Water Sorption and Solubility in Different Environments of Composite Luting Cements

An in vitro study

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The dimensional stability and structural integrity of composite luting cement are key factors of clinical performance and intraoral durability. The solubility both in water and saliva of a composite luting cement is influenced by the absorption of liquid by that cement. The aim of this study is to evaluate the solubility and water sorption of three different adhesive cements (Variolink, Nexus, Dualcim) in two different environments: artificial saliva (Artisial/Biocodex, France) and distilled water at different timed intervals. Water sorption is dependent on the chemical composition of the material, the time and the immersion medium.Water sorption was different for the three cements tested, at all three time periods, both in distilled water and in artificial saliva. ANOVA test results show that for all three materials used there are statistically significant differences (p <0.002) between the mean values for the three tested periods, both in terms of solubility and absorption. Between 7 and 14 days the water sorption continued to increase significantly for all materials tested, showing a stabilizing trend in most cases only between 14 to 21 days.

Key words: water sorption, absorption, solubility, artificial saliva, composite luting cements, adhesive cements

Water sorption and solubility of dental cements have been extensively evaluated in clinical and laboratory studies [1-3], given that the solubility and absorption may cause degradation of cement [4, 5].

Cement degradation leads to retention loss and/or fracture of the restoration [6, 7], to micro-leakage increasing [8] and promotes secondary caries [9]. Composite luting cements are very important for the adhesive cementation in fixed prosthodontics [10], but their behavior regarding water sorption and dimensional stability have not been adequately studied. Into a wet medium, the polymeric matrix of a composite luting agent may absorb water and increase its volume, resulting a decrease of both Young‘s modulus and mechanical strength [11].

Water sorption by the polymers is a diffusion-controlled process which mainly takes place in the resin matrix. Thus, the adhesive cements with a lower content of filler probably give a higher water sorption. The water absorbed by the polymer structure may cause detachment of the filler - matrix interface or hydrolytic degradation filler - resin interface. When the cement specimens are submerged in water, some of their components - such as the unreacted monomer or the filler - are dissolved in water, resulting in a loss of weight and volume which can be measured as the solubility [12]. Diluting those dissolved components of the cements can influence or change the original size of the cement specimens and their clinical performance and in the same time the aesthetic quality and even the biocompatibility of the restorations may change.

Experimental part

Three composite resin cements were included in this study was, respectively Variolink II, Nexus NX3 and Dualcim, whose compositions are detailed in table 1.

Table 1

CHEMICAL COMPOSITION OF THE MATERIALS USED IN THIS STUDY [13]

Composite cement	Monomers	Fillers
Dualcim	D1- Bis-GMA, TEGDMA, UDMA, DHEPT, CQ, DMAEM	70 wt % Aluminosilicate glass, colloidal silica, quartz
	D2- Bis-GMA, TEGDMA, UDMA, POB	70 wt % Aluminosilicate glass, colloidal silica, quartz
NX3	TEGDMA, Bis-GMA	70 wt% (47 vol%) fluoro-aluminosilicate glass
Variolink	Bis-GMA, UDMA, TEGDMA	73,4 wt % (46,7% vol) barium glass, ytterbium trifluoride, baroalumino fluorosilicate glass, mixed oxides

Bis-GMA- 2,2-bis (4- (2-hydroxy-3-metacrilopropoxi) phenyl) propane (synthesized ICCRR Cluj); UDMA- urethane-dimethacrylate (Merck); TEGDMA: triethylene glycol dimethacrylate (Aldrich) DMAEMA - N, Ndimetilaminometilmetacrilat (Aldrich); CQ - Camforochinonã (Merck), DHEPT- N, N dihidroxietil- para-toluidine

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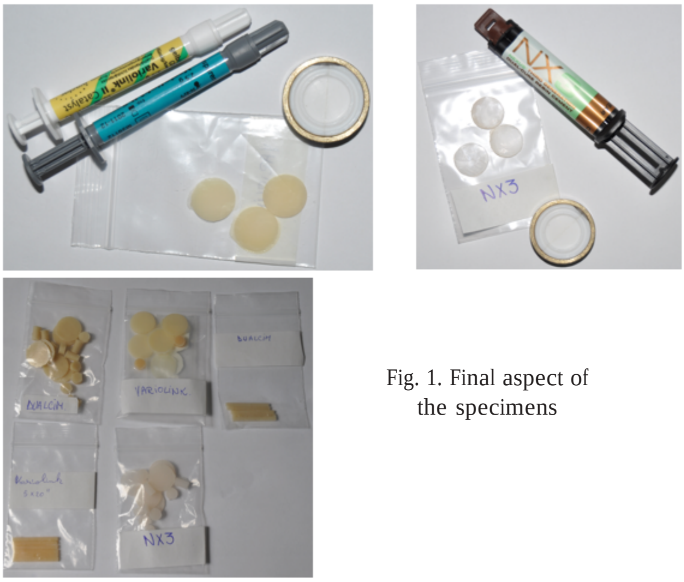

Fig. 1. Final aspect of the specimens

A metallic ring teflon matrix was used for fabrication of the specimens, which produced disc-shaped cement specimens with a 15 mm diameter and 1 mm thickness (fig. 1), according to ISO standard 4049/2002 [14]. The matrix was filled with the composite cement, then covered with a polyester strip and a glass plate, which were maintained under finger pressure until the completion of the fotopolymerisation process. The specimens' surface must be smooth and plane. Then the specimens were dehydrated in an oven at 37 ± 1°C for 24 h and weighted on a precision scale, recording a value $\mathbf{m_1}$. After this, the specimens were submerged in distilled water for 7 days and during this period they were weighted daily: each specimen was removed from the distilled water with tweezers, wiped with tissue paper and air dried for 15 seconds. After 1 minute from the removal from distilled water they must be weighed, recording a value $\mathbf{m_2}$. Then, the specimens were dehydrated again in the oven for 4 hours until they obtained a constant weight $\mathbf{m_3}$. This operation is repeated at 14 and 21 days. The water sorption value is calculated from each specimen according to the formula:

$$W_{sp} = (m_2 - m_3) / V$$

where:

$\mathbf{m_3}$ - specimen weight after 24 h of water immersion (μg);

$\mathbf{m_3}$- specimen weight after oven dehydration until a constant value (μg);

V - specimen volume (mm^3).

The value of water sorption for each of the cements investigated was considered to be the arithmetical mean of 5 measurements.

The protocol to evaluate the solubility of composite cements is similar to that described above for the water sorption. The difference is in the calculation formula that is obtained using experimental values expressed in mg/mm^3.

$$SL = (m_1 - m_3) / V$$

where:

$\mathbf{m_1}$ - specimen constant weight before water immersion (μg);

$\mathbf{m_3}$- specimen weight after oven dehydration until a constant value (μg);

V - specimen volume (mm^3).

In order to carry out the determination of solubility, we used the same materials as shown in table 1 and figure 1. The solubility values were recorded at 1, 2, 3, 4, 5, 6, 7, 14 and 21 days.

The results were statistically analyzed. They calculated the average values and standard deviations for the absorption of water and solubility of each composite and each of the two test environments, 7, 14, and 21 days.

Data were statistically analyzed. There were calculated the average values and standard deviations for the water sorption and solubility of each composite cement and each of the two test environments at 7, 14 and 21 days.

Data were statistically analyzed using ANOVA test for the three groups of materials and T test for comparison of each two groups of materials. Statistical significance is $p < 0.05$. To assess the correlation between solubility and water sorption Pearson index was used. For analysis and processing was used PASW Statistics 18 software.

Results and discussions

The results obtained from the analysis of water sorption and solubility in water and in artificial saliva for the materials studied are given in figures 2-5.

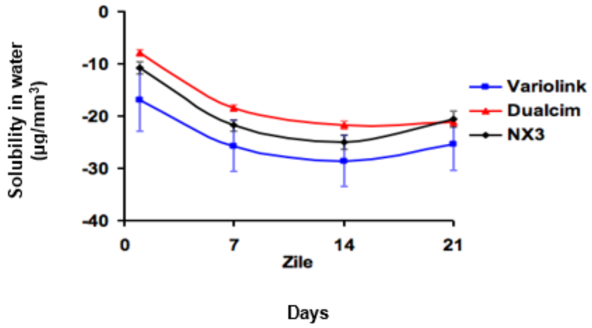

Fig. 2. Graphical representation of solubility values for samples immersed in water

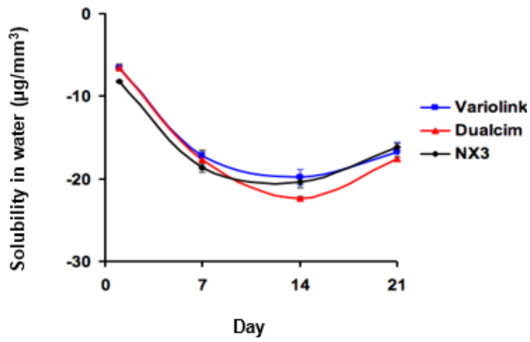

Fig. 3. Graphical representation of solubility values for samples immersed in artificial saliva

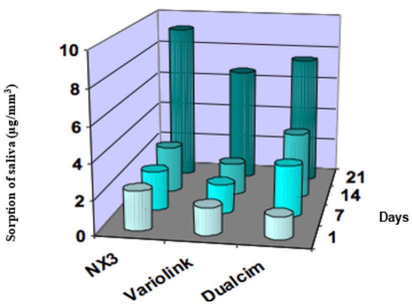

Fig. 4. Graphic representation of the values of water sorption of the samples immersed in water

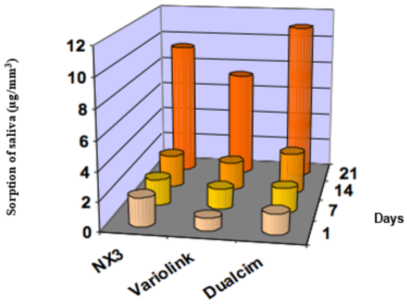

Fig. 5. Graphic representation of the values of water sorption of the samples immersed in artificial saliva

Table 2
MEAN VALUES, STANDARD DEVIATION AND STATISTICAL PARAMETERS FOR THE WATER SORPTION (mg / mm^3) AND SOLUBILITY IN WATER (mg / mm^3) AFTER 21 DAYS

	Cement Specimen	Mean (µg/mm^3)	SD	SEM	p (t test)			ANOVA	
					vs. NX3	vs. Variolink	vs. Dualcim	p	F
Water sorption	NX3	9.200	0.542	0.271	1.0000	0.0028	0.0052	**0.0001**	**30.24**
	Variolink	6.652	0.283	0.141	0.0028	1.0000	0.1027		
	Dualcim	7.501	0.542	0.271	0.0052	0.1027	1.0000		
Solubility	NX3	-20.52	3.009	1.504	1.0000	0.2768	0.6590	0.4736	0.8130
	Variolink	-25.47	9.729	4.864	0.2768	1.0000	0.4382		
	Dualcim	-21.23	1.497	0.748	0.6590	0.4382	1.0000		

SD - standard deviation, SEM - standard error of mean, p <0.05 (statistically significant)

In accordance with ISO standard 4029/2002 [14] the water sorption values must be less than 50 mg/mm^3 and solubility less than 5 g/mm^3. Water absorption values for all materials studied are within the ISO standard. Tables 2 and 3 show the mean values, standard deviation and statistical parameters for the water sorption and solubility in water and artificial saliva (mg/mm^3) after 21 days.

Statistical results show that for all three materials cementing there are significant differences (p <0.0001) for the 21 days between the average values of two tested mediums for the water sorption values (table 2).

Water absorption values of tested cement specimens are between 6.65 to 9.2 mg / mm^3. Low levels of water sorption were recorded for Variolink cement followed by Dualcim cement. Water sorption increased significantly for Nexus NX3 cementing material.

The solubility data obtained (table 2) after 21 days of testing are showing statistically significant differences in mean values for Dualcim cementing material.

Solubility values recorded in this study are negative and lie between - 20.52 and -25.47 mg/mm^3 at 21 days.

Average values for saliva absorption varies between 7.36 to 11.04 mg/mm^3 and the mean solubility in saliva after 21 days still stands at negative values between -16.13 and -17.55 mg/mm3.

The data obtained for solubility in saliva (table 3) after 21 days of testing show statistically significant different for the mean values of Nexus NX3 and Dualcim cementing materials.

Table 4 presents comparative statistical analysis for water sorption and solubility between the group immersed in water and the group immersed in saliva at 7 days. T-test results show that average values of the two groups did not differ statistically significant both in the case of solubility and water sorption for all three cementation materials tested, except the difference between the levels of water sorption for Dualcim cement.

Table 3
MEAN VALUES, STANDARD DEVIATION AND STATISTICAL PARAMETERS FOR SALIVA ABSORPTION (mg / mm^3) AND SOLUBILITY IN SALIVA (mg / mm^3) AFTER 21 DAYS

	Cement specimen	Mean (µg/mm^3)	SD	SEM	p (T test)			ANOVA	
					vs. NX3	vs. Variolink	vs. Dualcim	p	F
Absorption	NX3	9.341	0.326	0.163	1.0000	0.0272	0.0000	0.0000	63.50
	Variolink	7.360	0.653	0.326	0.0272	1.0000	0.0049		
	Dualcim	11.04	0.326	0.163	0.0000	0.0049	1.0000		
Solubility	NX3	-16.13	0.980	0.490	1.0000	0.4501	0.0304	0.4223	0.950
	Variolink	-16.70	2.288	1.144	0.4501	1.0000	0.4557		
	Dualcim	-17.55	0.462	0.231	0.0304	0.4557	1.0000		

SD - standard deviation, SEM - standard error of mean, p <0.05 (statistically significant)

Material		Medium	7 days		
			Mean	SD	P
NX3	Absorption	Water	2.2646	0.462	0.3189
		Saliva	1.8400	0.283	
	Solubility	Water	-21.13	1.819	0.1333
		Saliva	-18.68	0.980	
Variolink	Absorption	Water	1.6985	0.653	0.4950
		Saliva	1.4154	0.326	
	Solubility	Water	-25.76	9.800	0.1904
		Saliva	-17.26	0.980	
Dualcim	Absorption	Water	2.9723	0.542	0.0028
		Saliva	1.6985	0.462	
	Solubility	Water	-18.40	1.084	0.6770
		Saliva	-17.69	2.374	

Table 4
THE COMPARATIVE STATISTICAL ANALYSIS OF ABSORPTION AND SOLUBILITY BETWEEN THE GROUP IMMERSED IN THE WATER AND THAT IN SALIVA AT 7 DAYS

Material		Medium	21 days		
			Mean	SD	P
NX3	Absorption	Water	9.200	0.542	0.6376
		Saliva	9.342	0.327	
	Solubility	Water	-20.52	3.009	0.0565
		Saliva	-16.13	0.981	
Variolink	Absorption	Water	6.653	0.283	0.0796
		Saliva	7.360	0.654	
	Solubility	Water	-25.47	9.730	0.1911
		Saliva	-16.70	2.288	
Dualcim	Absorption	Water	7.502	0.542	0.0009
		Saliva	11.04	0.327	
	Solubility	Water	-21.23	1.498	0.0204
		Saliva	-17.55	0.462	

Table 5
THE COMPARATIVE STATISTICAL ANALYSIS FOR ABSORPTION AND SOLUBILITY BETWEEN GROUP IMMERSED IN WATER THAT IN SALIVA AT 21 DAYS

The comparative statistical analysis for absorption and solubility between the group immersed in water and that in saliva at 21 days is shown in table 5. The test results revealed that between the average values of the two groups there are statistically significant differences in the absorption and solubility for Dualcim cement.

ANOVA test results show that there are statistically significant differences ($p < 0.002$) between all three cementing materials tested for the mean values of the three test periods, both in terms of solubility and absorption. PostHoc Scheffe test shows the periods where the differences between mean values are statistically significant (table 6-9).

The comparative statistical analysis of difference between test periods for water solubility is shown in table 6. In case of water solubility, the differences are statistically significant for all three cements tested at 7 and 14 days, but no differences between 7 to 21 days are statistically significant for all three materials tested.

Between 7 to 21 days the differences are statistically significant for Nexus NX3 and Variolink cements, but for Dualcim cement there are statistically significant differences within 7-14 days.

Regarding the water sorption (table 7), the differences are statistically significant for composite cements Nexus NX3, Variolink and Dualcim between 7-21 days, but the range of 7-14 days the differences are not statistically significant for Nexus NX3 and Variolink cements. In the range 14 - 21 days the differences are statistically significant for all three cements tested.

Table 6
THE COMPARATIVE STATISTICAL ANALYSIS OF DIFFERENCE BETWEEN TEST PERIODS FOR WATER SOLUBILITY

Period	NX3			Variolink			Dualcim		
	Mean	SD	p	Mean	SD	p	Mean	SD	P
7 days	-21.79	1.98	0.0139	-25.76	9.80	0.0011	-18.40	1.08	0.0012
14 days	-25.05	2.63		-28.59	9.70		-21.65	1.55	
7 days	-21.79	1.98	0.2151	-25.76	9.80	0.1816	-18.40	1.08	0.0917
21 days	-20.52	3.00		-25.47	9.72		-21.23	1.49	
14 days	-25.05	2.63	0.0003	-28.59	9.70	0.0003	-21.65	1.55	0.7644
21 days	-20.52	3.00		-25.47	9.72		-21.23	1.49	

Table 7
THE COMPARATIVE STATISTICAL ANALYSIS OF DIFFERENCE BETWEEN TEST PERIODS FOR WATER SORPTION

Period	NX3			Variolink			Dualcim		
	Mean	SD	p	Mean	SD	p	Mean	SD	p
7 days	2.265	0.462		1.699	0.654		2.972	0.542	
14 days	2.689	0.283	0.2151	1.840	0.542	0.3910	3.822	0.849	0.0138
7 days	2.265	0.462		1.699	0.654		2.972	0.542	
21 days	9.200	0.542	0.0001	6.653	0.283	0.0004	7.502	0.542	0.0003
14 days	2.689	0.283		1.840	0.542		3.822	0.849	
21 days	9.200	0.542	0.0000	6.653	0.283	0.0004	7.502	0.542	0.0021

Table 8
STATISTICAL ANALYSIS OF DIFFERENCES BETWEEN THE COMPARATIVE TEST PERIODS FOR SOLUBILITY IN SALIVA

Period	NX3			Variolink			Dualcim		
	Mean	SD	p	Mean	SD	p	Mean	SD	p
7 days	-18.68	0.98		-17.27	0.98		-17.69	2.37	
14 days	-20.76	1.31	0.0081	-19.82	1.96	0.0138	-22.36	0.33	0.0350
7 days	-18.68	0.98		-17.27	0.98		-17.69	2.37	
21 days	-16.42	0.98	0.0000	-16.70	2.29	0.4501	-17.55	0.46	0.9244
14 days	-20.38	1.31		-19.82	1.96		-22.36	0.33	
21 days	-16.14	0.98	0.0001	-16.70	2.29	0.0003	-17.55	0.46	0.0001

The comparative statistical analysis of the differences between periods for solubility in saliva is shown in table 8. There are statistically significant differences between 7-14 days and 14-21 days for all three materials tested cement. Between 7 to 21 days are not statistically significant differences for any of cementing materials tested.

There are statistically significant differences in the period 14-21 days for Variolink cement and between 7-14 days for Dualcim cement regarding saliva absorption (table 9).

Table 9
STATISTICAL ANALYSIS OF DIFFERENCES BETWEEN THE COMPARATIVE TEST PERIODS SALIVA ABSORPTION

Period	NX3			Variolink			Dualcim		
	Mean	SD	p	Mean	SD	p	Mean	SD	p
7 days	1.840	0.283	0.2151	1.415	0.327	0.0000	1.699	0.462	0.0163
14 days	2.265	0.654		1.982	0.327		2.831	0.000	
7 days	1.840	0.283	0.0000	1.415	0.327	0.0000	1.699	0.462	0.0000
21 days	9.342	0.327		7.360	0.654		11.04	0.327	
14 days	2.265	0.654	0.0000	1.982	0.327	0.0001	2.831	0.000	0.0000
21 days	9.342	0.327		7.360	0.654		11.04	0.327	

For Nexus NX3 there is no statistically significant differences in any trial period and the same for Variolink cement between 7-14 days and 7-21 days.

The water penetrates into the resin based materials by various mechanisms, such as direct diffusion into the matrix resin, the penetration of the voids incorporated into the resin or cracks that are already present in the material generated by hydrolysis or the movement of water along the interface filler-matrix [15]. Therefore, differences in water sorption values observed for the three investigated resin cements have been found to be related to the type of resin used and the content of the filler.

For a given amount of material, as the volume fraction of the filler is higher, the less will be the volume occupied by the resin matrix and therefore a smaller degree of water sorption will be possible. Moreover, the nature of the monomers forming the matrix is hydrophobic. The cements investigated have Bis-GMA based composition, that has two hydroxyl groups per molecule and form hydrogen bonds with the absorbed water.

It is difficult to correlate absorption and solubility data with those obtained in other studies because the results will inevitably be different due to different laboratory work, different time periods and different units of measurement [16]. Moreover, comparisons are difficult to make because of reported differences in size specimens - a factor that will lead to different periods of time necessary for the water to infiltrate completely into the polymer matrix. A smaller specimen has a shorter balancing water period and more water-absorbing materials need more time to stabilise [17]. A weak point of the test is that the absorption of water involves an increase in weight of the samples, representing earnings of water.

However, in reality, it is the difference between the gain in water and the dissolution of low molecular weight monomers. Therefore, the true values of water sorption could be somewhat higher than those reported [16]. Moreover, it notes that the constant manipulation of a specimen can also cause wear surface, which leads to weight reduction. Therefore, studies that determine the water sorption and solubility of the resin materials are especially important for relative values, but numerical comparisons are not always possible.

In the same group of cement, water sorption may vary depending on the content and type of filler material. Although, there cannot be a significant absorption of water for the filler particles itself, the monomers absorb more water and the absorption of additional water is done most likely at the interface between the particles of the anorganic filler and the resin matrix. It appears that water diffuses through the resin and reacts with the filler along the filler-matrix interface [18]. Such a diffusion process requires a longer time compared to a reaction at the surface of the sample, held in the current study. Therefore, the observed differences in the water sorption could be attributed to the nature of filler particles.

Some factors that influence the solubility include concentration of the filler and mean particle size, the coupling agent, the nature of the filler particles and the type of solvent used [19].

The effect of the saliva in the properties degradation of the composite material is not investigated in many studies. It is known that the presence of a high content of proteins and enzymes, may increase the number of degradation reactions in the polymer matrix, but there are no studies showing that during the enzymatic degradation of polymers in dental materials, saliva would be the one to induce these changes. Since the reaction of material degradation is a long process, tests made by us have been followed in artificial saliva. Strength of materials depends on their inherent properties [20].

The behaviour of cementing materials in artificial saliva tracks trends seen in their behaviour in water; the small differences observed are not indicative of establishing the causes that generate them.

In one study, Vrochari et al. [21] have tested the absorption and solubility in water of some cements and found that Maxcem and Biscem had a high water sorption. RelyX Unicem and Multilink Sprint showed water sorption values only slightly higher than the control group. Regarding water solubility, the largest weight loss was found for Maxcem. Multilink Sprint showed no change in weight and Biscem proved to have low solubility.

In this study, when the medium was water, higher values of absorption were obtained for Nexus NX3 throughout the whole testing period. Negative values of the solubility of all three materials would probably be explained by elution of the components and the degree of conversion of the monomers, ranging different among these materials. Changing the size of the samples showed similar patterns of cement for both storage solutions. Overall, all cements presented had a reduced final volume after drying. This was thought to be due to loss of the unpolymerized components which are soluble in the storage solutions and will be replaced with water later.

Conclusions

The study shows a low water sorption for Dualcim and Variolink cements for the two tested mediums. Higher absorption values were obtained for Nexus NX3 throughout the whole test period, when the medium was water. Negative values of the solubility of all the three tested cementing materials could probably be explained by elution of the components and by the degree of conversion of the monomers, which varies differently for these materials.

Absorption was within the maximum value allowed by ISO 4049/2002 for all materials, but the testing period provided by this standard is plausible not to predict well enough the behavior of long-term absorption.

Absorption and solubility testing is technically difficult and does not accurately predict the intraoral behavior of cementing materials.

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