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Physical properties of different root canal sealers

Физичка својства различитих паста за пуњење канала корена

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SUMMARY

Introduction/Objective was to evaluate the push-out bond strength to root canal dentine and radiopacity of three different sealers: Adseal (Meta Biomed, South Korea), CeraSeal (Meta Biomed, South Korea) and control AH Plus (Dentsply, Germany).

Methods In nine dentin discs, 1 mm thickness, three holes, 1.2 mm diameter, were drilled in with a fissure carbide bur. Discs were immersed in 0.5% NaOCl and 10% citric acid respectively, for 60 seconds rinsed and dried. Every hole was filled with different sealer. Specimens were wrapped in gauze previously immersed in Hank's balanced salt solution at 37°C / seven days. The push-out test was performed using universal testing machine at a cross-head speed of 1mm/min. The radiopacity was tested (ISO 6876/2012 standard). Three sealer specimens, 5 mm in diameter and 2 mm thick were prepared and radiographed using radiovisiography system (CCD sensor, Trophy, France) with graded aluminum step-wedge. Gray-scale value was assessed using Adobe Photoshop CS7 (San Jose, USA).

Results Mean values of push-out bond strength were 5.21 ± 0.87 MPa (Adseal), 0.06 ± 0.02 MPa (CeraSeal), and 3.13 ± 0.38 MPa (AH Plus). A statistically significant difference in push-out bond strength was observed among all three sealer groups ($p < 0.05$). Adseal exhibited the strongest bond strength to root canal dentine. All sealers achieved radiopacity over 3 mm, with statistically significant difference among the groups ($p < 0.05$).

Conclusion The epoxy-based sealer Adseal showed higher bond strength compared to AH Plus and calcium silicate-based sealer CeraSeal, which, expectedly, showed the weakest dislocation resistance. All three sealers fulfilled the ISO standard to be distinguished on dental radiogram.

Keywords: root canal sealer; radiopacity; push out; calcium silicate

САЖЕТАК

Увод/Циљ Циљ је био да се да се испита јачина везе и рендгенконтрастност три различите пасте за пуњење канала корена: Адсеал на бази епокси смоле (Мета Биомед, Јужна Кореја), ЦераСеал на бази калцијум силиката (Мета Биомед, Јужна Кореја) и контролни АХ Плус (Дентсплу, Немачка).

Методе На девет дискова дентина, дебљине 1 мм, препарисана су три кавитета, пречника 1,2 мм, карбидним сврдлом. Дискови су потопљени у 0,5% *NaOCl* / 10% лимунске киселине, 60 секунди, затим испрани физиолошким раствором и осушени. Сваки кавитет је испуњен различитом пастом. Узорци су умотани у газу натопљеном вештаком ткивном течношћу на 37°*C* / седам дана. Тест смицања је изведен на универзалној машини при брзини наставка од 1 мм/мин. Радиопацитет је тестиран у складу са (ИСО 6876). Три узорка заптивача, 5 × 2 мм су припремљена и радиографисана коришћењем радиовизиографског система (ЦЦД сензор, *Trophi*, Француска) са градуисаним алуминијумским еталоном. Вредност сиве скале је процењена коришћењем *Adobe Photoshop CS7* (Сан Хозе, САД).

Резултати Средње вредности јачине везе биле су 5,21 ± 0,87 МПа (Адсеал), 0,06 ± 0,02 МПа (ЦераСеал) и 3,13 ± 0,38 МПа (АХ Плус). Све пасте су показале статистички значајну разлику у јачини везе ($p < 0,05$). Све пасте су остварile рендгенконтрастност преко 3 мм, са статистички значајним разликама између узорака ($p > 0,05$).

Закључак Паста на бази епокси смоле, Адсеал показала је јачу везу у односу на АХ Плус и ЦераСеал, који је, очекивано, показао најслабију отпорност на дислокацију. Све три пасте су остварile ренгенконтрастност прописану ИСО стандардом.

Кључне речи: паста за пуњење канала; радиоконтрасност; јачина везе; калцијум силикат

INTRODUCTION

Adequate root canal obturation should effectively seal the root canal system, preventing apical and coronal leakage and enable long-term success of endodontic treatment. Root canal sealers should have appropriate physical properties to achieve three-dimensional sealing [1]. One of the most important characteristics of an endodontic sealer is its capacity to adhere to radicular

dentine. Adequate adhesion minimizes gap formation at the sealer–dentine interface, which could otherwise permit fluid percolation [2], and improves resistance to material displacement during functional loading or clinical procedures [3]. Likewise, materials that fill the canal space should have adequate mechanical properties that will strengthen the root canal and compensate for the reduced resistance caused by instrumentation [4].

Radiopacity is a physical property that enables radiographic visualization of the root canal filling aiding in the assessment of its quality. Furthermore, adequate radiopacity is necessary for distinction of the root canal filling material from surrounding dental and periapical tissues and for detection of voids in the root canal sealers or at the interface sealer/dentine or sealer/core material.

Epoxy resin-based sealers are known for their favorable physical properties, including extended working and setting times, low solubility, high flowability, minimal polymerization shrinkage, and excellent adaptation to dentinal walls. Their adhesion to dentine is attributed to the formation of covalent bonds between epoxide rings and exposed amino groups within the collagen network [5, 6].

Calcium silicate-based sealers are derived from mineral trioxide aggregate (MTA), which is known for its favorable clinical and biological outcomes. These sealers are biocompatible [7], form a biomimetic apatite layer when interacting with phosphate-containing simulated body fluids [8, 9], release calcium [10], and exhibit excellent compatibility with various core materials due to their sufficient flow and optimal handling characteristics [11].

CeraSeal (Meta Biomed Co., Cheongju, Korea) is a premixed calcium-silicate-based sealer, but unfortunately, there is very little information in literature on its properties and performance in vitro and in vivo.

Thus, the aim of this study was to evaluate the bond strength to root canal dentine and radiopacity of Adseal (Meta, Biomed, Cheongju, South Korea), CeraSeal (Meta, Biomed, Cheongju, South Korea) and AH Plus (Dentsply, DeTrey, Konstanz, Germany). The null hypothesis were: 1) three is no statistically significant difference in the bond strength to root canal dentine among the tested sealers and 2) there is no statistically significant difference in radiopacity among the tested sealers.

METHODS

1. Three different root canal sealers were used in this study (Table 1):

- Adseal (Meta, Biomed, Korea) epoxy resin-based,

- CeraSeal (Meta, Biomed, Korea) calcium silicate-based,

- AH Plus (DeTrey, Konstanz, Germany) epoxy resin-based used as a control group.

2. Nine maxillary third molars from humans, extracted for orthodontic purposes, were cleaned of debris and preserved in a 0.2% thymol solution at 4°C for no more than six months. The teeth were embedded in acrylic (Duracryl plus, Spofa dental, KavoKerr, CA, USA) using standardized silicone molds measuring 10 × 10 × 15 mm, up to the cemento-enamel junction. The crowns were then cut off at the cemento-enamel junction with a diamond saw operating at a speed of 0.7 mm under coolant, aligned perpendicular to the tooth's long axis. A 1 ± 0.1 mm disk was sectioned from the middle segment of each tooth. Each disk had three standardized cavities, each 1.2 mm in diameter, prepared using a 1.2 mm fissure carbide bur (Dentsply/Maillefer, Ballaigues, Switzerland) in a fixed handpiece to ensure uniform cavity

preparation. After this, the disks were immersed in three different solutions – 0.5% sodium hypochlorite, 10% citric acid, and saline—for 60 seconds each and then blotted dry.

The cavities in each disk were randomly assigned to different groups and filled with the respective sealers, which were mixed according to the manufacturer's instructions using a probe in a vibrating motion. Any excess material was carefully removed with a plastic instrument.

The specimens were wrapped in gauze that had been soaked in Hank's balanced salt solution and incubated at 37°C for seven days. This procedure ensured that each disk contained all three sealers.

To assess the push-out bond strength of each tested sealer to the root dentine, a universal testing machine (PCE-FM 200) was used. Each disk was placed between two supports, ensuring that the dislocation of the sealer was not obstructed. A custom-made cylindrical stainless-steel indenter, 0.8 mm in diameter, applied force to the sealer at a speed of 1 mm/min until the sealer dislodged from the root canal space. The bond strength (σ, in MPa) was calculated using a specific formula:

$$\sigma = \frac{F}{2r\pi h}$$

where F is the maximum load (N) measured at fracture, r is cavity radius (0.6 mm) and h is specimen height (1 mm).

3. The radiopacity was tested in accordance with the International Organization for Standardization (ISO 6876). The sealers were mixed following manufacturer's instructions and three specimens, 5 mm in diameter, 2 mm high, were made for each tested material.

After the setting period, the specimens were radiographed using a radiovisiography system (CCD sensor, Trophy, France) with an exposure time of 0.04 seconds, a voltage of 60 kV, and

an amperage of 10 mA. The distance from the source to the object was 35 cm. Each sealer specimen was radiographed alongside an aluminum step-wedge, which was graduated from 1 to 10 mm in 1-mm increments. The gray-scale values for each step of the aluminum step-wedge and the tested materials were measured using Adobe Photoshop CS7 (San Jose, USA). The correlation between the logarithm of the aluminum thickness and its corresponding gray-scale value was utilized to calculate the equivalent thickness of aluminum for each root canal sealer specimen examined. Data were compared using Welch's ANOVA with Games–Howell post-hoc test ($\alpha = 0.05$).

Ethics: This study was approved by the Ethics Committee School of dental medicine Belgrade University No 36/15.

RESULTS

Regarding the push-out bond strength of all tested sealers, Adseal demonstrated the highest mean push-out values (Mean = 5.21 ± 0.87), followed by AH Plus (Mean = 3.13 ± 0.38), whereas Ceraseal showed the lowest adhesion performance (Mean = 0.06 ± 0.02) (Figure 2.). When comparing the push-out bond strength across all groups (Adseal, AH Plus, and Ceraseal), Welch's ANOVA confirmed a statistically significant difference among the materials ($p < 0.001$). Comparing the AH Plus and Ceraseal, the difference between these two sealers was statistically significant ($p < 0.001$).

Subsequent Games–Howell post hoc analysis revealed that Adseal had significantly higher push-out bond strength than AH Plus ($p = 0.011$) and Ceraseal ($p = 0.001$), while AH Plus also exceeded Ceraseal ($p < 0.001$), Table 2.

Regarding radiopacity, Welch's ANOVA revealed a statistically significant difference in radiopacity among the tested sealers ($p < 0.001$). Games–Howell post hoc analysis showed that Adseal exhibited significantly higher radiopacity compared with AH Plus ($p = 0.009$) and Ceraseal ($p = 0.001$). AH Plus also demonstrated higher radiopacity than Ceraseal, and the difference was statistically significant ($p = 0.001$). These findings indicate that all three materials differ in radiopacity, with Adseal being the most radiopaque sealer, followed by AH Plus and Ceraseal (Table 2. Figure 3.).

All sealers showed a statistically significant difference in bond strength (< 0.05).

All three sealers achieved radiopacity over 3mm Al. There was statistically significant difference in the values radiopacity of the tested sealers ($p < 0.05$) (Figure 3).

DISCUSSION

Significant differences between tested sealers were found regarding the push-out bond strength. Therefore, the first null hypothesis was rejected.

Push-out bond test is a method commonly used to evaluate the interfacial bond strength between endodontic materials and root dentine. The advantage of using the standard push-out test is that multiple slices can be derived from a single root specimen [12]. On the other hand, these slices are obtained by preparing the natural root canal of the tooth which often leads to difficulty in creating a reliable baseline due to the intricate intracanal anatomy [13]. In this study a novel set-up model, introduced by Scelza et al. (2008) was used in order to increase the internal validity of the push-out test by forming artificial standard canal-like holes in dentine discs [14].

Resistance to dislocation of root canal sealers is conditioned by various factors such as type of sealer, presence/absence of smear layer, irrigating solutions, shape of root canals (C factor), as well as number and size of dentinal tubules [14, 15, 16,17].

In this study, dentinal discs were immersed in NaOCl solution and then in 10% citric acid solution with the aim of removing the smear layer. There is no uniform position in the literature on the influence of the smear layer on the push-out bond strength of the calcium silicate-based sealers to root canal dentine. The use of acids such as EDTA can adversely affect the formation of CSH gel which is being produced during the hydration process of calcium silicate-based sealers [18].

AH Plus sealer was used as a control material in this study. This sealer epoxy resin sealer is dimensionally stable in the long term, is insoluble and has low toxicity [19]. Compared to other sealers, it has a superior dislocation resistance to root canal dentin [20, 21] and is considered the "gold standard" in endodontics.

Our study showed that epoxy resin-based sealers Adseal and AH Plus demontrated higher bond strength values than calcium silicate-based CeraSeal. High resistance to dislocation can be explained by the chemical composition of these sealers, i.e., by forming covalent bonds between open epoxy paste rings and amino groups present in dentin collagen [22] as well as low polymerization contraction [23]. Also, cohesion between paste molecules increases resistance to paste dislocation resulting in better adhesion [24].

In this study, Adseal proved to have the highest dislocation resistance to root canal dentine. Lee et al. investigated physicochemical properties of epoxy resin-based and bioceramic-based root canal sealers. Flow, final setting time, radiopacity, dimensional stability, and pH change were examined according to modified ISO 6876/2012 standards and ANSI/ADA's specifications number 57. AdSeal showed bigger expansion rate than the favorable rate

suggested by the international standards, which may partially explain very high values of bond strength in our study. The authors in the mentioned study recommended further investigation of the potential risk of inducing the vertical root fractures by the sealer expansion [25, 26].

Beautlin and al. [27] found that CeraSeal demonstrated similar values of push-out bond strength values compared to AH Plus. This experimental set up model used gutta-percha which may explain the differing distribution of bond strength values. On the other hand, a similar paper used gutta-percha while assessing bond strength and showed that bond strength values of Ceraseal were significantly lower than AH Plus [28]. These findings may be due to different protocols of root canal drying techniques used in the study. Our results are similar to the findings of Maharti et al. [29] who established that CeraSeal had lower dislocation resistance compared to AH Plus in a comparable set up model.

Radiopacity is an important feature of a sealer it helps to differ the sealer in an obturated root canal from other anatomical features on a radiogram. This property is essential to determine if there was insufficient or inadequate root canal filling or a sealer leakage. Following the International Organization for standardization ISO standards tested sealers fulfilled the standard norm of over 3 mm of radiopacity. Radiopacity of all three sealers did differ in a significant manner $p < 0.05$.

These results are in concordance with the findings of Zamparini et al, where CeraSeal and AH Plus demonstrated radiopacity values above 8 mm Al [30].

CONCLUSION

The epoxy-based sealer Adseal showed higher bond strength compared to AH Plus and calcium silicate-based sealer CeraSeal, which, expectedly showed the weakest dislocation resistance. All three sealers achieved the ISO standard values, to be distinguished on the dental radiogram.

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Conflict of interest: None declared.

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Table 1. Manufacturer and composition of the tested sealers

Sealer	Manufacturer	Composition
Adseal	Meta Biomed Co., Cheongju-si, Chungcheongbuk-do, South Korea	Base -epoxy resin -NS calcium phosphate -NS zirconium dioxide -NS calcium oxide -NS ethylene glycol salicylate Catalyst -N, n-dibenzyl-5 oxanonandiamin-1,9 -amantadine
CeraSeal	Meta Biomed Co., Cheongju-si, Chungcheongbuk-do, South Korea	-Calcium silicates, -zirconium oxide, -thickening agent
AH Plus	Dentsply, Konstanz, Germany	Paste A -bisphenol-A, -bisphenol-F calcium tungstate, -zirconium oxide, -silica iron oxide pigments Paste B -dibenzyldiamineamino adamantane tricyclodecane-diaminecalcium tungstate, -zirconium oxide, -silica, -silicone oil

Table 2. Push out bond strength and radiopacity

Sealer type	Push out MPa	p	Radiopacity (mm)	p
Adseal	5.21 ± 0.87	0.011*	6.26 ± 0.57	0.009*
CeraSeal	0.06 ± 0.02	0.001*	3.70 ± 0.17	0.001*
AH Plus	3.13 ± 0.38	p < 0.001*	4.80 ± 0.11	0.001*

Statistical significance *

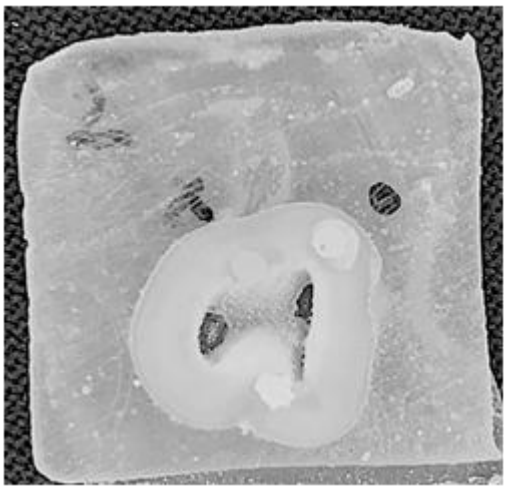

Figure 1. Dentine disk sample filled with tested sealers

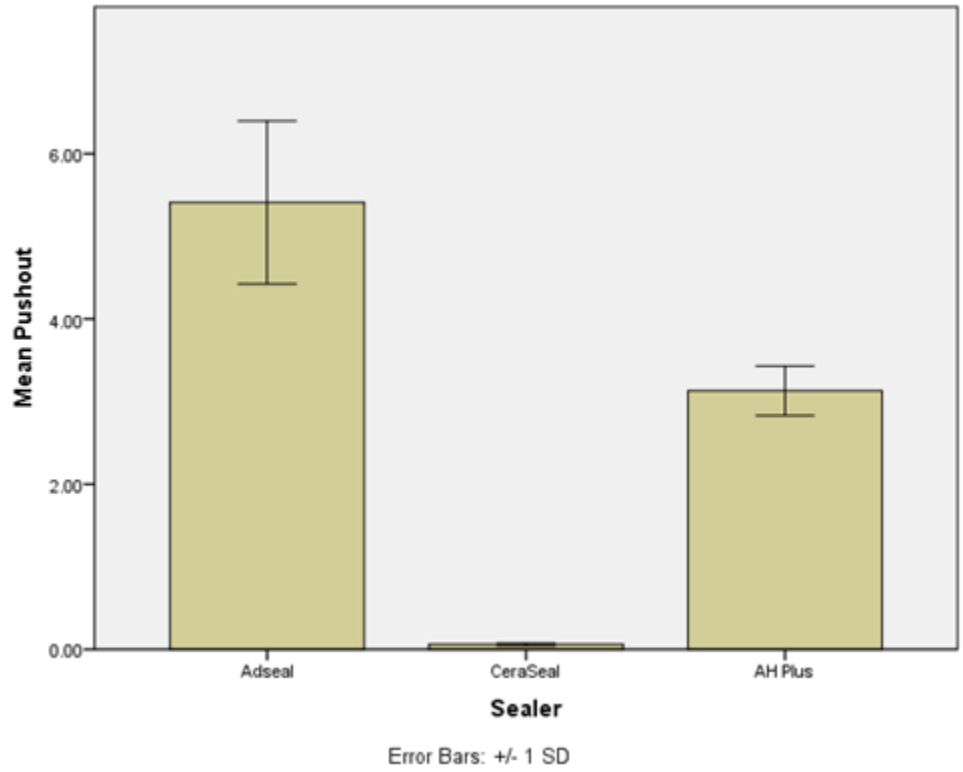

Figure 2. Push-out bond strength mean values (MPa) and standard deviation (SD)

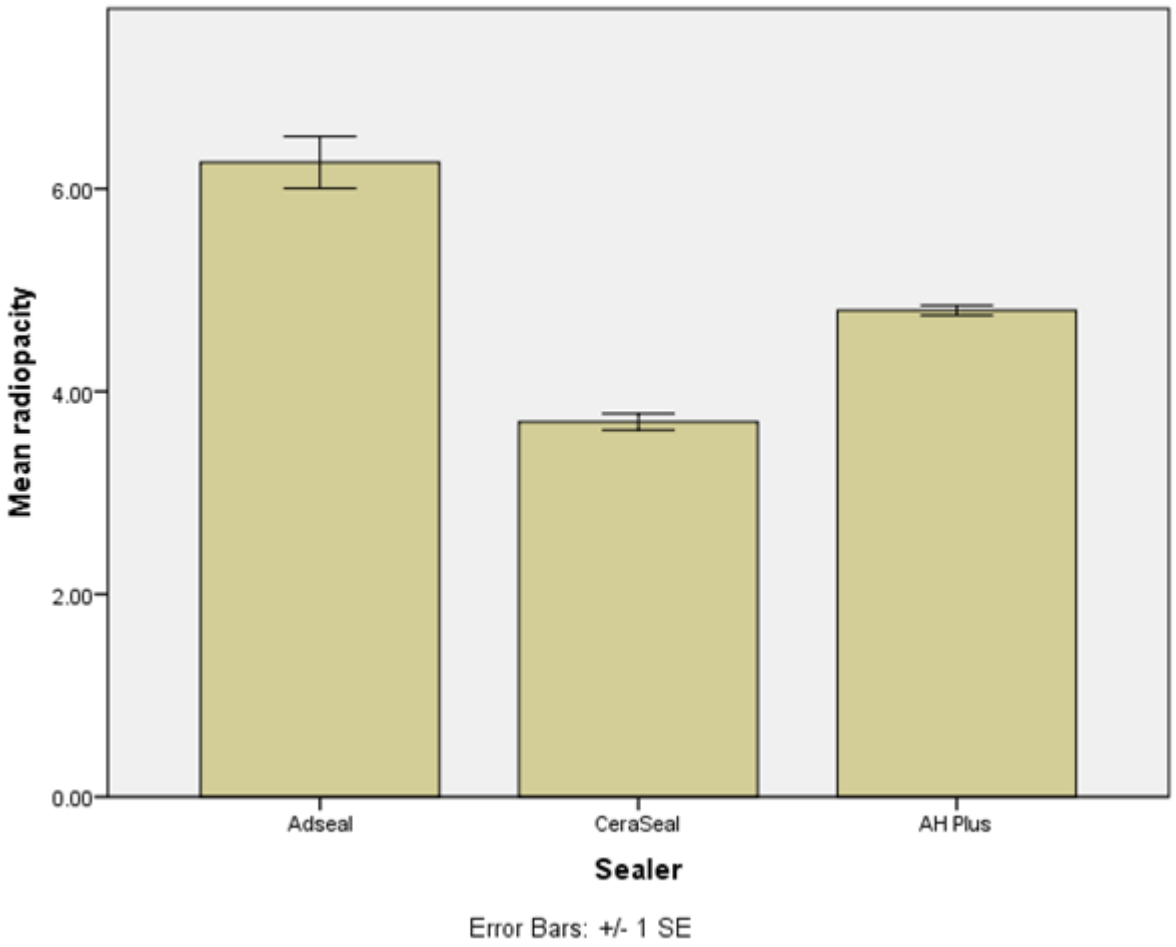

Figure 3. Different sealer radiopacity

