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RESEARCH ARTICLE
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Long-term water sorption/solubility of two dental bonding agents containing a colloidal dispersion of TiO2

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ABSTRACT

Aims (Purpose): The study's aim was to investigate the effect of colloidal dispersion of titanium dioxide (TiO2) nanoparticles at 4% by mass on the long-term water sorption and solubility of two commercial universal bonding agents.

Settings and Design: An in vitro study.

Methods and Material: A colloidal dispersion of TiO2 nanoparticles was prepared and incorporated into two commercial dental bonding agents, that is, Ambar Universal (FGM, Brasil) and G-Premio Bond Universal (GC, America) at 4% by mass. Forty bonding agents discs were prepared and divided into four groups of 10 discs each, that is, GA: Ambar Universal (control), GB: Ambar Universal (4% TiO2 incorporated), GC: G-Premio Bond universal (control), and GD: G-Premio Bond (4% TiO2 incorporated). The discs were prepared by dispensing the bonding agents into a silicone mold of 5 mm diameter and 1 mm depth. After the discs' desiccation, the cured discs were weighed and stored in distilled water to be evaluated for water sorption and solubility over 1-year storage period.

Statistical analysis used: Statistical analysis was performed by independent variable t-test performed using the IBM-SPSS software.

Results: The incorporated bonding agents groups (GB and GD) showed significantly lower ($P < 0.05$) water sorption and solubility after 1 year of water storage in comparison to the control bonding agents. Both GC and GD showed significantly lower water sorption and solubility than GA and GB.

Conclusions: Incorporation of the colloidal dispersion of TiO2 nanoparticles at 4% by mass into the universal bonding agents significantly reduced their water sorption and solubility in comparison to their control groups.

Keywords: *universal bonding agents; water sorption; solubility; colloidal dispersion; TiO2*

INTRODUCTION

The name “universal” or “multimode” bonding agents refers to the manufacturer’s assertion that these bonding agents can be used in any adhesion strategy depending on the clinical scenario and can be used with a variety of direct and indirect restorative materials.1

Hybrid layer formation starts with the infiltration of bonding agents resin monomers into the mineral-depleted water-rich dentin and exposed collagen matrix, followed by the subsequent in situ photo polymerization. The establishment of a stable hybrid layer will allow the establishment of a crosslinked three-dimensional polymer–collagen network capable of reducing microleakage, marginal staining, bacterial invasion, secondary caries formation, and pulpal irritation.2 However, because the dental bonding agents are based on chemistry that contains both hydrophilic and hydrophobic functional monomers, they will be chemically unstable when placed in contact with moist dentin substrate. This would result in instability of the hybrid layer which will lead to phase-separation of the monomers causing inadequate degrees of conversion.3

Water sorption is described as the absorption and diffusion of water into bonding agents’ monomers, resulting in dimensional changes, softening, and plasticization of the cured polymer network.4 This will affect their physical and mechanical characteristics.

While solubility of dental bonding agents is defined as the hydrolytic degradation of bonding agents’ monomers in the presence of water, which is caused by a chemical reaction with water that can break the covalent bonds between polymer networks, resulting in the loss of monomer mass, which has an adverse effect on the mechanical properties and stability of the resin polymer network.5

Titanium dioxide (TiO2) is a trace element with a high refractive index. TiO2 is also a chemically resistant substance that is thermally stable. Furthermore, due to their nano size, TiO2 nanoparticles have a huge surface area and are biocompatible. TiO2 nanoparticles have been utilized in dentistry to increase endodontically treated teeth’s fracture resistance, osseointegration of dental implants, and to enhance a material’s antibacterial potential.6,7

Therefore, the goal of this in-vitro study is to enhance and maintain the stability of the bonding agent’s polymer network in a wet environment through the incorporation of colloidal dispersion of TiO2 nanoparticles into these bonding agents by testing their effect on the water sorption and solubility of such bonding agents.

MATERIALS AND METHODS

Preparation and incorporation of the colloidal dispersion of TiO2 nanoparticles

For this study, a colloidal dispersion of TiO2 nanoparticles was prepared according to the patented protocols described by Cave and Mundell, in (2015).8 After preparation, the TiO2 colloidal dispersion was incorporated into two commercial universal dental bonding agents which are Ambar Universal (FGM, Brasil) and G-Premio Bond Universal (GC, America) at 4% by mass (0.20 g/5 g) utilizing the mass fraction formula.

TABLE 1. Chemical composition of the universal bonding agents used in this study.

Bonding agents	Manufacturer	Composition
Ambar Universal	FGM, Joinville, SC, Brazil	UDMA, HEMA, methacrylate hydrophilic monomers, methacrylate acid monomers, ethanol, water, silanized silicon dioxide, camphorquinone, ethyl 4-dimethylamino-benzoate, surfactant, sodium fluoride
G-Premio Bond Universal	GC, America	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica

Sample preparation

According to the ISO standardization 4049-10 in 2009 protocol,9 40 bonding agents disc samples were prepared using silicone rubber molds (5 × 1 mm) (Figure 1). At first, the disc space was filleted to half with the bonding agents, followed by gentle evaporation of the bonding agent’s solvent using hot air applied by the warm air tooth dryer.

Then the second half of the disc space was filled with the bonding agents, the solvent evaporated again, and was covered with a transparent strip and finally the bonding agents were light cured for 40 s.

FIGURE 1. The bonding agents’ discs after removal from the mold, confirming the dimensions (thickness and diameter) of the bonding agents discs.

Grouping

The 40 bonding agents' discs were divided into four groups of 10 discs each to test the water sorption and solubility as following:

Group I: Ten discs of the non-incorporated (Ambar Universal) (control group).

Group II: Ten discs of the 2% incorporated (Ambar Universal).

Group III: Ten discs of the non-incorporated (G-Premio Bond Universal) (control group).

Group IV: Ten discs of the 2% incorporated (G-Premio Bond Universal).

Testing procedure

The methodology for testing the water sorption and solubility of the bonding agents' groups was carried out in accordance with ISO standard 4049. After their removal from the molds, the bonding agents disc specimens were put in a desiccator containing silica gel (Figure 2), and then transferred to a pre-conditioning oven at 37°C for 1 day (Figure 2-43B) to evaporate any remaining solvents and unreacted monomers.10

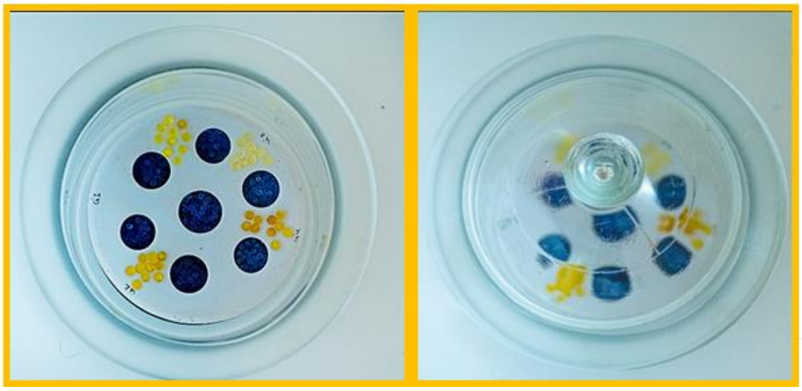

FIGURE 2. The bonding agents' discs are placed inside the desiccator containing fresh silica beads.

Following that, the specimens were weighed at a 1-day interval until a constant mass (named "m1") was achieved (i.e., the constant mass was recorded when the variance in any 1-day period was less than 0.2 mg). The thickness and diameter of the specimens were then measured using a digital caliper and rounded to the nearest 0.01 mm, and the volume (V) for each specimen was calculated using these values (in mm3). The disc specimens were then submerged for 1 year in a sealed glass vial containing 10 mL of distilled water (pH 7.2) at 37°C (Figure 3).11

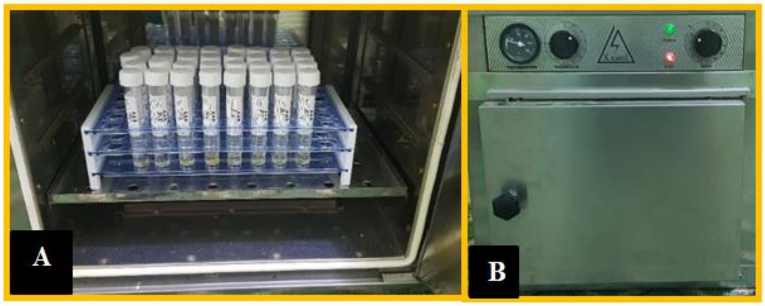

FIGURE 3. (A) The bonding agents' discs in vials containing distilled water. (A) The incubator that was used to store the vials at 37°C.

The disc specimens were rinsed in running water, rubbed gently with a soft absorbent paper to absorb excess moisture, and weighed in an analytical scale to get mass (m2) at the end of the 1-year storage period (Figure 2-45A) (Figure 2-45B). Finally, the specimens were dried in a desiccator with new silica gel and weighed every day until they reached a consistent mass (m3) (i.e., the same as described previously). The change in bonding agents' disc mass after the predetermined time period of water storage is calculated using the starting mass measured after the first desiccation process (m1) (i.e., 1-year). The following formulas were used to compute the water sorption and solubility:12

$\text{Water Sorption} = (m2 - m3)/V$

$\text{Solubility} = (m1 - m3)/V$

TABLE 2. Descriptive statistical results of water sorption values (µg/mm3).

	N	Minimum	Maximum	Mean	Std. deviation
GA	10	48.450	54.950	51.144	1.554
GB	10	26.155	36.390	31.074	1.588
GC	10	31.245	41.564	36.853	1.686
GD	10	21.660	33.640	27.055	1.456

RESULTS

Water sorption

Descriptive statistics

Table 2 and Figure 4 show the findings of descriptive statistics that comprised the minimum and maximum, mean values, and standard deviation values of water sorption for all the tested groups.

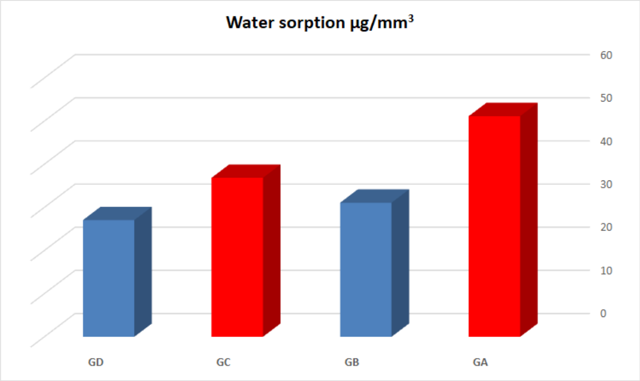

FIGURE 4. Mean values of water sorption for all the tested groups (µg/mm3).

The 4% TiO2-incorporated bonding agent groups had substantially lower mean water sorption values than the control non-incorporated bonding agents, as shown in Table 2 and Figure 4. The mean water sorption value of the control and 4% TiO2-incorporated G-Premio Bond Universal bonding agent is lower than the control and 4% TiO2-incorporated Ambar Universal groups. Ambar Universal has the greatest mean water sorption values.

Inferential statistics

The inferential statistics utilizing independent samples t-test demonstrated that there were statistically significant differences between the control and 4% TiO2-incorporated bonding agents groups for both bonding agents (Table 3). The test also demonstrated that the water sorption values of the G-Premio Bond Universal bonding agent groups (control and incorporated) are significantly lower than those of the Ambar Universal bonding agent groups.

TABLE 3. Independent samples t-test to compare the significance of difference in water sorption mean values of the tested bonding agent groups.

Groups	Mean difference	Standard error difference	P-value	Significance
GA versus GB	0.425	0.753	0.000	(HS)
GC versus GD	0.624	0.851	0.000	(HS)
GA versus GC	0.584	0.643	0.000	(HS)
GB versus GD	0.448	0.578	0.000	(HS)

Solubility

Descriptive statistics

Table 4 and Figure 5 show the findings of the descriptive statistics that comprised the minimum and maximum, mean values, and standard deviation values of water solubility for all tested groups.

TABLE 4. Descriptive statistical results of solubility values (µg/mm3).

	N	Minimum	Maximum	Mean	Standard deviation
GA	10	2.850	3.552	3.210	0.193
GB	10	0.982	1.952	1.737	0.215
GC	10	1.798	2.810	2.351	0.157
GD	10	0.851	1.821	1.478	0.185

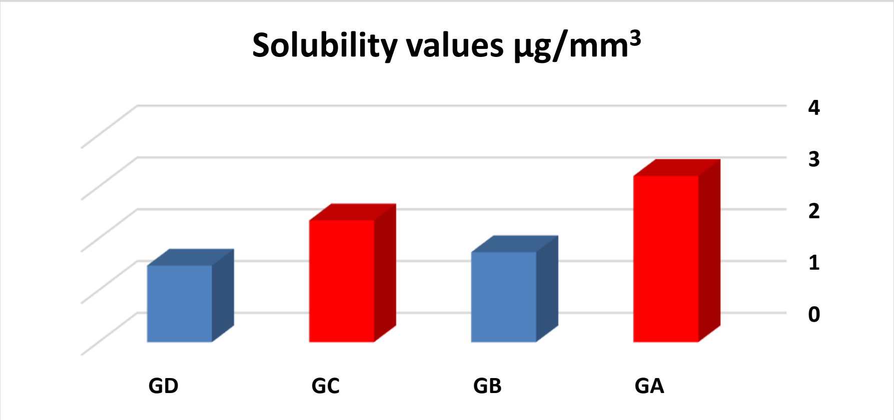

FIGURE 5. Mean values of solubility for the tested bonding agents groups (µg/mm3).

The 4% TiO2-incorporated bonding agents had lower solubility mean values than the control groups, as shown in Table 4 and Figure 5. Ambar Universal bonding agent with 4% TiO2 has higher solubility mean values than G-Premio Bond Universal bonding agent with 4% TiO2. The Ambar Universal bonding agent (control) had the greatest mean solubility values, while the G-Premio Bond Universal bonding agent groups (4% TiO2-incorporated) had the lowest mean solubility values.

Inferential statistics

The inferential statistics utilizing independent samples t-test demonstrated that there were statistically significant differences between the control and 4% TiO2-incorporated bonding agent groups for both bonding agents (Table 5). The test also demonstrated that the solubility values of the G-Premio Bond Universal bonding agent groups (control and incorporated) are significantly lower than those of the Ambar Universal bonding agent groups.

TABLE 5. Independent samples t-test to compare the significance of difference in solubility mean values of 2% AA-SPN incorporated bonding agents in comparison to the control groups.

Groups	Mean difference	Standard error	P	Significance
GA versus GB	0.475	0.110	0.000	(HS)
GC versus GD	0.324	0.104	0.000	(HS)
GA versus GC	0.557	0.108	0.000	(HS)
GB versus GD	0.583	0.125	0.000	(HS)

DISCUSSION

The hydrophilic monomer content (i.e., HEMA, 4-META, PENTA, 10-MDP) and hydrophobic monomers (i.e., Bis-GMA, UDMA) are chemically balanced in the composition of the contemporary universal bonding agents that allow them to intrinsically penetrate and infiltrate into the wet dentin surface.13 Therefore, the water sorption and solubility of dental bonding agents have been demonstrated to have a direct influence on the long-term performance of esthetic restorative materials. Dental bonding agents' resins are polymer-based biomaterials that are often utilized in restorative dentistry to the bond tooth structure to resin composites. Polymerization shrinkage, inadequate encapsulating of collagen fibrils, microleakage, and accumulation of dental biofilms are some of the common issues associated with contemporary dental bonding agents' resins.14

When compared to dental amalgams and other restorative materials, these variables have been shown to cause esthetic restorations to fail prematurely due to secondary caries and have shorter service lifetimes.15

In comparison to the control groups, the findings of this investigation revealed statistically significant variations in water absorption and solubility of the 4% TiO2-incorporated bonding agents. Both bonding agent groups met the ISO 4090 standard criteria for dental applications, which limit water sorption and solubility to a maximum of 40 g/mm3 and 7.5 g/mm3, respectively.

When compared to the control groups, the 4% TiO2-included bonding agents (both kinds) demonstrated a considerable reduction in water sorption solubility values. This might be attributed to the increase in the filler loading of the bonding agents after incorporating the TiO2 nanoparticles which probably might limit the polymer's water permeability by reducing the empty spaces within the polymerized polymer network, and therefore the polymer's swelling by water sorption. Furthermore, the incorporated nanoparticles filled the free polymer spaces that limited the extraction of unreacted monomer components from the polymerized resin network preventing monomer loss which would have a negative impact on the mechanical properties and longevity of these polymeric materials.16

When comparing the water sorption mean values of G-Premio Bond Universal and Ambar (Incorporated and control groups), G-Premio Bond Universal showed significantly lower values than Ambar Universal bonding agent. This is most likely due to the differences between G-Premio Universal and Ambar Universal formulated chemistry. The chemistry of Ambar Universal bonding agent is based on HEMA/UDMA monomers.17 The ester bonds in these monomers are responsible for the chemical breakdown of the polymer network, which starts with the ester bonds being hydrolyzed, releasing tiny alcohol molecules, and destroying the crosslinked structures formed during resin polymerization.18 HEMA and UDMA hydrophilic monomers have been shown to elute from methacrylate-based self-etch bonding agents in less than 24 h, indicating that unpolymerized monomers are easily extracted.19

CONCLUSIONS

Incorporation of the TiO2 nanoparticles at 4% by mass into the universal bonding agents significantly reduced their water sorption and solubility compared to their control groups. The Ambar Universal showed significantly higher water sorption and solubility than G-Premio Bond Universal bonding agents.

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