

Water sorption and water solubility of current luting cements: An in vitro study

David Alain Gerdolle, DDS, MS¹/Eric Mortier, DDS, MS²/
Bruno Jacquot, DDS, MS³/Marc M. Panighi, PhD⁴

Objective: To evaluate the water-sorption characteristics and the solubility behavior of 4 luting cements—2 composite resins (CRs), a polyacid-modified composite resin (PMCR), and a resin-modified glass-ionomer cement (RMGIC)—according to the ISO 4049 specifications. **Method and Materials:** Five disks (15 × 1 mm) of each material (Variolink II [CR1; Vivadent], Panavia F [CR2; Kuraray], Resinomer [PMCR; Bisco], and Fuji Plus [RMGIC; GC]) were prepared according to the manufacturers' instructions. Specimens were first desiccated until a consistent mass was obtained. Specimens were immersed for 7 days in distilled water and immediately weighed after this period. Then the disks were post-desiccated and weighed every day for 35 days. The water sorption and solubility of each specimen were calculated according to the change in its weight as observed before and after immersion and desiccation periods. **Results:** Fuji Plus exhibited the overall higher values of water sorption and solubility ($P < .001$). Of the 3 resin-based luting cements, Resinomer demonstrated significantly higher water sorption and solubility ($P < .001$), whereas Variolink II and Panavia F showed low values and were not significantly different ($P = .2$). **Conclusions:** Behavior of resin-based materials in water varies according to the composition characteristics. In particular, the high portion of hydrophilic chemical species, as well as the filler characteristics, provoke very high water sorption and solubility values. In this way, and within the limitations of this in vitro study, composite resin luting cements appear to be more suitable than compomers and resin-modified glass-ionomer cements to meet longevity requirements. (*Quintessence Int* 2008;39:265.e107–114)

Key words: dental polymers, ISO 4049 specifications, luting cements, solubility, water sorption

Dental composite materials (ie, acrylic resin-based polymer materials) constitute an important group of materials in modern prosthetic and esthetic dentistry.¹ Resin luting cements now account for a growing proportion of cement use, because of their ability to bond to dental tissues and their better mechanical properties compared with conventional luting agents.² Acceptable clinical longevity of composite materials has been

shown,^{3,4} although problems due to microleakage have been reported.⁵ Failures due to deteriorated mechanical properties and wear may be explained by the influence of moisture from the oral environment on the luting cement, leading to degradation and subsequent microleakage.⁶ In oral conditions, indeed, prosthodontic restorations are often close to the gingival crevice and in contact with the sulcular fluid. Therefore, the luting cement's water sorption and solubility behavior is of considerable importance from a clinical point of view.

Fluid uptake into the resin phase may have both beneficial and detrimental consequences. The dimensional changes, provoked by water uptake, may compensate for any initial polymerization contraction.⁷ Hygroscopic expansion may relieve the residual

¹Private practice, Vevey, Switzerland.

²Senior Lecturer, Conservative Dentistry Department, Faculty of Odontology, Nancy, France.

³Senior Lecturer, Biomaterials Department, Faculty of Odontology, Montpellier, France.

⁴Professor and Chairman, Conservative Dentistry Department, Faculty of Odontology, Nancy, France.

Correspondence: Dr D.A. Gerdolle, rue du Collège 3, 1800 Vevey, Switzerland. E-mail: davidgerdolle@bluewin.ch

Code	Material (manufacturer)	Type	Main components	Batch No.
CR1	Variolink II (Vivadent)	Composite resin	<i>Monomers</i> (26.6 wt%): bis-GMA (13 wt%), UDMA (6.6 wt%), TEGDMA (7 wt%) <i>Fillers</i> (73 wt%): silanized barium glass (38 wt%), ytterbium trifluoride (25 wt%), Ba-Al-F-silicate glass (5 wt%), silanized spheroid mixed oxide (wt 5%) <i>Additional contents</i> (0.4 wt%): catalysts, stabilizers, pigments	Base: C09470 Catalyst: C09915
CR2	Panavia F (Kuraray)	Composite resin	<i>Monomers</i> (21.5 wt%): bis-EMA, phosphate monomer MDP <i>Fillers</i> (78 wt%): silanized barium glass, silanized silicate glass <i>Additional contents</i> (0.5 wt%): sodium fluoride, BPO, N,N-diethanol p-toluidine	Paste A: 00085A Paste B: 00039A
PMCR	Resinomer (Bisco)	Polyacid-modified composite resin (compomer)	<i>Monomers</i> (41 wt%): bis-GMA (13 wt%), DPSDMA (10 wt%), TEGDMA (12 wt%), HEMA (6 wt%) <i>Fillers</i> (57 wt%): Ba-Al-F-silicate glass (27 wt%), Sr-silicate glass (25 wt%), amorphous silicate glass (5 wt%) <i>Additional contents</i> (2 wt%): sodium fluoride	
RMGIC	Fuji Plus (GC)	Resin-modified glass ionomer	<i>Powder</i> : fluoroaluminosilicate <i>Liquid</i> : copolymer of acrylic and maleic acid, HEMA, UDMA, water, initiator Powder/liquid ratio: 2/1	Powder: 070881 Liquid: 240781

(bis-GMA) bisphenol-A glycidyl methacrylate; (UDMA) urethane dimethacrylate; (TEGDMA) triethylene glycol dimethacrylate; (Ba) barium; (Al) aluminum; (F) fluorine; (bis-EMA) ethoxylated bisphenol-A glycol dimethacrylate; (MDP) 10-methacryloxy dihydrogen phosphate; (BPO) benzoyl peroxide; (DPSDMA) diphenylsulfone dimethacrylate; (HEMA) 2-hydroxyethyl methacrylate; (Sr) strontium.
* All information was provided by the manufacturers.

stresses set up inside the resin phase during polymerization shrinkage.⁸ Swelling of the resin matrix may also reduce both interfacial gap width and corresponding microleakage.⁵ If most polymerization occurs within the first minute, fluid sorption takes several days, and during that period, the gap would become contaminated by salivary microorganisms before material expansion can close the interfacial fissure.⁸ On the other hand, the fluids that penetrate the resin phase will act as a plasticizer and may lead to the degradation of the filler resin interface⁹; it may decrease the flexion strength and the elastic modulus.⁶ The overall result could gather together a decrease in mechanical properties, a microleakage improvement, and an esthetic failure by cement discoloration.

Conversion of monomers into a polymer network is never complete, either for the composite resins, the polyacid-modified compos-

ite resins (so-called compomers), or the resin-modified glass-ionomer cements. Consequently, chemical substances could be re-leased by the material into the environment, bringing into question the biocompatibility of dental restoration products.¹⁰ Residual monomers, fillers, activators, and inhibitors of polymerization or degradation products such as formaldehyde and methacrylic acid are thus able to leach out from resin-based materials.¹⁰⁻¹² These molecules are potentially hazardous to the surrounding soft tissues.¹³ Currently, there are 3 main types of luting cement used for bonded prosthodontics: composite resin (CR), polyacid-modified composite resin (PMCR), and resin-modified glass-ionomer cement (RMGIC). Although each type is chemically and physically different,² none is ideal nor can be applied in all clinical situations.¹⁴

Table 2 Data relating to water sorption and water solubility

Material	Mean water sorption (W_{sp}) and water solubility (W_{si}) ($\mu\text{g}/\text{mm}^3$)*		Mass gain (M_g) and mass loss (M_l) (%)	
	W_{sp} (SD)	W_{si} (SD)	M_g	M_l
CR1	17.9 (1) ^a	4.8 (1.1) ^a	0.85	0.23
CR2	21.6 (3.1) ^a	4.9 (1.4) ^a	1.16	0.27
PMCR	60.3 (4.3) ^b	7.9 (1.4) ^a	3.49	0.48
RMGIC	230.2 (48.9) ^c	21.7 (3.9) ^b	11.24	1.18

See Table 1 for more details of materials.

* Results with the same superscript letter are not statistically different.

The purpose of the present in vitro investigation was to determine the water sorption characteristics and solubility behavior of 3 resin-based materials (2 composite resins and a compomer) and compare them to the same characteristics of a resin-modified glass ionomer. The hypothesis was that regarding chemical compositions, there would be no significant difference among the resin-based luting cements, but the difference between the resin-based cements and the resin-modified glass-ionomer cement would be significant.

METHOD AND MATERIALS

The luting cements used in the present study are listed in Table 1. Five disks of each material were made according to the International Organization for Standardization (ISO) specification 4049:07-2000.¹⁵ Specimens were made in a jig consisting of a Teflon mold (15 mm in diameter by 1 mm in thickness) compressed between 2 glass slabs with 50- μm -thick polyester separating sheets; materials were packed into the molds, care being taken to minimize air inclusion. A visible light-polymerizing unit with an irradiating diameter of 9 mm (Helipar Highlight, 3M ESPE) was used to polymerize the cements. Light-activation energy was controlled to assure a minimum value of 600 mW/cm². The light tip was first directed over the center of the sample for 40 seconds and then irradiated in 8 peripheral overlapping sectors for 20 seconds each. The specimens were immediately stored in an incubator at 37 °C for 15 minutes. The excess material was then removed with a scalpel. The rim was trimmed and polished with 1,000-grit

silicon carbide grinding paper until the diameter of the final product was maintained within 14.9 ± 0.1 mm average diameter, obtained from 2 measurements at perpendicular planes with an electronic caliper (Digimatic, model 500-181U, Mitutoyo).

After that, the specimens were immediately placed in a desiccator together with silica gel (Silicagel, Prolabo), and the whole set was stored in an incubator maintained at $37^\circ\text{C} \pm 1^\circ\text{C}$. According to the ISO standard, disks were weighed every day for 35 days using an analytical balance (Model M-120, Denver Instrument) with a repeatability of 0.1 mg, until a constant mass (m_1) was obtained (ie, until the mass loss or gain of each sample was less than ± 0.2 mg in any 24-hour period). The volume (V) of the specimens was then determined by measuring with an electronic caliper the specimen diameter from 2 perpendicular planes and the thickness from 5 measurements, 1 at the center and 4 at equally spaced points on the specimen circumference. This initial cycle allows the "loosely bound" water to be removed, as recommended in the ISO standard test.

Each specimen was then suspended in a flask containing 40 mL of distilled water and stored in the incubator at $37^\circ\text{C} \pm 1^\circ\text{C}$. After 12 hours, the specimens were removed, washed briefly with water, wiped up with absorbent paper, and shaken in air for 15 seconds. Specimens were weighed 1 minute after removal from the incubator to record their mass. The same procedure was repeated at 24, 36, 48, 96, and 168 hours, when the final mass (m_2) was recorded. This second cycle gives a combination of water sorption and dissolution of the soluble components from the specimen.

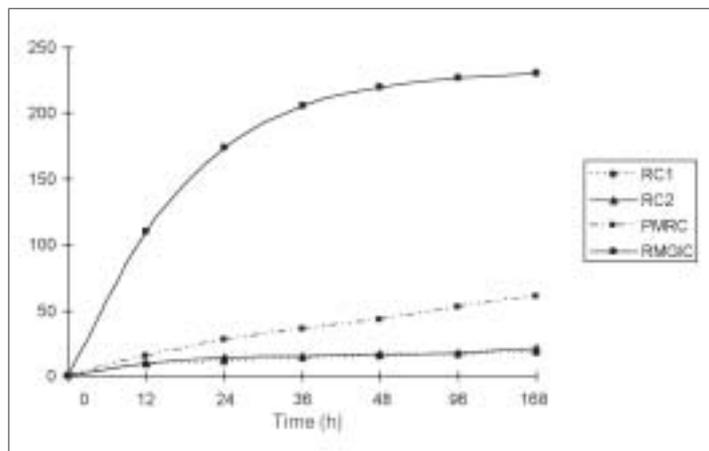


Fig 1 Water sorption kinetic.

After weighing, the disks were reconditioned at a constant mass (m_3) in a desiccator and incubator in the same manner as the stages of initial cycling described before. This third cycle allows measurement of the mass loss.

The values for sorption (W_{sp}) and solubility (W_{sl}) at 168 hours were calculated in $\mu\text{g}/\text{mm}^{-3}$ for each specimen using the following equations:

$$W_{sp} = \frac{m_2 - m_3}{V} \quad W_{sl} = \frac{m_1 - m_3}{V}$$

where m_1 is the mass (μg) before immersion in water; m_2 is the mass (μg) after immersion in water for 7 days; m_3 is the reconditioned mass (μg); and V is the volume of the specimen (mm^3). Statistical analysis of the results was conducted with Kruskal-Wallis tests and Bonferroni correction multiple comparison tests.

RESULTS

The results of the water sorption and solubility are presented in Table 2 for all the materials tested. The mean values and standard deviations were calculated for each series tested. According to the Kruskal-Wallis tests ($P = .05$), the luting cements absorbed and solubilized in a significantly different way from each other ($P = .001$). Bonferroni cor-

rection multiple comparison procedures ($P = .05$) show that RMGIC has significantly higher water sorption values than all the other materials ($P < .001$). In addition, the water sorption of the RMGIC was nearly 4 times that of the PMCR cement and 12 times that of the composite resin cements. The PMCR luting agent has significantly higher water sorption values than the CR1 and CR2 materials ($P < .001$), which are not different from each other ($P = .2$). Comparison of the water sorption values at 12, 24, 36, 48, 96, and 168 hours (Fig 1) showed that the water uptake occurred mostly in the first 24 hours for CR1, CR2, and RMGIC, whereas it was achieved in a more linear way for PMCR.

Regarding solubility, the RMGIC presented the highest solubility values ($P < .001$), whereas with the 3 resin-based luting cements (CR1, CR2, and PMCR), the solubility values were not significantly different ($P = .15$).

DISCUSSION

The water sorption measurement actually measured the net gain in weight of a specimen as a result of the ingress of water molecules and egress of monomers and other small molecules.¹⁶ From an atomic point of view, diffusion mechanisms are a stepwise migration of atoms from site to site. Generally, 2 patterns are known for the diffusion of water through polymeric materials.¹⁷ One is the pattern following the “free volumetric theory,” in which water diffuses through microvoids without any mutual relationship to the polar molecules in the material. The other pattern is called “interaction theory,” in which water diffuses through the material binding successively to the hydrophilic groups. In the case where there was a negative correlation between the diffusion and equilibrium water uptake, the latter pattern of diffusion was supposed to occur mainly.¹⁷

Recently, it has been assumed that both approaches could be valid, each one for a defined specimen family or both simultaneously. However, polymers absorb water to different degrees depending on their microstructural and molecular aspects, eg, polarity

of the molecular structure, presence of pendant hydroxyl groups capable of forming hydrogen bonds with water, degree of cross-linking of the continuous matrix, presence of residual water-attracting species, and type, dimension, volume, diffusivity, and solubility of filler particles.¹²

Thus, it was not surprising in the present study that the most significant water sorption occurred in the RMGIC luting cement. In fact, this kind of material has a dual-setting reaction involving mainly the acid-base reaction of conventional glass-ionomer cements and, at the same time, the free-radical polymerization process used in composite resin systems. The polymerization of these resin components has been claimed to protect against attack by moisture during initial setting.¹⁸ In that case, the polymerized structure contains numerous hydrophilic functional groups (mainly hydroxyethyl methacrylate [HEMA] monomers) in a highly entangled matrix and behaves as a synthetic hydrogel, which has the capability to imbibe further water and undergo substantial swelling. Hygroscopic expansion has been shown to occur with microfilled composite materials that only exhibit 2% to 4% water sorption, whereas the RMGIC tested in the present study exhibited 11.24% in weight.¹⁹ However, the fact that hydrogels have been found to swell more in distilled water than in aqueous solutions of electrolytes, such as sodium chloride, may imply that the amount of water sorption could be less marked clinically than *in vitro*.¹⁶

However, materials with less HEMA content are expected to present less water sorption. Compared to RMGIC, PMCR showed in the present study nearly 4 times less sorbed water. Polyacid-modified composite resin cements differ from resin-modified glass-ionomer cements in their composition and structure. They are fabricated of ion-leachable glass particles, identical to those of glass-ionomer cements, embedded in a polymeric matrix. The matrix is formed principally during the dual mode of polymerization of different kinds of monomer: modified methacrylates, such as bisphenol glycidyl methacrylate (bis-GMA), urethane dimethacrylate (UDMA), diphenylsulfone dimethacrylate (DPSDMA),

or triethylene glycol dimethacrylate (TEGDMA), and bifunctional monomers comprising 2 carboxylic groups and 2 double-bond functions.²⁰ The functional monomers are supposed to react concurrently with methacrylates by the common free-radical polymerization and with the cations liberated from glass fillers by an acid-base reaction. The acid-base reaction is initially limited in PMCR because of its anhydrous structure. Once water infiltrates the PMCR material, a belated acid-base reaction is likely to occur.²⁰ Water uptake in PMCR cements thus involves hydration of the glass filler within the polymerized material on top of the water absorbed in large part by the resin matrix.²¹ This phenomenon is probably responsible in the present study for the greater amount of water absorbed by PMCR than by the composite resins (CR1 and CR2; see Table 2).

CR1 and CR2 specimens clearly exhibited less water sorption than PMCR and RMGIC. These results are in agreement with other studies, and the hydrophobic nature of the constituent monomers has been reported to be the major factor.⁹ In addition, many other hydrophobic monomers, such as ethoxylated bisphenol-A glycol dimethacrylate (bis-EMA), have been developed as alternatives to bis-GMA or UDMA to reduce further water sorption values of composite resin materials. A lower water sorption is then achieved by the elimination of the pendant polar groups.²² In the present study, however, the presence of bis-EMA in the matrix of CR2 cement did not lead to lower water sorption values than CR1 cement, which contains bis-GMA and UDMA. Nevertheless, though low, water sorption did occur in composite resin cements. Water then permeates the hydrophobic matrix according to 3 mechanisms: direct diffusion into the material phase; penetration of microvoids or microdamages already present in the material or generated by water attack; and flow of water molecules along the filler-matrix interface.^{9,18} For the latter, it is known that the nature and quality of the links between fillers and matrix are also important: A weak adhesion between fillers and matrix phase may determine routes of a capillary diffusion, which carry and hold water.^{8,23} In the present

study, the fillers contained in CR1 and CR2 are silanized, and this may have contributed to reduce the water sorption for these cements.

The water uptake kinetic was also observed over 168 hours, according to the ISO 4049 requirements. The short-term experiment does not exhibit results different from those in other experiments testing materials for months.^{19,24} In particular, it was pointed out in the present study that the larger water uptake occurred within the very first hours of immersion for RMGIC, CR1, and CR2, whereas it followed a more continuous process for PMCR. This observation may reflect the maturity of RMGIC, CR1, and CR2 at the time of evaluation, even if the RMGIC specimens were predesiccated before immersion.

It is known indeed that RMGIC, like conventional glass-ionomer cement, contains water as an integral part of its structure, in addition to the resin matrix, and as a consequence, the predesiccation method of assessment of water sorption used in this study may lead to higher sorption values.²⁵ Nevertheless, during the predesiccation process, it is the "loosely bound" water that is lost by evaporation. The decrease in water sorption observed after 96 hours is then the result of the finishing of the setting reaction, by conversion of "loosely bound" water (ie, the amount of water soaked up after immersion) to "tightly bound" water. The acid-base reaction required to achieve cement maturity needs some days for RMGIC, as some of the water in the cement is replaced by water-soluble monomers.²⁵ CR1 and CR2, like most composite resin luting cements, exhibited little postcuring. This accounts for the minimal change in water sorption between 96 and 168 hours. Concerning PMCR, the regular water uptake may reflect the belated but continuous acid-base reaction occurring after the initial polymerization.²⁰ Thus, despite moderate initial water sorption values, the hygroscopic expansion of the PMCR might grow gradually and lead to failure of full-ceramic reconstructions.²⁶

Otherwise, the present solubility results are consistent with data found in the literature: Composite resins and compomers dissolve

less than resin-modified glass-ionomers.^{9,24,27} RMGIC is the luting cement with the highest solubility values, and this may be credited to the setting reaction between the fluoroaluminosilicate and the polyacrylic acid.²⁵ However, the solubility value of the resin-based materials could imply beneficial, as well as detrimental, biologic consequences. On one hand, and concerning PMCR, among the substances leaking out, released fluorides present a cariostatic effect, this phenomenon occurring particularly during the first week.²⁸ On the other hand, water is not known to be of any help in the fundamental chemistry of resin-based material, and a high solubility in water could lead to decreased material strength.²⁹ In fact, a major factor for resin-based material solubility seems to be the monomer conversion rate; residual monomers solubilize easily, and a lower rate of polymerization entails much more solubility.³⁰ In connection with that, the voids trapped in the bulk polymer contain oxygen that may inhibit polymerization and consequently facilitate solubility. In fact, solubility must inevitably depend on the resin matrix, its composition and polymerization, the filler nature, its size and dispersion within the matrix, as well as the interface characteristics between filler and matrix. Thus, the discrepancies in the solubility results, statistically different or not, may stem from physicochemical variations in the different types of polymer.

Finally, the water sorption and solubility properties of these 4 luting agents have also been discussed regarding their clinical handling. In particular, the 3 resin-based materials, conversely to RMGIC, must be used with a bonding agent, which may constitute the Achilles' heel of the bonding chain. Basically, bonding agents have been developed to improve the adhesion of polymer luting cements to dental hard tissues with the intention of thwarting the polymerization contraction and forming a gap-free interface. Furthermore, if prosthodontic durability must be envisaged, the tooth/luting cement interface is to be considered when the luting cement involves no major drawback. However, since a previous study³¹ had shown that bonding agents exhibited values of water sorption and solubility 30 to 150

times higher than that of the corresponding resin-based materials, the integrity of the interface is now becoming a question of major concern. Deterioration of bonding agents thus opens a gap interface where microorganisms may penetrate and colonize. If adhesives expose a very small surface area to the oral fluids, the fluids have a large contact with dentin. Dentinal tubules occupy about 20% to 40% of the midcoronal dentin surface, and water represents approximately 22% of the volume of dentin.³² Regarding water, 75% of its total amount is in the tubules, and 25% is bound in the mineralized matrix around mineral crystals and/or collagen.³³ With time, the tubular fluids may consequently damage the bond joint between dentin and the restoration. This observation may explain why the clinical results of resin-modified glass-ionomer luting cements seem to be as satisfying as composite resin cements,^{3,4} as their theoretical water behavior seems so questionable.

Further crucial points emerge from the present study. First, ISO 4049 specifications were conceived to obtain intrinsic polymers' water sorption and solubility values, and are used mainly for product control. In the case of RMGIC, the sequence of experimental events takes place during the setting procedure, especially during the acid-base reaction, which is working for several hours. This may distort final results. Clinically, polymers used in cavity restorations do not undergo desiccation before contact with oral fluids. Second, the use of distilled water alone as the means of mimicking oral conditions emerges as insufficient. The chemistry of the oral environment must be taken into account, although the effects differ according to the type of material being tested. Thus, solubility results that are greater than ISO 4049 values must be expected in a clinical situation. Moreover, to obtain clinically relevant outcomes, water sorption and solubility experiments need to be conducted in artificial saliva at different pH storage.³⁴ Third, results depend on exposure time. Although there is no indication of a single, universally acceptable time for testing, it could easily be considered that early values may not reflect later behavior. Therefore, the present data only indicate an early and theoretical deterioration of luting cements.

CONCLUSIONS

1. Due to its hydrophilic nature, as well as to the filler characteristics, the resin-modified glass-ionomer luting cement (Fuji Plus) exhibited the significantly highest values of water sorption and water solubility.

2. Of the 3 resin-based luting cements, the polyacid-modified composite resin, so-called compomer (Resinomer), demonstrated significantly higher water sorption and water solubility than the composite resin cements (Variolink II and Panavia F), which showed low values and were not significantly different from each other.

3. In this way, and within the limitations of this *in vitro* study, composite resin luting cements appear to be more suitable than compomers and resin-modified glass ionomers to meet longevity requirements.

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