

# Evaluation of sorption and solubility of dental adhesives submitted to different light-curing

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**Objective:** The aim of the study was to evaluate the influence of different photoactivation times (10, 15, 30 and 60 seconds) on the sorption and solubility of adhesives Ambar, Scotchbond Multi-Purpose (SMP), Single Bond 2-SB (SB2), Single Bond Universal (SBU) and Ambar Universal (AU). **Methods:** Disks were made with each adhesive (n = 10). They were dispensed into 6.0 x 1.0-mm silicone molds and polymerized. The sorption and solubility values were obtained

after 7 days of storage in distilled water at 37°C. **Results:** The results were analyzed by Kolmogorov-Smirnov, Kruskal-Wallis and Dunn's tests at 5% of significance level. According to the results of sorption, the activation time factor showed no statistical difference ( $p > 0.05$ ) compared to the other materials. For the solubility, statistical difference ( $p < 0.05$ ) was found for SMP at 10s, 30s and 60s time. Evaluating the materials within the same activation

time, SMP at 10s showed the lowest value of sorption and solubility, following these low values when activated for 15s and 30s. The highest solubility values were found for SB2, at 10, 15 and 30s. **Conclusions:** It can be concluded that the phenomena of sorption and solubility are closely related to the composition of the adhesive system and not to the time of photoactivation. **Keywords:** Light-curing of dental adhesives. Solubility. Adhesives.

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## INTRODUCTION

Adhesive systems are classified as conventional (Etch-and-Rinse) and Self-Etch.<sup>1,2</sup> Conventional systems can involve two or three clinical steps presenting the agents primer and bonding – isolated or combined in a single flask – with previous preparation of dental structures.

In contrast, Self-etch systems are an alternative based on the non-removal of acid monomers, also known as “all-in-one” adhesives for combining “etching, priming and bonding”, thus containing functioning, hydrophilic and hydrophobic acid monomers, water, and organic solvents in a single solution for clinical application.<sup>3,4</sup>

A demand for lower technical sensitivity, shorter clinical application time and lower occurrence of post-surgery sensitivity has led Self-Etch adhesive systems to become a promising approach modified to be highly hydrophilic and compatible to moist dentin substrate<sup>5</sup> in relation to conventional systems.<sup>6</sup> Another advantage is that the infiltration of adhesive resin tends to occur simultaneously to the self-conditioning process, despite controversies regarding this process.<sup>7,8</sup>

In 2011, the market launched Multi-Mode systems, also known as universal adhesives, projected from the concept of “all-in-one” adhesive systems, but also incorporating the versatility of being adaptable to diverse clinical conditions,<sup>9-11</sup> that is, an adhesive which can be applied in both senses, enabling the operator to decide on the best adequate bonding protocol for the cavity undergoing preparation.<sup>1,7,11</sup>

An increasing simplification tendency reflects a professional desire for efficiency and reduced clinical time; however, this simplification has been followed by a genuine technological advance,<sup>3,9-14</sup> since it has been induced worse

results in terms of durability of the adhesive bonds, influenced by increased water sorption and adhesive solubility.

Water sorption (WS) and solubility (SO) are phenomena which contribute to degrade the resinous component of the hybrid layer (they are formed by the dentin substrate and adhesive).<sup>8,12,15,16</sup> This layer on the dentine is especially subjected to degrading processes, which may end up compromising the stability of the merging of the resin composed and the tooth surface.

The phenomena of water sorption and solubility are closely related to the composition of adhesive systems<sup>8,15,16</sup> and the efficiency of the photoactivation process.<sup>17</sup> Resins are said to be composed with inadequate polymerization, which may increase water sorption and solubility. Such an incomplete polymerization of the adhesive generates regions of pores which function as water sorption channels and hydrophilic monomers, later leading to a hydrolytic degradation of the dentine-resin merging, since the adhesive procedure was not successful at promoting full monomer infiltration inside the collagen fibres.<sup>8</sup> The residual solvent may also lead to an adhesive system at a low polymerization degree, which favors the fluid movement within the adhesive layer.

A possible clinical solution to minimize the degrading phenomena of polymers through water sorption and solubility on the hybrid layer would be to increase the photoactivation time of adhesive systems.<sup>17,18</sup> Therefore, the stability achieved at the resin-dentine merging region could contribute to a clinical success of restoration. In this context, the goal of this paper was to assess the properties of sorption and solubility for different adhesive systems in relation to different photoactivation times. Our

hypothesis is that the different photoactivation times will not influence neither water sorption nor solubility of the adhesive systems tested.

## MATERIALS AND METHODS

### Preparation of the specimens for sorption and solubility testing

The studied factor was to assess the sorption and solubility of some adhesive systems, including Ambar (FGM, Joinvile, SC, Brazil), ScotchBond Multi-Purpose (SMP) (3M/ESPE, St. Paul, MN, EUA), Single Bond 2-SB (SB2) (3M/ESPE, St. Paul, MN, EUA), Single Bond Universal (SBU) (3M/ESPE, St. Paul, MN, EUA), and Ambar Universal (AU) (FGM, Joinvile, SC, Brazil) regarding different photoactivation times (10, 15, 30, and 60 seconds).

We produced 200 specimens divided in five groups (n=10) for each photoactivation time. To prepare the samples, we placed a polyester tape (Dentsply, Petrópolis, Rio de Janeiro, Brazil) on a glass plate and silicone matrix (A-Silicone - Express XT/3MESPE, St. Paul, MN, EUA) with standardized dimension of  $1.0 \pm 0.1$ mm in thickness and  $6.0 \pm 0.1$ mm in inner diameter for the polyester tape. The adhesives were equally distributed on the silicone molds ( $6\mu\text{L}$ ), and subsequently the air free from water/oil was smoothly sand-blasted for the period set by each manufacturer at a distance of 10cm to facilitate the solvent evaporation. In the case of SMP, we used only the flask containing adhesive, working as control to the experiment. To insert the adhesive SBU, we proceeded with friction using microbrush (Coltene, Vigodent, Rio de Janeiro, RJ, Brazil) for a 20-second period. The adhesive systems, except for SMP, were subjected to a soft air jet for 10s.

The material was covered with another polyester tape and pressed manually with a glass

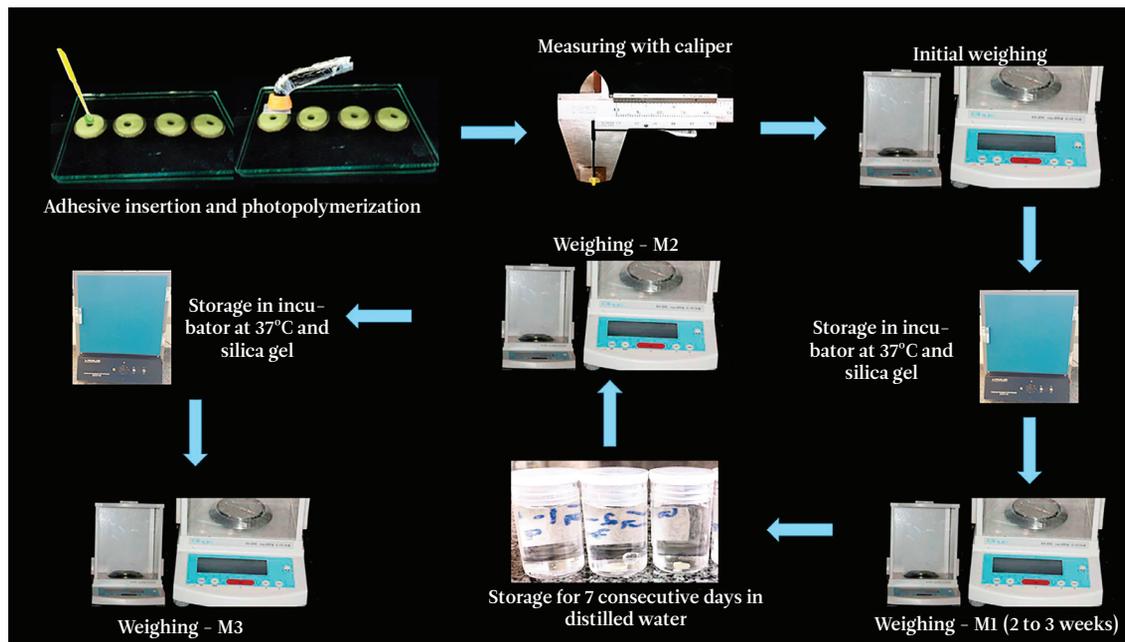
slide (Labmais, Bioprecisa, Curitiba, PR, Brazil) for the liquid samples to remain on a flat layer of uniform thickness. The samples produced followed the specifications by regulation ISO 4049/2009, but with a few modifications regarding the dimensions of the specimens to have their sizes adapted according to the diameter of the long leg of the curing light equipment Rad-di-cal (SDI, Victoria, Australia). The adhesives were photoactivated at times of 10, 15, 30, and 60 seconds under irradiance of  $1200 \text{ mW/cm}^2$ .

After the photoactivation, the specimens had their edges slightly regularized with silicon carbide sandpaper (3M, Sumaré, SP, Brazil) with granulation 1200 and stored in buffered transparent polyethylene jars of 10mL (Adria Laboratories, Londrina, PR, Brazil) in an environment without light for full adhesive polymerization. The storage time of the samples was seven days in distilled water, according to ISO 4049/2009.

### Sorption and solubility testing

The samples were stored in a desiccator (Dinâmica Química Contimerânea Ltda, Diadema, SP, Brazil) for the removal of free water. The desiccator was carried to the inside of a bacteriological incubator (ECB2, Biodont, Odontobrás, Ribeirão Preto, SP, Brazil) under controlled temperature of  $37^\circ\text{C}$ . After a twenty-four-hour interval, the samples were daily weighed using a digital analytical balance (JK-180, Chyo Balance Corp, Tokyo, Japan) to verify the variations in the masses until achieving a constant mass (m1), that is, up to the mass loss was below or equal to 0.1mg, over a period of three weeks (Fig 1).

The area of each disk was verified using a digital caliper (Mitutoyo, Sul Americana, São Paulo, Brazil) from the average of four spaced points at the beginning of the first to the end weighing, in order to calculate the volume of



**Figure 1:** Sequence of the sorption and solubility testing.

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the specimens. By the end of the three weeks, the specimens were stored in 1.0 mL of distilled water at  $37\pm 1^\circ\text{C}$  in bacteriological incubator for seven consecutive days, without weighing. After this period, the samples were washed under running water for ten seconds and dried out with absorbent paper for 20 seconds. The specimens were agitated in the air using a tweezer for 30 seconds to subsequently have their mass measured once again (m2). After the weighing, the samples were relocated in their respective recipients, but now without water, and carried to the desiccation chamber, without a lid, inside the incubator, at  $37\pm 1^\circ\text{C}$ , for seven days, until achieving a constant mass detected by weighing throughout the week on a daily basis. At day 8, these samples were measured again using a

caliper and weighed, establishing thus the m3 value (Fig 1).

The values obtained by measuring the samples were applied in specific formulations of water sorption (Wsp) and solubility (Wsl), in which m1 is the initial mass of the specimen before water immersion (in  $\mu\text{g}$ ), m2 is the mass after a seven-day immersion in distilled water (in  $\mu\text{g}$ ), m3 is the mass after reconditioning of the specimen without water immersion (in  $\mu\text{g}$ ) and V is the volume of the specimen in cubic millimeters (in  $\text{mm}^3$ ).

### STATISTICAL ANALYSIS

The results were subjected to the Kolmogorov-Smirnov normality test followed by Kruskal-Wallis and Dunn tests at 5% significance level.

## RESULTS

According to the results presented in Table 1, referent to sorption data, the factor of light application time (photoactivation) had no statistical difference ( $p > 0.05$ ) in relation to different types of materials. However, when assessing the materials within the same photoactivation time, the SMP, in 10 seconds, had a lower sorption value, being statistically different ( $p < 0.05$ ) in relation to the remaining materials over the same period. The adhesives SB2, SBU and Ambar reached the highest sorption values. Over a fifteen-second period, the adhesive Ambar presented the highest sorption value – statistically different ( $p < 0.05$ ) from the remaining adhesives over the same period.

The adhesive SMP remained with the lowest sorption values, which was corroborated over a thirty-second period – statistically different ( $p < 0.05$ ) in relation to the adhesives AU and SB2 (the latter, along with the SBU, had the highest sorption values). The highest sorption values were found in the adhesive SB2 for all photoactivation times.

According to the results presented in Table 2 regarding solubility data, the factor of light application time (photoactivation) presented statistical difference ( $p < 0.05$ ) for the material SMP in relation to the time of 10 and 30 seconds with the application of 60 seconds of light. Among the remaining materials, no statistical difference ( $p > 0.05$ ) occurred regarding the activation x solubility time.

When assessing the materials ranging the same photoactivation time, the SMP had the lowest solubility values – statistically different ( $p < 0.05$ ) in relation to the AU and SB2 over 10 seconds. The same material presented the lowest solubility values for 15 and 30 seconds, respectively – statistically different ( $p < 0.05$ ) from the adhesive AU for 15 second, and SB2, AU and Ambar for 30 seconds. No statistical difference ( $p > 0.05$ ) was indicated for the materials assessed regarding the time of 60 seconds of photoactivation. The highest solubility values were confirmed for the material SB2 at times of 10, 15, and 30 seconds of photoactivation.

**Table 1:** Values of the median and interquartile interval of sorption ( $\mu\text{g}/\text{mm}^3$ ).

ADHE-SIVES/ TIME	10s			15s			30s			60s		
	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3
Ambar	26.54	30.96 Aab	35.39	26.55	35.40 Ab	35.40	24.34	26.55 Aab	35.40	26.55	30.97 Aa	35.40
SMP	17.69	17.69 Aa	17.69	15.49	17.70 Aa	17.70	15.49	17.70 Aa	19.91	22.12	30.97 Aa	46.46
SBU	17.70	26.55 Aab	28.76	17.70	30.97 Aab	44.25	17.70	26.55 Aab	26.55	26.55	35.40 Aa	39.82
AU	26.55	39.82 Ab	46.46	17.70	35.40 Aab	44.25	35.40	35.40 Ab	53.10	26.55	35.40 Aa	44.25
SB2	26.55	35.40 Ab	44.25	17.70	26.55 Aab	37.61	33.19	35.40 Ab	53.10	17.70	35.40 Aa	35.40

Different capital letters in the line and low-case letters in the column indicate statistical difference through Dunn test.

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**Table 2:** Values of the median and interquartile interval of solubility ( $\mu\text{g}/\text{mm}^3$ )

ADHE-SIVES/ TIME	10s			15s			30s			60s		
	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3	Q1	ME-DIAN	Q3
Ambar	8.85	8.85 Aab	17.69	8.85	13.27 Aab	17.70	8.85	17.70 Ab	17.70	8.85	17.70 Aa	17.70
SMP	0.00	0.00 Aa	2.21	0.00	4.42 BCa	8.85	0.00	0.00 ABa	8.85	0.00	17.70 Ca	28.76
SBU	6.64	8.85 Aab	17.70	8.85	17.70 Aab	26.55	8.85	8.85 Aab	17.70	15.49	17.70 Aa	26.55
AU	15.49	17.70 Ab	19.91	8.85	17.70 Ab	17.70	15.49	17.70 Ab	28.76	17.70	17.70 Aa	17.70
SB2	6.64	8.85 Ab	17.70	8.85	17.70 Aab	26.55	8.85	8.85 Ab	17.70	15.49	17.70 Aa	26.55

Different capital letters in the line and low-case letters in the column indicate statistical difference through Dunn test. ( $\alpha=0.05$ )

## DISCUSSION

The simplification in the use of dentin adhesive systems has called attention regarding its long-term behavior,<sup>19</sup> since the water sorption and solubility rates presented by the adhesive systems after polymerization are crucial to the marginal quality of the restorations. The moist present in the oral environment has a major role in the process of chemical degradation of polymers, presenting a deleterious effect for the dentine-resin interface, compromising the longevity of the restoration.<sup>8,15,20</sup>

Recent papers have demonstrated that an inadequate polymerization of the adhesive may result in inferior physical-mechanical properties, in addition to higher sorption and solubility of the material<sup>21-23</sup>. In contrast, when dental adhesives are photoactivated for prolonged periods, the degree of polymerization increases while the permeability and nanoinfiltration are reduced.<sup>24</sup> Based on this information, our study suggested the use of prolonged activation time (15, 30, and 60 seconds) in relation to the time recommended by the manufacturer (10 seconds), in order to verify the inter-relationship between the extension of photoactivation periods, with capacity of sorption and solubility of conventional adhesives involving two or three clinical steps constituted of mono and bicomponents as well as “all-in-one” adhesives, containing functional acid monomers acting as universal systems.

According to the results found in our study, we observed that the different photoactivation periods had no statistical influence on the water sorption of different adhesives. Thus, our hypothesis should be partially accepted since for the phenomenon of sorption, mass alteration seems to be related to other factors and not to the efficiency of the photoactivation process. In contrast, the solubility of the materials tested

proved to be associated with the factor of photoactivation time for the material SMP, which had a lower solubility in relation to the periods of 10 and 30 seconds. Although, with an increase in the occurrence of light from 30 to 60 seconds, the solubility of the material consequently raised, which can be related to the increase in temperature caused by a longer photoactivation time. This could have been enough to cause higher mobility of radicals, increasing the frequency of non-reactive molecules collisions,<sup>24,25</sup> generating a higher number of available bonds with water due to an increase in kinetic energy<sup>26</sup> causing leaching of the non-reactive components.

When a composite is immersed in a solvent, two processes occur: initially it is the sorption of the solvent which causes a swelling and mass gain<sup>23,27</sup> followed by the leaching of the non-reactive components and possible loss in mass, volume and reduction of mechanical properties.<sup>8,17</sup> The solvent has a plasticization mechanism, spreading inside the polymeric network, causing the polymeric chains to be separated and generating an expansion. However, since during the polymerization reaction, it occurs the formation of a polymeric network with microspaces and free volume between the chains, part of the solvent is accommodated without alteration in the volume of the material. Subsequently, as far as attraction forces between the polymeric chains are exceeded, the matrix can be dilated, increasing the network volume and consequently promoting the softening of the resinous matrix.<sup>8,17,28,29</sup> Thus, it is possible to consider that the mechanism of sorption is self-limiting, that is, as far as the free volume between the chains is saturated by the solvent, the phenomenon tends to decrease.<sup>15,17,20</sup> The sorption of solvents is also followed by a loss of components, which

causes volume reduction.<sup>27</sup> When the material is immersed in water, some of the components, as non-reactive monomers, are released, resulting in mass loss, which characterizes the phenomenon of solubility. However, in addition to the release of non-reactive monomers, it may occur a dissolution of filler particles. The solvent absorbed by the matrix is probably accumulated over time in the charge-matrix interface, leading to the loss of their merge or even hydrolytic degradation of filler particles.<sup>7,8,15,20,27</sup>

When assessing the data of sorption and solubility for the same photoactivation time, the adhesives of a single flask SB2, SBU, Ambar, and AU, regarded as more hydrophilic materials, had higher sorption and solubility values, that is, absorbed more water because of the higher density of their hydrophilic sites and plasticized more rapidly allowing a greater flow and accommodation of substances between the chains when using a light occurrence for 10, 15, and 30 seconds; thus, more hydrophilic adhesives absorb more water and lose more substances to the environment over a shorter period (seven days of storage in distilled water).

Some authors believe that the monomeric component, depending on the concentration, influences the sorption and solubility of the material, especially if this component is the 2-hydroxyethyl methacrylate (HEMA), which provides a higher capacity of water absorption and solubility.<sup>30</sup> The mixtures containing HEMA can form hydrogels with water, and even after polymerization, the HEMA attracts water<sup>20</sup> and promotes the formation of the same product (hydrogels), which weakens the mechanical strength of the

adhesive. In the case of materials which obtained an increase in sorption and solubility, as the SB2 and Ambar, all presented the monomer HEMA in their compositions. Regarding the universal adhesives SBU and AU, as a result of including a high concentration of acid monomers and water to enable the ionization of these monomers and solubilization of calcium and phosphate, the polymers became very hydrophilic and favored an increase in sorption and solubility of the material in a moist media.

It is important to consider that even though most of the adhesive systems tested had the same components (HEMA, Bis-GMA, water, and ethanol), the quantity of each component differs considerably among the materials and the manufacturers do not provide specific data since by assessing each photoactivation time and concluding that both the simplified and universal materials had higher values of sorption and solubility, we can justify that the phenomena of sorption and solubility are related to the composition of the adhesive systems and not only to the photoactivation periods.

## CONCLUSIONS

The different photoactivation periods had no influence on water sorption of the adhesives; however, for solubility, the material SMP had a lower value for the periods of 10 and 30 seconds. When assessing the same light occurrence time regarding the materials, the simplified and universal adhesive systems presented higher water sorption and solubility, justifying that the phenomena of sorption and solubility are closely related to the composition of the adhesive systems and not to photoactivation time.

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