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In Vitro Evaluation of Sorption and Solubility in Orthodontic Composite Resins

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ABSTRACT

Introduction: Sorption and solubility are important mechanical properties when evaluating a composite resin for orthodontic use, since chemical degradation is a phenomenon that occurs in materials for dental use due to exposure to saliva and other liquids present in the oral cavity. The aim of this in vitro study was to evaluate some sorption characteristics (sorption, solubility) of five resins for orthodontic use: Transbond XT, Orthocem, Orthobond Plus, Ortholink VLC and Orthocem UV-Trace. **Methods:** Five specimens of each resin (n=5) were prepared. After fabrication, the samples were stored in water and the properties measured for 28 days. **Results:** Sorption and solubility were statistically analyzed by two-way ANOVA (time and resin) followed by the post hoc Tukey's test. The Transbond XT resin obtained the lowest values for sorption and solubility at all times. **Conclusion:** The mechanical properties of resin materials used in orthodontics differ according to their chemical composition. The resin that showed the best properties in all tests conducted in this study was Transbond XT, which considered the gold standard in research related to bonding materials for brackets and orthodontic accessories.

Keywords: Orthodontics, Resin Cements, Solubility.

INTRODUCTION

To comprehend the characteristics and limitations of different orthodontic adhesives, several physical properties have been studied. Among these, the characteristics related to polymerization stand out. Orthodontic adhesives, predominantly resin-based composites, face inherent physical restrictions caused by incomplete polymerization [1,2]. Normally, monomers are not completely consumed in the polymerization reaction, resulting in the retention of unreacted monomers that are ready to be released (leached) into the oral environment [3]. This presence of residual monomers is a factor that triggers an increase in the resin's porosity through sorption and solubility [4-6].

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Sorption and solubility are important in bonding orthodontic attachments because orthodontic resins are constantly exposed to chemical agents in saliva, liquid substances of different pH levels, mouthwashes, and food. Over time, this exposure can lead to a degradation effect [7,8].

Chemical degradation is generally caused by oxidation or hydrolysis, where water plays an important role in this process [9]. This results in weight loss and can be measured as solubility or leaching [9,10]. In turn, water sorption is a diffusion phenomenon that occurs primarily in the organic matrix of the resin. The accumulation of water can cause hydrolytic corrosion of the network structure, resulting in the debonding of silanized fillers and compromising the durability of the bond [9,11]. Clinically, this can lead to premature failure of the orthodontic attachment, requiring rebonding and increasing the treatment time for the patient [11]. Several factors contribute to these processes, such as the exposure time and the particle composition of a resin [12-14].

The methods for calculating the water sorption and solubility of dental materials, consist of immersing them in aqueous media [7,8,15,16]. The standardized evaluation of sorption and solubility of dental materials adheres to the ISO 4049 standard [15,17,18]. The method consists of immersing desiccated samples in distilled water for seven days to determine the amount of absorbed water (sorption) and then obtaining the final weight after re-desiccation until a constant weight is achieved [15,17,18].

Notwithstanding innovations in filler architectures and bioactive monomers, including the promising use of superparamagnetic nanoparticles in advanced dentistry [19-21], the contemporary literature reveals a scarcity of data on sorption and solubility in commercial orthodontic resin composites [22,23], underscoring a critical evidence gap for clinical translation.

Thus, this in vitro study evaluated the water sorption and solubility of five commercial orthodontic composite resins in distilled water, following the guidelines of ISO 4049. The null hypothesis was that there would be no intermaterial differences in sorption and solubility values among the tested materials.

MATERIAL AND METHODS

Five commercial composite resin for bracket adhesion were tested: Transbond XT, Orthocem, Orthobond Plus, Ortholink VLC and Orthocem UV-Trace. The composition of the materials used for the different tests performed is specified in Table 1.

For sorption and solubility measurements, five disk specimens were prepared for each material (n=5) in a cylindrical metal matrix [5.0 mm diameter and 2.0 mm thickness Odeme Prod Odont Ltda (Joaçaba, SC, Brazil - Figure 1)].

Figure	Material	Manufacturer	Chemical Composition
	Transbond XT	3M Unitek (Monrovia, EUA)	Bis-GMA, TEGDMA Bis-EMA Bisphenol A Dimethacrylate Bis (2- hydroxyethyl ether), silane treated silica, quartz microparticles (80% by weight).
	Orthocem	FGM (Joinville, SC, Brazil)	Methacrylic monomers such as BisGMA, TEGDMA and phosphated methacrylic monomers, stabilizer, camphoroquinone, co-initiator and silicon dioxide nano filler (55% by weight)

	Orthobond Plus	Morelli (Sorocaba, SP, Brazil)	Acrylic copolymer adhesive paste. Bisphenol A Dimethacrylate Glycerolate, Triethylene Glycol Dimethacrylate, inorganic filler (quartz with an average size of 45 μm), red solvent, photoinitiators and stabilizers, acrylic copolymer adhesive paste
	Ortholink VLC	Orthometric (Marília, SP, Brazil)	Bis-GMA-Dimethacrylate, Bisphenol A glycerolate, amine, light stabilizer, photoinitiator, synthetic amorphous silica, Teg-DMA-Dimethacrylate Triethylene Glycol, inorganic filler.
	Orthocem UV-Trace	FGM (Joinville, SC, Brasil)	Methacrylic monomers such as BisGMA, TEGDMA and phosphated methacrylic monomers, stabilizer, sodium fluoride, camphoroquinone and co-initiator, inorganic fillers of nanometer silanized silicon dioxide and luminescent pigment.

Solid petroleum jelly was applied to the base of the mold using a microbrush (KG Sorensen, Cotia, SP, Brazil), and a polyester strip was then positioned in contact with the mold base. After isolation of the mold, the composite resins were inserted using a No. 1 titanium resin spatula (Indusbelo, Salvador, BA, Brazil) until complete filling of the mold was achieved. Subsequently, a glass slide was positioned over the resin surface, and the material was light-cured using an LED-based light-curing unit for 40 seconds with a tip irradiance of 1000 mW/cm^2 (VALO, Ultradent Products, South Jordan, UT, USA). The adopted radiant exposure (4.8 J/cm^2) allowed specimen removal from the mold without deformation. Finally, the specimens were carefully removed using a Lecron spatula (SS White, Vasco da Gama, Rio de Janeiro, RJ, Brazil) to eliminate excess resin material (Figure 1).

Subsequently, the specimens were sanded with water sandpaper sequentially 600-800 and 1000-1200 to perform the finishing and flattening for subsequent testing.

The ISO 4049 specification employed in the determination water sorption (S) and solubility (SL) [15,17,18]. Immediately after polymerization, the specimens placed in silica gel dissectors, which transferred to a preconditioned oven at 37°C and left for 7 days. After that, they washed at 24 h intervals until the mass variation between two consecutive days was less than 0.2 μg. A digital caliper (Figure 2) was used to measure the thickness and diameter of the specimens, with an accuracy of 0.01 mm, to calculate the volume (V in mm^3).

Each specimen was weighed preliminarily (m1), then each specimen was placed in a micro tube (sealed Eppendorf) with 10 mL of distilled water (pH 7.2) at 37ºC. After intervals of 1, 8 and 24 h and 7, 14 and 28 days of storage time, each specimen removed from the oven and left at room temperature for 30 min. Then, they were washed in running

water, gently cleaned with soft and absorbent paper, and weighed on an analytical balance (m2), returning to the micro tubes with 10 mL of fresh distilled water.

After the storage time of 28 days, the specimens were dried in desiccators containing silica gel at 37 °C, which remained at rest for 10 days or until the mass variations were less than 0.2 µg, obtaining a constant mass (m3). Once the weightings m1, m2 and m3 were obtained at the respective times, the S and SL were measured with the following formulas:[16] *S = (m2 - m3) / V and SL = (m1 - m3) / V*

RESULTS

Sorption mean values (um/mm^3) and standard deviations are presented in Table 1. Sorption was different for the tested materials ($p<0.05$).

Table 1. Means and standard deviation of the obtained sorption (ug/mm3) data

	1h	8h	24h	7d	14d	28d
Transbond XT	0.10±0.04 AB	0.10±0.04 A	0.15±0.03 A	0.11±0.04 A	0.11±0.04 A	0.11±0.04 A
Orthocem	0.27±0.10 CD	0.30±0.10 BC	0.48±0.07 C†	0.45±0.04 E†	0.47±0.04 E†	0.48±0.07 D†
Orthobond Plus	0.23±0.09 BD	0.25±0.07 B	0.30±0.10 B	0.27±0.07 BC	0.27±0.07 BC	0.24±0.06 B
Ortholink VLC	0.34±0.03 D	0.35±0.02 C	0.35±0.05 B	0.31±0.03 CD	0.31±0.03 CD	0.31±0.04 BC
Orthocem UV-Trace	0.19±0.09ABC	0.22±0.06 B	0.37±0.06. B†	0.37±0.05DE†	0.39±0.05DE†	0.40±0.04 D†

(*) Equal letters indicate statistically similar results between columns (Tukey's test, $p<0.05$). Symbol (†) represents statistically different results compared to the 1 h (initial) sorption time.

Figure 1 shows the results obtained for water sorption at the six different times. The Transbond XT resin group was the one that obtained the lowest sorption values at all times (ranging from 0.10 ± 0.04 to 0.15 ± 0.03 µg/mm^3) when compared to the other resins, as well as no significant differences in values over the evaluated period. Orthobond Plus and Ortholink VLC resins reached similar values among themselves (peaking at 0.30 ± 0.10 and 0.35 ± 0.05 µg/mm^3, respectively, at 24 hours), and did not undergo significant changes in mass over time. Orthocem and Orthocem UV-Trace resins showed the highest values of water sorption over time.

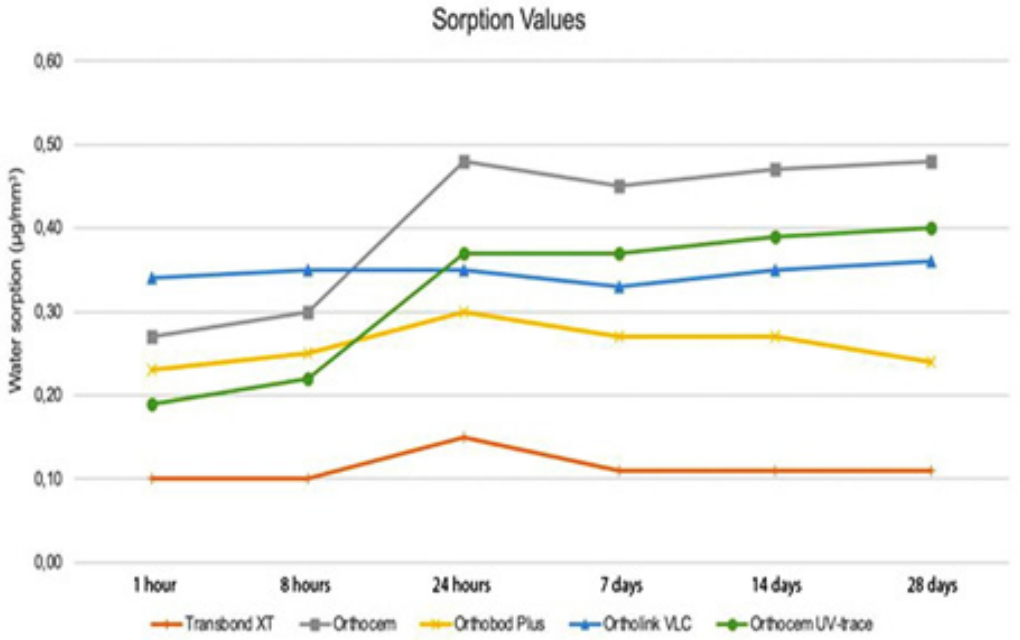

Figure 1. Sorption data averages (ug/mm^3).

Table 2 shows the solubility results of the tested materials. It can be observed that Ortholink VLC resin reached the highest value (0.35 ± 0.03 μg/mm^3) when compared to Transbond XT, which on the other hand, obtained the lowest rate (0.12 ± 0.05 μg/mm^3). The other resins, Orthocem (0.28 ± 0.09 μg/mm^3), Orthobond Plus (0.21 ± 0.08 μg/mm^3), and Orthocem UV-Trace (0.18 ± 0.08 μg/mm^3), demonstrated intermediate solubility values.

Table 2. Averages and standard deviation of the solubility data obtained

Resin	Solubility (ug/mm^3)
Transbond XT	0.12 ± 0.05 A
Orthocem	0.28 ± 0.09 BD
Orthobond Plus	0.21 ± 0.08 ABC
Ortholink VLC	0.35 ± 0.03 CD
Orthocem UV-Trace	0.18 ± 0.08 AB

Notes: (*) Equal letters indicate statistically similar results (*Tukey's* test, p<0.05).

Figure 2 shows the solubility variations between the different resins tested, where it is evident that the Transbond XT resin presents the lowest solubility.

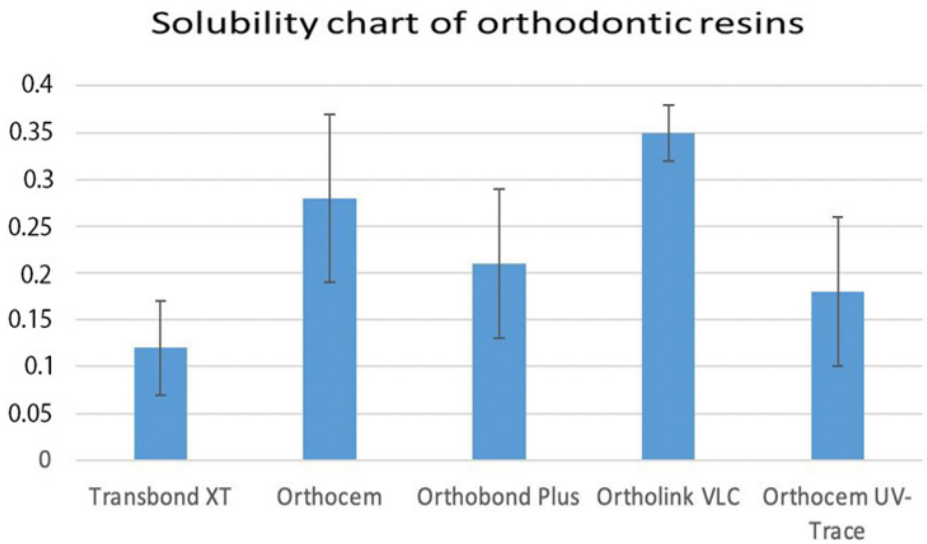

Figure 2. Solubility of the orthodontic resins tested.

Statistical analysis

To detect normality between the groups the Shapiro Wilk test was applied, and the homogeneity of variances was tested using Levene's test. Sorption was analyzed by ANOVA two-way (time and resin). Solubility was analyzed by one-way ANOVA (resin). In order to observe the differences between the groups evaluated, for sorption and solubility, the Tukey's test performed afterwards. All calculations performed using the statistical software SPSS® (Statistical Package for the Social Science) version 20.0 (IBM, Inc Chigago, Illinois USA).

DISCUSSION

The observed sorption and solubility patterns in this study reveal marked inter-material disparities among the tested orthodontic resins. Transbond XT consistently demonstrated the lowest values (0.10–0.15 μg/mm^3 across 1h to 28d; p>0.05), a finding that aligns with its established hydrophobic formulation and high inorganic filler loading (up to 80 wt%). This high filler content reduces the volume of the organic matrix available for water uptake, while the combination of Bis-GMA and UDMA monomers—which are less hydrophilic than other low-viscosity monomers—restricts water ingress via reduced hydrophilic domains [1,24]. This stability

mirrors reports by Pelourde et al. (2018), who attributed such resilience to superior silane coupling that preserves filler-matrix integrity against hydrolytic assault [24].

Regarding the mechanisms of water sorption, several factors determine the diffusion coefficient, including resin composition, porosity, and the nature of the monomeric units. One of the most common monomers in composite resins is TEGDMA, which is a relatively hydrophilic compound with a high water sorption capacity due to its ethoxy groups [2, 10]. According to Venz and Dickens, the performance of common monomers regarding water uptake follows the order: TEGDMA > Bis-GMA > UDMA [10]. In this study, the resins with the highest water sorption indices were Orthocem and Orthocem UV-Trace (up to 0.48 μg/mm^3 at 28d; $p<0.05$). This behavior is likely attributable to their lower inorganic content (55%) and greater residual monomer elution. These findings are consistent with recent analyses where UV tracers and higher TEGDMA concentrations correlated with accelerated porosity amplification and time-dependent mass gain [23,25].

Solubility profiles closely paralleled the sorption trends, as the same parameters affecting water uptake—such as the degree of conversion and monomer chemistry—interfere with the leaching of unreacted components. Transbond XT's minimal mass loss (0.12 ± 0.05 μg/mm^3) parallels recent in vivo saliva monitoring, which detected rapid tapering of monomer elution (HEMA/poly-EGDMA) within 24 hours [26]. Conversely, Ortholink VLC's higher solubility (0.35 ± 0.03 μg/mm^3) echoes studies linking high solubility to potential cytotoxicity and degradation of the bonding interface [27]. These variances decisively reject the null hypothesis, pinpointing compositional determinants—filler volume fraction, monomer hydrophilicity, and silane efficiency—as pivotal modulators of material longevity [2].

From a clinical perspective, an ideal orthodontic resin should exhibit low sorption and solubility to prevent oxidation and staining during the bonding of metal or ceramic brackets, which is crucial considering the high aesthetic demands and the risk of overtreatment in contemporary dentistry [28], as well as to avoid filler-matrix decohesion that leads to debonding. While all tested resins remained within the ISO 4049:2019 thresholds for restorative materials (<40 μg/mm^3 for sorption and <7.5 μg/mm^3 for solubility) [29], the elevated values in the Orthocem variants and Ortholink VLC signal a higher vulnerability to chronic degradation compared to the "gold standard" Transbond XT. Recent innovations in self-etch systems (e.g., 10-MDP variants) have shown up to 35% reduction in leaching compared to conventional systems, yet commercial analogs often still exhibit formulation gaps that may impact long-term intraoral performance [26].

This investigation unequivocally rejects the null hypothesis, revealing significant inter-material disparities that underscore compositional hydrophilicity and filler-matrix interfaces as fundamental for long-term stability. The resilience of Transbond XT supports its preference for sustaining durable orthodontic bonding. Conversely, high-sorption adhesives require caution or adjunctive protocols to mitigate porosity-induced failures [24]. These outcomes advance material selection paradigms by linking *in vitro* metrics to real-world performance amidst evolving bioactive innovations [22].

Study limitations include the static *in vitro* immersion without thermal cycling, enzymatic challenge, or biofilm simulation. Nonetheless, the temporal granularity of this study (up to 28 days) enhances translational fidelity compared to shorter protocols [23]. Future research should integrate dynamic intraoral simulations—encompassing thermomechanical fatigue and biofilm interactions—to refine predictive models and catalyze the development of next-generation adhesives with sub-ISO degradation profiles [23]. Furthermore, the surface degradation and porosity of these resins could be thoroughly evaluated using advanced texture analysis methods [30].

CONCLUSION

The mechanical properties of resins used in orthodontics present differences according to their chemical composition. The resin that showed the best properties in sorption and solubility tests was Transbond XT, which demonstrated the lowest sorption values (ranging from 0.10 to 0.15 μg/mm^3) and the lowest solubility (0.12 μg/mm^3) across all evaluated periods. Conversely, the highest water sorption was observed in Orthocem (0.48 μg/mm^3 at 28 days), while Ortholink VLC exhibited the highest solubility (0.35 μg/mm^3).

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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