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Original Article

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The effect of acidic drinks on the solubility and water absorption properties of composite resins with different structures

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Abstract

Aim: The aim of this in vitro study was to examine the changes occurring over time in water absorption and solubility values in acidic solution environments of composite materials with different structures.

Methods: A total of 90 disc-shaped samples (8 mm diameter, 2 mm thickness) were prepared using three different composite resins: Kaiser Bulk Fill Composite (Dr. Müller, Remar Technik GmbH, Lübbecke, Germany), Kaiser Nano Composite (Dr. Müller, Remar Technik GmbH, Lübbecke, Germany), and GC Flowable Composite (GC Corp., Tokyo, Japan). After leaving the samples in an incubator for 24 hours, baseline weights were recorded. The samples were then placed in cola, energy drink, and distilled water solutions for 7 and 15 days. From the measurements taken at the end of these periods, the water absorption and solubility values were calculated. In the statistical analyses of the study, the Shapiro-Wilk test, ANOVA, Kruskal-Wallis test, Bonferroni and Dunn-Bonferroni post hoc tests, dependent samples t-test, and Wilcoxon signed-rank test were used.

Results: At the end of 7 and 15 days, the solubility values of Kaiser Nano Composite were determined to be significantly lower than those of GC Flowable Composite and Kaiser Bulk Fill Composite ($p < 0.05$). Among the immersion solutions, the energy drink significantly increased the solubility of Kaiser Bulk Fill Composite compared with distilled water on days 7 and 15 ($p < 0.05$). In the comparison of the water absorption values, no statistically significant difference was determined between the materials ($p > 0.05$).

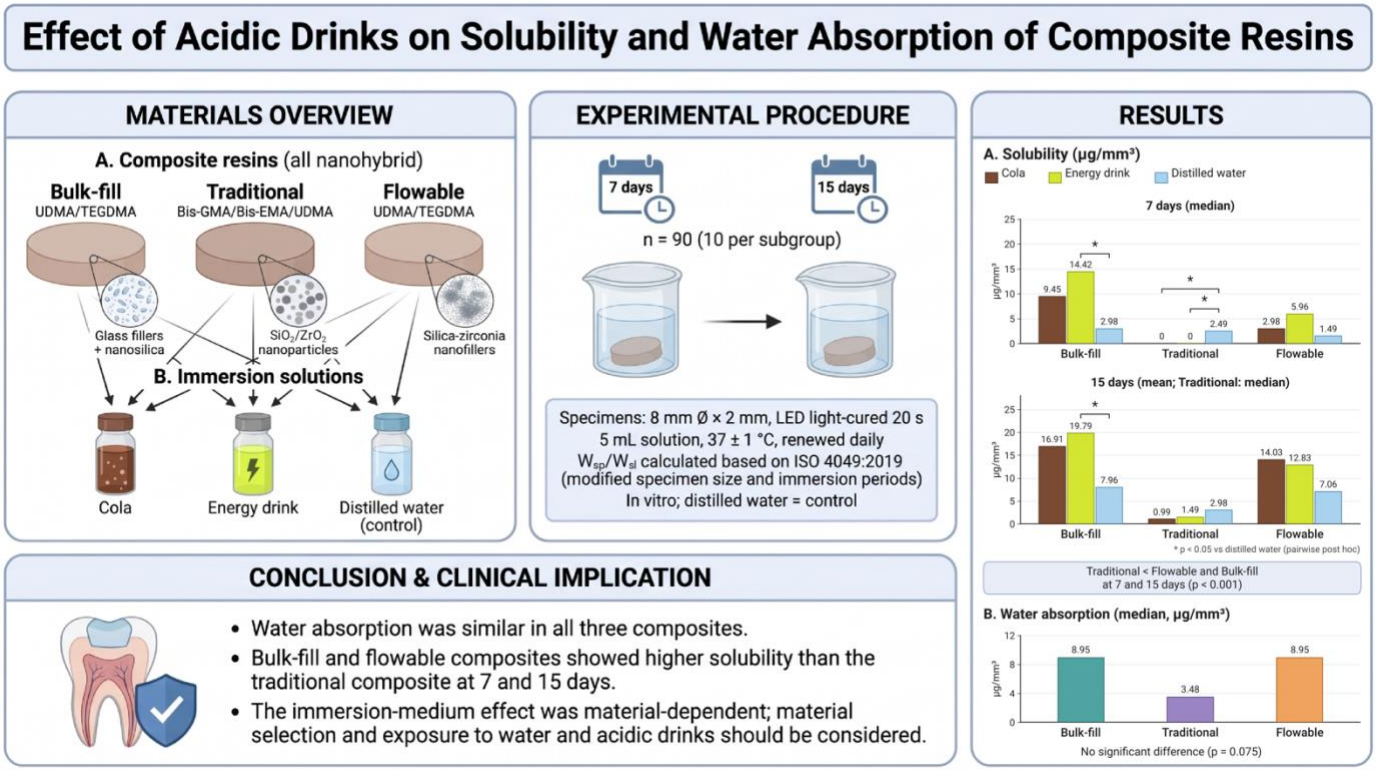
Conclusion: Differences in the structural properties of restorative materials can significantly affect the water absorption and solubility in water behaviors of these materials depending on time and environment.

Keywords: Acidic beverages, bulk-fill composite, composite resin, energy drinks, solubility, water absorption

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Graphical abstract



Introduction

Composite resins are frequently used in restorative dentistry because of their superior aesthetic properties and ease of use, allowing direct restoration to be performed. These materials have acquired an important place in clinical applications as they show strong bonding strength to dental tissue, are economically suitable, and offer a conservative treatment approach compared to full crowns (1). In contrast to metal-based restorations, composite restorations are distinguished by their high aesthetic properties. The most important disadvantage of the material is that it causes microleakage as a result of shrinkage occurring during polymerization. This can lay the ground for impaired marginal adaptation and the formation of secondary decay (2, 3). In addition, composite resin restorations are not as resistant to chewing forces as amalgam fillings, and for a successful application, the tooth surface must be completely dry before restoration (4).

Composite resins are formed from a modified methacrylate or acrylate-based resin matrix. Triethylene glycol dimethacrylate (TEGDMA), bisphenol A-glycidyl methacrylate (Bis-GMA), and urethane dimethacrylate (UDMA) are among the most commonly used monomers in this matrix structure. Bis-GMA can limit the flowability of composites because of high molecular weight and viscosity, whereas TEGDMA reduces viscosity with the addition of a co-monomer and increases the processability of the material. Inorganic filler particles, such as silica, quartz, or glass, are included in the formulation to reduce shrinkage during polymerization and increase the radiopacity of composite resins. These filling materials not only increase mechanical resistance but also affect the clinical performance and long-term stability of composite resin (5, 6).

Despite the advantages of composite resin, such as aesthetics and ease of shaping, significant clinical problems can emerge owing to water absorption and solubility. High rates of these characteristics can lead to dimensional changes in the material and negatively affect long-term performance. Moreover, by causing color instability in restorations, weakened marginal integrity, and reduced compatibility with biological tissues, increased water absorption and solubility can result in negative outcomes regarding aesthetics and biocompatibility. This weakens the physical and chemical properties of composite resins. Moisture in the oral environment, hydrolysis, and enzymatic reactions negatively affect the surface integrity of composite resins, thereby causing deterioration and wear. By facilitating the development of secondary decay, this can lead to a decrease in the clinical performance of the restoration (7–9).

To a great extent, water absorption in composite resins occurs because of the chemical composition of the material and factors such as polymerization and monomer concentration. Water absorption occurs through the diffusion of water molecules to the polymer matrix, filling the gaps between polymer chains and consequently causing dimensional changes with swelling in the material. The type and composition of composite resins are the primary factors determining the level of water absorption. In addition, the chemical structure of the resin monomers used plays an important role in this process, and especially monomers such as Bis-GMA that contain hydroxyl groups in the molecular structure, become more sensitive to water absorption because of their hydrophilic properties (10–13).

Resin-based materials are widely used in dentistry because of their aesthetic properties, mechanical strength, and cost advantages (1). The aim of this in vitro study was to examine and compare the water absorption and solubility values of composites containing nanoparticles, which are increasingly being preferred in clinical applications, and to evaluate the effect of acidic drinks (cola and energy drink) on their solubility compared with distilled water. The null hypotheses of the study were that (i) these materials would exhibit solubility and water absorption values at the same rate and (ii) the immersion solution would not affect the solubility of the materials.

Materials and Methods

The sample size was determined by an a priori power analysis. In addition, a sensitivity power analysis (α = 0.05, power = 0.80) showed that the comparisons between composite groups (n = 30 per composite) could detect an effect size of $f \geq 0.33$ (power = 0.93 for f = 0.40), whereas the comparisons between solution groups within each composite (n = 10 per subgroup) could detect only large effects ($f \geq 0.60$).

Three different composite resins were used in this study: Kaiser Bulk Fill Composite (Dr. Müller, Remar Technik GmbH, Lübbecke, Germany), Kaiser Nano Composite (Dr. Müller, Remar Technik GmbH, Lübbecke, Germany), and GC Flowable Composite (GC Corp., Tokyo, Japan) (Table 1). In the preparation of the samples, stainless-steel molds, 8 mm in diameter and 2 mm in thickness, were used. A total of 90 samples were prepared, with 10 samples in each of the nine subgroups formed by the three composite resins and the three immersion solutions (cola, energy drink, and distilled water).

To standardize the sample preparation, all procedures were performed by a single researcher. At this stage, a transparent band was placed on a glass slide, then a stainless-steel mold was placed carefully on it. The composite resins were placed in the stainless-steel molds in a single layer of 2 mm thickness using a manual instrument to ensure no air pockets. A transparent band was placed over the upper surface of the molds, and the excess material was removed by applying light pressure with a glass slide. The polymerization process was performed for 20 s using an LED light device (Woodpecker LED.B, Guilin Woodpecker Medical Instrument Co., Ltd., Guilin, Guangxi, China; manufacturer-reported irradiance 1000–1200 mW/cm^2 in the 20-s mode). During this process, the light source was held stable at the closest possible distance to the sample surface.

After the polymerization process was completed, a graded polishing process was applied to the sample surfaces that had been exposed to light. During this process, rough, moderate, fine, and super-fine grain discs were used (Sof-Lex™ XT, 3M ESPE, St. Paul, MN, USA). Each disc was applied to the surface for approximately 10 s at low speed, and the operator maintained fixed pressure throughout the process. A new disc was used for each sample.

Calculation of the water absorption and solubility in water values

The prepared samples were kept in an incubator (EN025, Nüve, Ankara, Türkiye) at 37 ± 1 °C for 24 h in a desiccator containing silica gel to allow for complete polymerization. Each sample was then weighed on analytical scales sensitive to 0.0001 g (Precisa XB 220A, Precisa Gravimetrics AG, Dietikon, Switzerland). The measurements were repeated every 24 h until the weight loss within any 24-h period was less than 0.1 mg. This value was then accepted as the fixed baseline weight and recorded as M_1 (μg). The diameter and thickness of the samples that had reached a fixed weight were measured using calipers, and the mean volume of each sample was calculated in mm^3 using the formula $V = \pi r^2 h$.

The samples were placed in plastic bottles filled with 5 mL Coca-Cola (The Coca-Cola Company, Atlanta, GA, USA), Red Bull (Red Bull GmbH, Fuschl am See, Austria), and distilled water (Tekkim Kimya San. ve Tic. Ltd. Şti., Bursa, Türkiye) in an incubator at 37 ± 1 °C. The solutions were changed every 24 h. At the end of 7 and 15 days, the samples were removed from the solutions and carefully dried with absorbent drying paper to remove excess liquid from the surface. Each sample was then weighed again on a sensitive scale, and the value was recorded as M_2 (μg). Before weighing the samples in the cola and energy drink groups, each sample was immersed in distilled water for 1 min and then subjected to a standard ultrasonic bath for 5 min to prevent incorrect mass measurement of potential sugar/pigment remnants. The blotting procedure was then repeated.

The same samples were then placed in a desiccator at 37 ± 1 °C, and a cycle of weight measurements was performed until a fixed mass criterion was obtained (≤0.1 mg difference), and the final value was recorded as M_3 (μg). Based on ISO 4049:2019, with modified specimen dimensions (8 mm × 2 mm instead of 15 mm × 1 mm) and immersion periods (7 and 15 days), water absorption (W_{sp}) and solubility (W_{sl}) were calculated using the

following formulae, and the results were recorded as μg/mm³ (14-17).

$$W_{sp} = (M_2 - M_3) / V$$
$$W_{sl} = (M_1 - M_3) / V$$

All weight measurements were performed by the same operator using the same scales after stabilization of the measurement room temperature.

Statistical analysis

Data obtained in the study were analyzed statistically using SPSS Statistics for Windows version 25.0 (IBM Corp., Armonk, NY, USA).

Conformity of continuous variables to normal distribution was examined using the Shapiro-Wilk test. Descriptive statistics were stated as mean ± standard deviation (SD) values if the distribution was normal and as median (minimum-maximum) values if the distribution was not normal. In the comparisons of more than two groups and when a normal distribution was observed, the ANOVA test was used, and when the distribution was not normal, the Kruskal-Wallis test was applied. When there was an overall significance, the Bonferroni test was applied in subgroup analyses after the ANOVA test, and the Dunn-Bonferroni test after the Kruskal-Wallis test. When comparing the solubility values on days 7 and 15, the dependent samples t-test was applied when there was normal distribution of data, and the Wilcoxon signed-rank test when the distribution was not normal. The solubility difference score was calculated for each sample as the difference between the day-15 and day-7 solubility values. The type I error rate was set at 5%.

Table 1. The composite resin materials used in this study and their basic properties.

Material (manufacturer)	Lot number	Material type	Main components
Kaiser Bulk Fill Composite Dr. Müller, Remar Technik GmbH, Lübbecke, Germany	2023008278	Nanohybrid bulk-fill composite	UDMA, TEGDMA, Ba-Al-silicate glass fillers, nanosilica particles
Kaiser Nano Composite Dr. Müller, Remar Technik GmbH, Lübbecke, Germany	2023007936	Nanohybrid traditional composite	Bis-GMA, Bis-EMA, UDMA, SiO_2/ZrO_2 nanoparticles
GC Flowable Composite GC Corp., Tokyo, Japan	2411151	Nanohybrid high-filler flowable composite	UDMA, TEGDMA, silica-zirconia nanofillers

Results

A statistically significant difference was observed between the composite groups for both the 7th-day and the 15th-day solubility values ($p < 0.001$ for both time points). In the subgroup analyses, the solubility value of the traditional composite was lower than those of the flowable and bulk-fill composites at both time points ($p < 0.001$ for both comparisons). No significant difference was found between the values of the flowable composite and bulk-fill composite groups ($p = 0.400$) (Table 2).

A statistically significant difference was observed between the solution groups for the 7th-day solubility values of the traditional composite ($p = 0.005$). In the subgroup analyses, the 7th-day solubility value in the distilled water group was found to be significantly higher than those of the other groups ($p = 0.006$).

In the traditional composite, no significant difference was observed between the solution groups for the 15th-day solubility values ($p = 0.051$). No significant difference was observed between the solution groups in respect of the solubility difference scores of the traditional composite ($p = 0.684$).

A statistically significant difference was observed between the solution groups for the median 7th-day solubility values of the bulk-fill composite ($p = 0.020$). In the subgroup analyses, the median 7th-day solubility value in the energy drink group was significantly higher than that in the distilled water group ($p = 0.027$). No significant difference was observed in the other paired comparisons ($p > 0.05$) (Table 3).

A statistically significant difference was observed between the solution groups for the mean 15th-day solubility values of the bulk-fill composite ($p = 0.032$). In the subgroup analyses, the mean 15th-day solubility value in the energy drink group was found to be significantly higher than that in the distilled water group ($p = 0.036$). No significant difference was observed in the other paired comparisons ($p > 0.05$). There was seen to be no significant difference between the solution groups in terms of the solubility difference scores of the bulk-fill composite ($p = 0.667$) (Table 3).

No significant difference was observed between the groups according to the water absorption values ($p = 0.075$) (Table 4).

Table 2. Comparison of the solubility measurements of the composite groups.

	Composite groups			
	Flowable Composite	Traditional Composite	Bulk-Fill Composite	*p*-value
7th day	2.98 (0-41.78)	0 (0-4.97)	6.96 (0-40.78)	**<0.001**[a]
15th day	9.95 (0.99-42.77)	0.99 (0-11.94)	11.94 (0.99-45.76)	**<0.001**[a]
Solubility difference score	3.98 (0-25.86)	0.99 (0-8.95)	3.98 (0.99-31.83)	**<0.001**[a]

Data are stated as median (minimum-maximum) values.
a: Kruskal-Wallis test

Table 3. Comparisons of solubility measurements according to composite types and solution groups.

	Solution groups			
Flowable Composite	**Cola**	**Energy drink**	**Distilled water**	***p*-value**
7th day	2.98 (0.99-41.78)	5.96 (0-15.92)	1.49 (0.99-12.93)	0.069[a]
15th day	14.03 ± 12.52	12.83 ± 6.75	7.06 ± 5.19	0.182[b]
Solubility difference score	2.49 (0-11.94)	3.98 (0.99-25.86)	4.48 (0.99-8.95)	0.759[a]
Traditional Composite				
7th day	0 (0-0.99)	0 (0-0.99)	2.49 (0-4.97)	**0.005**[a]
15th day	0.99 (0-1.99)	1.49 (0-4.97)	2.98 (0-11.94)	0.051[a]
Solubility difference score	0.99 (0-1.99)	0.99 (0-4.97)	0.99 (0-8.95)	0.684[a]
Bulk-Fill Composite				
7th day	9.45 (0-40.78)	14.42 (1.99-32.83)	2.98 (0.99-7.96)	**0.020**[a]
15th day	16.91 ± 11.81	19.79 ± 11.35	7.96 ± 4.71	**0.032**[b]
Solubility difference score	3.98 (0.99-14.92)	3.98 (0.99-31.83)	4.48 (0.99-7.96)	0.667[a]

Data are stated as mean ± standard deviation and median (minimum-maximum) values.
a: Kruskal-Wallis test, b: ANOVA

Table 4. Comparisons of water absorption values of the composite groups.

	Composite groups			
	Flowable Composite	Traditional Composite	Bulk-Fill Composite	*p*-value
Water absorption value	8.95 (3.98-31.83)	3.48 (0.99-18.9)	8.95 (4.97-18.9)	0.075[a]

Data are stated as median (minimum-maximum) values.
a: Kruskal-Wallis test

Discussion

In this study, comparisons were made of the water absorption and time-related solubility levels of composite resins with nanofillers but different structures. According to the criteria defined in ISO 4049:2019, the water absorption and solubility levels of all restorative materials used in clinical practice must be below a certain level. However, it is known that even if these values remain below the threshold, they can be differentiated at a significant level (18). The current study results showed an increase in solubility values from day 7 to day 15 in all groups, and these results are consistent with the findings of previous studies (7). Because the solubility values differed significantly between the composite groups, whereas the water absorption values did not, the first null hypothesis was rejected for solubility but could not be rejected for water absorption. Because the immersion solution significantly affected solubility only for some materials and time points, the second null hypothesis was partially rejected.

The study results showed that the solubility value of the traditional composite was significantly lower than that of the flowable and bulk-fill composites on day 7, whereas the bulk-fill composite exhibited a relatively higher solubility value on day 15. This could be attributed to the low degree of polymerization conversion due to the use of a thick layer in the polymerization of bulk-fill composites (19, 20). There could also be a greater build-up of water absorption and solubility between 1 week and 1 month in bulk-fill composites than in conventional composites, and a statistically significant increase was observed due to the layer thickness and the time immersed in the solution (21).

Previous studies have shown that hydrophilic monomers, such as Bis-GMA and TEGDMA, in composite resins increase the water permeability of the matrix and the material's solubility in solutions, because they reduce cross-link density (18). Although it may seem paradoxical that high solubility values were observed in some of the current study's distilled water groups compared to cola and energy drink solutions, it has been reported that full desorption may not always be reflected after drying during the measurements because water bound to or remaining on the surface causes masking of the net mass loss (7). In addition, pigment and sugar remnants formed in acidic drinks may be held on the surface of the material, causing an increase in the mass. Therefore, although changes in microhardness and roughness reflect actual chemomechanical erosion, they may diverge from the net solubility value (22, 23).

In addition to the importance of the acid type, it has been shown that the chelation property of citric acid in energy drink content can cause greater deterioration of the filler–matrix interface than phosphoric acid in cola (22, 23). In parallel with this, the current study results showed significantly higher solubility values on days 7 and 15 in the energy drink group than in the distilled water group for the bulk-fill composite, whereas the difference between the cola and distilled water groups was not significant. However, this pattern was not uniform: in the traditional composite, the day-7 solubility value was significantly higher in the distilled water group than in the acidic drink groups ($p = 0.005$), indicating that the effect of the immersion medium was material-dependent.

In respect of water absorption, no significant difference was determined between the current study groups. Water absorption was observed at a similar volumetric level in the three materials used. However, water absorption alone is not fully predictive of performance and should be read in conjunction with mechanical and optical markers (10).

Continued contact of the surface of composite resins with acidic drinks leads to a decrease in surface microhardness and an increase in roughness. Consequently, these changes increase the risk of long-term clinical failure by leading to loss of brightness, plaque retention, and deterioration in color stability (24, 25). Therefore, supporting solubility measurements that assess mass loss according to ISO 4049:2019 with tests such as microhardness, surface morphology, and volumetric material loss via profilometry will help capture the real effect of erosive solutions more accurately (22).

When the current study results are clinically examined, there is a need to optimize the adhesive protocol and polymerization technique and duration to protect the mechanical integrity in long-term acid-moisture exposure because of the affinity of the composite matrix to water and the degree of polymerization conversion failure in deep cavities of bulk-fill and flowable composite systems (23). Long-term water-storage studies have also shown that the sorption and solubility of bulk-fill and conventional composites depend on material composition and continue to develop over time, which may affect the durability of the restoration (26).

The fact that the samples used in the current study had standard measurements and that the preparation/polishing protocol was performed by the same operator increased the reliability of the results obtained. However, there were also some limitations that should be considered, primarily that no measurement was performed of the pH and titrational acidity of the drink solutions, no thermal/abrasion cycle was added, and only mass-based solubility was measured; moreover, with a weighing criterion of 0.1 mg and a specimen volume of approximately 100 mm^3, the smallest detectable change corresponded to approximately 1 $\mu g/mm^3$, so some between-group differences were close to the measurement resolution (27). In addition, the ultrasonic cleaning step was applied only to the cola and energy drink groups, which may have introduced a systematic difference relative to the distilled water group. Finally, with 10 samples per subgroup, the comparisons between solutions within each material were powered to detect only large effects, so non-significant differences between solutions should be interpreted with caution.

Conclusion

Composite resin materials with different structures exhibited similar water absorption values under the conditions tested.

The flowable and bulk-fill composite resins tested exhibited higher solubility values than the traditional composite resin at both 7 and 15 days.

The effect of the immersion solution on solubility was material-dependent; the energy drink increased the solubility of the bulk-fill composite compared with distilled water. Because of this material–environment interaction, both material selection and the anticipated exposure of the restoration to water and acidic drinks should be considered when planning clinical protocols.

Disclosures

Peer-review: Externally peer-reviewed.

Author Contributions: Concept – A.Y.; Design – A.Y., T.T.; Supervision – S.C.; Materials – T.T., S.C.; Data collection and/or processing – H.O.; Analysis and/or interpretation – A.Y., T.T., S.C.; Literature search – S.C., H.O.; Writing – A.Y., S.C.; Critical review – A.Y., T.T., S.C., H.O.

Declaration of Generative AI and AI-Assisted Technologies: During the preparation of this work, FigureLabs was used to create the graphical abstract, and the AI figure-generation feature in BioRender was used for subsequent text edits to the figure. The author(s) then reviewed, edited, and verified all visual and scientific content for accuracy and take(s) full responsibility for the final published material.

Conflict of Interest: The authors declare that there is no conflict of interest.

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