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Study Regarding some Physico-chemical Properties of Composite Resins for Direct Restorations

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Abstract

Aim: This study investigated the physicochemical properties i.e. water sorption (Wsp) and solubility (Wsl) of one experimental composite in comparison with four commercially available materials used for direct restorations. *Material and Methods:* Disc-shaped specimens of each material were prepared and investigated according to the ISO standard 4049:2000. *Results*: The experimental composite had similar water sorption values with the trade-mark composites after 1 day storage. Significant differences regarding Wsp were found after 7 days and respectively 1 month of immersion ($p<0.05$). Similarities between the experimental composite and the trade-mark materials were observed regarding Wsl. *Conclusion*: The Wsp and Wsl values of the experimental material as well as of the trade-mark composites are in accordance with ISO standard 4049:2000, that is Wsp < 40 $\mu g/mm^3$ and Wsl <7.5 $\mu g/mm^3$.

Keywords: Water sorption; Solubility; Composite resin; Direct restoration

Introduction

Composite resins are considered the most frequently used restorative materials because of the increasing esthetic demands of the patients [1-3]. Over the years these resin-based composites continued to develop in the attempt to achieve a restorative material with optimal mechanical, physical and esthetic properties [4-6].

Composite resins consist of a mixture of resin matrix based on different monomers and filler particles [7]. They are usually classified on the basis of their filler particle size in macrofill, hybrid and microfill. Recently, after the introduction of nanotechnology in dentistry [8,9], a new class of composites, so called nanocomposites is available [10]. Thus, the new classification includes nanofilled, nanohybrid and microhybrid composites [10-12].

The properties of dental composites are determined by their chemical composition, particle size distribution and amount of the filler [12] and by the chemical structure of the organic matrix [13]. The flexural strength, degree of polymerization, hardness or wear resistance are some of the main characteristics of composite resins [11]. Water sorption and solubility of resin based materials are also important, since they influence the mechanical properties of the dental materials and longevity of composite restorations [11,14].

In a wet oral environment, composites absorb water and release unreacted monomers and inorganic ions. The water uptake in dental composites has deleterious effects on their mechanical /physical properties because of the hydrolytic breakdown of the bond between the silane –filler particles, filler-matrix debonding or hydrolytic degradation of the filler. In the same time, residual monomers and other components eluted from composites into the oral environment, weaken their mechanical properties. On the other hand, the absorbed water may cause a hygroscopic expansion of the composite that could compensate the polymerization shrinkage and improve the restoration seal [9]. The dimensional changes of resin composites immersed in water depend on the material characteristics [15].

The water sorption and solubility of some composite resins for indirect restorations (inlays) were reported in our previous works [16,17]. The present research is dedicated to the study of some physicochemical properties of dental materials used for direct restorations. In this respect, the water sorption and solubility of one experimental direct composite resin were investigated in comparison with four similar trade-mark materials.

Material and Methods

Investigated Materials

Five different dental composites, one experimental and four trade-mark materials were used in this study. The materials and their main characteristics are summarized in Table 1.

Table 1. Tested materials and their main characteristics

Material	Manufacturer	Code	Type	Composition *
Experimental material	ICCRR, Cluj-Napoca, Romania	C14	Nano-hybrid	**Matrix**: Bis-GMA, TEGDMA **Filler**: Barium-based glass; Quartz; Colloidal silica; Zinc- based glass **Filler content**: 80 wt%
Kalore	GC Corporation, Tokyo, Japan	KA	Nano-hybrid	**Matrix**: DX-511 Monomer (DuPont), DMA, UDMA **Filler**: Prepolymerized filler, containing strontium-based glass and lanthanoid fluoride; Glass microfiller, formed of fluoro aluminosilicate glass and strontium-based glass; Nanosilica; **Filler content**: 82 wt%
Beautifil- II	Shofu Inc, Kyoto, Japan	BF	Nano-hybrid	**Matrix**: Bis-GMA, TEGDMA **Filler**: Multi-functional glass; S-PRG filler based on fluoroboraluminosilicate glass **Filler content**: 83.3 wt%;
Venus	Heraeus-Kulzer, Hanau, Germany	VS	Micro-hybrid	**Matrix**: BIS-GMA,TEGDMA **Filler**: Barium-aluminium-fluoride glass; Highly dispersive silicon dioxide; **Filler content**: 79wt%
Premise	Kerr Corporation, Orange, USA	PD	Nano-hybrid	**Matrix**: Bis-EMA, TEGDMA **Filler**: Prepolymerized filler; Barium-based glass; Silica nanoparticles; **Filler content**: 84wt%

*Composition according to references [18] for Kalore, [19] for Beautifil II, [20] for Venus and [21] for Premise; Bis-GMA=Bisphenol-A glycidyl methacrylate, TEGDMA= triethylene glycol dimethacrylate, UDMA=Urethane dimethacrylate,DMA= dimethacrylate, Bis-EMA= ethoxylated bisphenol-A dimethacrylate, S-RPG= surface reaction type pre-reacted glass-ionomer

Specimen Preparation

For each investigated material 5 disc-shape specimens (15mm in diameter, 1 mm in height) were prepared by placing the composite resin into a special disc-shaped mold. The composite resin was then covered with plastic foil and light-cured for 20 seconds using a light activation unit (Demi LED Curing Unit, Kerr Corporation, Orange, CA, USA). Then, the specimens were removed from the mold and subjected to the water sorption and solubility tests.

Water Sorption and Solubility Protocol

The water sorption (Wsp) and water solubility (Wsl) were determined according to the ISO standard 4049:2000 [22]. The specimen discs were stored in a desiccator at 37°C for 24 hours, then stored in another desiccator at 23°C for 2 hours and then 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. Then they were kept in distilled water at 37°C for 1 month when all measurements were repeated.

The Wsp and Wsl values ($\mu g/mm^3$) were calculated with the following formulas:

$$\mathrm{Wsp} = \frac{\mathrm{m}_2 - \mathrm{m}_3}{\mathrm{V}} \tag{1}$$

and

$$\mathrm{Wsl} = \frac{\mathrm{m}_1 - \mathrm{m}_3}{\mathrm{V}}, \tag{2}$$

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 [22].

Statistical Analysis

Data were statistically analyzed using SPSS 18.0 and Statistica 8.0 software. Normal distribution of data was tested using the EasyFit Program with Kolmogorov-Smirnoff test at a level of significance of 0.05. For normally distributed data, mean values and standard deviations were reported. Water sorption and solubility comparison in dynamics were done with Student test for independent samples for different materials, in accordance with the equality and inequality of the variances, respecting the normality assumption. The specimens in each group were compared using ANOVA test and respecting the normality assumption. All tests were applied at a $p \leq 0.05$ level of significance.

Results

The water sorption and water solubility of the five materials were measured after different periods of immersion, namely 24 hours, 7 days and 1 month. The Wsp and Wsl mean values (Mean) and standard deviations (StDev) as well as the minimum (Min) and Maximum (Max) values of the materials are presented in Table 2.

Regarding water sorption, after 24 hours of immersion the tested materials had statistically similar values (p ANOVA =0.2848), but statistically significant differences were observed after 7 days and 1 month of storage as ANOVA test revealed a significance level of 0.032 and <0.0001 respectively. A Bonferroni test, applied to see between which of the materials there were significant differences, showed that after 7 days of immersion the experimental composite C14 absorbed water similarly to the trade-mark materials. Statistical differences were observed only between PD and VS composites (p=0.0282). After 1 month of storage, statistical significant differences were found between VS and the other investigated composites, PD (p<0.0001), KA (p=0.0002), BF (p=0.0091) and C14 (p=0.0014).

Table 2. Descriptive statistics for the investigated materials

Material code	Stat	Wsp			Wsl		
		1 day	7 days	1 month	1 day	7 days	1 month
PD	Mean	7.9264	9.8514	12.7955	-4.3029	-9.2852	-12.7955
	StDev	1.3278	3.6823	1.1740	0.6455	1.8606	1.6311
	Min	5.6617	6.7941	11.8896	-5.0955	-11.3234	-14.1543
	Max	9.0587	14.1543	14.7205	-3.3970	-7.3602	-10.7573
VS	Mean	8.6058	4.8691	7.0205	-4.4161	-10.3043	-17.3248
	StDev	3.8691	1.4764	2.1410	0.7382	0.7382	1.6795
	Min	3.9632	3.3970	4.5294	-5.0955	-11.3234	-19.2498
	Max	14.7205	6.7941	9.0587	-3.3970	-9.6249	-15.2866
KA	Mean	6.4544	8.3793	11.7764	-6.2279	-18.0042	-25.5909
	StDev	0.6455	1.5711	0.4737	0.5662	1.9776	1.3517
	Min	5.6617	6.7941	11.3234	-6.7941	-20.3822	-27.1762
	Max	7.3602	10.1911	12.4558	-5.6617	-15.8528	-23.7792
BF	Mean	9.5117	8.8323	10.9837	-4.9823	-13.8146	-25.5909
	StDev	0.4737	1.1740	1.4211	1.0128	2.0648	3.9713
	Min	9.0587	7.3602	9.6249	-6.2279	-15.8528	-29.4409
	Max	10.1911	10.1911	12.4558	-3.9632	-11.3234	-21.5145
C14	Mean	7.9264	7.9264	10.3043	-3.9632	-9.1720	-19.1366
	StDev	2.4021	2.6856	0.8398	0.6934	3.8274	2.5129
	Min	6.2279	5.6617	9.0587	-5.0955	-13.5881	-21.5145
	Max	11.8896	12.4558	11.3234	-3.3970	-5.6617	-16.4190

where PD = Premise; VS =Venus ; KA =Kalore; BF =Beautifill II ; C14 =Experimental composite; Wsp = water sorption; Wsl = water solubility; Stat = statistics;

Regarding water solubility, the investigated materials differed significantly after 24 hours, 7 days and 1 month of immersion, respectively (p ANOVA < 0.05). After 24 hours of immersion, the experimental material C14 had similar solubility with the other investigated materials. Statistical differences were observed for KA composite and PD, VS, and BF respectively ($p<0.05$). After 7 days of immersion, C14 differed significantly from BF ($p=4.83\cdot10^{-2}$), while after 1 month of immersion C14 differed significantly from VS ($p=2.82\cdot10^{-4}$), BF ($p=4.32\cdot10^{-3}$) and PD ($p=6.04\cdot10^{-7}$). Regarding the other materials, after 24 hours of immersion KA differed significantly from VS ($p=1.03\cdot10^{-2}$), and PD ($p=5.90\cdot10^{-3}$) respectively, after 7 days KA also differed from VS ($p=3.83\cdot10^{-4}$) and PD ($p=8.06\cdot10^{-5}$). After 1 month of immersion differences in solubility were observed between PD and KA ($p=6.04\cdot10^{-7}$), PD and BF ($p=5.13\cdot10^{-3}$), VS and KA ($p=2.82\cdot10^{-4}$) and VS and C14 ($p=2.82\cdot10^{-4}$).

Table 3 summarizes the comparison in dynamics for water sorption and solubility for each investigated material after the different periods of immersion.

The experimental composite C14 absorbed significantly more water between the seven days period and 1 month period of immersion ($p=1.14\cdot10^{-4}$). Regarding the solubility, statistically significant differences ($p<0.05$) were observed between all periods of immersion.

PD absorbed more water between 7 days and 1 month period ($p=1.82\cdot10^{-5}$), and between 1 day and 1 month period of immersion ($p=1.09\cdot10^{-2}$). The solubility values also differed between the 1 day and 7 days, respectively between the 1 day and 1 month periods.

VS had more water uptake between 7 days and 1 day period ($p=3.78\cdot10^{-2}$) and between 7 days and 1 month period ($p=9.33\cdot10^{-5}$). For KA statistical significant differences were observed between 7 days and 1 month of immersion ($p=4.62\cdot10^{-7}$) and 1 month and 1 day ($p=2.31\cdot10^{-4}$) respectively, while BF absorbed significant more water between 7 days and 1 month period ($p=1.94\cdot10^{-4}$). Significant differences ($p<0.05$) were noticed regarding solubility between all intervals of immersion for VS, KA and BF respectively.

Table 3. Comparison in dynamics of Wsp and Wsl between the values from 24 hours, 7 days and 1 month immersion

Material	Compared groups	Wsp		Wsl	
		Mean of diff	p-value	Mean of diff	p-value
PD (n=5)	1 day – 1 month	-4.8691	$1.09 \cdot 10^{-2}$	4.9823	$2.35 \cdot 10^{-3}$
	1st day – 7th day	-1.9250	$2.64 \cdot 10^{-1}$	8.4926	$5.86 \cdot 10^{-4}$
	7th day – 1 month	22.6469	$1.82 \cdot 10^{-5}$	3.5103	$8.26 \cdot 10^{-2}$
VS (n=5)	1 day – 1 month	1.5853	$3.79 \cdot 10^{-1}$	5.8882	$4.46 \cdot 10^{-4}$
	1st day – 7th day	3.7367	$3.78 \cdot 10^{-2}$	12.9087	$1.61 \cdot 10^{-4}$
	7th day – 1 month	22.1939	$9.33 \cdot 10^{-5}$	7.0205	$1.07 \cdot 10^{-4}$
KA (n=5)	1 day – 1 month	-5.3220	$2.31 \cdot 10^{-4}$	11.7764	$1.55 \cdot 10^{-4}$
	1st day – 7th day	-1.9250	$6.24 \cdot 10^{-2}$	19.3631	$2.17 \cdot 10^{-5}$
	7th day – 1 month	33.9703	$4.62 \cdot 10^{-7}$	7.5867	$2.30 \cdot 10^{-3}$
C14 (n=5)	1 day – 1 month	-1.4720	$9.78 \cdot 10^{-2}$	8.8323	$1.11 \cdot 10^{-3}$
	1st day – 7th day	0.6794	$3.05 \cdot 10^{-1}$	20.6086	$4.46 \cdot 10^{-4}$
	7th day – 1 month	34.4232	$1.14 \cdot 10^{-4}$	11.7764	$1.99 \cdot 10^{-4}$
BF (n=5)	1 day – 1 month	-2.3779	$1.49 \cdot 10^{-1}$	5.2088	$3.15 \cdot 10^{-2}$
	1st day – 7th day	0.0000	1.00	15.1734	$1.41 \cdot 10^{-4}$
	7th day – 1 month	27.0630	$1.94 \cdot 10^{-4}$	9.9646	$5.15 \cdot 10^{-4}$

where PD = Premise; VS =Venus ; KA =Kalore; BF =Beautifill II ; C14 =Experimental composite; Wsp = water sorption; Wsl = water solubility; n=sample size; Mean of diff=mean of difference

The comparative water sorption results, median, 25%-75% percentiles, minimum, maximum for each material are depicted in Figure 1.

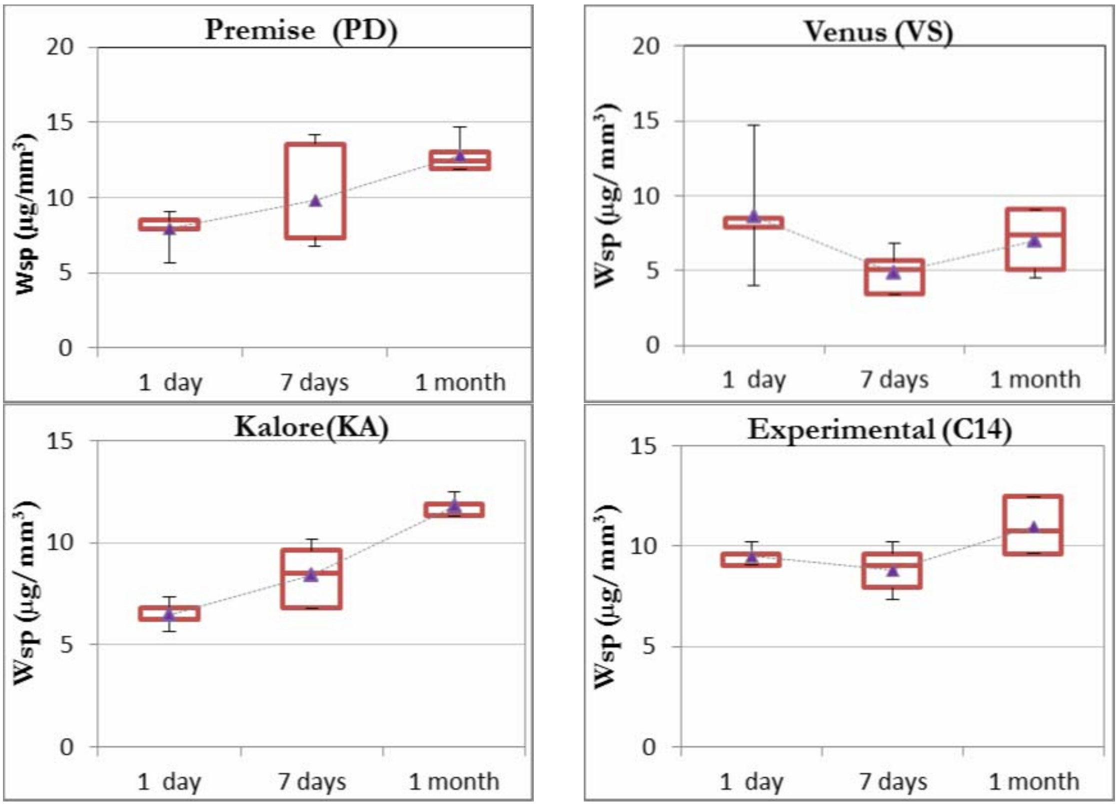

Figure 1. Water sorption (in µg/mm³) for the investigated materials

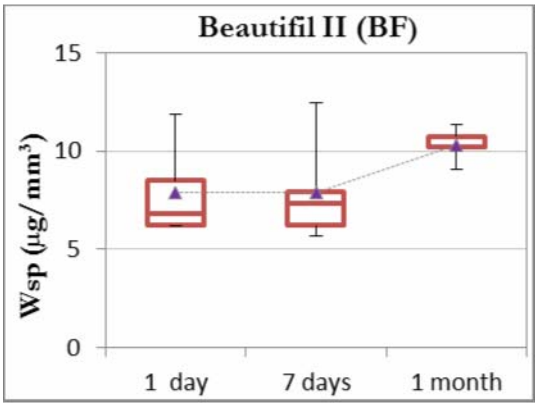

Figure 1. (*continuation*) Water sorption (in µg/mm^3) for Beautifil II

The graphics show the variations in water sorption for each investigated material after 24 hours, 7 days and 1 month of immersion. The Wsp values are ranged between approximately 5- 12 µg/mm^3.

Discussion

There are many factors that influence the water sorption of resin based materials, like the hydrophilicity of the organic matrix or the composition of the inorganic filler [11, 14, 15, and 23]. Most of the commercially available composites contain Bis-GMA based organic matrix. The high viscosity of this monomer requires the addition of some diluent monomers, like TEGDMA. These monomers associated with the presence of hydroxyl groups of Bis-GMA molecule, favor the increase of the water sorption [14, 24]. Various methods have been employed in order to reduce the hydrophilicity of Bis-GMA based systems, like using Bis-EMA or UDMA monomers, that are less hydrophilic [14, 25].

The experimental composite (C14) has an organic matrix based mainly on Bis-GMA and TEGDMA monomers. From the trade-mark composites, only BF and VS composite contain organic matrices based on Bis-GMA and TEGDMA. The matrix of the other materials, namely KA and PD consists in DX-511 Monomer (DuPont), DMA and UDMA, and Bis-EMA and TEGDMA, respectively. These differences could explain the results for Wsp values. C14 had Wsp values similar to the trade-mark materials. Although , after 24 hours of storage, C14 absorbed more water than the trade mark composites (9.5117±0.4737), no significant differences were noticed ($p>0.05$). After 7 days of immersion, C14 had similar water uptake with the trade-mark composites, as no statistical significant differences were observed between them. After 1 month of storage, C14 differed significantly in comparison with VS composite ($p=0.0014$). The experimental composite had significant higher Wsp values after 1 month (10.9837±1.4211) than after 7 days storage (8.8323±1.1740).

The amount of inorganic filler influences the Wsp and Wsl values of composite resins, by reducing the volume of polymers available for absorption [11, 15]. In the same time, the size, shape and type of filler influence the Wsp and Wsl values.

The investigated materials used in this study are nano-hybrid materials except the VS composite which is micro-hybrid. The nano-hybrid materials contain silica nanofiller that consists from both non-aggregated particles and agglomerated ones (nanoclusters) [23]. The porous nature of silica nanoclusters favors a higher water uptake. The agglomerated silica provides a large surface area allowing fluids to accumulate around the filler-polymer interface and leading to a higher water sorption [23]. This could explain the results in this study for water sorption. The experimental nano-hybrid composite C14 had similar Wsp values with the trade-mark composites. VS composite differed statistically in comparison with the others composites ($p<0.05$), probably because of the different type of filler. In this case, the micro-hybrid filler is formed from nano-sized particles dispersed through irregular shaped micro-particles.

The water solubility of the composite resins refers to the release of residual monomers or oligomers, and the leaching of ions from the filler surface [10,23]. Negative solubility values were recorded for both the experimental material and the trade-mark composites. There could be some explanations for these negative results, such as: incomplete removal of the absorbed water through desiccation [12], the fact that the real solubility is masked by a rather high value for the water sorption [26] or the occurrance of some chemical reactions between water and the composite[12].

Regarding the water solubility, significant differences were reported for all the investigated materials ($p<0.05$).

Conclusions

The water sorption and solubility of one experimental composite resin (C14) in comparison with four trade-mark materials namely Premise (PD), Venus (VS), Beautifil II (BF) and Kalore (KA) for direct restorations were investigated.

All the investigated materials present water sorption (Wsp) and water solubility (Wsl), but the values are in accordance with the ISO 4049:2000 that is Wsp less than 40 $\mu g/mm^3$ and Wsl less than 7.5 $\mu g/mm^3$, at the end of storage period. The water sorption results are directly related with both the chemical composition of the organic matrix and the amount and particle dimensions of the inorganic filler.

The experimental composite has similar water sorption with the commercial dental materials. After 1-30 days storage in water, C14 has similar water uptake with the trade-mark composites, as no statistical significant differences were observed between them. On the contrary, for the water solubility values, significant statistical differences were observed between all the investigated materials. The obtained solubility values are negative, due to the incomplete removal of water during desiccation or to the fact that the real solubility is masked by a rather high value for the water sorption.

Conflict of Interest

The authors declare that they have no conflict of interest.

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