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Mass variation of dentin adhesive system stored in different solutions

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Abstract - The aim of this study was to evaluate the mass variation of different adhesive systems in different storage conditions. Samples were prepared using a standard matrix (ISO 4049) with three dentin adhesive systems (n=8): Bond 1 (Jeneric-Pentron), Scotch Bond Multi-Purpose Plus (control, 3M ESPE) and Single Bond (3M ESPE). Samples were cleaned, dried for 24 hours in an oven at 37ºC and 23° for 1 hour, and then weighed. Initial mass was recorded (m0) on a high precision analytic balance (GENAKA - AG 200) and stored in saline solution, Milli-Q water, and lactic acid (pH=4). Mass variation was evaluated after 2,5 (m_1), 10 (m_2), and 31 days (m_3). A paired T-test was used for comparisons between m0 and m3. One-way ANOVA and the Holm-Sidak method were used for comparisons among mass variations. Bond One showed a statistically greater mass alteration than Scotch Bond (m0>m3) (p=0.009). Bond One presented a statistically greater mass variation when stored in Mili-Q and the statistically lowest mass variation in lactic acid (p<0.01). The statistically highest mass variation of Single Bond and Scotch Bond were found in lactic acid (p<0.05). Based on the methodology applied in this study, adhesive systems undergo mass variation according to the solutions used for storage.

Keywords - Dental materials, degradation, adhesive systems.

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Introduction

Mechanical adhesion, which relies on penetration of one material into a different material at a microscopic level, is responsible for micromechanical interlocking with dental tissues and is the main mechanism used to improve bond strength of dental adhesive systems. Thus, the fundamental principle of adhesion to tooth substrate is based on an exchange process (1) by which inorganic tooth material is exchanged for low viscosity composites, having the ability to bond to this substrate.

Since the development of dental adhesives, manufacturers have been making advances in material chemistry according to the evidence shown in laboratorial bonding effectiveness tests, like bond-strength tests and measurement of marginal sealing effectiveness. In recent years manufacturers have been advocating new adhesive systems due to their user-friendly techniques. In addition to the traditional "etch&rinse" strategy systems, "self-etch" systems were introduced last decade as a new pattern for underlying adhesion (2). Primer and adhesive are joined in the same bottle in one-bottle adhesives systems in the "etch&rinse" strategy, while for "self-etch" adhesives, all-in one (one blister) and two-step adhesive systems (two bottles) are common subclasses of materials.

Better clinical performance of dentin adhesive systems can be attributed to the use of hydrophylic components associated with volatile solvents (3-5). These systems were initially presented in two bottles (three step systems or fourth generation adhesives). Following the interest in user-friendly techniques, manufacturers have been advocating one-bottle adhesive systems (two steps or fifth generation

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adhesives) where primer and adhesive are placed in the same bottle (1).

One of the greatest concerns in the development of adhesive systems is chemical stability and durability in the oral environment. Degradation of some components may result in several problems that affect adhesive restorations (6,7). Decrease in adhesive failure and microleakage are two important issues needing to be solved (8,9). In addition, the release of chemical components can affect biological compatibility (10-13). Further research is needed on the degradation behavior of adhesive systems to better understand the factors involved and to develop further clinical strategies.

The aim of this study was to verify the alteration in mass of adhesive systems stored for 31 days in different storage solutions.

Materials and Methods

Three adhesive systems were evaluated: Bond One, Jeneric-Pentron (B1), Single Bond, 3M ESPE (SB), and Scotch Bond Multi-purpose, 3M ESPE (SBMP). Table 1 provides information about the material used. The latter was used as a control. According to materials, three groups were created (n=24).

A metallic mould 15.0 (±0.1) mm in diameter and 0.5 (±0.05) mm deep (ISO 4049 – 1988) was used for preparation of the disks. During confection of the specimens with B1 and SB, the mould was filled with adhesive and allowed to rest for 60 seconds for primer evaporation before photo activation. A mylar strip and a glass microscope slide were interposed between the hole of the mould and the tip of the photo curing unit. Photo-activation was performed for 20 s, through a glass microscope slide and a mylar strip, for all specimens. The light photo curing unit used was XL 3000 (3M ESPE), with an irradiance higher than 450 mW/cm^2. The volume of the specimens was checked with a digital caliper.

Materials and methods were based on solubility methodology, according to specifications of ISO 4049 (14). Specimens were removed from the mould, cleaned with distilled water and neutral detergent, and placed in a desiccator at 37°C for 24 hours. Specimens were then kept at 23°C for one hour before being weighed on an analytical balance (Genaka - AG 200) with an accuracy of ± 0,2 mg. After initial weights (mo) were recorded, specimens were identified and immersed in three different solutions: saline, miliqui water, and lactic acid (pH=4). According to combinations of Adhesive systems and solutions, nine groups (n=8) were created. Specimens were weighed at four different times: initially (m_0) and at 2.5 (m_1), 10(m_2), and 31(m_3) days of storage in solutions. Desiccator cycles were repeated before weighing of specimens at each storage time. Mass variations were calculated based on differences in initial weight for each specimen (m_0) and weight at specified storage times (m_1, m_2, and m_3).

Results

In general, the pattern of mass variation showed a trend for mass to decrease as compared to initial mass, for specimens immersed in solutions. Mass variation is illustrated in Figures 1, 2, and 3.

The pattern of mass variation in Mili-Q water showed a slight trend for Single Bond and Scotch Bond to increase in weight after 2,5 days (m_0–m_1). For Bond One all solutions presented the trend for the adhesive systems to decrease in weight. Degradation with lactic acid at pH=4 seems to have been more intense for Scotch Bond and Single Bond, as compared to the other 2 solutions. For all simulations, the change in weight may represent degradation of the adhesive systems tested.

Table 1. Description of dentin adhesive systems used

Adhesive	Batch number	Manufacturer	Composition
Single Bond	1 FW	3M/ESPE, St. Paul, MN, USA	*Bis-GMA, HEMA, dimetacrilates, polialckenoic acid, water, ethanol, and photoinitiator.*
Scotchbond Multi-Purpose	1 MJ	3M/ESPE, St. Paul, MN, USA	Bis-GMA, HEMA, hexafluorphosfate, and photoinitiator.
Bond 1	22853	Jeneric Pentron, Wallingford, EUA	PMGDM, HEMA, photoinitiator, and acetone.

Table 2. Mass variation after 31 days.

Adhesive	Solution	Initial mass (mg)	Mass after 31 days (mg)	Mass Variation (m_0-m_3)	P value (significant when p<0.05)	Power of Paired T-test with α=0.05*
Scotch Bond	Mili-Q	^{A}79.4 (3.02)	^{B} 77.48 (3.96)	1.95 (0.95)**b**	P=0.027	0.74
	Saline	^{A} 81.18 (4.59)	^{A} 80.58 (5.51)	0.6 (0.94)**b**	P=0.29	0.09
	Lactic acid	^{A} 77.1 (6.92)	^{B} 73.77 (7.1)	3.32 (1.28)**a**	P=0.014	0.91
Single Bond	Mili-Q	^{A} 41.33 (3.1)	^{B} 39.93 (3.23)	1.4 (0.47)**b**	P=0.009	0.96
	Saline	^{A} 41.78 (3.82)	^{B} 39.55 (3.06)	2.22 (1.12)**b**	P=0.028	0.73
	Lactic acid	^{A} 42.8 (2.69)	^{B} 38.75 (4.06)	4.05 (1.49)**a**	P=0.012	0.93
Bond One	Mili-Q	^{A}66.13 (11.17)	^{B} 60.05 (9.67)	6.07 (1.68)**a**	(P=0.005	0.99
	Saline	^{A} 62.15 (7.85)	^{B} 57.9 (9.12)	4.25 (2.34)**b**	P=0.036	0.65
	Lactic acid	^{A} 42.52 (1.86)	^{B} 40.7 (1.42)	1.83 (0.45)**c**	P=0.004	0.99

Different letters in rows represent statistically significant differences (p<0.05) using Paired T-test.
Different small letters in columns, for the same adhesive, represent statistically significant differences (p<0.05) according to ANOVA and the Holm-Sidak method.
*When the power of the test performed is less than the desired power (0.8), negative findings should be interpreted cautiously.

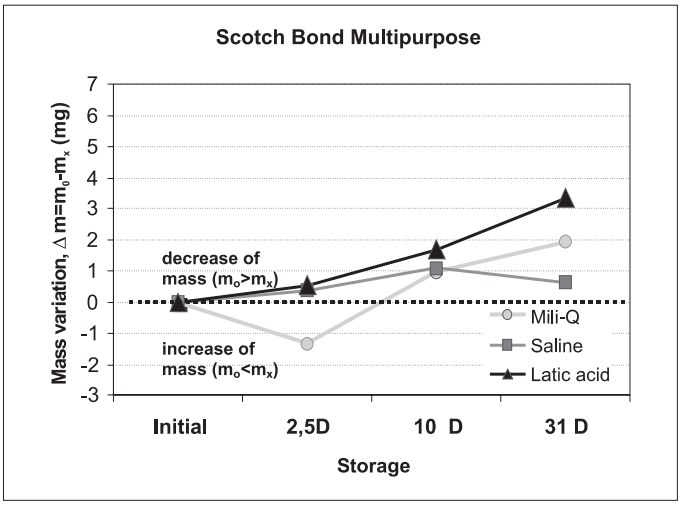

Figure 1. Show the mass variation patterns of the adhesives according to solutions at specific storage times.

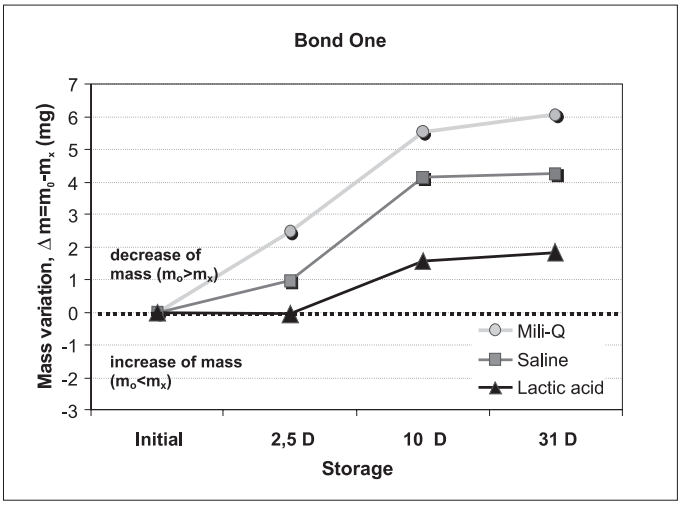

Figure 2. Show the mass variation patterns of the adhesives according to solutions at specific storage times.

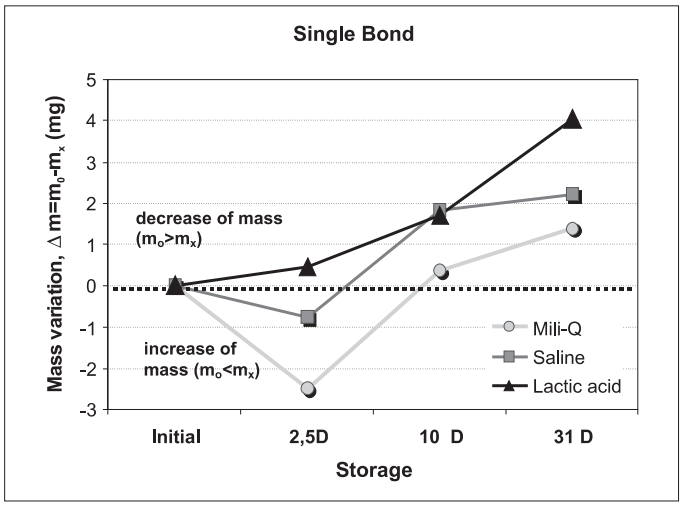

Figure 3. Show the mass variation patterns of the adhesives according to solutions at specific storage times.

Total mass variations (m_0–m_3) of each adhesive used were grouped (independently of storage solution), and analyzed with One-way Analysis of Variance and the Holm-Sidak multiple comparison method. Bond One exhibited statistically more mass alteration than Scotch Bond (control) ($p=0.009$, power of test =0.55). Mass variation of Single Bond was similar to that of Scotch Bond.

After 31 days of storage, Bond One showed the statistically highest mass variation (decrease) in Mili-Q water and saline ($p<0.001$), while Single Bond and Scotch Bond were similar to each other. When stored in lactic acid, mass variation of Single Bond was statistically higher ($m_0<m_3$) than Bond One ($p<0.039$), while Scotch Bond produced intermediate values.

Comparisons performed between the solutions used showed that for Bond One, the statistically highest and smallest mass variation were found for Mili-Q water and lactic acid, respectively ($p<0.05$). Scotch Bond and Single Bond showed statistically higher mass variations when stored in lactic acid ($p<0.05$). Storage of specimens of these two adhesives in saline and Mili-Q water exhibited similar mass variations after 30 days.

Discussion

Two of the adhesive systems evaluated, Bond One and Single Bond 3M ESPE (SB), are classified as one-bottle, two-step adhesive systems (acid + primer & adhesive), while the other, Scotch Bond Multi-purpose, is classified as a three-step system (acid + primer + adhesive) (2). Chemical composition and pH are related to strategy of clinical use of the adhesives and consequently to packaging of the adhesives (traditional or simplified systems).

Chemical stability is an important feature for dental materials since mechanical properties are affected by the degradation of adhesive systems; consequently, microleakage, recurrent caries, and marginal discoloration may occur with the breakdown of the interface seal (1,15).

Variation of mass may be correlated with water absorption and solubility of material. A poor conversion degree associated with exposure to oral fluids can increase the level of molecules leached from composites. Some substances have an adverse effect on vital dental tissues (11,16).

Ethanol, water, and acetone are common solvents found in one-bottle adhesives. It is necessary for primer components to have hydrophilic features for hybridization of dental tissues to occur; however, this feature may be partially kept after photoactivation, promoting the appearance of water inside the adhesive layer, which is described as water treeing (7,17).

A polymer can be degraded in two ways; by hydrolysis (passive degradation) and by enzymatic reaction (active degradation) (18). Thus, swelling of dissolution polymers can reduce mechanical integrity. Specifically for adhesives, degradation can be partially explained by hydrophilic components of adhesives and pH. Water movement across the cured adhesive layer may occur in the presence of increased concentrations of dissolved inorganic ions, and uncured water-soluble and hydrophilic resin monomers, within the oxygen inhibition layer of the cured adhesive. This concentration difference may establish an osmotic pressure gradient, causing water to move from a region of low solute to a region of high solute concentration (7). This, in turn, may result in osmotic blistering of microscopic water droplets along the uncured surface responsible for degrading the adhesive layer.

According to mass variation patterns, after 31 days a significant loss of mass can be identified for almost all the specimens in storage solutions. This variation of mass can be interpreted as degradation of material. Further studies are needed to better understand lixiviated substances and their effect on biological tissues in different storage solutions.

For composites, the pH level is relevant to mass alteration (18,19). Single Bond and Bond One are one-bottle adhesive systems with the adhesive and primer in the same bottle. The presence of hydrophilic components in the primer and pH may contribute to initial incorporation of solutions, accelerating aging and degradation. Bond One showed the lowest mass variation in lactic acid, pH=4, while Single Bond exhibited the highest mass variation. This fact may be associated with chemical interactions due to the solvent used. Bond One has acetone and Single Bond has ethanol and water as solvents. Generally, the more similar the storage solution is to the adhesive solvent, the greater will be the degradation of polymers, such as composites and adhesives.

Bond One undergoes more loss of mass than Scotch Bond, independently of the situations simulated in this study. The Scotch Bond system has adhesive and primer in different bottles, so the last layers are more stable against external fluids. This fact can be used in some clinical situations like hybridization of dental tissues for hypersensitivity treatment or for protection of hybridized layers against adverse interactions of simplified adhesives and chemical or dual cure resin cements.

Resumo

DONASSOLO, Tiago Aurélio, OSINAGA, Prudêncio Willy Rodo, DEMARCO, Flávio Fernando e PIVA, Evandro. Variação de massa de sistemas adesivos dentinários armazenados em diferentes soluções. Oral Sci., mai./ago. 2005, vol.1, no.1, p.27-32.

O objetivo desse estudo foi avaliar a variação de massa de diferentes sistemas adesivos em diferentes condições de armazenagem. As amostras foram preparadas utilizando uma matriz padronizada (ISO 4049) com três sistemas adesivos (n=8): Bond One (Jeneric-Pentron), Scotch Bond Multi-Purpose Plus (controle, 3M ESPE) e Single Bond (3M-ESPE). As amostras foram limpas, secas em um forno por 24 horas (37ºC) e por uma hora (23°C), e em seguida pesadas. A massa inicial foi registrada (m0) em uma balança analítica de alta precisão (GENAKA - AG 200) e armazenada em solução salina, água Milli-Q, e ácido lático (pH=4). A variação da massa foi avaliada após 2,5 (m1), 10 (m2) e 31 dias (m3). Um teste T pareado foi utilizado para comparações entre m0 e m3. Análise de variância (ANOVA) a um critério e o método Holm-Sidak foram utilizados para comparações entre as variações de massa. Bond One mostrou uma maior variação de massa quando comparado com Scotch Bond (m0>m3) (p=0,009). Bond One apresentou, estatisticamente, maior variação de massa quando armazenado em Mili-Q e menor variação de massa em ácido lático (p<0,01). A maior variação de massa do Single Bond and Scotch Bond foi verificada quando submetidos ao ácido lático (p<0,05). Com base na metodologia aplicada nesse estudo, os sistemas adesivos demonstraram variação de massa conforme as soluções usadas para armazenagem.

Palavras-chave: Materiais Dentários, degradação, sistemas adesivos.

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