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Influence of the final irrigation solution on the push-out bond strength of calcium silicate-based, epoxy resin-based and silicone-based endodontic sealers

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Abstract

The aim was to evaluate the influence of different irrigation solutions on the push-out bond strength (POBS) of three different sealers (AH Plus, BioRoot RCS, GuttaFlow2). Root canals of 180 single-rooted human teeth were instrumented with F360 up to size 45.04. All canals were irrigated with 5 ml NaOCl 3% and 5 ml EDTA 17%. The canals were finally irrigated with either 5 ml NaOCl 3%, CHX 2%, EDTA 17%, citric acid 20% or NaCl 0.9% ($n = 36$) with a contact time of 5 min and obturated using matching gutta-percha cones according to the single-cone technique in combination with one of the sealers ($n = 12$). After 8 weeks of incubation, the roots were embedded in resin. Two slices of 1 mm thickness were obtained representing the middle third of the root. Dislodgement resistance was measured and POBS was calculated. Specimens were examined under 4× magnification to determine the mode of bond failure. Statistical analysis was performed using two-way ANOVA and Student–Newman–Keuls test for POBS and Chi-square test for the mode of failure. POBS was significantly affected by the factor "sealer" ($P < 0.001$) and by the interaction "sealer/irrigation solution" ($P < 0.01$). AH Plus revealed significantly higher POBS than BioRoot RCS and GuttaFlow2 ($P < 0.05$). The POBS of GutttaFlow2 was not affected by the irrigation protocol ($P > 0.05$). The POBS of AH Plus was positively influenced by EDTA and NaOCl. EDTA had a negative effect on the POBS of BioRoot RCS. The POBS of GuttaFlow2 was not influenced by the irrigation solutions.

Keywords AH Plus · BioRoot RCS · GuttaFlow2 · Irrigation solutions · Push-out bond strength

Introduction

Three-dimensional obturation of the root canal system is along with instrumentation and disinfection one crucial step of a successful root canal treatment. The sealer's function is to establish a bacteria and fluid-tight seal at the sealer–dentin and sealer–core interfaces and to provide resistance against dislocation of the obturation when it comes to deformation of the root dentin under occlusal loads.

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AH Plus (Dentsply, Konstanz, Germany) is an epoxy resin-based sealer and well investigated [1]. Chemical bonding to the amino groups of dentinal collagen [2, 3] and high resistance against dislodgement were reported [3, 4].

BioRoot RCS (Septodont, St. Maur-des-Fossés, France) is a calcium silicate-based sealer (Table 1) with a good biocompatibility [5]. The release of calcium hydroxide after hydration and the contact with phosphate from tissue fluids leads to precipitation of calcium phosphate or calcium carbonate on the surface [6, 7]. The formation of hydroxyapatite on BioRoot RCS after contact with phosphate-buffered saline solution was reported [7]. Calcium silicates form an interfacial layer at the dentin called the "mineral infiltration zone" with increased mineralization [8, 9].

GuttaFlow2 is a silicon-based sealer (Coltène/Whaledent, Langenau, Germany; Table 1) and was introduced in 2012 to replace GuttaFlow [10]. Both displayed good biocompatibility [11]. Data about the sealer–dentin interaction are sparse. Regarding the pure silicone sealer RoekoSeal (Coltène/Whaledent) and GuttaFlow, inconsistent findings

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Table 1 Composition of the sealers used

AH Plus	*Epoxide paste* Diepoxide, calcium tungstate, zirconium oxide, aerosil, pigment	*Amine paste* 1-Adamantane amine, *N,N′*-dibenzyl-5-oxa-nonandiamine-1,9, TCD-Diamine, calcium tungstate, zirconium oxide, aerosil, silicone oil
BioRoot RCS	*Powder* Tricalcium silicate, zirconium oxide, povidone	*Liquid* Aqueous solution of calcium chloride with polycarboxylate
GuttaFlow2	Gutta-percha powder, polydimethylsiloxane, platinum catalyst, zirconium dioxide, micro-silver particles, coloring	

of the sealing ability [12, 13] and a lack of chemical interaction between RoekoSeal and the dentinal collagen were reported [3].

Most study designs only consider instrumentation, irrigation or obturation, which are obviously strongly associated with each other. However, the combination of different irrigation protocols and obturation materials is of clinical relevance [14]. Push-out bond strength (POBS) is a relevant prognostic factor to evaluate the link of a root canal sealer to the canal wall and the core material [15].

The aim of this study was to evaluate the POBS and the mode of failure of AH Plus, BioRoot RCS, and GuttaFlow2 after using different irrigation solutions. The null hypotheses were as follows: there are no differences between the sealers after the use of different irrigation solutions regarding (1) POBS and (2) the mode of failure.

Materials and methods

Based on data of a previous study [4] power calculation using G*Power 3.1 (Heinrich Heine University, Düsseldorf, Germany) indicated that the sample size for each group should be at least 11. Thus, 12 canals were used for each experimental group.180 human single-rooted mandibular premolars with only one straight (curvature < 5°) root canal were included. This was checked by viewing their buccal and proximal radiographs in an imaging software (ImageJ, NIH, MD, USA) as described previously [16]. To exclude cracks, all roots were observed with a stereomicroscope under 20× magnification (Expert DN, Müller Optronic, Erfurt, Germany). The working length was obtained by measuring the length of the initial instrument [size 10 C-Pilot file (VDW, Munich, Germany)] at the major apical foramen minus 1 mm. All teeth were cut in a way that a working length of 18 mm was established. Patency of the canal was determined with K-files ISO 10 (VDW). Only teeth whose canal width near the terminus was approximately compatible with ISO 15 were included. This was checked with silver points of sizes 10 and 15 (VDW). All root canals were instrumented with NiTi F360 files (Komet, Lemgo, Germany) up to size 45.04 using the file sequence 25.04, 35.04 and 45.04 in the torque-limited electric motor VDW.

Gold (VDW) according to the manufacturer's instructions (300 rpm, torque 1.8 Ncm). After three pecking motions, the root canal was irrigated with 2.5 ml NaOCl 3%.

The specimens were randomly divided into 15 groups (*n* = 12) (Table 2).

The final irrigation protocol was 5 ml NaOCl 3% and 5 ml EDTA 17% (contact time 5 min) using a 30-g open-ended needle (NaviTip, Ultradent, South Jordan, USA). Thereafter, the canals were irrigated with either 5 ml NaOCl, 5 ml CHX, 5 ml EDTA, 5 ml citric acid or 5 ml NaCl according to the groups (contact time 5 min). Finally, the canals were dried with paper points.

All canals were obturated using F360 gutta-percha cones of size 45.04 (Komet) according to the single-cone technique. The sealers were mixed according to the manufacturers' instructions and applied into the canal using gutta-percha cones. A heated plugger was used to remove the coronal excess gutta-percha. The teeth were radiographed in buccal and proximal view to verify correct obturation. The canal orifices were sealed with Cavit G (3M ESPE, Seefeld, Germany). The specimens were placed in an incubator

Table 2 Irrigation protocols

Group	Sealer	Irrigation solution
A1	AH Plus	NaOCl 3%
A2		CHX 2%
A3		EDTA 17%
A4		Citric acid 20%
A5		NaCl 0.9%
B1	BioRoot RCS	NaOCl 3%
B2		CHX 2%
B3		EDTA 17%
B4		Citric acid 20%
B5		NaCl 0.9%
G1	GuttaFlow2	NaOCl 3%
G2		CHX 2%
G3		EDTA 17%
G4		Citric acid 20%
G5		NaCl 0.9%

NaOCl sodium hypochlorite, *CHX* chlorhexidine, *EDTA* ethylenediaminetetraacetic acid, *NaCl* sodium chloride

(Heraeus, Hanau, Germany) at 37 °C and 100% humidity for 2 months. Complete setting of all sealers under these conditions was verified in a pilot study. All treatment procedures were carried out by the same operator who was proficient in the techniques used.

The roots were embedded into acrylic resin vertically (Technovit 4071, Heraeus) and sectioned horizontally with a 0.25-mm-low-speed saw (Leitz, Wetzlar, Germany) beginning with a distance of 7 mm from the apex under permanent water cooling. Two slices of 1 mm thickness were obtained representing the middle third of the root.

The specimens were placed in a metallic jig with a hole underneath. A standard size plunger with a tip of 0.6 mm diameter was used to apply the vertical load onto the gutta-percha core of the filling. The diameter of the plunger tip was dimensioned according to the gutta-percha point diameter at 6 mm from the tip to ensure an equal distribution of the load on about 75–85% of the gutta-percha cone diameter without touching the sealer phase of the root canal filling. The vertical load in an apical to coronal direction was generated by a universal testing machine (Lloyd LF Plus/Nexygen, Ametek, Berwyn, USA) at a speed of 1 mm/min. The failure load was recorded in Newton when an abrupt reduction of the load was measured.

The lateral surface of the root canal of each specimen was calculated by the truncated cone formula $M = (R + r) \cdot \pi \cdot m$. The POBS of each specimen was then calculated and expressed in N/mm^2 (equivalent to MPa).

Photographs of each specimen were taken with a laser microscope (VK-X100, Keyence, Osaka, Japan) under 4× magnification. The mode of failure was evaluated by two blinded operators in three categories: adhesive failure (no material left on canal wall), cohesive failure (material present on entire canal wall) and mixed failure (material in patches on canal wall) (Fig. 1). If disagreement existed, a joint meeting of all authors was made until a consensus was reached. A joint meeting was necessary in only four cases.

Statistical analysis of POBS values was performed using two-way ANOVA and the post hoc Student–Newman–Keuls test ($P < 0.05$) as data were distributed normally (Kolmogorov–Smirnov test). Statistical analysis of the mode of failure of each sealer was performed using Chi-square test.

Results

All specimens had measurable adhesion to the root dentin and no premature failure occurred. No significant difference occurred between the two section levels ($P < 0.05$). Thus, the data of the section levels were pooled per group.

Regarding the POBS, two-way ANOVA indicated that the results were significantly affected by the factor "sealer" ($P < 0.001$) and by the interaction "sealer/irrigation solution" ($P < 0.01$).

Irrespective of the irrigation protocol, AH Plus revealed significantly higher POBS than the other sealers ($P < 0.05$). Groups A1 and A3 showed higher POBS values than group A4, while groups A2 and A5 displayed the significantly lowest POBS values of the AH Plus groups ($P < 0.05$; Table 3).

Regarding BioRoot RCS, the groups B2, B4 and B5 reached significantly higher POBS values than the groups G1–G5 ($P < 0.05$). Group B3 did not show significant differences compared to the groups G1–G5 ($P > 0.05$). Among the groups B1–B5, significant differences only existed between the groups B2 and B3, while EDTA caused lower POBS values than CHX ($P < 0.05$; Table 3).

Regarding GuttaFlow2, all groups showed significantly lower POBS values than the groups A1–A5, B1, B2, B4 and B5 ($P < 0.05$) without significant differences between the groups G1–G5 ($P > 0.05$; Table 3).

AH Plus predominantly displayed cohesive failure mode irrespective of the irrigation protocol. Chi-square test did not reveal significant differences between the groups A1–A5 ($P > 0.05$) but significant differences between the groups
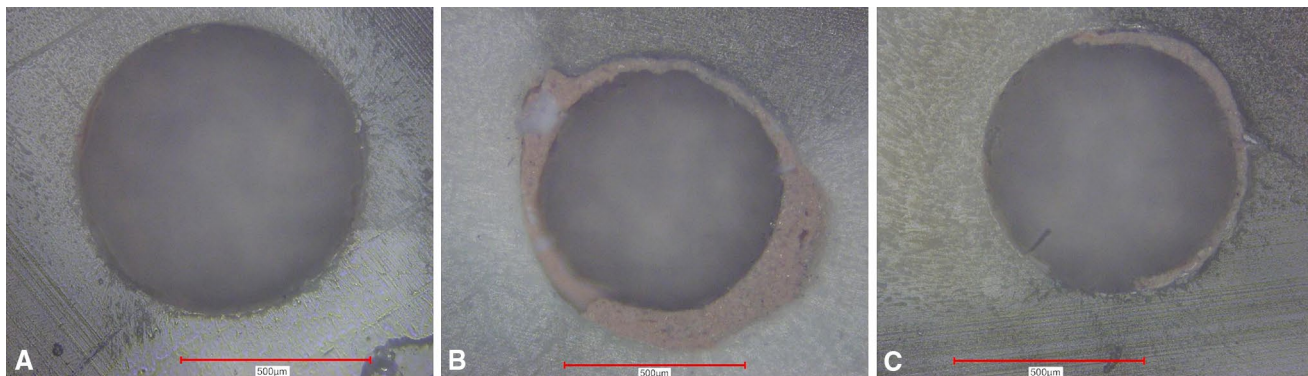

Fig. 1 Images obtained by laser microscopy at 4× magnification for analysis of the mode of failure; examples for adhesive (**a**), cohesive (**b**) and mixed (**c**) failure types are given

Table 3 Means and standard deviations of push-out bond strength of all groups (in N/mm^2)

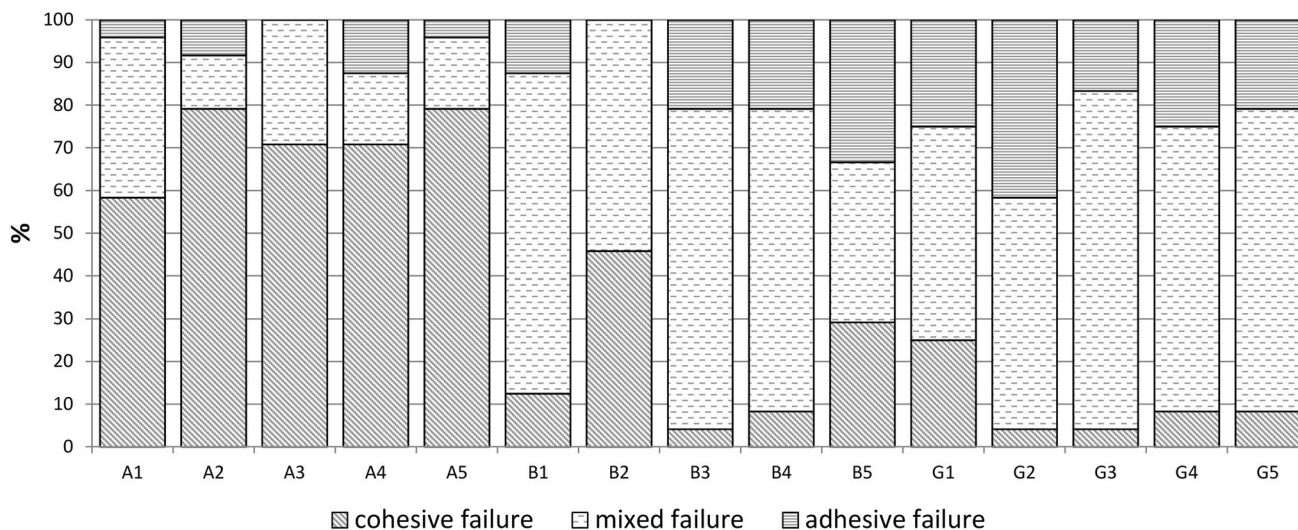| Groups	
AH Plus	
A1 (NaOCl)	9.44 ± 2.92[a]
A2 (CHX)	5.77 ± 1.89[c]
A3 (EDTA)	8.78 ± 2.74[a]
A4 (citric acid)	7.81 ± 2.01[b]
A5 (NaCl)	6.50 ± 2.04[c]
BioRoot RCS	
B1 (NaOCl)	3.75 ± 0.85[d,e,f]
B2 (CHX)	3.94 ± 1.61[d]
B3 (EDTA)	2.67 ± 0.95[e,f]
B4 (citric acid)	3.29 ± 1.08[d,e,f]
B5 (NaCl)	3.42 ± 1.39[d,e,f]
GuttaFlow2	
G1 (NaOCl)	1.88 ± 0.63[f,g]
G2 (CHX)	1.44 ± 0.68[f,g]
G3 (EDTA)	1.38 ± 0.57[f,g]
G4 (citric acid)	1.89 ± 0.88[f,g]
G5 (NaCl)	1.62 ± 0.52[f,g]

Values with different superscript letters were statistically different at $P = 0.05$ (ANOVA and Student–Newman–Keuls test)

B1–B5 ($P < 0.05$). Groups B1, B3 and B4 mainly displayed mixed failure mode whereas the group B2 displayed mixed and cohesive failure mode in nearly equal halves. Group B5 showed adhesive, mixed and cohesive failure mode in nearly equal thirds. Chi-square test revealed no significant differences between the groups G1–G5 ($P > 0.05$) displaying predominantly mixed and adhesive failures (Fig. 2).

Discussion

Push-out tests have been widely used to evaluate the dislodgement resistance of root canal filling materials [14, 17]. After the final irrigation with NaOCl or EDTA, AH Plus displayed the highest POBS values whilst BioRoot RCS displayed the lowest POBS after EDTA. GuttaFlow2 was not affected by irrigation solutions. Thus, the null hypotheses (i) and (ii) were rejected.

When evaluating POBS values, the bond of the sealer either to the root canal wall or to the core material is assessed [18]. The most crucial step of the experimental setup is the ratio of the pin diameter and the specimen's diameter. A ratio of less than 0.6 [15, 18] and ratios higher than 0.85 have been reported to influence the POBS test [18]. In the present study, the pin diameter was designed to be within a range 75–85% of the gutta-percha cone diameter. Moreover, different experimental protocols that were established in the past concerning root canal preparation (diameter and taper), root canal obturation (cold versus warm obturation techniques), the tooth type and portion, slice thickness, load velocity, and other parameters can be an explanation for the variability in results [14, 17, 19]. In the present study, a reproducible instrumentation was sought by a preparation up to size 45 with a continuous taper of 0.04. Regarding obturation techniques, lateral condensation and warm vertical compaction may exert a certain impact on the POBS [20, 21] and are less reproducible than the single-cone technique. Therefore, the single-cone obturation using matching gutta-percha cones was performed in the present study. Recently, a standardization of the push-out test was demanded to investigate special

Fig. 2 Distribution of failure modes in all groups (in %)

questions regarding the sealer–dentin interface [14]. In some studies, the root canal filling was established without the use of a core material such as gutta-percha [22, 23] to eliminate a possible confounding factor. In the present study, three different types of sealers were investigated and, therefore, the use of a core material is to be regarded as a constant. The different sealer types are poorly comparable due to their differing hardness when used without a standard core material.

Predominantly superior POBS of AH Plus compared to other sealer types have been reported [3, 19, 23–28]. In accordance with the present findings, superior POBS of AH Plus compared to BioRoot RCS was reported [4]. To the best of our knowledge, no other study assessing the POBS of BioRoot RCS is yet available. Superior POBS of AH Plus compared to silicone sealers has been reported [3, 25]. No data comparing the POBS of AH Plus and GuttaFlow2 can be provided.

An impact of the final irrigation protocol on the POBS has been reported for AH Plus [29]. The removal of the smear layer using EDTA after the use of NaOCl enforced the POBS of AH Plus compared to other irrigation protocols [30, 31]. The highest POBS was found when NaOCl was used as final irrigant after the use of EDTA, compared to other irrigant combinations [32]. The sole use of NaOCl did lead to lower POBS whereas higher POBS after the sole use of EDTA or the combination of EDTA and NaOCl was found [33]. These studies corroborate the present findings. Irrespective of the irrigation protocol, AH Plus predominantly displayed cohesive failure mode which is in accordance with previous studies [4, 34, 35] and this finding provides further evidence for the relatively strong bond of AH Plus to root dentin.

There is only one study regarding the POBS of BioRoot RCS showing inferior POBS compared to AH Plus [4], which corroborates the present findings. There is no information about the influence of irrigation solutions on the POBS of BioRoot RCS. In the present study, the use of the chelating agent EDTA as a final irrigant significantly reduced the POBS of BioRoot RCS. Contradictory results were obtained in another study in as far as different chelating agents had no effect on the POBS of the calcium silicate-based sealer Total Fill BC (FKG, La Chaux-des-Fonds, Switzerland) [23]. However, as no control group was included in the latter study [23], interpretation of the results is limited. BioRoot RCS predominantly displayed mixed failure mode after the use of chelating agents or NaOCl in the present study, which is in accordance with a previous report [4]. The use of final irrigation solutions that are unable to modify the root dentin surface, such as CHX and NaCl, leads to a higher proportion of cohesive failures.

Data concerning GuttaFlow2 are sparse as no study evaluating the POBS of this sealer is yet available. The results of one study assessing the POBS of GuttaFlow in comparison to AH Plus are in accordance with the present findings [25]. Regarding RoekoSeal, inferior POBS compared to AH Plus and a lack of chemical interaction with root canal dentin have been reported [3]. Thus, it can be postulated that there seems to be no chemical and only minor mechanical interaction between silicone sealers and root canal dentin. The higher proportion of adhesive failures in the present study correlates with the low POBS of GuttaFlow2.

Obviously, AH Plus has high resistance to dislodgement and the use of chelating agents and NaOCl has a positive impact on the POBS of AH Plus. The complete exposure of the amino groups of the dentinal collagen due to the removal of the smear layer and debris may increase the number of covalent bonds between the epoxy resin and amino groups resulting in a stronger link of AH Plus to root canal dentin. The long-term substantivity of CHX [36] might hinder the access of AH Plus to the dentinal collagen and thus lead to a lower POBS.

EDTA influenced the POBS of BioRoot RCS negatively. The reduction of calcium at the sealer–dentin interface or a degradation of the calcium silicate fraction in the sealer might hinder the formation of the "mineral infiltration zone" postulated by Atmeh et al. [8]. This may result in a weaker interaction between the root canal wall and the sealer.

Conclusion

AH Plus showed higher POBS than BioRoot RCS and GuttaFlow2. The POBS of AH Plus was positively influenced by EDTA and NaOCl. EDTA had a negative effect on the POBS of BioRoot RCS. The POBS of GuttaFlow2 was not influenced by the irrigation solutions.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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