Physical Properties of Different Orthodontic Composites

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ABSTRACT

Objective: The aim of this study was to compare the water sorption, solubility, and compressive strength of 3 traditional orthodontic composites and 1 flowable composite after 1, 7, and 30 days of immersion in water.

Materials and Method: The traditional orthodontic composites used in this study were ORTHO Bracket Paste (Bisco, Schaumburg, IL, USA), Transbond XT (3M, Monrovia, CA, USA), and Light Bond (Reliance, Itasca, IL, USA). The flowable composite used was Tetric EvoFlow (Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were subjected to water sorption and solubility tests based on the ISO 4049 and ADA No. 8 requirements. Mechanical tests were performed with an Instron Universal Test Machine.

Results: There were no significant differences in solubility among the 4 materials after 1 and 7 days ($p > 0.05$). Tetric EvoFlow had a lower compressive strength than the traditional orthodontic composites ($p > 0.05$) and exhibited significantly higher water sorption than the traditional orthodontic composites after immersing them in water for 1, 7, and 30 days ($p < 0.001$).

Conclusion: The results revealed that the flowable composite had higher solubility and water absorption but less compressive strength than the traditional orthodontic composite resins used in this study. (*Turkish J Orthod* 2014;27:28–33)

KEY WORDS: Composite, Orthodontic, Strength, Water

INTRODUCTION

Orthodontic composite materials consist of filler particles, a polymer matrix based on different monomers and/or oligomers of various kinds of acrylates, and additives. Because of problems such as inadequate polymerization or the presence of oral liquids, the orthodontic composite can degrade or erode.[1,2]

The degradation process is related to the degree of cross-linking in the polymerized matrix, the chemical structure of the monomers and oligomers, the environmental impact, or the conversion level.[3] Water plays an important role in degradation because it diffuses into the material and triggers the oxidation and hydrolysis stages of degradation.[4] Water sorption in orthodontic composites is a type of diffusion-controlled process and, according to Bra-den,[5] it occurs in the resin matrix. Water diffusion also leads to erosion of the unreacted monomers and filler particles in the composite material.[6,7]

Degradation and erosion of the composite material leads to mechanical and clinical problems.[8,9] Thus, the aim of this study was to understand the degradation and erosion behaviors of orthodontic composite materials that have not been researched before.

MATERIALS AND METHODS

The 4 traditional and commercially available orthodontic composites used in this study were ORTHO Bracket Paste (Bisco, Schaumburg, IL, USA), Transbond XT (3M, Monrovia, CA, USA), Light Bond (Reliance, Worth, IL, USA), and the flowable composite Tetric EvoFlow (Ivoclar Vivadent, Schaan, Liechtenstein) (Table 1). The immersion

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Table 1. Orthodontic composite materials used in this study[a]

Group Name	Composite	Manufacturer	Components
Group BO	Bisco Ortho Bracket Paste	Bisco, Schaumburg, IL, USA	Fused silica, UDMA, TEGDMA
Group XT	Transbond XT	3M, Monrovia, CA, USA	Silane treated quartz, BisGMA, BisBMA, silane treated silica, Diphenyliodonium hexafluorophosphate
Group LB	Light Bond	Reliance, Itasca, IL, USA	BisGMA, fused silica, amorphous silica, sodium floride
Group TEF	Tetric Evo Flow	Ivoclar Vivadent, Schaan, Liechtenstein	BisGMA, UDMA, DDMA, prepolymer, barium glass, ytterbium trifluoride, inorganic fillers

[a] UDMA indicates urethane dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; BisGMA, bisphenol-A glycidyl dimethacrylate; DDMA, decamethylene dimethacrylate; BisBMA, bisphenol-A benzyl-dimethacrylate.

media used was distilled water. A split-ring mold was fabricated to prepare specimen discs 1 ± 0.1 mm thick and 15 ± 0.1 wide. The excess at the edges of the specimens was eliminated with a scalpel blade and lightly rotated and finished against 1000–1500 grit silicon carbide abrasive paper to remove irregularities and obtain smooth surfaces. All the specimens were prepared at room temperature (23 ± 1°C) according to the manufacturer's instructions. They were then subjected to water sorption and solubility tests based on the ISO 4049 and ADA No. 8 requirements. The samples were carefully handled during preparation to minimize the inclusion of entrapped air bubbles, and the material was confined between 2 opposing surfaces to minimize exposure to oxygen from the atmosphere.

Changes in the weight of the specimens were determined after the samples were immersed in water for 1, 7, and 30 days. Specimens were weighed using an analytic balance with a precision of 0.001 g (GR-200, AD Company Limited, Toshi-maku, Tokyo, Japan). After the initial dry mass (m$_1$) of each specimen was measured, they were placed in a 24-well plate filled with distilled water (pH 7.2) and then dried in an oven at 37°C for 1, 7, or 30 days, after which the well plates were removed from the oven and the specimens taken out. They were gently wiped with soft absorbent paper and weighed using the analytic balance. The specimens were then spread out on a metallic mesh resting inside a desiccator, which contained anhydrous calcium chloride underneath the mesh. To remove moisture, a suction vacuum (Cole-Parmer, Chicago, IL, USA) was applied through a hose attached to the top cover of the desiccator. After 24 hours, the speci-mens were taken out from the desiccator and dry weight measurements were taken (Table 2).

The water sorption and solubility of the orthodontic resins were calculated according to BS EN ISO 4049:2000 using the following formulas:[10,11]

$$\text{Water sorption}(S) = \frac{m_2 - m_3}{V}$$

$$\text{Solubility}(SL) = \frac{m_1 - m_3}{V}$$

where m_1 is the initial dry mass (in milligrams) before water immersion, m_2 is the mass after water immersion, m_3 is the mass of the dried specimens after water immersion, and V is the specimen volume in mm^3.

The percentage of mass change of the specimen ($M_g\%$) is the apparent amount of the liquid sorbed by the specimen. It was determined after 30 days of sorption according to the following formula:[12]

$$M_g\% = \frac{m_2 - m_1}{m_1} \times 100$$

where m_2 represents the weight of the saturated specimen after 30 days of sorption.

The percentage of solubility (SL%), which repre-sents the amount of unreacted monomers that may have been extracted by water after a 30-day immersion period, was calculated by the following

Table 2. Measurement times

m1	Dry mass value at the begininng
m2	Mass value after 24 h in distilled water
m3	Dry mass value at 1 d
m4	Mass value after 7 d in distilled water
m5	Dry mass value at 7 d
m6	Mass value after 30 d in distilled water
m7	Dry mass value at 30 d

Table 3. Group descriptive statistics for water sorption and solubility

	m1 ± SD	m2 ± SD	m3 ± SD	m4 ± SD	m5 ± SD	m6 ± SD	m7 ± SD
Group BO	0.43 ± 0.05	0.43 ± 0.05	0.42 ± 0.05	0.43 ± 0.05	0.43 ± 0.05	0.43 ± 0.05	0.42 ± 0.05
Group XT	0.44 ± 0.05	0.44 ± 0.05	0.44 ± 0.05	0.44 ± 0.05	0.44 ± 0.05	0.44 ± 0.05	0.44 ± 0.05
Group LB	0.43 ± 0.05	0.43 ± 0.05	0.43 ± 0.05	0.44 ± 0.05	0.43 ± 0.05	0.44 ± 0.05	0.43 ± 0.05
Group TEF	0.38 ± 0.07	0.38 ± 0.06	0.38 ± 0.06	0.38 ± 0.07	0.38 ± 0.07	0.38 ± 0.07	0.38 ± 0.07

m: mass value (as defined in Table 2).

formula:[13]

$$SL\% = \frac{m_1 - m_3}{m_1} \times 100$$

The percentage amount of water absorbed after 30 days was calculated by the following formula:[14]

$$S\% = M_g\% + SL\%$$

The diameter of each specimen was measured from 3 perpendicular planes using a digital caliper (0–20 cm, Mitutoyo Corp, Tokyo, Japan). The thickness was measured at 5 points on the specimen, 1 at the center and 4 at equally spaced points using a micrometer gauge with an accuracy of 0.02 mm (0–25 mm, Moore Wright, Sheffield, UK). These measurements were taken in order to determine the volume of the specimens (V) using the following equation: $V = \pi r^2 h$, where r is the mean radius of the specimen and h is the mean thickness.[15]

For compressive strength (CS) tests, stainless steel cylindrical split-ring molds with a height of 6 mm and an internal diameter of 4 mm were used according to ISO 9917-1.[16] Cylindrical forms of the cements studied were prepared for each group. Twelve cylindrical specimens of each cement were made to test for CS, and the same number were made to test for diametral tensile strength using a 2-part metallic matrix measuring 6 mm in height and 4 mm in diameter, according to American Dental Association specifications. All specimens were prepared at room temperature (23 ± 1°C), according to the manufacturer's instructions, and mechanical

tests were performed with an Instron Universal Test Machine, model 4444 (Instron Corp, Norwood, MA, USA).

The CS values were calculated in MPa according to the following formula:[17]

$$\text{Compressive strength(MPa)} = 4F/\Pi d^2$$

where F is the fracture load (in Newtons, N) and d is the sample diameter.

The descriptive statistics of the groups are shown in Table 3. The data for water sorption and solubility were statistically analyzed using one-way analysis of variance (ANOVA; SPSS 15.0, SPSS Inc, Chicago, IL, USA). *Post hoc* tests were calculated at the $p = 0.05$ significance level. For the compressive tests, we applied the Tukey test to determine which cement groups were statistically different (Table 4) because the F-test for each one-way ANOVA detected significant statistical differences ($p \leq 0.05$) among the means of each group for each mechanical test.

RESULTS

The mean values and standard deviations of the solubility and water sorption for the orthodontic cements after immersion are summarized in Table 3. The ANOVA revealed a significant 2-factor interaction ($p < 0.05$) between the independent variables (materials and immersion times) of sorption.

The highest sorption (S) was exhibited by group TEF after a 30-day immersion period. The lowest sorption was exhibited by group XT. The highest

Table 4. Descriptive statistics for compression strength

Group	Mean (SD)	Minimum	Maximum	Tukey
Group BO	210.41 (37.58)	140.16	249.5	a
Group XT	198.91 (44.16)	116.25	238.16	a
Group LB	186.83 (38.08)	143.50	251.58	a
Group TEF	89.16 (26.83)	37.91	127.08	b

Table 5. Mean (standard deviation) sorption and solubility characteristics after immersion in distilled water[a]

	S (μg/mm^3) After Immersion at			After 1 Month of Immersion			
	1 Day	1 Week	1 Month	SL (μg/mm^3)	Mg (%)	SL (%)	S (%)
Group BO	7.7 (0.29)[c]	10 (0.59)[a]	10.6 (0.71)[c]	1.18 (0.11)[c]	0.55 (0.12)[a]	0.14 (0.03)[c]	0.69 (0.27)[c]
Group XT	4.14 (0.55)[b]	8.29 (0.61)[c]	9.48 (0.75)[c]	1.18 (0.32)[c]	0.43 (0.45)[a]	0.16 (0.02)[c]	0.59 (0.12)[a]
Group LB	5.33 (0.95)[a]	9.48 (0.98)[a]	11.25 (1.05)[a]	2.37 (0.17)[a]	0.52 (0.76)[a]	0.1 (0.01)[a]	0.62 (0.35)[a]
Group TEF	5.30 (0.7)[a]	28.4 (0.66)[b]	19.5 (0.52)[b]	4.14 (0.26)[b]	0.67 (0.21)[b]	0.9 (0.23)[b]	1.57 (0.34)[b]

[a] Superscript letters indicate homogenous subsets (within which $p > 0.05$) where comparison has been made with respect to cements for each sorption and solubility.
S: sorption SL: solubility Mg: Mass

SL% was that of group TEF, whereas the lowest SL% was that of group LB. The highest M_g% was that of group TEF, whereas group XT exhibited the lowest M_g% (Table 5).

There were no significant differences in water sorption between group BO, group XT, and group LB over 1, 7, and 30 days ($p > 0.05$). There were no significant differences in solubility among the 4 materials over 1 and 7 days ($p > 0.05$); however, group TEF had significantly higher solubility than other traditional orthodontic composites after a 30-day immersion period ($p < 0.001$).

For CS tests, we observed statistically significant differences only for group TEF (Table 4). Group L exhibited the highest CS, whereas group TEF exhibited the lowest ($p < 0.05$).

DISCUSSION

Water molecules affect the degradation and erosion of composite materials, which takes place via hydrolytic reactions.[2,4] Thus, the water sorption and solubility of orthodontic composite materials is very important. Water diffusion in orthodontic composites occurs in the resin matrix and is a diffusion-controlled process. According to Braden et al.,[18] the diffusion coefficient of water decreases with decreasing amounts of water in the resin matrix. One can therefore infer that a decrease in the rate of water uptake is to be expected with increasing immersion time.

Several water sorption and solubility studies have been conducted with different composite materials. Unfortunately, it is difficult to compare them as they were carried out over different immersion times, they used samples of different sizes, and the results were presented in different units.[19] The smaller the specimen, the shorter the time needed to achieve equilibrium with water and the molecules, which absorbed more water took longer to stabilize.[20,21]

One of the fallacies of water sorption studies is the assumption that an increase in specimen weight corresponds to an increase in water content, when in actuality, it is the difference between the increase in water content and the dissolution of low-molecular-weight organics. Therefore, the true water sorption values are larger than those reported.[22] According to Pham and Ferracane,[23] improper handling of the samples can cause minute wear of the surface, leading to a decrease in weight.

We observed that water sorption increased as a function of time for almost all the immersion times and groups studied (Table 5). However, the materials had different sorption values, and this could be attributed to differences in the components of the materials studied (Table 1). Water sorption mainly depends on the resin structure; generally, hydrophilic molecules increase water sorption. Thus, the resin-based oligomer matrix urethane dimethacrylate (UDMA) is more hydrophilic than monomer bisphenol A systems such as bisphenol-A glycidyl methacrylate (BisGMA) and bisphenol-A benzyl methacrylate (BisBMA),[20,24] results that are similar to those found in our study, in which group TEF and group BO showed higher water sorption than the other materials. The lowest sorption was recorded for group XT because it contains BisGMA and BisBMA. According to a similar study,[25] a UDMA-based composite sorbs less water than a BisGMA-based composite. Group BO contains triethylene-glycol dimethacrylate (TEGDMA) and BisGMA. It was reported that composites based on TEGDMA alone had higher diffusion coefficients than composites based on both TEGDMA and BisGMA.[5] In another study, it was reported that although TEGDMA does not contain hydroxyl groups, it exhibits some affinity to water depending on the number of hydroxyl groups within the matrix that are available to form hydrogen bonds with water.[26]

It was difficult to compare and calculate the solubility of the commercial orthodontic adhesives used in this study because it is important to know the exact amount of each component; unfortunately, that information was not provided by most of the manufacturers. The highest solubility was obtained for group TEF, whereas the lowest was observed for group LB, which indicates that the more hydrophilic resin exhibits higher solubility. For the other groups, however, the more hydrophilic composites exhibited water sorption lower than the others. Our results are in accordance with a study performed by Malacarne et al.[27] On a molecular level, the extent and rate of water sorption–solubility in polymer networks are determined by 2 factors: resin polarity and network topology. Resin polarity is the concentration of polar sites available to form hydrogen bonds with water. Network topology is related to the cohesive energy density of the polymer network.[28]

The CS test is a reliable representation of the mechanical integrity of different materials. In the CS tests, 2 axial forces are applied to the test material in opposite directions.[29] Thus, the CS test can be used to test dental materials and is very important for determining mastication resistance.[30]

In this study, we found that the flowable composite had lower CS than the other composites. This result is in agreement with a similar study,[31] where the contents of the composites affected their mechanical behavior. Generally, mechanical strength tends to increase with an increase in filler content.[31] Because most flowable composites contain a small amount of filler, decreased mechanical strength is not surprising.

The mechanical tests can give information about the performance of products. For example, when they are used to evaluate band cementation, their adherence to enamel and metal, their durability under mastication, and the properties of the cements used.[32] Loss of cement material can be affected by water sorption and solubility or low CS, leading to increased risk of caries.[33]

CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that the water sorption and solubility of orthodontic composites are dependent on the composite's composition. The results also revealed that flowable composites have higher water solubility and water sorption and lower CS compared with the traditional orthodontic composite resins used in this study.

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