechanical properties.[6,8-11]

Although resin composites are well-established as esthetically accepted restorative materials, biodegradation is one of the weak points encountered in the materials. These materials may absorb significant amounts of liquids, when exposed to different aqueous environments, affecting the restorations' physical, chemical and mechanical properties.[12-16] Two different mechanisms can influence hygroscopic/hydrolytic behavior of resin composite restorations; the first one is the water uptake that produce material expansion and swelling, to be followed by the second mechanism in which softening and leaching of the materials, leading to degradation of the final restorations.

One of the challenging factors for restoration durability is the reduction in resistance to resin hygroscopic/hydrolytic changes. Through the available reviews, the data available comparing the sorption/solubility behavior of bulk-fill resin composites[16-22], are currently limited. Hence, out of the previous introduction, a laboratory study to determine the effect of different consumed liquids, alkaline drinking water, ethyl alcohol and lemon juice on Sorption/solubility behavior of two different bulk-fill resin based composites**,** may be of value.

MATERIALS AND METHODS

Materials selection and specimen preparation

In this study, two dental resin composites were selected, on the basis of filler content, matrix monomer variations and flowability, within the major category of bulk-fill resin based composites: Venus bulk fill flowable resin composite (VF) in a universal shade and Sonic Fill bulk fill (SF) in A_2 shade (table 1). Materials' description are presented in Table 1. Three storage media (Table 2) were selected representing some of the usual daily intakes, including Flo alkaline spring water (AW), 75% ethyle alcohol (EA) and lemon juice (LJ).

Total 120 disc specimens, 5mm in diameter and 4mm in hight, were constructed in two groups (n=60) relative to the bulk-fill based resin composites. In group 1, Venus bulk fill resin composite was

TABLE (1) Restorative Materials used

Material	Composition	Manufacturer
Venus bulk fill flowable resin composite	UDMA, EBPDMA Filler (65wt%, 38vol%): Ba-Al-F-Si Glass, ytterbiumtrifluoride SiO2	Heraeus Kulzer, Hanau, Germany
Sonic Fill bulk fill resin composite	Bis-GMA, TEGDMA, Bis-EMA, SIMA Filler (84wt%, 66.8vol%) SiO2 glass	Kerr Hawe S.A, Bioggio, Switzerland

Bis-GMA, bisphenol-glycidyl-methacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, bisphenol-a-ethoxydimethacrylate; SIMA, siloxane-dimethacrylate; UDMA, urethane-dimethacrylate; EBPDMA, ethoxylated bisphenol A dimethacrylate.

TABLE (2) Storage media

Storage media	Description/ preparation	Manufacturer/source
Flo alkaline spring water	Mineral alkaline water with dissolved minerals: each 100 ml contains 29 mg Mg, 2mg K, 73mg Ca and 298 HCO_3; at pH=8.1	Flo Water, Family owned artesian Spring, Bruce County, Ontario, Canada
Ethyl Alcohol	75% Ethanol/water solution	Scharlab SL, Barcelona, Spain
Lemon juice Citrus aurantiifolia	Freshly squeezed juice, Composed of: citric acid, malic acid, ascorbic acid, polyphenols, terpenes, and tannins; at pH=2.	Prepared in Faculty of Dentistry, Mansura university.

used and Sonic-fill resin composite in group 2 (Table 1). All specimens were constructed in a single increment of 4 mm thickness, in a split teflon mold. For group 2, SF material was sonically activated by a SonicFill handpiece, to convert the material to a low flowable consistancy during insertion. After activation, in order to ensure complete loading of the mold and prevent formation of oxygen inhibited layer during polymerization, each specimen, in the two tested groups, was compressed between two transparent celluloid strips, supported with microscopic glass slides with the composite material was packed under a constant weight of 500g. The weight and the glass slides are removed just before light curing of the specimens. All the specimens were subjected to LED curing at WL 430-480nm and intensity of 1200 mW/cm^2 (*Elipar S10, 3M ESPE AG, Seefeld, Germany*) for 20 seconds, with additional10 sec at the bottom. The intensity of the photo-curing unite was periodically verified to assure even curing of the specimens, with the help of a radiometer (*Ecel RD-7, Ribeirao preto SP, Brazil)*. To ensure optimum polymerization, all specimens were then stored in distilled water at 37±1°C in light-proof containers for 24h.

Thereafter, all specimens were desiccated in container incorporated a dehydrated silica gel (Quimidrol Comercio e Industria ImportacaoLtda, Joinvilla, SC, Brazil) and incubated at 37±1°C for 24 h. During desiccation, specimens were periodically weighed on an analytical balance accurate to ± 0.0001 g (*ae adam AEP 650G 1 FOX Hollow Road, Oxford CT 06478 USA)*, until the loss of the specimen mass was less than± 0.0001g in a period of 24 hours, giving a constant mass ***(m_1)***. After complete dehydration, each specimen dimensions were measured to calculate its volume ***(V)*** in mm^3.

The specimens in each group were further divided into 3 subgroups (n=20) according to the storage media i.e. alkaline drinking water (subgroup a), 75% ethyl alcohol (subgoup b) and lemon juice (subgroup c). Specimens in each subgroup were subjected to four weeks storage, in an incubator at 37±1°C, in the 3 different media. The storage media were changed every 24 hours. After storage, the specimens were removed, washed and light dried using paper napkins. All specimens were reweighed to obtain the mass after sorption ***(m_2)***. The specimens were desiccated again to obtain constant mass ***(m_3)*** following the steps discussed for ***m_1***. Sorption and solubility properties in water were calculated according to the formula proposed by ISO 4049- 2009. Water sorption (***W_{sp}***) was calculated in micrograms per cubic millimeter applying the following equation ***$W_{sp}= m_2- m_3/V$,*** wherein ***m_2***: was the samples' mass in micrograms after immersion in water for four weeks. ***m_3***: was the reconditioned samples' mass in micrograms after desiccation at the second time. ***V***: was the samples' volume in mm^3. Water solubility (***W_{sl}***) value was calculated in micrograms per cubic millimeter applying the following equation ***$Ws_l=m_1-m_3\backslash V$,*** Wherein ***m_1***: was the samples' mass in micrograms before immersion in water for four weeks.

Statistical analysis

The collected data obtained for each subgroup were tabulated and subjected initially to ANOVA (two- way) test, followed by Multiple Comparison post Hoc Test (Tukey) , $P< 0.05$.

RESULTS

Statestical results of fluid sorption and solubility for the two bulk-fill resin composites are displayed in Tables 3-6. For fluid sorption, two-way ANOVA indicated significant difference for both factors, resin composites and immersion media($p < 0.05$), table 3. Tukey's test showed that VF significantly absorb fluids than SF, regardless the storage media. The mean weight changes in the VF resin composite showed increasing fluid sorption after storage in AW (14.359), EA (15.214) and LJ (20.681). There are no significant differences between groups stored in AW and EA. There are significant differences between either AW or EA and LJ. For SF, fluid sorption are increased from AW (7.328), to be followed by EA (9.465) and LJ (12.545) table 4.

For fluid solubility, two-way ANOVA indicated significant difference for the resin composite and immersion media factors ($p < 0.05$), table 5. Tukey's test showed that solubility of VF was significantly higher than SF, when stored in each immersion medium. The mean weight loss in the VF resin composite showed increasing solubility after immersion in AW (4.102), LJ (6.919) and EA (7.151), with significant differences between AW storage and the other media. The mean weight loss for SF revealed increasing solubility

TABLE (3) Two-Way ANOVA of Sorption test Results (Both factors significantly influenced the results)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2208.563[a]	5	441.713	458.054	.000
Intercept	21125.044	1	21125.044	21906.552	.000
Var1	1460.426	1	1460.426	1514.453	.000
Var2	720.293	2	360.146	373.470	.000
Var1 * Var2	27.845	2	13.922	14.437	.000
Error	109.933	114	.964		
Total	23443.541	120			
Corrected Total	2318.496	119			

TABLE (4) Mean values of fluid sorption (mg) in Venus and Sonic-fill resin composites, after storage in different media

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean	
					Lower Bound	Upper Bound
VF_AW	20	14.3745[b]	.92298	.20638	13.9425	14.8065
VF_EA	20	15.2145[b]	1.10817	.24779	14.6959	15.7331
VF_LJ	20	20.6810[a]	1.14627	.25631	20.1445	21.2175
SF_AW	20	7.3145[e]	.90929	.20332	6.8889	7.7401
SF_EA	20	9.4565[d]	.75228	.16822	9.1044	9.8086
SF_LJ	20	12.5675[c]	.99966	.22353	12.0996	13.0354
Total	120	13.2681	4.41397	.40294	12.4702	14.0659

Different superscript letters indicate significant differences between pairs within the same column. (Tukey's, P<0.05)
Different superscript numbers in rows indicated significant differences between values within the same group. (Tukey's, P<0.05)

TABLE (5) Two-Way ANOVA of Solubility test Results (Both factors significantly influenced the results)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	526.061[a]	5	105.212	118.613	.000
Intercept	2394.133	1	2394.133	2699.077	.000
Var1	362.269	1	362.269	408.411	.000
Var2	134.448	2	67.224	75.787	.000
Var1 * Var2	29.344	2	14.672	16.541	.000
Error	101.120	114	.887		
Total	3021.315	120			
Corrected Total	627.182	119			

TABLE (6) Mean values of fluid solubility (mg) in Venus and Sonic-fill resin composites, after storage in different media

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
VF_AW	20	4.2415[b]	.95547	.21365	3.7943	4.6887	2.79	5.41
VF_EA	20	7.1515[a]	1.05854	.23670	6.6561	7.6469	5.42	9.09
VF_LJ	20	7.2195[a]	.92399	.20661	6.7871	7.6519	5.97	9.10
SF_AW	20	1.8190[c]	.95717	.21403	1.3710	2.2670	.61	4.00
SF_EA	20	3.9480[b]	.90590	.20257	3.5240	4.3720	1.96	5.12
SF_LJ	20	2.4205[c]	.83553	.18683	2.0295	2.8115	.85	4.11
Total	120	4.4667	2.29574	.20957	4.0517	4.8816	.61	9.10

Different superscript letters indicate significant differences between pairs within the same column. (Tukey's, P<0.05)
Different superscript numbers in rows indicated significant differences between values within the same group. (Tukey's, P<0.05)

after immersion in AW (1.585), LJ (2.421) and EA (3.948). VF revealed higher significant fluid solubility when stored in water, than in EA or LJ. EA cause significantly higher solubility of SF resin composite, than AW or LJ storage media, Table 6.

DISCUSSION

Revolutions in the resin composite and adhesive technologies have increased their clinical use. In this context, bulk-fill resin composites were developed to overcome the worst flaw in incrementally applied resin composites, the time consumption and technique sensitivity. Bulk-fill resin composites used in this study induce a depth of cure reaching 4 mm due to improved material viscosity.

During planning for a restoration, both the patient and the clinician are seeking for a durable restoration with acceptable esthetic. As resin composite restorations are usually exposed to several challenges, including the deleterious effects of fluids sorption/solubility; their physical and mechanical properties may decline. These circumstances can permanently compromise their performance, leading to clinical drawbacks.[22]

In the current study, the values of fluids sorption/solubility of VF resin composite were significantly higher than that of SF composite, regardless the storage media. This can be attributed to the composition differences between the materials. The increased resin matrix volume is the keyword of the obtained results. VF resin composite tested in this study has the lower filler content, 38vol%, when compared with SF, 66vol%. The increased monomeric volume resulted in increased water sorption, as it is mainly promoted by the resin matrix.[17,23] Likewise, the hydrophilicity of the resin composites is determined by the monomers contained in the resin matrix. Water sorption of different monomers is decreased as follow, TEGDMA, Bis-GMA, UDMA, then Bis-EMA.[18,19,21] Although SF included hydrophilic monomers, TEGDMA and Bis-GMA in its composition, the presence of the less hydrophobic monomer Bis-EMA in conjunction with hydrophobic siloxan[24] containing monomer SIMA, does result in reduction of the water sorption of SF.

Water penetrates in the resin composites into two distinct forms, the grain unboubded form freely present between the nanopores created during polymerization and the polymer chains; and the bound form, chemically interact with the hydrophilic groups in the polymer chains, via hydrogen bond.[25,26] Hydroxyl groups, in bis-GMA and UDMA monomers, formed these strong hydrogen bonds with water molecules, that also could explain the high value of water absorbency of VF. These monomers favored water sorption and increased the hydrolytic degradation velocity.[18,20,22] It is thought that resin-based composites with metallo-silica glasses, existing in the VF bulk-fill resin composite, are less stable than that with SiO2 filler, present in SF. Metal ions, melted in these metallo-silica glasses, are more readily leached into water, and be replaced by the smaller H^+ ions in the silicone and oxygen network.[27]

Alshali et al,[21] are in conformity with the present study and concluded that Water sorption and solubility of resin-composites are material-dependent and highly affected by the filler loading and hydrophilicity of the resin matrix. Misilli and Gonulol [22] disagree with these results and reported that the xtra power mode for SonicFill resulted in the worst performance in terms of water sorption. SonicFill revealed decrease in solubility, irrespective of the curing modes.

Different storage media were compared in this study, including Flo alkaline spring water (pH=8.1) was used to represent the alkaline medium. A 75% ethyl alcohol was used to simulate the alcohol containing mouth washes. Lemon juice (pH=2) was used in this study to represent the acidic beverages commonly consumed. From the results of this study, the mean fluids sorption/solubility of the two tested materials showed increasing order of water sorption from AW, EA to LJ, regardless the resin composite. Whilst for fluid solubility, it was found to become AW, LJ to EA.

The deteriorating and softening effects of alkaline spring water resin composite is due to its high alkalinity (pH=8.1). Resin composites underwent moderate softening after storage in alkaline media.[28,29] The sorption/solubility effect of the alkaline media on resin composite is due to its interaction with OH^- ions during the hydrolytic process, accelerating resin degradation. Ester bonds in dimethacrylate resin polymers are susceptible to hydrolytic degradation, with the formation of methacrylic acid and formaldehyde as by-products. Hence, expecting that the alkaline media would catalyse these reactions, is reasonable.[30-32] In addition, the metal ions of the filler particles may be excessively hydrolyse in the excess OH^- ions. These alkaline reactions may compromize the hydrolytic stability of silane coupling agents in the presence of water.[33]

Lemon juice is a widely consumed acidic beverage (pH=2). The sorption/solubility behavior of resin-based restorative materials increased when exposed to acidic soft drinks.[34,35] This can be attributed to the capability of acidic solutions to soften resin-based restorative materials. Acid challenge leads to massive micromorphological changes in the matrix of the resin-based restorative materials. These changes are significantly greater than that noticed with water, the case in this study.[36,37]

Ethanol is usually added to mouthrinses to act as a solvent, taste enhancer and as an antiseptic. Organic solvents like ethanol have the possibility for resin degradation and softning[38], by removing the polymer structures, such as unreacted monomer, oligomers and linear polymers[39], plasticizing the resin matrix[40,41] and facilitating the fluids sorption/solubility.[42,43] The solubility of monomers in organic solvents was higher than that in water.[44] The results of the present study was in agreement with Leal J P et al[43] who concluded that the sorption and solubility of tested composites were higher in mouthwashes containing alcohol.

According to ISO standard 4049-2009, the maximum acceptable and critical values of sorption (Wsp) are 40 µg/mm^3 and solubility (Wsl) 7.5 µg/mm^3 for polymeric restorative materials. An increase in these values above the critical points adversely affects the physical and mechanical properties of these materials. All the results of this study are within these ranges.

CONCLUSIONS

Within the conditions of this *in-vitro* study, the following conclusions were drawn:

1. The sorption and solubility behavior of the bulk-fill resin composites is material dependent, as Venus flowable bulk-fill resin composite tested in this study showed higher fluids sorption/solubility tendency than that of Sonicfill bulk-fill resin composite.
2. Regarding to the storage media, pH of the solutions seems to have an influence on the sorption/solubility behaviour of composite resin materials. Both acidic and alkaline media increase sorption/solubility behaviour of the bulk-fill resin composites.

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