

Long-term water sorption/solubility of two dental bonding agents containing a colloidal dispersion of titanium dioxide

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ABSTRACT

The aim was to analyze the influence of the incorporation of 4% by mass of colloidal dispersion of titanium dioxide (TiO₂) nanoparticles on the long-term water sorption and solubility of two commercial universal bonding agents. *In vitro* studies. A colloidal dispersion of TiO₂ nanoparticles was formulated and blended into two commercial dental bonding agents, i.e., Ambar Universal (FGM, Brasil) and G-Premio Bond Universal (GC, America) at 4% by mass. Forty bonding agent discs were fabricated and segregated into four bonding agent groups of 10 discs each, i.e., 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 bonding agent discs were developed by dispensing the bonding agents into a silicone cast of 5 mm diameter and 1 mm depth. After bonding agent discs were desiccated, the cured discs were weighed and kept in distilled water to be evaluated for water sorption and solubility over 1 year storage period. Statistical analysis was performed by independent variable *t*-test performed using the IBM SPSS software (Chicago, IL: SPSS Inc). The incorporated bonding agent groups (GA and GB) showed significantly lower ($P < 0.05$) water sorption and solubility following 1 year of water storage in comparison to the control bonding agents. Both GC and GD demonstrated remarkably lower water sorption and solubility than GA and GB. Incorporation of the colloidal dispersion of TiO₂ nanoparticles at 4% by mass into the universal bonding agents has significantly reduced their water sorption and solubility contrast to their control groups.

Key words: Colloidal dispersion, solubility, titanium dioxide, universal bonding agents, water sorption

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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 diverse direct or indirect restorative materials.^[1]

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Hybrid layer formation starts with bonding agents resin monomers infiltrating into the mineral-depleted water-rich dentin and into the exposed collagen matrix, followed by the subsequent *in situ* photo-polymerization. The establishment of a stable hybrid layer permits the formation of a cross-linked 3-D polymer-collagen network to reduce microleakage, marginal staining, bacterial incursion, secondary caries formation, and pulpal irritation.^[2] Although the dental bonding agents are based on chemistry that contains both hydrophilic and hydrophobic functional monomers, they could become chemically unsteady when kept in contiguity with a moist dentin substrate. This could result in instability of the hybrid layer which might cause 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 affects their physical and mechanical characteristics. While solubility of dental bonding agents is elucidated 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 impact on the mechanical characteristics 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.^[6] TiO₂ nanoparticles have been used in dentistry to increase endodontically treated teeth' fracture resistance, osseointegration of dental implants, and enhance a material's antibacterial potential.^[7]

Therefore, the goal of this *in vitro* analysis was to ameliorate and prolong the stability of the bonding agent's polymer network in a wet environment through the incorporation of a colloidal dispersion of TiO₂ nanoparticles into those bonding agents by testing their impact on the water sorption and solubility of such bonding agents.

MATERIALS AND METHODS

Preparation and incorporation of the colloidal dispersion of titanium dioxide nanoparticles

For this study, the colloidal dispersion of TiO₂ nanoparticles was prepared according to the patented protocols described by Cave and Mundell (2015).^[8] After preparation, the TiO₂ colloidal dispersion was incorporated into two commercial universal dental bonding agents [Table 1] which are the Ambar universal (FGM, Brasil) and G-Premio Bond

Universal (GC, America) at 4% by mass (0.20 gm/5 gm) utilizing the mass fraction formula.

Sample preparation

According to the ISO standardization 4049-10 in 2009 protocol,^[9] 40 bonding agents disc samples were prepared using a silicone rubber molds (5 mm × 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 covered with a transparent strip and finally, the bonding agents were light cured for 40 s.

Grouping

The forty bonding agent discs were categorized into four batches of 10 disks each to test the water sorption and solubility as follows:

Group I: 10 bonding agent discs of the nonincorporated (Ambar Universal) (control group).

Group II: Eight bonding agent disks of the 2% incorporated (Ambar Universal).

Group III: Eight bonding agent disks of the nonincorporated (G-premio Bond Universal) (control group).

Group IV: Eight bonding agent disks of the 2% incorporated (G-premio Bond Universal).

Testing procedure

The methodology for testing the water sorption and solubility of the bonding agent groups was carried out in accordance with ISO standard 4049. Following removal from the molds, the bonding agents disc specimens are put in a desiccator comprising silica gel [Figure 2] and then kept in an oven at 37°C for one day [Figure 2] to evaporate any remaining solvents and unreacted monomers.^[10]

Following that, the specimens were weighed at 1-day intervals until a constant mass (named as "m1") was



Figure 1: The bonding agent discs after removal from the mould confirming the dimensions (thickness and diameter) of the bonding agents discs

achieved (i.e., the constant mass was recorded when the variance in any 1-day period was <0.2 mg). The thickness and diameter of the specimens were then recorded utilizing a digital caliper and the volume (V) for each specimen was estimated 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 a temperature of 37°C [Figure 3].^[11]

The discs were rinsed in running water, rubbed delicately with absorbent paper to absorb excess moisture, and weighed in an analytical scale to get a mass (m₂) at the end of the 1-year storage period. Finally, the discs were dried in a desiccator with new silica gel and reweighed every day till they reached a consistent mass (m₃) (i.e., the same as described previously). The change in the bonding agent's disc mass after the predetermined period of water storage was computed utilizing the starting mass measured following 1st desiccation (m₁) (i.e., 1 year). Water sorption

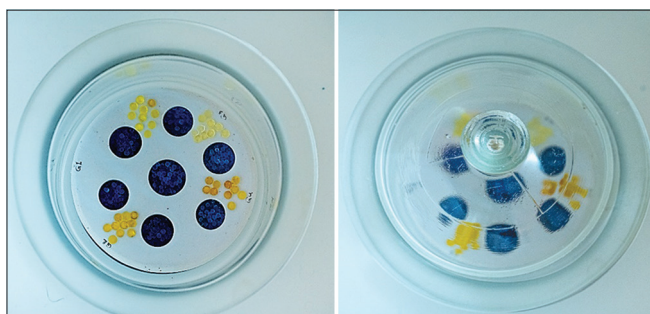


Figure 2: The bonding agent discs are placed inside the desiccator containing fresh silica beads

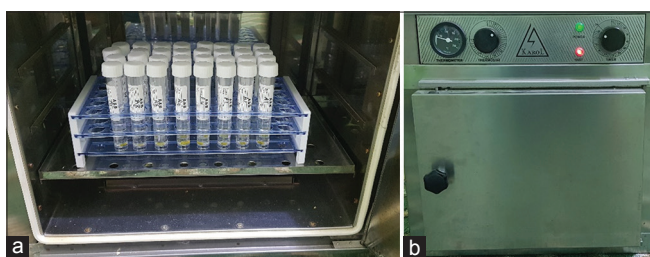


Figure 3: (a) The bonding agents discs in vials containing distilled water. (b) The incubator that was used to store the vials at 37°C

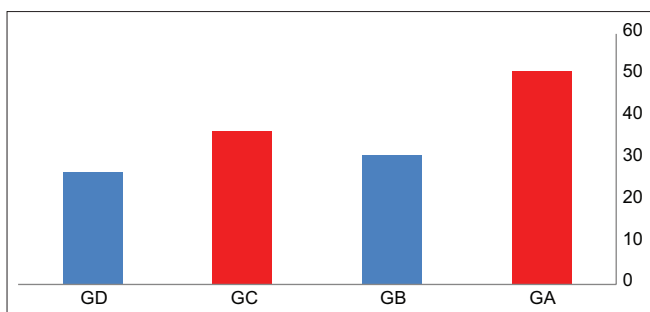


Figure 4: Graph showing the mean values of water sorption of all tested groups (µg/mm³)

and solubility were computed by the below-mentioned formulas:^[12]

$$\text{Water Sorption} = (m_2 - m_3)/V$$

$$\text{Solubility} = (m_1 - m_3)/V$$

RESULTS

Water sorption

Descriptive Statistics

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

The 4% TiO₂ incorporated bonding agent groups had substantially lower mean water sorption values than the control nonincorporated bonding agents, as shown in Table 2. The mean water sorption value of the control and 4% TiO₂ incorporated G-premio bond universal bonding agents was 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 exhibited that there were statistically significant differences among the control and 4% TiO₂ incorporated bonding agent groups for both bonding agents [Table 3]. The test

Table 1: Chemical constitution of the universal bonding agents used in this investigation

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

FGM: Company name, SC: South Colombia, GC: Company name, UDMA: Urethane dimethacrylate, HEMA: Hydroxyethyl methacrylate, MDP: Methacryloyloxydecyl dihydroxy phosphate, MET: Methacryloyloxyethyl trimellitic acid, MEPS: Methacryloyloxyalkyl thiophosphate methacrylate

Table 2: Descriptive statistical results of water sorption values (µg/mm³)

	n	Minimum	Maximum	Mean	SD
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

SD: Standard deviation, GA: Group A, GB: Group B, GC: Group C, GD: Group D

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.

Solubility

Descriptive statistics

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

The 4% TiO₂ incorporated bonding agents had lower solubility mean values than the control groups, as shown in Table 4. Ambar Universal bonding agents with 4% TiO₂ have higher solubility mean values than G-Premio bond universal bonding agents with 4% TiO₂. The Ambar Universal bonding agents (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 manifested that there were statistically significant differences among 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) were significantly lower than those of the Ambar universal bonding agent groups.

DISCUSSION

The hydrophilic monomer content (i.e., HEMA, 4-META, PENTA, and 10-MDP) and hydrophobic monomers (i.e., Bis-GMA and 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 qualities of dental bonding agents have been demonstrated to directly influence the long-term performance of esthetic restorative materials.

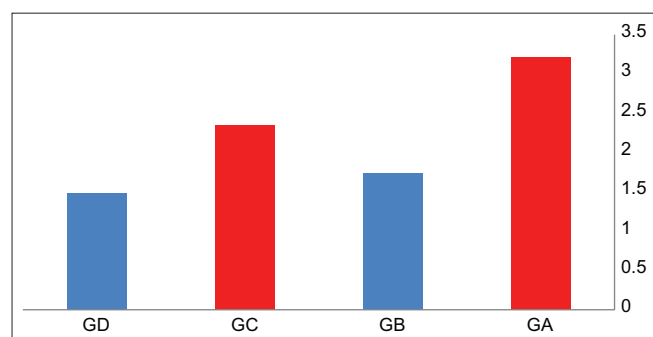


Figure 5: Graph showing the mean values of solubility for the tested groups (µg/mm³)

Dental bonding agent resins are based on polymer biomaterials that are often utilized in restorative dentistry to bond tooth structure to resin composites. Polymerization shrinkage, inadequate encapsulation of collagen fibrils, microleakage, and accumulation of dental biofilms are a few common issues correlated with contemporary dental bonding agent resins.^[14] In contrast 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 contrast 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

Table 3: Independent samples *t*-test

Groups	Mean difference	SE difference	P	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

SE: Standard error, HS: Highly significant, GA: Group A, GB: Group B, GC: Group C, GD: Group D

Table 4: Descriptive statistical results of solubility values (µg/mm³)

	n	Minimum	Maximum	Mean	SD
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

SD: Standard deviation, GA: Group A, GB: Group B, GC: Group C, GD: Group D

Table 5: Independent samples *t*-test for comparison of the significance of difference in solubility mean values of 2% AA-SPN incorporated bonding agents in comparison to the control groups

Groups	Mean difference	SE	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

SE: Standard error, HS: Highly significant, GA: Group A, GB: Group B, GC: Group C, GD: Group D

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 could limit the extraction of unreacted monomer components from the polymerized resin network, preventing monomer loss which would negatively impact the mechanical characteristics and durability 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 demonstrated remarkably lower values than Ambar universal bonding agents. This is most likely due to the differences between G-premio universal and Ambar universal formulated chemistry. The chemistry of Ambar universal bonding agents 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 cross-linked structures formed during resin polymerization.^[18] HEMA and UDMA hydrophilic monomers have been shown to elute from methacrylate-based self-etch bonding agents in <24 h, indicating that unpolymerized monomers are easily extracted.^[19]

CONCLUSIONS

Incorporating 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 bond universal demonstrated significantly higher water sorption and solubility than G-Premio bond universal bonding agents.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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