MATERIALE PLASTICE
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https://doi.org/10.37358/Mat.Plast.1964

An Analysis of Water Sorption of Some Composites Used in Dentistry

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Abstract: *This study aimed to investigate the water sorption levels of 3 different condensable bulk fill composites, two flowable bulk fill composites, two microhybrid composites, one nanofilled composite, and one nanohybrid composite material polymerized with the same beam device on the 14th and 30th days.We evaluated nine different composite materials. The materials were prepared as cylindrical blocks (n=7) with a diameter of 7 mm and a thickness of 2 mm and polymerized. Each sample was kept in distilled water for specified times and measured by the ISO 4049 method.We used The Kruskal-Wallis test to compare more than two independent groups where numerical variables had no normal distribution. The Dwass-Steel-Critchlow-Fligner test evaluated differences between the groups if the non-parametric tests were applied. We used The Wilcoxon test to compare the numerical variables without normal distribution (water sorption values in the 14th and 30th-day measurements for each cement material).For water sorption, there was no statistically significant difference between the water sorption values of the resin composite materials on the 14th and 30th days. No statistically significant difference was found in comparing water sorption values between the 14th and 30th measurements for each resin composite material. There is a need for more extended studies by keeping the conditions constant for the evaluated composites and for bulk fill composites to be evaluated with 4mm samples.*

Keywords: *Composite resin, water sorption, bulk fill composite, nanofilled composite*

1. Introduction

Caries lesions observed in the teeth and gingival diseases that occur together or are detected alone are among the most common chronic diseases in the world. Tooth decay causes pain, nutritional disorders, and cosmetic discomfort to the patient, and if not treated, it leads to a process that progresses to tooth loss [1, 2].

Although there are different treatment options in the treatment of dental caries, the most commonly used method for restoration today is the use of composite materials. In addition to existing composite materials, bulk-fill composite materials, which can provide polymerization in thicknesses of more than 2 mm, have been put into clinical use in recent years. These composite materials ensure the polymerization of 4 or 5-mm composite material at once. In this way, the patients reduce the time spent in the dentist's chair, and contamination during layering does not happen [3, 4].

Nanofilled composites with smaller filler particles have been in the dental market for some time to eliminate the deficiencies in the surface properties of microfill composites. In addition, hybrid composites have been produced to eliminate the deficiencies of their mechanical properties with better polishability [5, 6]. The filler in nanofilled composites can degrade over time; one of the most important reasons for this is the water absorption of the material. To avoid this problem in the composite materials available in the market, it must be evaluated with in vitro studies. Similarly, the water absorption properties of not only nanofilled but also newly produced bulk-fill composites should be evaluated [5,7]. The high water absorption values of resin composites cause the rapid color change of the composite material, decrease in wear resistance, insufficient fracture strength, and inadequacy in mechanical properties such as the release of unreacted monomers. At the same time, it can cause microleakage by damaging the connection between the tooth and the filling [8].

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The degree of polymerization also dramatically affects the water absorption of the material. The degree of polymerization may decrease as the distance to the light that will provide polymerization increases, that is, in the deeper parts of the restoration. As a result, water absorption values and mechanical properties decrease. Considering that bulk fill composite materials have higher polymerization degrees, such as 4-5 mm, it is thought that a better polymerization will be achieved in layers of 2 mm, and water absorption will be less [9-11]. There are studies showing that bulk-fill composites have a higher degree of polymerization than conventional composites. However, in the results of these studies, it has been stated that water absorption is higher in regions far from the polymerization light in thicknesses of more than 2 mm. It is appropriate to apply a maximum of 3 mm composite layers when bulk-fill composites are used [12].

This study aims to examine the water absorption levels of 3 condensable bulk-fill composite materials, two flowable bulk-fill composite materials, two microhybrid composites, one nanofilled composite, and one nanohybrid composite polymerized with the same beam device at the 14th and 30th-day intervals.

2. Materials and methods

2.1. Material selection

This study used three condensable bulk-fill composite materials, two flowable bulk-fill materials, two microhybrid composites, two nanohybrid material. The types and properties of the materials used in the study are shown in Table 1.

Table 1. Characteristics of the materials used

GROUPS	MATERİALS	TYPE OF MATERIAL
Group 1	Bulk Fill Composite System Applied With Sonic System	Bulk-Fill Composite
Group 2	Bulk Fill Composite Material With Additional İvocerin İnitiator	Bulk-Fill Composite
Group 3	Nanofilled Bulk Fill Composite Material	Bulk-Fill Composite
Group 4	Flowable Bulk Fill Composite Material With İvocerin İnitiator	Flow Bulk-Fill Composite
Group 5	Flowable Bulk Fill Composite Material Produced With Smart Dentin Replacement Technology	Flow Bulk-Fill Composite
Group 6	Universal Microhybrid Composite Material	Microhybrid Composite
Group 7	Universal Sub-Micron Hybrid Composite Material	Nanohybrid Composite
Group 8	Universal Nanohybrid Composite Material	Nanofilled Composite
Group 9	Supra-Nano Hybrid Composite Material	Nanofilled Composite

2.2. Application tages of the tudy

The materials were polymerized with LED light devices to form cylindrical blocks (n=7) with a diameter of 7 mm and a thickness of 2 mm. We selected the A2 colors of the materials in each sample and polymerized them with a 400-550 nm LED light source. Water sorption was evaluated according to ISO 4049 criteria. The samples were dried in a desiccator at 37°C for 24 h to evaporate the water they contained. The dry weights of the samples were measured with a precision balance, and the fixed mass weights were determined as “M1” mass in micrograms (μg). Each sample was kept in distilled water for 14 days and 30 days, respectively, at 37±1°C and stored in an oven. All seven samples for each group were placed in water after the measurements on the 14th day, and the measurements were repeated on the 30th day. After these periods, the samples removed from the water were measured again, and these measurement weights were determined as “M2”. They were again exposed to 37°C for 24 h in a desiccator to regain their constant mass weights. Then another measurement was made, and these weights were recorded as “M3”.

We calculated the volumes of the sample's cubic millimeters (mm^3) according to sample diameters and heights, and calculated water sorption (WS) values with the equation (Formula 1) shown below.

M2= Weight of samples after soaking in water (µg)
M3= Weight of samples after desiccator (µg)
V= Sample volume (mm^3)

$$WS\ (\mu g/mm^3) = (M2-M3)/\ V \qquad (1)$$

2.3. Statistical analysis

For descriptive statistics, mean ± standard deviation was used to give continuous data with normal distribution. Median with minimum-maximum values was applied for continuous variables without normal distribution. Numbers and percentages were used for categorical variables. The Shapiro-Wilk, Kolmogorov-Smirnov, and Anderson-Darling tests analyzed the normal distribution of the numerical variables.

The Kruskal Wallis test was used to compare more than two independent groups where numerical variables had no normal distribution. The Dwass-Steel-Critchlow-Fligner test evaluated differences between the groups if the non-parametric tests were applied.

The Wilcoxon test was used to compare the numerical variables without normal distribution (water sorption values in the 14th and 30th-day measurements for each cement material).

Jamovi (Version 2.2.5.0) and JASP (Version 0.16.1) were used for statistical analysis. The significance level (p-value) was determined at 0.05 in all statistical analyses.

3. Results and discussions

The water absorption values of the materials in each group were recorded on the 14th and 30th days. In each group, there are materials that show water absorption after being kept in water. In addition, there are also materials that do not show any difference in the values measured at the end of the 14th and 30th days in almost every group.

Table 2 shows The water sorption values of the examined materials on the 14th day. The highest median water absorption level was recorded for group 4 (11,693 µg/mm^3), while the group 7 was the material with the lowest (1,299 µg/mm^3). There was no statistically significant difference between the water absorption values of the resin composite materials (p=0.146).

Table 2. 14th day water sorption values of resin composite materials

	14th day Water Sorption (µg/mm^3) §	***p-values***
Resin Composites Materials		
Group 1 (n=7)	5,197 [0 – 15,591]	0.146
Group 2 (n=7)	2,598 [0 – 16,89]	
Group 3 (n=7)	1,299 [0 – 24,685]	
Group 4 (n=7)	11,693 [0 – 22,087]	
Group 5 (n=7)	6,496 [0 – 6,496]	
Group 6 (n=7)	7,795 [0 – 33,78]	
Group 7 (n=7)	1,299 [0 – 5,197]	
Group 8 (n=7)	7,795 [0 – 20,788]	
Group 9 (n=7)	6,496 [0 – 9,095]	

§: median [min-maks]
Kruskal Wallis-H test

Table 3 shows the water sorption values of the materials examined on the 30th day. group 9 was determined as the material with the highest median water sorption value (9,095 µg/mm^3). No statistically significant difference was found in comparing the water sorption values of the resin composite materials on the 30th day (p=0.555).

Table 3. 30th day water sorption values of resin composite materials

	30th day Water Sorption (µg/mm^3) §	***p-values***
Resin Composites Materials		
Group 1 (n=7)	2,598 [0 – 7,795]	0.555
Group 2 (n=7)	6,496 [0 – 16,89]	
Group 3 (n=7)	2,598 [0 – 5,197]	
Group 4 (n=7)	5,197 [0 – 10,394]	
Group 5 (n=7)	2,598 [0 – 11,693]	
Group 6 (n=7)	1,299 [0 – 7,795]	
Group 7 (n=7)	5,197 [0 – 10,394]	
Group 8 (n=7)	2,598 [1,299 – 7,795]	
Group 9 (n=7)	9,095 [0 – 111,733]	

§: median [min-maks]
Kruskal Wallis-H test

Figure 1 shows the changes in the water absorption values of the resin composite materials on the 14th and 30th days.

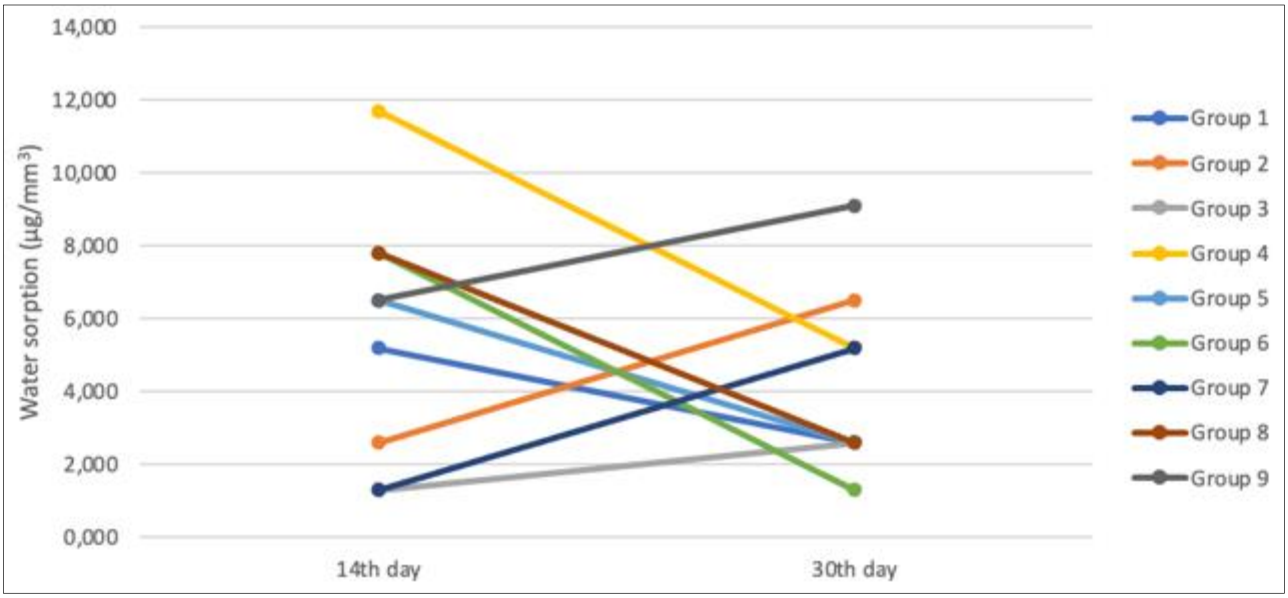

Figure1. Water sorption of each material in the 14th and 30th days

No statistically significant difference was found in the comparison of water absorption values between the 14th and 30th measurements for each resin composite material (p>0.05) (Table 4).

Table 4. Comparison of 14th day and 30th day water sorption values of resin composite materials

	14th day Water Sorption µg/mm^3) §	**30th day Water Sorption (µg/mm^3) §**	***p-values***
Rezin Composite Materials §			
Group 1 (n=7)	5,197 [0 – 15,591]	2,598 [0 – 7,795]	0.297

Group 2 (n=7)	2,598 [0 – 16,89]	6,496 [0 – 16,89]	0.349
Group 3 (n=7)	1,299 [0 – 24,685]	2,598 [0 – 5,197]	0.670
Group 4 (n=7)	11,693 [0 – 22,087]	5,197 [0 – 10,394]	0.059
Group 5 (n=7)	6,496 [0 – 6,496]	2,598 [0 – 11,693]	0.674
Group 6 (n=7)	7,795 [0 – 33,78]	1,299 [0 – 7,795]	0.109
Group 7 (n=7)	1,299 [0 – 5,197]	5,197 [0 – 10,394]	0.074
Group 8 (n=7)	7,795 [0 – 20,788]	2,598 [1,299 – 7,795]	0.271
Group 9 (n=7)	6,496 [0 – 9,095]	9,095 [0 – 111,733]	0.469

Resin composites are now indispensable materials in dental practice for both anterior and posterior restorations. However, many factors coexist in the oral environment, and environmental conditions change in a dynamic process. Restorative materials applied to the teeth react to these changing environmental conditions in many ways, such as water absorption, dissolution, and coloration. The water absorption of the different composite materials we examined in this study causes the bonds in the composite resin to deteriorate and the mechanical properties of the composite, such as color and wear resistance, to weaken over time. For this reason, an ideal restoration material is expected to have minimal water absorption. Water sorption is considered an evaluation criterion directly related to the clinical success of the composite [13,14].

Water absorption occurs in the organic matrix. It is seen that the degree of conversion occurring in the material's structure and the binding density of the molecules with each other are directly related. As the amount of inorganic filler increases, water absorption decreases [15].

As the amount of water absorption of resin composites increases, the deterioration in their structures will increase [16]. The presence of electropositive metal ions (such as barium and zinc) and hydrophilic materials (such as hydrophilic acid phosphate groups, HEMA) in composite resins makes the materials more susceptible to water absorption [17-19].

A recent study comparing the properties of bulk-fill and conventional composite resins found that the bulk-fill material showed less water absorption than other materials due to more hydrophobic molecules such as Bis-GMA, UDMA, and Bis-EMA [20].

In an article on the water absorption properties of Bis-EMA-based light-cured dental resins and composites, it was observed that water absorption increased over time. The reason for this result was thought by the author to have already desorbed some of the monomers that did not enter what was trapped in the discs [21].

The output wavelength of the standard light curing unit is generally in the visible range of 445 nm to 480 nm. However, photoinitiators Ivocerin are not efficiently activated at these wavelengths, resulting in a lower degree of conversion (DC) [22]. In 2015, Kolpakova concluded that Ivocerin-initiated composite resin takes longer to reach maximum irradiance, increasing slowly after approximately 9 seconds of irradiation [23].

In the study in which the 7-day water absorption of three composite materials with nanosized, microsized and milisized filler content was compared, it was observed that the water absorption values of all three composites were lower than the values required by the ISO 4049 standard, and no significant difference was observed when compared with each other. In this study, it was concluded that the size and distribution of the filler particles affected the water absorption minimally and did not create a significant difference [24]. In a recent study, when the water absorptions of a microhybrid composite and two nanofilled composites were evaluated on the first and thirteenth days, it was concluded that the water absorption of one of the nanofilled composites was significantly higher than the other two composites. Therefore this composite could show a weaker clinical performance [25].

In the study of Par et al., in which they examined the degree of polymerization of bulk fill composites, it was shown that the long-term polymerization of flowable bulk fill composite material produced with smart dentin replacement technology continued for up to 7 days after polymerization and that unreacted components were present in this process, which may cause solubility and water absorption [26].

In our study, when flowable bulk fill composite material produced with smart dentin replacement technology was compared in terms of water absorption between the 14th and 30th days, it was seen that the 30th day showed less water absorption rates, supporting this study.

Water sorption in composite materials is diffusion-controlled in the resin matrix. Therefore, the diffusion coefficient decreases with the concentration of water in the matrix. So in our study, we expect water absorption to decrease over time [27]. However, there are materials where we see increased water absorption on day 30 compared to day 14. One of them is Universal Sub-Micron Hybrid Composite Material in group 7. This material contains Bis-EMA in addition to Bis-GMA and exhibited behavior that supports the work of Sideridou I. Composite material in group 2, which increased on the 30th day, contains ivocerin initiator in addition to campharoquinone. Studies on the degree of polymerization of Ivocerin have shown that it polymerizes in a longer time than camphoroquinone. For this reason, we think that we have observed that water absorption does not decrease over time, but rather increases. Studies on the degree of polymerization of Ivocerin have shown that it polymerizes in a longer time than camphoroquinone. A composite material in group 2, which increased on the 30th day, contains ivocerin initiator and campharoquinone.For this reason, we have observed that water absorption does not decrease over time but increases.

Most of the composite resins available in the market contain hydrophobic and hydrophilic components in different ratios. When the contents reported by the manufacturers for the bulk fill composites, flowable bulk fill composites, nanofilled and mitohybrid composites used in our study are evaluated, it is seen that there is a certain amount of hydrophobic Bis-GMA, UDMA, Bis-EMA in all of them. The average water absorption values were found to be lower than the maximum water absorption value of 40 $\mu g/mm^3$ determined in ISO 4049. It was thought that the effect of hydrophobic components in the low water absorption values detected was large.

We think that if a similar study was planned with composite resin materials with reduced content of hydrophobic materials and increased content of hydrophilic compounds, high water absorption values and statistically significant differences between composites in this study could be seen.

In a recent study evaluating the water absorption and solubility in water as a result of fast and slow polymerization of bulk fill composites, it was seen that the degree of polymerization, that is, the formation of cross-links of the polymer network, is more important than the density of the inorganic and organic content of the composite resin [28]. In this respect, when current composites are evaluated, it is seen that studies that compare the polymerization degrees of composites are necessary. Our study needed to have been more comprehensive to observe in this regard.

In addition, 2mm samples were prepared for bulk fill composites since a study was planned with conventional composites. However, for a study conducted only with bulk fill composites, the preparation of 4 mm samples recommended by the manufacturers, supporting the production objectives, and advancing the study in this direction may lead to more exciting results. As a result of our work with 2 mm discs, we have experienced that all the materials we use have a water absorption of less than 40 $\mu g/mm^3$ and meet ISO standards.

In addition to these results, there is a need for more long-term studies with different composite materials.

4. Conclusions

As a result of comparing the 14th and 30th-day values of nine different composite materials whose water absorption was evaluated, no significant difference was found between each other. When the water absorption values of the same composites between 14 and 30 days were compared, no statistically significant difference was found.

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Manuscript received: 8.01.2023