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Effect on adhesion of a nanocapsules-loaded adhesive system

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Abstract: This study aimed to evaluate the *in situ* degree of conversion, contact angle, and immediate and long-term bond strengths of a commercial primer and an experimental adhesive containing indomethacin- and triclosan-loaded nanocapsules (NCs). The indomethacin- and triclosan-loaded NCs, which promote anti-inflammatory and antibacterial effects through controlled release, were incorporated into the primer at a concentration of 2% and in the adhesive at concentrations of 1, 2, 5, and 10%. The *in situ* degree of conversion (DC, n=3) was evaluated by micro-Raman spectroscopy. The contact angle of the primer and adhesive on the dentin surface (n = 3) was determined by an optical tensiometer. For the microtensile bond strength μTBS test (12 teeth per group), stick-shaped specimens were tested under tensile stress immediately after preparation and after storage in water for 1 year. The data were analyzed using two-way ANOVA, three-way ANOVA and Tukey's *post hoc* tests with α=0.05. The use of the NC-loaded adhesive resulted in a higher *in situ* degree of conversion. The DC values varied from 75.07 ± 8.83% to 96.18 ± 0.87%. The use of NCs in only the adhesive up to a concentration of 5% had no influence on the bond strength. The contact angle of the primer remained the same with and without NCs. The use of both the primer and adhesive with NCs (for all concentrations) resulted in a higher contact angle of the adhesive. The longitudinal μTBS was inversely proportional to the concentration of NCs in the adhesive system, exhibiting decreasing values for the groups with primer containing NCs and adhesives with increasing concentrations of NCs. Adhesives containing up to 5% of nanocapsules and primer with no NCs maintained the *in situ* degree of conversion, contact angle, and immediate and long-term bond strengths. Therefore, the NC-loaded adhesive can be an alternative method for combining the bond performance and therapeutic effects. The use of an adhesive with up to 5% nanocapsules containing indomethacin and triclosan and a primer with no nanocapsules maintained the long-term bond performance.

Keywords: Indomethacin; Triclosan; Drug Carriers; Dental Bonding.

Introduction

Nanocapsules (NCs) composed of a polymeric capsule and an oil core containing a drug promote controlled drug release.[1] Consequently, the bioavailability of the encapsulated drugs lasts twice as long and

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promotes a higher efficiency over time compared with the free drug.[2] Other advantages of encapsulated drugs include deeper penetration of the drug into narrow spaces, lower adverse effects and higher drug stability due to the nanometer size of the particles and the low reactivity of the encapsulated drug with the medium.[3,4,5] Due to these favorable conditions, nanocapsules containing drugs have been applied in dental adhesive systems, promoting certain therapeutic effects, such as anti-inflammatory effects in the pulp tissue and antimicrobial effects to protect against caries, without compromising the physicochemical properties of the adhesive.[6,7,8]

Nanocapsules applied to dentin as formulations with water, primer or adhesive promote drug diffusion up to 0.75 mm into dentin.[6,7,8] In the hybrid layer, the nanocapsules incorporated in the adhesive were seen to be dispersed over the intertubular dentin surface and penetrate into the dentinal tubules.[6,9] The well-dispersed particles and spherical shape of the NCs avoid stress concentration in the adhesive, thus improving its properties.[9] The chemical affinity between the polymer of the NCs and the monomers of the adhesive system leads to reliable physicochemical properties.[6,7,8,9] The diffusion of nanocapsules in the hybrid layer did not result in a decrease in the immediate bond strength.[7,9] Nonetheless, the influence of the nanocapsules on the long-term bond strength has not yet been elucidated.

Therefore, this study aimed to evaluate the *in situ* degree of conversion, the contact angle, and the immediate and long-term bond strengths between a primer and adhesive containing indomethacin-and triclosan-loaded nanocapsules.

Methodology

The present study is an experimental *in vitro* study. Nanocapsules containing indomethacin and triclosan were formulated and added to a commercial primer (Scotchbond) and an experimental adhesive, as described below.

Preparation of indomethacin- and triclosan-loaded NCs

Indomethacin- and triclosan-loaded NCs were prepared by interfacial deposition through a preformed polymer technique, producing particles with an average size of 159 nm[8]. The reagents were obtained from Sigma Chemical (St. Louis, USA). The organic phase was composed of a polymer (MMA-co-MAA), Eudragit® S100 (0.50 g), indomethacin (0.025 g), triclosan (0.025 g), medium-chain triglycerides (0.81 mL), sorbitan monostearate (0.19 g) and acetone (125 mL). The aqueous phase contained polysorbate 80 (0.385 g) and water (250 mL). The organic phase was added via a funnel to the aqueous phase under magnetic stirring at 25°C. The acetone and excess water were removed using a rotary evaporator (Rotavapor II, Buchi, Flawi, Switzerland) consisting of a B-740 recirculating chiller (Buchi, Flawi, Switzerland) and a U-700 vacuum pump (Buchi, Flawi, Switzerland). The suspension containing NCs was spray-dried (B-290, Buchi, Flawi, Switzerland) using hydrophilic fumed silicon dioxide (Aerosil® 200) with a content of 1.5% in the suspension as an adjuvant to prevent aggregation on the internal walls of the equipment. The inlet temperature in the drying chamber was approximately 150 ± 4°C, and the outlet temperature was 107 ± 4°C.

Formulation of the adhesive resin and primer containing indomethacin- and triclosan-loaded NCs

Experimental dental adhesives were formulated using 66/33 wt% bisphenol A glycol dimethacrylate (BisGMA)/2-hydroxyethyl methacrylate (HEMA). Camphoroquinone (CQ) and ethyl 4-dimethylaminobenzoate (EDAB) were added at a concentration of 1 mol% as a photoactivation system. The dried indomethacin- and triclosan-loaded NCs were added at 1, 2, 5 and 10 wt% (groups AD1%, AD2%, AD5%, and AD10%, respectively). The group with no added particles was designated the control (group AD0%). Two weight percent of the indomethacin- and triclosan-loaded NCs was incorporated in a commercial primer (Scotchbond MP, 3M-ESPE, Seefeld, Germany), and one group was maintained with no NCs (groups PR0% and PR2%). These concentrations were chosen based on previous studies[7,8]. All formulations were mixed and ultrasonicated (CBU 100/1 LDG, Plana, São Paulo, Brazil) for 30 minutes.

Preparation of teeth

To analyze the *in situ* degree of conversion, contact angle and microtensile bond strength, bovine permanent incisors were transversally sectioned to expose the dentin surface. SiC paper (600 grit) was used for 30 s under wet conditions to create a smear layer on the dentin surface. The dentin surface was etched with phosphoric acid for 15 s, washed, and partially dried using absorbent paper. The primer, with or without NCs, was applied, and the solvent was evaporated for 10 s. The adhesives with different concentrations of NCs were applied and photoactivated for 20 s using a light-emitting diode (Radii cal, SDI, Bayswater, Australia). Composite build-up was performed (Z350, 3M ESPE, St Paul, USA) in two increments of 2 mm.

In situ degree of conversion (DC)

Thirty bovine incisors (n = 3) were prepared as described in the section "Preparation of teeth." The restored teeth were transversely cut and analyzed by micro-Raman spectroscopy (SENTERRA, Bruker Optics, Ettlingen, Germany). The spectra were acquired starting from dentin, and the observed peaks were associated with the adhesive. The Raman spectrum of each uncured adhesive was also collected to identify the reference and reaction peaks needed for the degree of conversion calculations by comparing these spectra with the spectrum of the cured adhesive on dentin. The phenyl C=C peak observed at 1,610 cm^{-1}, which remained stable and unmodified during polymerization, was selected as the reference peak, while the vinyl C=C peak at 1,640 cm^{-1} was selected as the reaction peak. The DC of the adhesive within the hybrid layer was calculated using the ratio between the reaction and internal reference peak areas as the ratio of polymerized to unpolymerized adhesive.

Contact angle

Thirty bovine incisors (n = 3) were embedded in acrylic resin and prepared as described in the section "Preparation of teeth." The teeth were mounted on an optical tensiometer (Theta, Biolin Scientific, Stockholm, Sweden). The primer (3 μL) and adhesives (3 μL) with different concentrations of NCs were dropped onto the dentin surface at a speed of 60 mm/min through a microsyringe. Images were captured at a speed of 12 frames/s up to 20 s after drop deposition using a micro-video system. OneAttension software (Biolin Scientific, Stockholm, Sweden) provided the values of the contact angle 10 s after drop deposition by Young's equation.

Microtensile bond strength (μTBS)

Two hundred and forty bovine permanent incisors were divided into 20 groups (12 teeth per group) based on the concentration of NCs in the primer and adhesive and the length of evaluation (24 hours or 12 months). The teeth were prepared as described in the section "Preparation of teeth." After storage in distilled water at 37°C for 24 h, the teeth were sectioned into four to six beams (area of 0.5 mm^2) with a slow-speed saw. The beams in the longitudinal groups were stored in distilled water, which was changed every month for one year. After 24 hours or 12 months, the specimens were fixed to a microtensile device and tested on a mechanical testing machine (DL-2000, EMIC Equipment and Systems for Essay Ltda, São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min until failure. Analysis of the fractographic failure mode was performed using an optical microscope (400x magnification) and classified as an adhesive and mixed cohesive in dentin or cohesive in resin. Premature failures were also reported.

Statistical analysis

Statistical analysis was performed using two-way ANOVA (nanocapsule and primer concentration) and Tukey's *post hoc* tests for the *in situ* degree of conversion and contact angle. Three-way ANOVA (nanocapsules, primer and time) and Tukey' *post hoc* were used to analyze the microtensile bond strength. All tests were performed at $\alpha = 0.05$ using SigmaPlot.

Results

The use of the NC-loaded primer resulted in no influence on the *in situ* degree of conversion. However, the use of the NC-loaded adhesive resulted in a higher *in situ* degree of conversion. The values of DC varied from 75.07 ± 8.83% to 96.18 ± 0.87% (Table 1).

The contact angle varied from 22.97 ± 4.04 to 54.69 ± 5.19 degrees for the primer with NCs and from 21.23 ± 7.85 to 25.19 ± 2.47 degrees for the primer without NCs. Regarding the contact angle of the adhesive, there were no significant differences among the different concentrations of NCs in the adhesive. The primer containing NCs did not influence the contact angle when the adhesive without NCs was used. The use of both primer and adhesive with NCs resulted in a higher contact angle than the use of NCs in only the adhesive ($p < 0.05$), ranging from 50.71 ± 15.23 to 54.69 ± 5.19 degrees (Table 1).

The microtensile bond strength results and the mode of failure are shown in Tables 2 and 3, respectively. The NCs in only the adhesive did not influence the immediate μTBS. The longitudinal μTBS was inversely proportional to the concentration of NCs in the adhesive system. The groups of the primer with NCs and the different concentrations of NCs in the adhesive showed a decreased longitudinal microtensile bond strength ($p< 0.05$). The microtensile bond strength showed differences between the immediate and longitudinal results for the groups with NCs in the primer. Adhesive/mixed fractures were more frequently identified in all groups followed by the cohesive fracture mode in dentin (Table 3). Premature failure occurred only in the immediate bond strength tests with a low frequency.

Discussion

The present study confirms the favorability of adding nanocapsules to an adhesive system for maintaining reliable long-term bond performance. The procedure allows the dental adhesives to prevent the inflammatory process of pulp tissue in deep cavities and protect against caries through controlled drug release due to the use of nanocapsules.[7,8] In the present study, the NC-loaded primer and adhesives had no influence on the *in situ* degree of conversion or the immediate bond strength. Nonetheless, the use of a primer and adhesive combination with NCs increased the contact angle and decreased the bond strength after one year of aging.

Table 1. Degree of conversion (DC, in percent, %) *in situ* and contact angle (in degrees).

Adhesive	DC *in situ* (%)		Contact angle (degrees)	
	PR 0%	PR 2%	PR 0%	PR 2%
0%	80.58 ± 6.58^{Ba}	75.07 ± 8.83^{Ba}	23.35 ± 3.50^{Aa}	22.97 ± 4.04^{Aa}
1%	96.02 ± 1.11^{Aa}	92.84 ± 3.75^{Aa}	24.74 ± 5.31^{Aa}	54.69 ± 5.19^{Ab}
2%	92.61 ± 0.55^{Aa}	88.26 ± 5.79^{Aa}	25.19 ± 2.47^{Aa}	50.71 ± 15.23^{Ab}
5%	93.54 ± 0.47^{Aa}	93.85 ± 0.61^{Aa}	22.40 ± 5.52^{Aa}	53.27 ± 16.49^{Ab}
10%	94.57 ± 0.44^{Aa}	96.18 ± 0.87^{Aa}	21.23 ± 7.85^{Aa}	53.56 ± 8.54^{Ab}

Different capital letters in the same column denote a statistically significant difference. Values followed by different lower-case letters in the same row denote a statistically significant difference. PR: primer.

Table 2. Immediate (24 hours) and longitudinal (12 months) microtensile bond strength (μTBS, in MPa).

Adhesive	mTBS (24 hours)		μTBΣ (12 μοντησ)	
	PR 0%	PR 2%	PR 0%	PR 2%
0%	36.71 ± 10.36^{aA*}	38.79 ± 7.36^{aA}	31.30 ± 11.34^{abA*}	28.37 ± 14.30^{aA}
1%	33.26 ± 4.12^{aA}	31.19 ± 8.45^{bB}	35.05 ± 4.25^{abA}	17.49 ± 5.70^{bB}
2%	31.96 ± 4.50^{aA}	30.09 ± 5.75^{bB}	30.31 ± 8.39^{bA}	20.36 ± 4.22^{bB}
5%	36.83 ± 9.35^{aA}	29.23 ± 2.07^{bB}	40.63 ± 5.66^{aA}	17.11 ± 6.41^{bB}
10%	39.10 ± 6.45^{aA}	30.05 ± 4.28^{bB}	29.78 ± 14.03^{bB}	13.92 ± 6.92^{bB}

In each column, values marked by different lowercase letters denote a significantly difference ($p < 0.05$). In each row, values marked by different capital letters are significantly different ($p < 0.05$), comparing immediate and longitudinal periods. *indicates no significant difference between groups with primer with and without in the same period ($p > 0.05$). Three-way ANOVA multiple comparisons by Tukey post hoc. PR: primer; AD: adhesive.

Table 3. Mode of failure distribution (in percent, %) for immediate and longitudinal microtensile bond strength specimens.

Groups	Mode of failure (%)							
	A/M		CD		CR		PF	
	immediate	longitudinal	immediate	longitudinal	immediate	longitudinal	immediate	longitudinal
PR 0% AD 0%	35 (76.09)	32 (71.11)	6 (13.04)	10 (22.22)	4 (8.70)	3 (6.66)	1 (2.17)	0 (0.00)
PR 0% AD 1%	28 (63.64)	30 (65.22)	6 (13.64)	11 (23.91)	10 (22.72)	5 (10.87)	0 (0.00)	0 (0.00)
PR 0% AD 2%	26 (55.32)	39 (68.42)	10 (21.28)	14 (24.56)	10 (21.28)	4 (7.02)	1 (2.13)	0 (0.00)
PR 0% AD 5%	25 (67.57)	34 (69.39)	11 (29.73)	14 (28.57)	1 (2.70)	1 (2.04)	0 (0.00)	0 (0.00)
PR 0% AD 10%	21 (52.50)	37 (71.15)	12 (30.00)	13 (25.00)	5 (12.50)	2 (3.85)	2 (5.00)	0 (0.00)
PR 2% AD 0%	27 (75.00)	34 (72.34)	7 (19.44)	11 (23.40)	2 (5.55)	2 (4.25)	0 (0.00)	0 (0.00)
PR 2% AD 1%	25 (65.79)	24 (60.00)	6 (15.79)	13 (32.50)	5 (13.16)	3 (7.50)	2 (5.26)	0 (0.00)
PR 2% AD 2%	29 (67.44)	21 (58.33)	12 (27.91)	12 (33.33)	2 (4.65)	3 (8.33)	0 (0.00)	0 (0.00)
PR 2% AD 5%	29 (69.05)	23 (60.53)	11 (26.11)	10 (26.32)	2 (4.76)	5 (13.16)	0 (0.00)	0 (0.00)
PR 2% AD 10%	23 (57.50)	32 (71.11)	8 (20.00)	6 (13.33)	5 (12.50)	7 (15.55)	4 (10.00)	0 (0.00)

PR: primer; AD: adhesive; A/M: adhesive/mixed fracture mode; CD: cohesive fracture mode in dentin; CR: cohesive fracture mode in resin; PF: premature failures.

Networks with high cross-linking densities experience low hydrolytic degradation due to the availability of fewer ester groups for attack and the reduced free volume, which prevents water sorption.[10] The adhesive with NCs increased the polymeric cross-linking density.[9,11,12,13,14] The increased conversion is due to the chemical affinity between the methacrylate groups of the adhesive and the polymeric capsule. The *in situ* analysis corresponds to the most challenging conditions that exist inside the hybrid layer[12,15,16] and can predict the adhesive performance *in vivo*[17] due to a positive correlation with the μTBS.[15] Therefore, the results of the *in situ* DC in the present study suggest that it is possible to predict the low hydrolytic degradation and reliable μTBS. The incorporation of NCs in only the adhesive also resulted in no alteration of the contact angle. Low contact angles indicate an equilibrium between the phases, leading to a high bond strength,[17,22] which occurred when NCs were only in the adhesive at a concentration of up to 5%.

Although the adhesive showed a reliable DC and μTBS, the use of a primer with nanocapsules and adhesive that also contained nanocapsules could likely cause more extensive hydrolytic degradation, promoting a reduction in the bond strength. This can be explained by the contact angle, since in these groups, it represents a lower penetration of the adhesive into the tissues, making it more susceptible to degradation. The primer and adhesive containing the NCs exhibited higher contact angles due to the interference of the high content of massive organic particles at the interface between the two media. The NCs in the adhesive system resulted in a high interfacial energy because of the low surface free energy of the primed dentin using NCs in the primer and the high liquid surface free energy of the adhesive with NCs. The altered contact angle results in low infiltration of the adhesive into the dentin[21], which in turn results in more extensive degradation of the exposed collagen fibrils and, consequently, a lower longitudinal bond strength.

The measurement of the immediate and long-term bond strengths can predict the long-term clinical performance.[23,24] Incorporating NCs into the primer and adhesive did not compromise the immediate bond strength, as has been observed in previous studies,[7,9] indicating the reliable distribution of NCs in the hybrid layer.[6,9] Previous studies that tested commercial and experimental adhesives showed similar bond strength results.[25,26]

Storage in water satisfactorily mimics clinical restoration degradation[23,27] since it infiltrates, promotes the swelling of and reduces the forces between polymer chains,[10] causing a decrease in the mechanical properties, the elution of monomers and a consequent

weakening of the bond.[10,23,27] The introduction of up to 5% NCs in the adhesive had no influence on the longitudinal bond strength. A decrease in the bond strength of approximately 17% can be expected for the other three-step systems; however, this decrease was not significant.[25,26] The increased strength of the hydrolytic degradation is due to the final, separate and more hydrophobic resin layer.[26] The high content of NCs in both the primer and adhesive decreased the longitudinal bond strength, possibly due to the high water uptake by the outer hydrophilic portion of the polymeric capsule of the NCs.[27,28] Moreover, the altered thermodynamic equilibrium between the adhesive and dentin when using both primer and adhesive containing NCs (expressed by the higher contact angles) may have influenced the longitudinal bond strength. The lower surface energy and wettability of the dentin surface cause lower penetration of the adhesive, resulting in a higher portion of non-protected dentin whose collagen fibrils are susceptible to enzymatic degradation as well as porosity in the hybrid layer, leading to more extensive hydrolytic degradation.[27,28] Therefore, the incorporation of NCs in only the adhesive showed better adhesion performance.

The results of the failure analysis of the μTBS are similar to the *in vivo* failure patterns, showing the relation between the collagen fibrils in the demineralized, non-protected dentin and the degradation of the polymeric matrix of the adhesive.[23,27] The distribution of the fracture pattern in the present study was very similar to that in previous results.[7,15] The stress concentration at failure mainly started at the interface for all groups, indicating the degradation of the collagen fibrils and the polymeric matrix in the hybrid layer.

Conclusions

Based on the results of the present study, it is possible to conclude that adhesives containing up to 5% nanocapsules and a primer without NCs had no influence on the *in situ* degree of conversion, contact angle, and immediate and long-term bond strengths. Therefore, the NC-loaded adhesives can be an alternative method for combining the bond performance and therapeutic effects.

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References

1. Mora-Huertas CE, Fessi H, Elaissari A. Polymer-based nanocapsules for drug delivery. Int J Pharm. 2010 Jan;385(1-2):113-42. https://doi.org/10.1016/j.ijpharm.2009.10.018
2. Groo AC, Bossiere M, Trichard L, Legras P, Benoit JP, Lagarce F. In vivo evaluation of paclitaxel-loaded lipid nanocapsules after intravenous and oral administration on resistant tumor. Nanomedicine (Lond). 2015 Mar;10(4):589-601. https://doi.org/10.2217/nnm.14.124
3. Bernardi A, Zilberstein AC, Jäger E, Campos MM, Morrone FB, Calixto JB et al. Effects of indomethacin-loaded nanocapsules in experimental models of inflammation in rats. Br J Pharmacol. 2009 Oct;158(4):1104-11. https://doi.org/10.1111/j.1476-5381.2009.00244.x
4. Paese K, Jäger A, Poletto FS, Pinto EF, Rossi-Bergmann B, Pohlmann AR et al. Semisolid formulation containing a nanoencapsulated sunscreen: effectiveness, in vitro photostability and immune response. J Biomed Nanotechnol. 2009 Jun;5(3):240-6. https://doi.org/10.1166/jbn.2009.1028
5. Bernardi A, Frozza RL, Meneghetti A, Hoppe JB, Battastini AM, Pohlmann AR et al. Indomethacin-loaded lipid-core nanocapsules reduce the damage triggered by Aβ1-42 in Alzheimer's disease models. Int J Nanomedicine. 2012;7:4927-42. https://doi.org/10.2147/IJN.S35333
6. Priyadarshini BM, Selvan ST, Lu TB, Xie H, Neo J, Fawzy AS. Chlorhexidine nanocapsule drug delivery approach to the resin-dentin interface. J Dent Res. 2016 Aug;95(9):1065-72. https://doi.org/10.1177/0022034516656135
7. Genari B, Leitune VC, Jornada DS, Camassola M, Pohlmann AR, Guterres SS et al. Effect of indomethacin-loaded nanocapsules incorporation in a dentin adhesive resin. Clin Oral Investig. 2017 Jan;21(1):437-46. https://doi.org/10.1007/s00784-016-1810-7

8. Genari B, Leitune VC, Jornada DS, Camassola M, Arthur RA, Pohlmann AR et al. Antimicrobial effect and physicochemical properties of an adhesive system containing nanocapsules. Dent Mater. 2017 Jun;33(6):735-42. https://doi.org/10.1016/j.dental.2017.04.001
9. Ouyang X, Huang X, Pan Q, Zuo C, Huang C, Yang X et al. Synthesis and characterization of triethylene glycol dimethacrylate nanocapsules used in a self-healing bonding resin. J Dent. 2011 Dec;39(12):825-33. https://doi.org/10.1016/j.jdent.2011.09.001
10. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater. 2006 Mar;22(3):211-22. https://doi.org/10.1016/j.dental.2005.05.005
11. Sadek FT, Calheiros FC, Cardoso PE, Kawano Y, Tay F, Ferrari M. Early and 24-hour bond strength and degree of conversion of etch-and-rinse and self-etch adhesives. Am J Dent. 2008 Feb;21(1):30-4.
12. Navarra CO, Cadenaro M, Armstrong SR, Jessop J, Antoniolli F, Sergo V et al. Degree of conversion of Filtek Silorane Adhesive System and Clearfil SE Bond within the hybrid and adhesive layer: an in situ Raman analysis. Dent Mater. 2009 Sep;25(9):1178-85. https://doi.org/10.1016/j.dental.2009.05.009
13. Leitune VC, Collares FM, Trommer RM, Andrioli DG, Bergmann CP, Samuel SM. The addition of nanostructured hydroxyapatite to an experimental adhesive resin. J Dent. 2013 Apr;41(4):321-7. https://doi.org/10.1016/j.jdent.2013.01.001
14. Collares FM, Portella FF, Leitune VC, Samuel SM. Discrepancies in degree of conversion measurements by FTIR. Braz Oral Res. 2014;28:9-15.
15. Hass V, Dobrovolski M, Zander-Grande C, Martins GC, Gordillo LA, Rodrigues Accorinte ML et al. Correlation between degree of conversion, resin-dentin bond strength and nanoleakage of simplified etch-and-rinse adhesives. Dent Mater. 2013 Sep;29(9):921-8. https://doi.org/10.1016/j.dental.2013.05.001
16. Degrazia FW, Leitune VC, Garcia IM, Arthur RA, Samuel SM, Collares FM. Effect of silver nanoparticles on the physicochemical and antimicrobial properties of an orthodontic adhesive. J Appl Oral Sci. 2016 Jul-Aug;24(4):404-10. https://doi.org/10.1590/1678-775720160154
17. Navarra CO, Cadenaro M, Frassetto A, Fontanive L, Di Lenarda R, Breschi L. Degree of conversion of self-etch adhesives: in situ micro-raman analysis. Oper Dent. 2016 Sep-Oct;41(5):501-10. https://doi.org/10.2341/15-165-L
18. Wege HA, Aguilar JA, Rodríguez-Valverde MA, Toledano M, Osorio R, Cabrerizo-Vílchez MA. Dynamic contact angle and spreading rate measurements for the characterization of the effect of dentin surface treatments. J Colloid Interface Sci. 2003 Jul;263(1):162-9. https://doi.org/10.1016/S0021-9797(03)00114-0
19. Aguilar-Mendoza JA, Rosales-Leal JI, Rodríguez-Valverde MA, González-López S, Cabrerizo-Vílchez MA. Wettability and bonding of self-etching dental adhesives. Influence of the smear layer. Dent Mater. 2008 Jul;24(7):994-1000. https://doi.org/10.1016/j.dental.2007.11.013PMID:18295326
20. Tani C, Manabe A, Itoh K, Hisamitsu H, Wakumoto S. Contact angle of dentin bonding agents on the dentin surface. Dent Mater J. 1996 Jun;15(1):39-44. https://doi.org/10.4012/dmj.15.39 PMID:8940537
21. Owens DK, Wendt RC. Estimation of the surface free energy of polymer. J Appl Polym Sci. 1969;13:1741:7.
22. Rosales-Leal JI, Osorio R, Holgado-Terriza JA, Cabrerizo-Vílchez MA, Toledano M. Dentin wetting by four adhesive systems. Dent Mater. 2001 Nov;17(6):526-32. https://doi.org/10.1016/S0109-5641(01)00014-8
23. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res. 2005 Feb;84(2):118-32. https://doi.org/10.1177/154405910508400204
24. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A et al. Relationship between bond-strength tests and clinical outcomes. Dent Mater. 2010 Feb;26(2):e100-21. https://doi.org/10.1016/j.dental.2009.11.148
25. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K et al. Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res. 2003 Feb;82(2):136-40. https://doi.org/10.1177/154405910308200212
26. De Munck J, Mine A, Poitevin A, Van Ende A, Cardoso MV, Van Landuyt KL et al. Meta-analytical review of parameters involved in dentin bonding. J Dent Res. 2012 Apr;91(4):351-7. https://doi.org/10.1177/0022034511431251
27. Hashimoto M, Ohno H, Sano H, Kaga M, Oguchi H. In vitro degradation of resin-dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy. Biomaterials. 2003 Sep;24(21):3795-803. https://doi.org/10.1016/S0142-9612(03)00262-X
28. Prathap M, Dhachinamoorthi D, Silambarasi T, Parthiban S, Vikneswari A, Karthick K. Effect of hydrophilic and hydrophobic polymers on metformin hydrochloride sustained release matrix tablets. International Journal of Biological & Pharmaceutical Research. 2012;3:266-74.
29. Almeida LF, Souza SE, Sampaio AA, Cavalcanti YW, Silva WJ, Del Bel Cur AA et al. Effect of collagen matrix saturation on the surface free energy of dentin using different agents. J Contemp Dent Pract. 2015 Jul;16(7):531-6. https://doi.org/10.5005/jp-journals-10024-1717