

In vitro Study on Water Absorption of Dental Restorative Materials

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Abstract: Among the properties of materials for direct coronal restorations, water absorption is particularly important, influencing the mechanical properties, color and adhesion of the restorations made. The aim of this study was to compare the water absorption of six dental restorative materials, two composite, two compomers and two glass ionomers. Water absorption was determined on disk samples. 30 discs were prepared, 5 of each studied material, with a diameter of 15 mm and a thickness of 1 mm. The water absorption test was carried out based on the ISO 4049 specification. The composites and compomers showed a water absorption within the standard limits (40 μ g/mm³). Glass ionomers show high water absorption, therefore to be used as coronal restorative materials they need to be coated with a protective varnish.

Keywords: water absorption, dental restorative materials, mechanical properties

1. Introduction

The search for durable dental restorative materials that have physical and mechanical properties similar to dental hard tissues is an ongoing challenge for researchers in the field [1]. The ability of the restoration to fit the cavity walls perfectly and to seal them influences the durability of the treatment. One of the goals of restorative dentistry is the ability of materials to bond completely with hard dental tissues [2].

Manufacturers of dental restorative materials try to improve their mechanical and physical properties, such as bending and compression strength, modulus of elasticity, coefficient of thermalexpansion or water absorption [3]. Dental restorative materials must be dimensionally stable, insoluble, when exposed to fluids in the oral environment [4, 5].

Water absorption and solubility of fillers are extremely important because they often come into contact with the gingival region and the fluid in the gingival sulcus [6].

There are two mechanisms that occur when dental restorative materials are exposed to oral fluids: weight gain due to water absorption and weight loss due to dissolution in water [7]. Water absorption of restorative materials damages the chemical structures and this can be seen by increasing their surface smoothness [8]. Due to changes in their biological structure, marginal closure is lost and discolorations appear [9].

A disadvantage of all composite resins is their hygroscopic expansion associated with water absorption in the oral cavity [10,11]. Studies have shown that composite resins and poly(methyl methacrylate) denture bases absorb water and, in the early stages, this absorption follows Fick's low of

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diffusion [12-14]. There are few studies on resins that are used as adhesives for bonding hydrated dentin [15].

The ISO 4049 standard states that for resin-based materials, water absorption values must be equal to or less than 40 micrograms per cubic millimeter, and solubility values must be less than 7.5 micrograms per cubic millimeter [15, 16].

Restorative materials that release fluoride ions could cause increased resistance to solubility, and would help remineralize the tooth [17, 18]. In vivo and in vitro studies have shown that fluoride ions stimulate osteoblastic proliferation. Thus, research has been done to find a composite that consists of a modified aluminosilicate glass matrix and that contains large amounts of fluorine. Calcium fluoroaluminosilicate glass powder treated with fluorine shows an increase in mechanical strength [15].

The essential characteristics of the classes of materials used in dental fillers are summarised in Table 1.

Table 1. Essential characteristics of the classes of materials used in dental finers							
Material class	Characteristic 1	Characteristic 2	Characteristic 3				
Composite	non-reactive glass	monomer					
Compomer	reactive fluoride releasing glass	acidic monomer	water from the environment				

polyacid

Table 1. Essential characteristics of the classes of metarials used in dental fillers

reactive fluoride releasing glass

A compomer is a combination of a glass ionomer and a composite because it contains a reactive fluoride glass and an acid as well as a monomer. The major difference between glass ionomers and componers is that in the glass ionomer the acid is present as a polymer, while in the componer the acid is present as a monomer. The polymer is formed by the polymerization of monomers in the restorative material during curing. Another difference is that the componer does not contain water and the reaction between the glass and the acid monomer occurs while the compomer takes up water from the environment. As can be seen from Table 1, glass ionomer properties develop slowly, only after the compomer has first been used and polymerized as a composite.

2. Materials and methods

Glass ionomer

2.1. Materials

The water absorption test was carried out based on the ISO 4049/2019 specification, the materials used being the following: two composites, two compomers and two glass ionomers. Composites are: Point 4 of the Kerr company, a microhybrid composite, and Radopacril, an experimental composite of the "Raluca Ripan" Institute from Cluj-Napoca, Romania, both being light-curable. Kerr Point 4 composition: resin: Bis-GMA (bisphenol A-glycidyl methacrylate), TEGDMA (triethylene glycol dimethacrylate) and Bis-EMA (bisphenol A diglycidyl methacrylate ethoxylated); filler: Barium glass and Silica; average particle size 0.4 microns; loading 76% (w/w) or 57% (by volume). Radopacril composition: resin: Bis-GMA, TEGDMA, DMAEM (dimethyl aminoethyl methacrylate); filler: Strontium glass, Colloidal silica.

In our study, among the componers, we used Dyract AP, a second generation componer, and Dyract Extra, componer of the 3rd generation, light-curable componers from Dentsply Sirona. An entirely new monomer has been developed for Dyract that contains both the polymerizable groups of a composite resin and the acidic groups of a glass ionomer polymer. The average glass particle size in Dyract AP has been reduced by 0.8 microns. The second generation, Dyract AP, brought greater strength and less wear, greater fluoride release and a better polish. The organic matrix was modified by adding a small amount of strongly crosslinked monomer. Composition of Dyract AP: Polymerisable resins, TCB resin (Carboxylic acid modified dimethacrylate), Strontium-fluoro-silicate glass, Strontium fluoride.

Dyract Extra has as its matrix a blend of several methacrylate resins, such as Ethoxylated Bisphenol-A-dimethacrylate, Urethane dimethacrylate (UDMA), Triethyleneglycol dimethacrylate (TEGDMA), Trimethylolpropane trimethacrylate (TMPTMA), Carboxylic acid modified dimethacrylate (TCB). TCB resin serves to give the resin mixture a high cohesion, reduces its hydrophobicity, and increases the rate

water



of fluoride release. The matrix also contains a combination of the photoinitiator Camphoroquinone and the accelerator Dimethylaminobenzoic acid ethyl ester, and the concentrations of these have been carefully optimised to provide a long clinical working time (reduced sensitivity to ambient light) as well as high depth of cure. The filler component of Dyract Extra is the same strontium fluoride glass that is used in both Dyract and Dyract AP. It also contains iron oxide and titanium dioxide pigments.

From glass ionomers the proposed materials were Kavitan Plus from Spofa and Ketac Molar from 3M ESPE, both materials being self-curing. Kavitan Plus is a glass ionomer cement (polyalkenoate glass) having the composition: powder: fluorosilicate glass; liquid: aqueous solution of acrylic and itaconic acid copolymers. Ketac Molar is a powder/liquid system and is supplied in both manual mixing and automatically mixed capsule (Aplicap) systems. We used the manual mixing system. In the hand-mixed version, no polycarbonate acid is added to the powder, unlike Aplicap capsule products. The liquid in the hand-mixed version contains a higher concentration of acid. For all versions of Ketac Molar, after mixing the components the same acid concentration is obtained (Table 2).

Table 2. The powder and liquid relationship and the percentage proportion of the acid components in powder

Product	Powder/liquid ratio	Acid in powder	Acid in liquid	
Ketac Molar hand-mixed	3.0:1	0 %	100 %	
Ketac Molar Aplicap	3.4:1	25 %	75 %	

2.2. Tools and equipment

For the preparation of samples from the materials proposed in the study, we used: a teflon mold with an inner dimension of 15 mm in diameter and a thickness of 1 mm, consisting of two halves, clamped with the help of a metal ring to keep them in contact, with the help of which the samples are created; two plastic sheets to help fix the material inside the disc during photopolymerization; two glass plates that compress the mold; external power source (photopolymerization lamp); tank capable of maintaining a water temperature of 37°C; analytical balance with an accuracy of 0.05 mg; micrometer with an accuracy of 0.01 mm; pliers; plastic gloves, to avoid contamination of the samples during the test.

2.3. Preparation of samples

Depending on each type of material, the samples are prepared differently. Thus, for materials whose setting reaction does not require an external energy source (self-curing), mix the powder with the liquid using a plastic spatula, on the support indicated by the manufacturer and in the indicated proportion. After homogenizing the mixture, it is placed inside the mold until the space is full, after which the plastic film is placed, the whole assembly being kept under pressure between the two glass plates. After complete curing, the assembly is disassembled, recovering the primary sample, the excess of which is removed. Five such samples were made from both Kavitan Plus and Ketac Molar (Figure 1).

For the materials whose setting reaction requires an external energy source (light-curing lamp), the preparation of the samples proceeded as follows: the material is extracted from the syringes, condensed in the mold, and when the space is full the plastic film is applied, leveling the mass of material, and then it is kept under pressure with the help of a glass plate. This entire assembly is exposed to light-curing in nine portions of the future specimen for 40 s, on this side of the specimen (Figure 2).



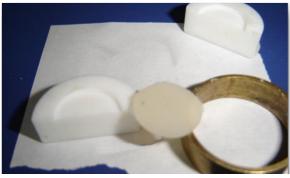


Figure 1. The test piece removed from the mold before being placed in the thermostatic bath



Figure 2. a) Portions subjected to light-curing; b) Exposure of the sample to light-curing

After the completion of these nine periods, the sample is removed from the mold, turned on the opposite side and exposed to the blue light of the lamp, using the same procedure. Using this method, five samples were made from the Point 4 and Radopacril composites, as well as from Dyract AP and Dyract Extra compomers.

2.4. Making determinations

These samples were maintained in the thermostatic bath with water at a temperature of 37 degrees Celsius for seven days. The samples are maintained using a grid, where the samples are kept at least 3 mm apart from each other, with a minimum of 10 mL of liquid required for each sample. After the seven days, the samples are removed from the thermostatic bath and washed with water until all impurities are completely removed. Then air dry for 15 s. The next step consists in weighing the samples: this mass determined in micrograms will be denoted by M_2 , the initial mass of the samples being denoted by M_1 .

The calculation of the water absorption value noted WA (equation 1) is expressed in micrograms per cubic millimeter. It will be done for each of the five samples from each material. Following the measurements, the results for the weighed samples were listed in Table 3. Equation 1 presents the calculation formula for water absorption: M_2 is the mass of the specimen after immersion in water for 7 days, while M_1 is the mass of the specimen before the experiment; V is the volume of the sample.

$$WA(\frac{\mu g}{mm^3}) = \frac{M_2(\mu g) - M_1(\mu g)}{V(mm^3)}$$
 (1)

Table 3. The mass of the five samples for each restorative material studied. $M_1 = \text{mass}$ of sample before the experiment; $M_2 = \text{mass}$ of sample after the seven days; D = difference between M_2 and M_1

	M1	0.4022	0.4062	0.4415	0.4555	0.4775
Point 4	\mathbf{M}_2	0.4044	0.4085	0.4437	0.4578	0.4803
	D	0.0022	0.0023	0.0022	0.0023	0.0028
Radopacril	M1	0.4129	0.4180	0.4210	0.4100	0.4095



	M1	0.4022	0.4062	0.4415	0.4555	0.4775
Point 4	M_2	0.4044	0.4085	0.4437	0.4578	0.4803
	D	0.0022	0.0023	0.0022	0.0023	0.0028
	M_2	0.4147	0.4194	0.4250	0.4150	0.4137
	D	0.0018	0.0014	0.0040	0.0050	0.0042
	M1	0.4436	0.5016	0.4416	0.4600	0.4440
Dyract AP	M_2	0.4484	0.5058	0.4458	0.4653	0.4474
	D	0.0048	0.0042	0.0042	0.0053	0.0034
	M1	0.5131	0.4891	0.4381	0.5133	0.4892
Dyract Extra	M_2	0.5160	0.4938	0.4422	0.5180	0.4939
	D	0.0029	0.0047	0.0041	0.0047	0.0047
	M1	0.3680	0.4610	0.3780	0.4130	0.3080
Kavitan Plus	M_2	0.3823	0.4775	0.3924	0.4278	0.3201
	D	0.0143	0.0165	0.0144	0.0148	0.0121
	M1	0.3490	0.4661	0.3907	0.4081	0.3915
Ketac Molar	M_2	0.3543	0.4723	0.4002	0.4161	0.3955
	D	0.0053	0.0062	0.0095	0.0080	0.0040

3. Results and Discussions

After performing the arithmetic mean for each material and calculating according to the formula mentioned above (equation 1) the values presented in Table 4 and Figure 3 emerged.

Table 4. Water absorption values of the studied materials

Restoration material	Point 4	Radopacril	Dyract AP	Dyract Extra	Kavitan Plus	Ketac Molar
WA (μg/mm ³)	13.75	30.00	26.17	22.07	81.94	57.81

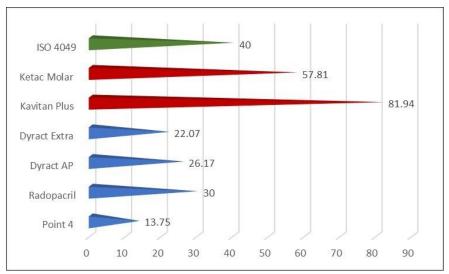


Figure 3. Water absorption of restorative materials studied by comparison with the ISO 4049 standard

For water absorption in restorative materials, a value of 40 µg/mm³ is reported as a standard limit [19]. Water absorption, as can be seen in Table 4, is very high in the case of the glass ionomers studied. Water absorption in the case of the Point 4 composite has the lowest value among the studied materials, which indicates the good clinical behavior of these fillers. Compomer restorations and Radopacril composite also comply with ISO 4049 for water absorption.

The higher values for water absorption negatively influence the mechanical properties of the resin matrix dental restorative materials, reducing the strength through the effect of water on the adhesion between the organic resin and the filler through the hydrolysis of the bonds made through the silane coupling agent [9,20].

After the curing reaction initiated by light-curing to componers, it is necessary to absorb a small

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amount of water to trigger the acid-base reaction, characteristic of glass ionomers. The higher level of water absorption in the case of componers is beneficial because it helps to trigger the acid-base reaction with the subsequent release of fluoride [21].

Absorption of water molecules in composite resin is made in three ways. The first is represented by water penetration in the gaps in the restorative material [22]. The second way is diffusion in the spaces between inorganic fillers. Third way is that water molecules penetrate by direct flow in the spaces between matrices and fillers in composite resin [23, 24]. The behavior of resin-based materials regarding absorption of water, shows variations depending on their characteristics [25-28]. Composite resins with a higher filler ratio absorb less water [24, 29]. The mechanical and chemical properties of composite resins, including water absorption, are influenced by the type and degree of filler, the inorganic silane and the distribution of particles in the organic phase [30-36]. If in the condensation stage, before polymerization, air bubbles are incorporated into the organic matrix, causing the appearance of macro/micropores, water/saliva can be absorbed without changing the volume of the restoration [37].

Water absorption at the level of glass ionomer cements decreases the marginal integrity of the restoration [38, 39]. In vitro studies have shown that glass ionomer cements absorb more water than composites [40]. Resin-modified glass ionomer cements showed higher water absorption than conventional glass ionomer cements due to the hydrophilicity of 2-hydroxyethyl methacrylate (HEMA) [CH₂=C(CH₃)COOCH₂CH₂OH] [41-46]. Fillers made of glass ionomers need to be covered with a protective varnish, as indicated by each manufacturer to prevent water absorption in the obturation and subsequently its degradation in the first hours after curing [47-52].

4. Conclusions

The tested composites and compomers met the corresponding requirements of ISO 4049, with no significant differences between them in terms of water absorption. Glass ionomers, on the other hand, showed increased water absorption, so if they are used with a protective surface varnish, with the correct indication, we believe that their clinical use will last longer.

By testing water absorption, the future behavior of the studied materials is predicted. Knowing the mechanical properties and physical performances can predict the in vivo behavior of the materials under study.

Acknowledgments: We thank the dental materials research team at the "Raluca Ripan" Institute in Cluj-Napoca, led by Dr. Cristina Prejmerean and Dr. Marioara Moldovan, for their help in carrying out this study.

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Manuscript received: 25.04.2023