Acta of Bioengineering and Biomechanics
Vol. 11, No. 3, 2009

Original Paper

Shear bond strength of epoxy resin-based endodontic sealers to bovine dentin after ozone application

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The idea of using ozone to disinfect root canals is of recent origin. The wide acceptance of epoxy resin-based sealers lead us to investigate whether ozone can influence the adhesion to the dentin. In this study, we tested the shear bond strength of AH Plus and EZ Fill.

Forty freshly extracted bovine teeth were randomly divided into 5 groups. 16 of these samples were treated with ozone for 60 seconds (HealOzone, Kavo). 8 samples were conditioned with the G Bond bonding system. The groups tested were: (1) AH Plus, (2) AH Plus and ozone, (3) EZ Fill, (4) EZ Fill and ozone, (5) AH Plus and G Bond. 48 hours after being prepared the specimens were tested for shear bond strength.

Statistical analysis showed significant differences between materials (AH Plus > EZ Fill) and significant, positive influence of ozone and bonding agent on the shear bond strength.

Key words: *epoxy resin-based endodontic sealer, ozone treatment, shear bond strength*

1. Introduction

The thorough cleansing and shaping of the root canal system and placing a hermetic seal are considered to be the key to success in endodontic therapy. Although obturation may not necessarily be the most critical stage, it should achieve the highest clinical standards.

Root canal sealers work as lubricants for gutta-percha cones, adhesives for gutta-percha and dentin, assist in filling irregularities in canal walls and fill additional canals [1], [2]. Some of their physicomechanical properties are specified in international standards such as EN-ISO 6876:2001 but these exclude, for example, adhesion to the canal wall. The ability to adhere to dentin seems to be an important feature of a root canal filling material [1], [3]. Adhesion appears desirable for two reasons. In a static situation, it should prevent fluid percolation between the gutta-percha and the canal wall [4]. In a dynamic situation, it should prevent dislodgement of the root filling, thereby reducing the risk of contamination [5].

A proper bond between the filling and the canal walls should provide an effective barrier against reinfection. Epoxy resin-based sealers have proven their capability in this respect in many in vitro and in vivo investigations [6]–[9].

Although few materials have seriously challenged the combination of gutta-percha and a sealer in the majority of clinical situations, research continues to find alternatives. New synthetic polymers have been introduced recently and the results of laboratory tests are promising [10]–[12]. However, many practitioners still find a combination of warm gutta-percha and an epoxy resin-based sealer to be their method of choice.

In comparison with introducing new, adhesive materials to fill root canals (successful dentin adhesives have been available since the 1980's), the ozone

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Received: March 30th, 2009
Accepted for publication: August 28th, 2009

treatment of caries seems an innovative idea. Some recent studies have shown that ozone can be also useful in endodontics [13]–[15]. By using a HealOzone (Kavo, Germany) device and a silicone cup with a detachable, flexible cannula, the gas can be safely applied to the canal. However it is too early to justify the clinical application of this procedure [16].

The aim of present study was to investigate a possible method of improving adhesion of epoxy resin-based sealers to canal wall. The null hypothesis was that ozone cannot increase adhesion.

2. Materials and methods

We chose two contemporary resin-based materials: AH Plus (Dentsply De Trey, Germany, lot number 0611003128) and EZ Fill (Essential Dental Systems, USA, lot number 080706). The materials were purchased from independent sources.

AH Plus is an epoxy resin-based cement derived from AH 26, which was introduced in 1954 [2]. AH Plus is a paste/paste type material. Paste A (epoxide paste) consists of two epoxy resins, calcium tungstate and zirconium dioxide. Paste B (amine paste) also contains calcium tungstate, zirconium oxide plus silicon dioxide.

EZ Fill consists of a powder and a liquid. The powder contains silver and bismuth oxides as primary radiopaquing agents, and the liquid consists of a bisphenol–A epoxy resin.

The study was carried out on forty freshly extracted, caries free, bovine lower incisors. Superficial bovine dentine has been shown to be a uniform and suitable alternative to human teeth [17]. Although using bovine teeth leads to values that are slightly lower than those obtained with human dentin, the results are suitable for comparison [17]. In order to achieve a smooth, flat dentin surface for shear bond strength test, labial sites were wet-ground with 600-grit silicon carbide paper in long axis direction (acc. to ISO 11405).

Prior to the test the bovine teeth were stored in distilled water at 37 °C for 24 hours to achieve thorough wetting [18]. They were then embedded in Futura Self acrylic resin and placed in square teflon templates (3 cm × 3 cm × 2 cm).

All the samples were rinsed several times with 2% NaOCl and finally in 0.9% NaCl. The dentin was then gently dried with an air syringe.

In an attempt to achieve at least eight usable measurements, 9 specimens for each group were prepared. The samples were divided into 5 equal groups:

Group 1 – AH Plus, Group 2 – AH Plus and ozone application, Group 3 – EZ-Fill, Group 4 – EZ-Fill and ozone application, Group 5 – AH Plus and the application of G Bond (GC Corporation) – a single component, self-etching light-cured adhesive, activated for 10 sec with an Elipar (3M Espe) curing light unit (figure 1). In this study, we decided to choose the adhesive agent as a control group. The use of an agent improved bond strength of AH 26 [19], [20].

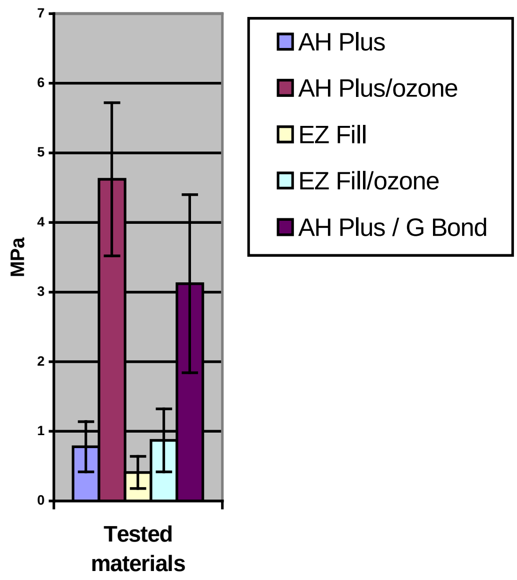

Fig. 1. Shear bond strength

The ozone was applied to the dentin by the HealOzone device (Kavo, Germany) after placing a silicone cup firmly over the flat tooth surface embedded in acrylic resin for 60 sec (according to the manufacturer's recommendations for endodontic procedures).

AH Plus was prepared with a mixing syringe (AH Plus Jet), and EZ-Fill was mixed according to the manufacturers' instructions. The material being tested was placed in a 4-mm diameter round hole, prepared in a Elite Double (Zhermapol, Poland) silicone mould which was placed on the tooth surface, in the centre of dentin. The materials being tested formed cylinders, ca. 2 mm high and 4 mm in diameter, with a circular contact area with dentin, ranging from 11 to 14 mm^2. Only one disc of the material tested was placed on each tooth.

After the initial set, the samples were transferred to an incubator at 37 °C, for 48 hours. Silicone forms were removed just before testing. The diameter was then measured with an accuracy of 0.001 mm by means of an electronic caliper.

The shear strength tests were performed on a Hounsfield H5KS universal testing machine (figure 2) with a 500 N cross-head and a cross-arm rate of 0.5 mm per minute. Bond strength refers to the force per unit area required to break the bond between the adhesive and dentin and is measured in megapascals (MPa), that is, in Newtons per square millimeter.

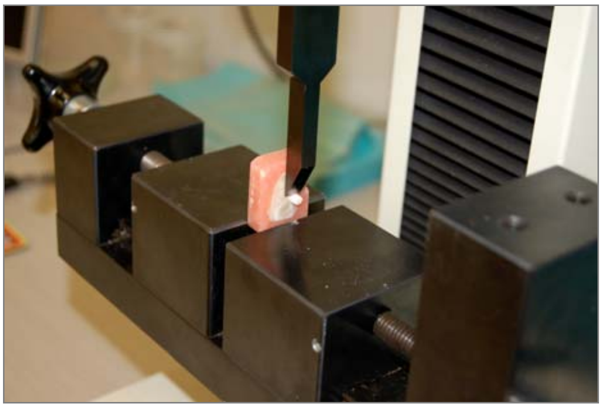

Fig. 2. Sample prepared on the universal testing machine

ANOVA was used to examine the data. The Student's *t*-test was used to measure the difference between the pairs of groups (in bonding to bovine dentin between the sealers). Significance was established at the 5% level.

Additionally we decided to compare the composition of both materials using X-ray diffraction method. We performed the X-ray study applying a D8 Advance powder diffractometer (Bruker AXS). The monochromatic parallel beam of CuKα radiation was used to collect diffraction patterns. Flat samples of sealers were prepared on microscope slides. The samples were placed on the diffractometer and the diffraction patterns were collected in 2θ range of 3–60°. Results are presented in figure 3.

3. Results

Figure 1 shows the mean shear bond strength in MPa with the standard deviation.

The mean shear bond strengths ranged from 0.41 for EZ-Fill without ozone to 4.62 for AH Plus with ozone, i.e. AH Plus had significantly greater bond strength than EZ-Fill ($p < 0.05$). The application of ozone increased significantly the bond strength of

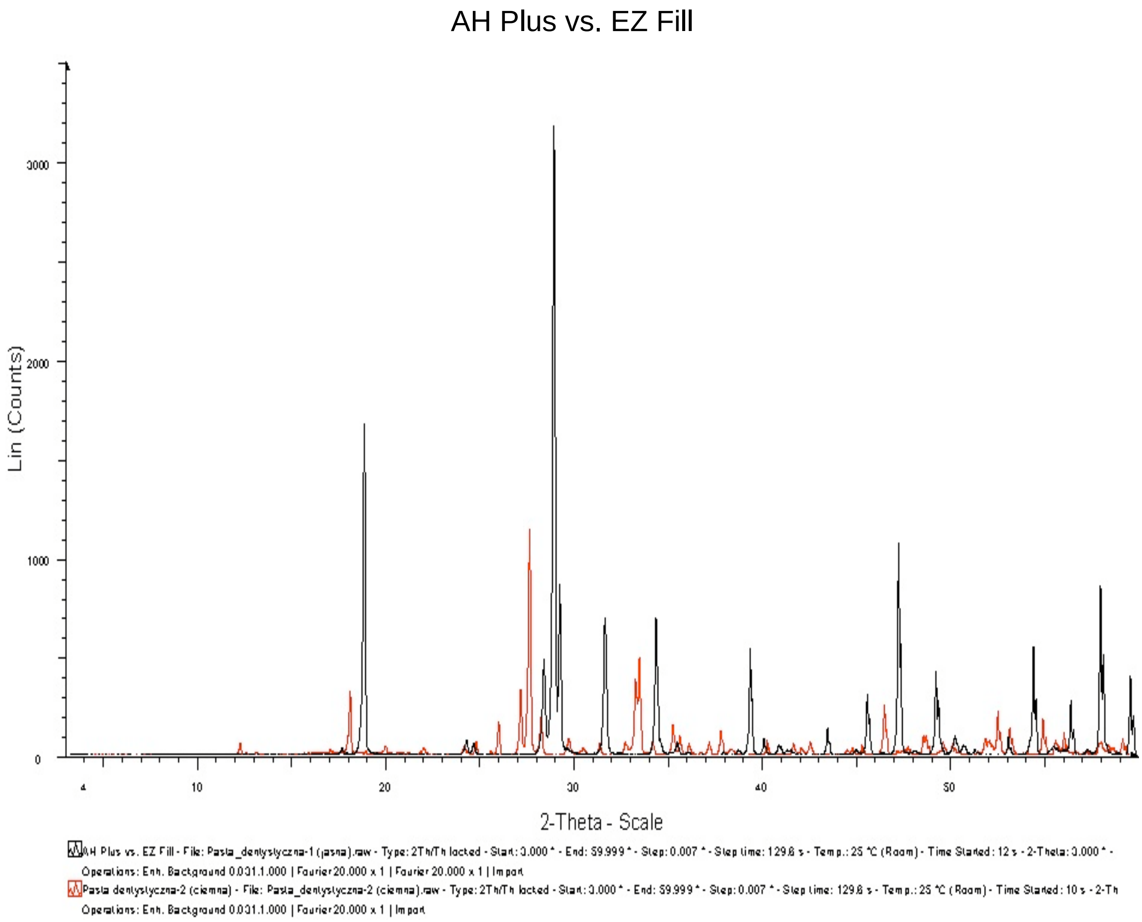

Fig. 3. Comparison of chemical composition of materials tested using X-ray diffraction

both materials. Treating dentin with a single component light-cured adhesive was almost as effective as applying ozone ($p = 0.18$).

4. Discussion

Root canal sealers are subject to International Standards and national regulations regarding their physical properties, but there is no consensus among researchers on adhesion testing. In other words, these tests are not standardized.

No test model reproduces clinical conditions precisely and attempts to duplicate them result in models which are complicated, hard to reproduce and even harder to interpret.

The methods to measure shear strength seem to be the simplest, most effective and reproducible. They were initially developed to evaluate the bonding of endodontic sealers to dentin and gutta-percha [21], [22].

Over the last few years new obturating materials and sealers based on dentin adhesion technologies borrowed from restorative dentistry have been developed in an attempt to seal the root canal system better than with the conventional materials. However, effective bonding in the environment of the root canal is a challenge [23]. Even in some recent in vitro studies methacrylate resin sealers achieved lower adhesion values than epoxy resin-based ones [24].

Are there any methods available which will improve the adhesion of commonly used sealers? Removal of the smear layer may enhance the ability of sealing materials to fill dentinal tubules [25]. This increases their bond to dentinal walls [19], [26], [27] and may also reduce leakage and subsequent bacterial penetration [1], [3], [9]. Smear layer acts as a reservoir for microorganisms and limits the extension of sealer tags to dentinal tubules. This reduces adhesion by micromechanical forces [19].

In our study, AH Plus achieved the highest bond strength, which was significantly higher than that with EZ-Fill being also an epoxy resin-based material. The application of ozone to the surface of bovine dentin for 60 sec significantly increased the shearing force and bond strength of both materials.

It is hard to explain this beneficent action of gaseous ozone. It is a strong oxidizing agent and probably may promote new chemical residues to react with epoxy resin. In theory, ozone accelerates polymerization of Bis GMA resins which is in agreement with the results of testing the bond strength of Panavia F 2.0 after treating samples with ozone by BITTER et al. [28]. However, adhesion of the other resin cement used in that study was significantly reduced by ozone. Another study revealed no significant effect of ozone on bond strength to dentin [29].

As has been shown in BITTER et al. [28] study, dentin shows changed topography after ozone application. This can lead to changes in dentin roughness. Bonding mechanisms depend here on frictional retention, at least to certain extent. An increased surface inside the canal could generate a higher bond strength, provided that a complete setting of the adhered material takes place (which can be interfered with gas residuals trapped inside the dentin tubules), and the root canal dentin is sufficiently hybridized. The authors plan to investigate the surface of the ozone-treated dentin by using X-ray photoelectron spectroscopy.

The application of a self-etching adhesive was not supported by the manufacturer's instructions for use of both materials but rather based on the null hypothesis of the compatibility of resin-based sealers and dentin adhesives. Statistical analysis did reveal that its use significantly increased the bond strength. This is compatible with previous observations regarding AH 26 [19]–[22]. However, performing a proper bonding procedure in a root canal may be difficult. Penetration with an uniform application of a curing light is limited in the root canal system. Also the use of an air blower for drying the agent in the apical one-third of the canal can be a bad idea [23].

EZ-Fill performed poorly in our tests and the results differed from those of COHEN et al. [6], [30]. The low bond strength we found came to us as a surprise, since EZ-Fill is advertised as having a formula similar to AH26, which has achieved high bonding strength in some studies [7], [19], [21], [22]. We decided then to compare both materials by X-ray diffraction. The results showed no resemblance between the materials tested. This may explain the significant difference in adhesion test results.

Unfortunately, we were unable to find any other authors who have evaluated the bond strength of EZ-Fill. However, the application of ozone did significantly increase the adhesion of this material, but not to the levels achieved with AH Plus.

So far, no strict correlation has been found between leakage of the root-canal filling and the bonding strength of the sealer to dentin in vitro [4], [21], [27], but it seems that high adhesion can slightly increase the fracture resistance of endodontically treated roots [12]. The idea to create a monoblock in the root canal is excellent in theory, but easier said than done [30]. However, it may be possible to improve the conditions for contemporary materials to adhere to the canal wall.

Ozone, used here as a dentin conditioner in the root canal, was shown to increase the adhesion of the both materials tested, with AH Plus being significantly better than EZ-Fill.

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