

Water Sorption and Solubility of an Experimental Dental Material: Comparative Study

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Abstract

Aim: The physico-chemical properties of an experimental composite resin used for indirect dental restorations were investigated. The goal was to evaluate the variation of water sorption and solubility of the composite specimens during a 7 day period of immersion and to assess the effect of an additional post-curing treatment on these properties. *Material and Methods:* The specimens were prepared and investigated according to the ISO standard 4049:2000. Two group of specimens were studied i.e. light-cured and post-cured (group A) samples and light-cured (group B) samples. *Results:* Significant statistical differences were observed regarding water sorption in both groups ($p < 0.05$), the mean values being higher in the first days of immersion. Regarding the water solubility, negative values were obtained, that varied throughout the entire period of immersion; the solubility is masked by the higher values of water sorption. *Conclusion:* All the specimens present relative high water sorption values especially during the first three days of immersion. The additional post-curing treatment decreases the water sorption and solubility values.

Keywords: Composite resins; Water sorption; Solubility; Statistical data.

Introduction

The resin-based composite technology was one of the most significant contributions to restorative dentistry [1]. Nowadays, resin-based composites are the most frequently used biomaterials in restorative dentistry especially due to the increased aesthetic demands [2-5].

Composite materials consist of a resin matrix, filler (glass, quartz, ceramic) and a matrix-filler coupling agent. In the oral aqueous environment, resins absorb water and release unreacted monomers [6,7]. Usually, the organic matrix consists of a mixture of base monomers, especially dimethacrylates [8], while the inorganic fillers have different particle type, size, shape and morphology [9].

The mechanical and physico-chemical properties of the composite resins are influenced by the monomers structure and the filler content [10]. Generally, relative high filler content leads to

improvement of the mechanical and physico-chemical properties while reducing the polymerisation shrinkage, thermal expansion coefficient and water sorption [6,7].

Water sorption is diffusion –controlled process that can cause the chemical degradation of the material, followed by the break of inorganic filler-organic matrix link or the release of the residual monomers [11]. This phenomenon will determine a decrease of material’s mechanical properties, reducing the restoration durability [12,13]; the organic matrix properties can be permanently altered, and the qualities of the material compromised. But more concerning are the biological effects on organism caused by the compounds released from composite restoration through the phenomena of solubility [14].

In this study, the physico-chemical properties of an experimental composite resin used for indirect dental restorations were investigated. The goal was to evaluate the variation of water sorption and solubility of the composite specimens and to assess the effect of an additional post-curing treatment on these properties.

Material and Method

The indirect composite resin Barodent, manufactured by the Raluca Ripan Institute for Research in Chemistry, Babeş Bolyai University Cluj-Napoca, România is the experimental material under investigation. Barodent has an organic matrix based on Bis-GMA, TEGDMA and UDMA monomers, and an inorganic filler (about 65%wt) formed from barium oxide based glass, colloidal silica and quartz.

Twelve disc shaped specimens were prepared by filling a mould of 15 mm in diameter and 1 mm in thickness with the un-polymerised composite resin. Half of the specimens (Group A) were light-cured and additionally post-cured, while the other half of the specimens (Group B) were only light-cured. The basic polymerization was performed using a halogen light-curing unit Optilux 501®/ Kerr Corp (20 seconds/layer) while the post-curing treatment was performed according to the manufacturer’s protocol, at 135°C, under pressure in nitrogen atmosphere.

The water sorption (W_{sp}) and water solubility (W_{sl}) were determined according to the ISO standard 4049:2000 [15]. All the specimens were placed in a desiccator at 37°C for 24 hours, stored in another desiccator at 23°C and weighted to an accuracy of ± 0.1 mg using an AW220M (Shimadzu Corporation, Japan) balance. This cycle was repeated until a constant mass (m_1) was obtained. The specimens were immersed in distilled water at 37°C. At time intervals of 24 hours, the specimens were removed, dried, weighted and re-immersed, during one week.

The W_{sp} and W_{sl} values ($\mu\text{g}/\text{mm}^3$) were calculated with the following formulas [15]:

$$W_{sp} = \frac{m_2 - m_3}{V} \quad (1)$$

$$W_{sl} = \frac{m_1 - m_3}{V} \quad (2)$$

where: m_2 is the specimen’s weight after immersion in water; m_3 is the specimen’s weight after being kept in desiccator until constant weight; m_1 is the specimen’s weight before immersion in water; V is the specimen’s volume [15].

Statistics

Statistic analysis was performed using SPSS 13.0 and Microsoft EXCEL applications. Normal distribution was tested with Shapiro-Wilk test. For comparison of two means of a continuous variable on paired samples, paired-sample t-test or Wilcoxon test were used. For comparison of two means of a continuous variable in the case of independent samples, t-test or Mann-Whitney test were used. The significance level was $p=0.05$.

Results

The water sorption and solubility values were determined and compared for each day, during a 7 day period of immersion.

The water sorption and solubility values for the specimens from Group A, subjected to post-curing treatment are presented in table 1. After the first day of immersion, the water sorption was 16.04 ± 2.05 whereas at the end of the process, the value was 11.13 ± 1.78 ($p=0.03$). Significant differences regarding water sorption were observed only between the first three days ($p<0.05$) and also between days 4 and 5 ($p=0.02$). As for the water solubility, negative values were obtained, which varied from -5.28 ± 1.92 to -15.29 ± 3.51 . Significant differences were noticed for almost all intervals of immersion.

The results for water sorption and solubility for the Group B of specimens prepared without post-curing treatment are presented in table 2. After the first day, the water sorption was 16.04 ± 2.05 whereas the final value was 12.46 ± 1.52 ($p=0.03$). Significant differences were observed in the first three days ($p=0.02$ and 0.03) and the last two days of immersion ($p=0.03$). As for the water solubility, this varied between -2.83 ± 0.88 and -10.38 ± 0.59 ; the differences are significant through the entire period of immersion, with the exception of days 4 and 5 ($p>0.05$).

Table 1. Mean values and standard deviation for water sorption and solubility for Group A (n=6) specimens ($\mu\text{g}/\text{mm}^3$)/day

Day	Water sorption (Wsp)			Water solubility (Wsl)		
	Mean	SD	p	Mean	SD	p
Day 1	16.04	2.05	<u>0.03</u>	-5.28	1.92	<u>0.03</u>
Day 2	8.49	0.51		-14.72	1.01	
Day 2	8.49	0.51	<u>0.03</u>	-14.72	1.01	<u>0.03</u>
Day 3	11.51	0.29		-13.97	1.78	
Day 3	11.51	0.29	1.00	-13.97	1.78	0.06
Day 4	11.51	0.29		-13.21	2.39	
Day 4	11.51	0.29	<u>0.02</u>	-13.21	2.39	<u>0.03</u>
Day 5	12.64	0.29		-16.42	2.21	
Day 5	12.64	0.29	0.46	-16.42	2.21	0.11
Day 6	13.02	1.83		-13.02	2.82	
Day 6	13.02	1.83	0.11	-13.02	2.82	<u>0.03</u>
Day 7	11.13	1.78		-15.29	3.51	
Day 1	16.04	2.05	<u>0.03</u>	-5.28	1.92	<u>0.03</u>
Day 7	11.13	1.78		-15.29	3.51	

Table 2. Mean values and standard deviation for water sorption and solubility for Group B (n=6) specimens ($\mu\text{g}/\text{mm}^3$)/day

Day	Water sorption (Wsp)			Water solubility (Wsl)		
	Mean	SD	p	Mean	SD	p
Day 1	16.04	2.05	<u>0.03</u>	-2.83	0.88	<u>0.02</u>
Day 2	9.25	2.11		-10.19	1.52	
Day 2	9.25	2.11	<u>0.02</u>	-10.19	1.52	<u>0.03</u>
Day 3	10.38	2.05		-11.70	1.46	
Day 3	10.38	2.05	0.11	-11.70	1.46	<u>0.03</u>
Day 4	12.83	4.09		-9.44	1.63	
Day 4	12.83	4.09	0.23	-9.44	1.63	0.46
Day 5	14.53	1.17		-11.89	3.54	
Day 5	14.53	1.17	0.23	-11.89	3.54	<u>0.03</u>
Day 6	15.10	1.55		-7.36	1.01	
Day 6	15.10	1.55	<u>0.03</u>	-7.36	1.01	<u>0.02</u>
Day 7	12.46	1.52		-10.38	0.59	
Day 1	16.04	2.05	<u>0.03</u>	-2.83	0.88	<u>0.02</u>
Day 7	12.46	1.52		-10.38	0.59	

The variation of the water sorption (mean values, 95% confidence interval) for the investigated Groups A and B during the 7 days immersion time is comparatively depicted in Figure 1. The graph illustrates that the specimens from both groups have a similar behaviour regarding the water sorption phenomenon. The additional post-curing treatment decreases the mean W_{sp} values, but the differences are not significant. Between Group A and B, W_{sp} values are significantly different only in Day 5 ($p=0.007$); in the other days, W_{sp} values are not significantly different i.e. Day 1 $p=1.00$; Day 2 $p=1.00$; Day 3 $p=0.32$; Day 4 $p=0.32$; Day 6 $p=0.06$ and respectively Day 7 $p=0.20$.

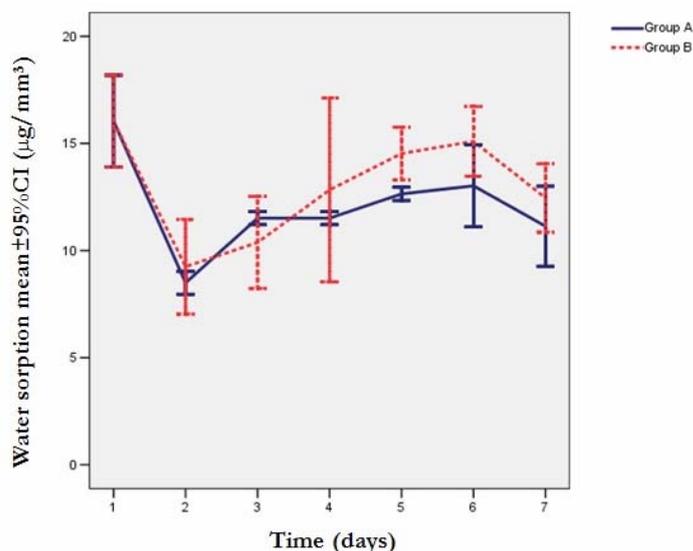


Figure 1. Water sorption for Groups A and B of specimens

The variation of the water solubility (mean values, 95% confidence interval) for the investigated Groups A and B during the 7 days immersion time is depicted in figure 2. It is obvious that, the specimens from both groups have a similar behaviour regarding the water solubility. The additional post-curing treatment decreases the mean W_{sl} values. Between Group A and B, W_{sl} values differ significantly as follows: Day 2 $p=0.007$, Day 3 $p=0.02$, Day 4 $p=0.049$, Day 6 $p=0.0009$ and Day 7 $p=0.01$. No significant differences between W_{sl} values for Group A and B are observed in the other days (Day 1 $p=1.00$ and Day 5 $p=0.11$).

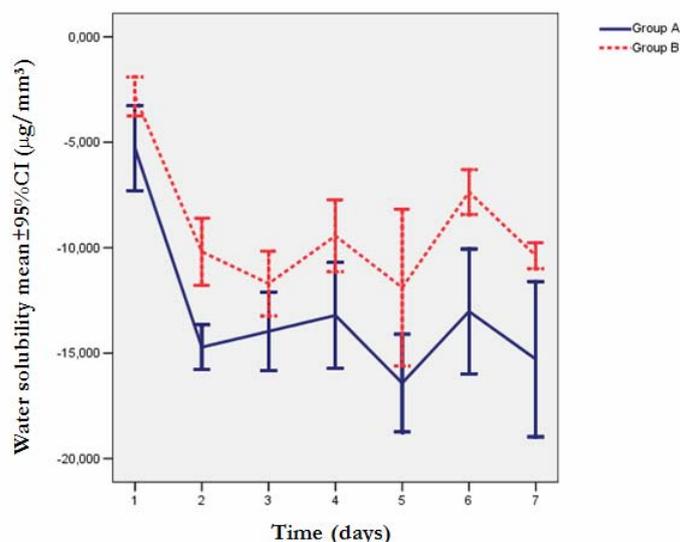


Figure 2. Water solubility for Groups A and B of specimens

Discussion

As stated in other research studies, the mechanical and physical properties of composite resins are influenced by the materials composition, i.e. the organic matrix type, the amount and type of filler [16,17].

The water sorption in the polymer matrix determines an increase in the total mass of the composites. The nature of the organic matrix i.e. the chemical structure of the monomers and their hydrophilicity influences the quantity and degree of hygroscopic expansion for any resin based restorative material [18]. Barodent composite has an organic matrix based on Bis-GMA and TEGDMA monomers, which are hydrophilic [19-21]. The values for the water sorption in Group A and B could be explained by the hydrophilic character of the matrix.

The amount and the type of inorganic filler influence the water sorption and solubility of composites, by decreasing the volume of polymers available for water sorption. Moreover, the composite resins containing filler based on silica or quartz are considered inert in water [14]. Although the experimental material under investigation has filler based on colloidal silica and quartz, the water sorption values are rather high. This could be probably explained by the relative reduced amount of filler.

For both groups of specimens, the water sorption value is higher in the first day of immersion; afterward, the samples absorb constantly water during the immersion period.

Specimens in Group A have lower values for water sorption than the specimens in Group B, but the differences are not significant ($p > 0.05$). This could be explained by the effect of the additional post-curing treatment that allows a higher degree of conversion of the polymers. As a result, the amount of un-polymerised monomers susceptible for water sorption is smaller. Moreover, the protocol for specimen preparation in disk shape, with a height of only 1 mm, is in the favour of an optimal polymerisation.

The solubility behaviour of composite resin materials is also affected by the organic matrix and filler types. All the investigated specimens have significant solubility values for almost the entire period of immersion. There are statistical significant differences between the specimens of the two groups ($p < 0.05$). The negative values obtained suggest that the composite material is more susceptible to water sorption, associated with an increase in weight of the specimens, which could mask the real solubility. This fact could be explained by the hydrophilicity of the organic matrix [22].

One can mention that the water sorption and solubility values are in accordance with the ISO 4049:2000, that is water sorption less than $40 \mu\text{g}/\text{mm}^3$ and solubility less than $7.5 \mu\text{g}/\text{mm}^3$.

Conclusions

The statistical analysis allowed the assessment of the experimental data regarding some of the physico-chemical properties of one experimental dental material i.e. the water sorption and solubility. The investigation was performed on specimens prepared by two different polymerisation protocols.

All the specimens present relative high water sorption values, especially at the beginning of the immersion period. The additional post-curing treatment decreases the water sorption and solubility values, in association with a better polymerisation degree. The solubility values were negative. All specimens exhibit a certain degree of solubility, but the real mass loss is masked by the higher water sorption.

However the water sorption and solubility values are in accordance with the ISO 4049:2000. The experimental dental material has optimal physico-chemical properties for an adequate behavior in the oral aqueous environment, making it suitable for indirect composite restorations.

List of abbreviations

Bis-GMA: Bisphenol A-glycidyl-methacrylate
TEGDMA: Triethylene glycol dimethacrylate
UDMA: Urethane dimethacrylate

Conflict of Interest

The authors declare that they have no conflict of interest.

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