EVALUATION OF CONTACT ANGLE BETWEEN ROOT CANAL SEALERS AND DENTIN TREATED WITH CALCIUM HYDROXIDE AND IRRIGATION SOLUTIONS

by

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DEDICATION

This thesis is dedicated to my beloved parents, Dr. Jurai and Songchai Nakaparksin, who made all this possible, for their endless encouragement and patience.

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INTRODUCTION

Calcium hydroxide ($Ca(OH)_2$) has been used in various endodontic treatments such as direct and indirect pulp capping,[1-5] internal root resorption,[6] external root resorption,[7-9] intracanal medication,[10-12] apexogenesis,[13,14] apexification,[15-17] repair perforation,[18, 19] horizontal root fracture,[20] vertical root facture[21]and as a root canal sealer.[22, 23] $Ca(OH)_2$ can be categorized as a setting or non-setting material, with the latter commonly used as a paste for intracanal disinfection during root canal therapy.[24] $Ca(OH)_2$ is mixed with a vehicle to form the paste. Three types of vehicles are used in general; aqueous, viscous or oily.[25] The antimicrobial action of $Ca(OH)_2$ is caused by the release of hydroxyl ions in an aqueous environment, causing pH levels to reach up to approximately 12.5.[24] Hydroxyl ions have a lethal effect on the bacterial cells due to their highly oxidizing free radicals that can destroy bacteria by damaging the cytoplasmic membrane, proteins, and bacterial DNA.[25]

The ideal intracanal application time for $Ca(OH)_2$ to disinfect a root canal is still controversial, and clinical studies show conflicting results.[10,11,26-28] Safavi et al. reported negative culture with *Streptococcus faecium*-infected dentin after submerged in $Ca(OH)_2$ for 2 hours.[29] Cvek et al. found no bacteria growth from 90 percent of samples that were medicated with $Ca(OH)_2$ for 3 months.[26] Bystrom et al. were able to eradicate all microorganisms when applying $Ca(OH)_2$ for 4 weeks.[11] At 2 weeks, Reit et al. found that infection could still be found in 26 percent of the root systems treated with $Ca(OH)_2$.[27] Sjogren et al. were able to eliminate all microorganisms after intracanal medication with $Ca(OH)_2$ for 1 week.[10] Orstavik et al. discovered positive cultures in 34.8 percent of canals after 1 week of medication.[30] Similarly, Barbosa et al., found 26.7 percent of the cases still presented microorganisms in the canal after 1 week of $Ca(OH)_2$ application.[28]

In the US, the trend of using $Ca(OH)_2$ during root canal therapy has been increasing.[31] In a 1977 survey, there was no report of using root canal medication,[32] but a decade later, $Ca(OH)_2$ showed an increase to 24.7 percent.[33] Recently, according to a 2009 survey by Lee et al., $Ca(OH)_2$ is the most frequently used intracanal medication in the United States, ranging from 39.8 percent to 66 percent of all root canal therapies depending on the clinical situation.[31] The use of $Ca(OH)_2$ as an intracanal medicament has been increasing worldwide as well. In Sweden, a survey showed that up to 90 percent of general dentists used $Ca(OH)_2$ as an intracanal medication.[34] In Japan, 87.7 percent of dental care facilities also used $Ca(OH)_2$ as an intracanal medication,[35] whereas 60 percent of primary care dentists in England routinely used non-setting $Ca(OH)_2$ for the same purpose.[36]

Despite the advantages of $Ca(OH)_2$ as an intracanal medicament, its long-term use was suggested to have an adverse effect on mechanical properties of the dentin and subsequently render the roots more susceptible to fracture.[37] An *in-vitro* study demonstrated a reduction in root fracture strength after 2 months of $Ca(OH)_2$ dressing. A recent systematic review revealed reduction in the mechanical properties of radicular dentin when non-setting $Ca(OH)_2$ was used for five weeks or longer in the majority of *in-vitro* studies.[38]

As a factor of long term success in endodontic treatment, achieving a three-dimensional fluid tight seal can prevent communication between the oral cavity and periradicular tissues.[39] In root canal obturation, solid-core root filling materials alone are not usually able to fill irregularities of the root canal. Hence, root canal sealer is used to assist in sealing gaps between the root canal wall and root filling material, as well as any root irregularity.[40,41] Various types of root canal sealer are available, such as these based on zinc oxide-eugenol, epoxy resin, silicone, MTA, calcium silicate phosphate, methacrylate resin based and calcium phosphate.

In the US, zinc oxide eugenol was found to be the most commonly used sealer (74 percent of endodontists) followed by epoxy based resin (25 percent).[31] A similar trend has also appeared in England, as Tubli-Seal, a zinc oxide eugenol based sealer, is used by a majority of the primary dental care dentists.[36] Zinc oxide eugenol sealer was proposed to have minimal shrinkage (0.14 percent) in comparison with a resin based sealer.[42] It also demonstrated antimicrobial effect on various root canal bacteria up to 7 days after mixing.[43] However, zinc oxide eugenol tended to have the highest solubility when compared to contemporary sealers.[44]

To achieve maximum three-dimensional seal during obturation, proper adaptation of the root canal sealer to the root canal wall is essential.[45,46] Flowability and wettability of root canal sealers may affect their ability to adapt to the root canal wall.[47,48] Contact angle can be used to indicate wetting behavior of any liquid.[49] Different dentin conditions can affect surface tension of the dentin, which in turn, affects the contact angle of sealers on root canal dentin. It is interesting that no study has investigated the wettability of sealer on dentin treated with $Ca(OH)_2$ by means of evaluating the contact angle. The purpose of this study, therefore, was to investigate the effect of $Ca(OH)_2$ treated dentin on the wetting behavior of two root canal sealers.

Specific aim

To investigate the effect of $Ca(OH)_2$ application on chemical composition and cleanliness, as well as the wettability of dentin treated for two and four weeks with two root canal sealers and different irrigation.

HYPOTHESIS 1

Null

Dentin treated with $Ca(OH)_2$ will have no effect on wettability of root canal sealers regardless of the treatment time.

Alternative

Dentin treated with $Ca(OH)_2$ will significantly decrease wettability of root canal sealers as the treatment time increases.

HYPOTHESIS 2

Null

Chemical composition will not be impacted by calcium hydroxide treatment, regardless of the treatment time.

Alternative

Chemical composition will show decreases in carbon and nitrogen, and increases in calcium and phosphate with calcium hydroxide treatment in a time dependent manner.

HYPOTHESIS 3

Null

Cleanliness will not be impacted by calcium hydroxide treatment, regardless of the treatment time.

Alternative

Cleanliness will decrease with calcium hydroxide treatment in a time dependent manner.

REVIEW OF LITERATURE

HISTORY OF ROOT CANAL SEALER

Accomplishment of ideal root canal treatment is attributed to several crucial factors such as proper instrumentation, biomechanical preparation, obturation, and post-endodontic restoration. The goal of this treatment is to prevent bacterial contamination of the periradicular tissue by predictably providing adequately cleaned, shaped, and filled root canal systems. Long term predictability of endodontic treatment is dependent on achieving a fluid proof seal of the root canal system to periradicular tissue.[39] Any residual bacteria should be entombed in the root canal filling. A bacteria-tight apical seal should be designed to last long term with sealed portals to prevent reentry of microorganisms, which cause reentry recontamination and lead to endodontic failure. Procedural errors such as loss of length, canal transportation, perforations, loss of coronal seal, and vertical root fracture may have occurred and have been shown to adversely affect the apical seal.[50] Evidence suggests that root canal systems cannot be completely cleaned and disinfected. Obturation of the radicular space is necessary to eliminate leakage.[50] Obturation prevents coronal leakage and bacterial contamination, seals the apex from the periapical tissue fluids, and seals the remaining irritants in the canal. In endodontic obturation, root filling materials alone are not usually able to fill irregularities of the root canal. Hence, root canal sealer is used to assist in sealing gaps between the root canal wall and root filling material, as well as any root irregularity.[40,41] Ideally, root canal sealer should have good adhesion to the canal, seal completely, be radiopaque, mix easily, have no shrinkage, show no stain on tooth structure, be bacteriostatic, set slowly, be insoluble, tissue tolerant, and easy to remove by common solvent.[50] Commercially available root canal sealers are categorized according to chemical components: zinc oxide-eugenol based, epoxy resin based, silicone based, MTA based, calcium silicate phosphate based, methacrylate resin based, and calcium phosphate based.

Zinc Oxide Eugenol Sealer

Early sealers were modified zinc oxide-eugenol (ZOE)-based cements which were widely used over the world.[51] ZOE-based sealers have a setting reaction between the zinc ion in the zinc oxide and eugenol. ZOE sealers have antimicrobial properties on different microorganisms, including *E. faecalis*, even 7 days after mixing.[43] ZOE sealer demonstrated a volumetric expansion which aids in sealing the canal.[42] However, ZOE-based sealers were inferior to other types of sealer in terms of their relatively high solubility.[42]

Epoxy Resin Sealer

Epoxy resin sealer consists of 2 pastes; epoxide paste and amine paste. Epoxy resin has been shown to have great stability in solution with lowest weight loss compared to different sealers in water and in artificial saliva at different pH.[42] Epoxy resin sealer demonstrated better penetration into micro-irregularities because of its creep capacity and long setting time, which increases the bonding between the root canal and sealer.[52] However, allergic reactions have been reported with unset epoxy based resin sealer and are thought to be due to the presence of formaldehyde (3.9 ppm).[51] Although pure epoxy resin is mutagenic, a study in 2002 by Saleh IM et al., reported no mutagenic effects from aqueous extraction of epoxide and amine paste.[46]

Silicone-based Sealer

Silicone based sealer was first introduced in 1984. Polymethylsiloxane is used as a silicone matrix with less than 30-nm gutta-percha particles embedded in the silicone. Silicone sealer shows comparatively minimal leakage, is non-toxic, but has no antibacterial activity. Silicone has limited dimensional change while setting at 0.15 percent to 0.6 percent with low water sorption.[51] The presence of silicone was shown to cause this type of sealer to have poor wettability on the root dentin surface.[53]

MTA-based Sealer

Mineral trioxide aggregate (MTA) was developed by Dr. Mahmoud Torabinejad. It is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, calcium sulfate, and bismuth oxide.[51] MTA-based sealer produces calcium hydroxide, which is released and induces formation of hydroxyapatite structures.[54] Sealers based on MTA have been reported to be non-resorbable, biocompatible with periradicular tissues, and to stimulate mineralization along the apical- and middle-thirds of the canals.[54] MTA-based sealer demonstrates comparable sealing ability to resin-based sealer.[51] However, this type of sealer does not bind to dentin or core materials.[51] Because of the low tensile strength of MTA and lack of bonding to dentin, MTA-based sealer can theoretically weaken the dentin.[55]

Calcium Silicate Phosphate-based Bioceramic Sealer

Bioceramic materials were introduced to endodontics in the 1990s, first as a retrograde filling material and then as root repair cement, root canal sealers and coatings for gutta-percha cones.[56] The popularity of bioceramic sealers has increased because of their advantages of being biocompatible, non-toxic, and non-shrinking. They have a short setting time and are highly alkaline with calcium ion release and chemical stability within a biological environment.[57] Bioceramic sealer also has the ability to form hydroxyapatite, reinforcing the set cement.[58]

An example of bioceramic sealer is BC Sealer (Brasseler USA, Savannah, GA) which contains zirconium oxide, tricalcium silicate, dicalcium silicate, colloidial silica, calcium silicates, calcium phosphate, and calcium hydroxide.[59] Zirconium oxide is used as a radiopacifier.[60] For BC Sealer to initiate and complete its reaction, moisture in dentinal tubules is required.[61] The antibacterial effect of BC Sealer comes from its high pH and active calcium hydroxide release. BC Sealer has nanosized particle size that can penetrate dentin tubule as deep as 1.5 mm using warm vertical condensation technique.[62] Nanosized particle of BC sealer is less than 2 µm; therefore, BC sealer can be easily delivered in 0.012 capillary tip.[56] A study by Chang et al. demonstrated temperature-dependent changes in rheological properties of bioceramic sealer. They found an increase in flowability when heat is applied up to 112.5°C; however, the negative effect was found when heated beyond 112.5°C. BC Sealer demonstrated higher film thickness of 22 µm when compared with AH plus, ThermaSeal, Guttaflow. [81]

Methacrylate-resin-based Sealer

Methacrylate resin based sealer is a bondable sealer which will bond to core material and dentin, in turn, forming a monoblock. However, the early generation gutta-percha did not bind with the sealer unless coated with a polybutadiene di-isocyanate-methacrylate adhesive. Currently, self-adhesive type root canal sealers are available to eliminate this problem.[51] However, a recent study has shown that a methacrylate resin based sealer contains more voids and gaps than a conventional sealer and gutta-percha.[63] Methacrylate resin based sealers also exhibited high leakage due to degradation of the polymers over time.

CALCIUM HYDROXIDE AS INTRACANAL MEDICAMENT

Despite the fact that Dr. B.W. Herman introduced $Ca(OH)_2$ in endodontic treatment in 1920, it only has gained popularity during the past few decades.[31,64] $Ca(OH)_2$ is a white odorless powder with a molecular weight of 74.08. It is used as an antibacterial agent due to its being a strong base due to the dissociation of Ca^{2+} and OH^- ions.[65] The advantages of $Ca(OH)_2$ are its initially being bactericidal, then bacteriostatic. It promotes healing and repair, has a high pH that stimulates fibroblasts, neutralizes low pH of acids, stops internal resorption, is inexpensive and easy to use.[65] As an intracanal medication, it is mixed with vehicles to form a high pH paste. Three types of vehicles are used for preparing $Ca(OH)_2$ pastes: aqueous, viscous and oily.[66] Aqueous vehicles include water, saline, anesthetics and Ringer's solution. These vehicles promote a high degree of solubility.[66] Viscous vehicles are water-soluble media that release Ca^{2+} and OH^- ions for more extended time periods. Viscous vehicles are glycerin, polyethyleneglycol and propylene glycol.[66] Oily vehicles have the lowest solubility due to their hydrophobic property. Examples of these vehicles are olive oil, silicone oil, camphor and metacresyl acetate. Safavi et al. reported the conductivity of $Ca(OH)_2$ in pure glycerin and propylene glycol is essentially zero and may impede the effectiveness as root canal dressing. $Ca(OH)_2$ is a slow working antiseptic. One showed a 24-hour contact period is required to completely kill enterococci.[50] An *in-vitro* study by Safavi et al. demonstrated a minimum of 2 hours to eliminate *S. faecium* in bovine infected dentin.[29] A clinical study demonstrated a 92.5-percent reduction in bacteria after 1 week of intracanal medication, whereas sodium hypochlorite alone resulted in a 61.9-percent bacteria reduction.[50]

CONTACT ANGLE

The contact angle is the angle, conventionally measured through the liquid, where a vapor-liquid interface meets a solid surface. Wettability studies usually involve the measurement of contact angles as the primary data, which indicate the degree of wetting when a solid and liquid interact. Small contact angles ($\ll 90°$) correspond to high wettability, while large contact angles ($\gg 90°$) correspond to low wettability. The theoretical description of contact angle comes from thermodynamic equilibrium between the three phases: the liquid phase (L), the solid phase (S), and the gas or vapor phase (G). If the solid–vapor interfacial energy is denoted by γ_{SG} , the solid–liquid interfacial energy by γ_{SL}, and the liquid–vapor interfacial energy by γ_{LG}, then the equilibrium contact angle θ_C is determined from these quantities by Young's Equation:

$$\gamma_{SG} - \gamma_{SL} - \gamma_{LG} \cos \theta_C = 0$$

Contact angle can be affected by surface tension of the sealer and surface condition of the dentin.[49] In order for a root canal sealer to act as a binding agent between root canal filling materials and canal wall, the root canal sealer must have an adequate adhesion to the root canal wall.[67] The measurement of contact angles can help indirectly assess the sealing ability of endodontic sealers.[68] However, surface modifications during irrigation and medication of root canal dentin may alter its chemical and structural components, which in turn, have an effect on wettability.

Studies have shown significant changes in wettability after dentin conditioning with different irrigating agents.[49,69] Ballal et al. demonstrated an increase in contact angle when EDTA was used as a final irrigant, but irrigation with NaOCl improved wettability.[69] Ballal et al. explained that the use of EDTA caused a thin layer of demineralized dentin fibrils on the dentin surface.[69] The presence of this layer is responsible for the poor wetting behavior of EDTA irrigated dentin.[70] Attal et al. suggested that high concentration EDTA could have caused mild etching of the dentin surface, leading to the exposure of the more hydrophobic collagen fibers. This exposure could have resulted in reduced wettability.[71] However, Tummala et al. suggested no significant difference in contact angle when ZOE sealer was measured after dentin irrigation with EDTA, NaOCl or both. The authors explained that the poor surface wetting of zinc oxide-eugenol sealer could be due to the increased viscosity of the sealer.[49]

The literature, up to this date, mainly focuses on the effects of radicular dentin irrigation on the contact angle (wettability) of different types of sealers. Only one study has treated dentin with $Ca(OH)_2$ before contact angle measurement within the context of endodontic regeneration.[72] The study proposed a significant increase in contact angle of a blood surrogate after dentin treatment with $Ca(OH)_2$ for four weeks compared with dentin irrigation with NaOCl and EDTA alone.[72] The study also found significant change in surface chemistry of dentin treated with $Ca(OH)_2$ in comparison with untreated dentin using energy-dispersive X-ray (EDX). EDX is an analytical technique used for elemental analysis. A high-energy beam such as electrons, protons, or a beam of x-ray is introduced to the sample, causing atoms within the sample to excite and emit energy in the form of x-rays. The number and energy of x-rays emitted from the sample can be measured by an energy-dispersive spectrometer, allowing the elemental composition of the sample to be measured.

MATERIALS AND METHODS

HUMAN DENTIN SPECIMEN PREPARATION

One hundred and fifty-six intact caries-free human third molars were collected and stored in 0.1-percent thymol solution at 4°C. Two-mm slices were cross-sectioned from the crowns parallel to the occlusal surface (Figure 1) and as close to the pulp chamber as possible using a low-speed saw (IsoMet; Buehler, Lake Bluff, IL) (Figure 2) under constant irrigation. Deep coronal dentin rather than radicular dentin was used in this study because it provided a wider surface area, as well as having similar density and cross-sectional area of dentin tubules in comparison to radicular dentin.[73] Circumferential enamel was removed using 1200-grit silicon carbide paper (Struers, Cleveland, OH) (Figure 3) under constant water irrigation. The non-pulpal side of each disc was flattened with 500-grit silicon carbide abrasive paper using a Struers polishing unit (Struers, Cleveland, OH). The pulpal side of each disc was flattened using 500-grit, 1200-grit, 2400-grit, and 4000-grit silicon carbide papers (Figure 4). The specimens were then placed under running deionized water for 3 minutes, sonicated in deionized water for 3 minutes and replaced under running deionized water for an additional 3 minutes. Next, the specimens were polished with a 0.3-mm diamond polishing spray (Struers, Cleveland, OH) before they were immersed under running deionized water for 3 minutes, followed by sonicating in deionized water for 3 minutes, and re-immersed in running deionized water for an additional 3 minutes. To remove the smear layer, the specimens received final sonication with 6.0-percent NaOCl (Clorox, Oakland, CA) and 17-percent EDTA (Henry Schein, Melville, NY) (Figure 5) for 5 minutes.

HUMAN DENTIN SPECIMEN TREATMENT

The polished dentin discs were randomized into twelve experimental groups (G1-12) (n=13), as described in Table I. Samples were placed individually in 2-mL conical sample cups (Fisher Scientific, Pittsburgh, PA) with the pulpal surface facing outward to receive the treatment (Figure 6). The first half of the experimental group (G1-G6) was prepared to test with Tubli-Seal. G1 received 0.1 mL of sterile water for two weeks followed by the chemical irrigation protocol consisting of 2-minute continuous flow irrigation with 10 mL of 6.0-percent NaOCl in a 10 mL syringe and 27-G needle followed by 10 mL of 17-percent EDTA in a similar manner. G2 and G3 were treated for two weeks with 0.1 mL of $Ca(OH)_2$ (UltraCal XS, Ultradent, South Jordan, UT, USA) (Figure 7) followed by either 2 minutes of irrigation with 10 mL of sterile water in a 10 mL syringe and 27-G needle or the chemical irrigation protocol previously described prospectively. G4-6 were treated identically to the first three experimental groups except for extending the treatment time from two weeks to four weeks (Figure 8). G7-12 speciments were prepared to test with BC sealer in a similar manner to G1-6.

All experimental groups were incubated at 37°C with approximately 100-percent humidity during the two or four week treatment with $Ca(OH)_2$ or sterile water (Figure 9, Figure 10). The two- and four-week application times of $Ca(OH)_2$ were selected because they are within the range of intracanal medicament application time (2 weeks to 4 weeks) during root canal therapy.[11, 27] The chemical irrigation protocol was selected because it is one of the common irrigation protocols used after $Ca(OH)_2$ application.[74]

CONTACT ANGLE MEASUREMENT

After treatment, contact angle between the treated dentin surfaces and a zinc oxide eugenol based sealer (Tubli-Seal Xpress, SybronEndo, Orange, CA, USA)(G1-6) or calcium silicate based sealer (BC Sealer, Brasseler, USA)(G7-12) was measured. Zinc oxide eugenol was selected for this study because it is the most common type of sealer used during root canals.[31] Furthermore, the Tubli-Seal brand was selected because it is available in automix syringes that facilitate the delivery of a standardized 1:1 ratio. Calcium silicate-based sealer was selected in the current study because it is one of the most recently introduced root canal sealers that offer numerous biological and chemical advantages.[56,75] Additionally, it is available in premix syringes that can minimize the potential errors that might occur during manual mixing (Table II).

Prior to contact angle measurement, each specimen was air-dried from a 6-inch distance for 2 seconds at 5 bar pressure. Then, a 2-μL drop of sealer was vertically dispensed on the treated dentin surface using a capillary tip with built in piston (CP10, Gilson, Middleton, WI, USA) that was attached to a positive displacement pipette (Microman, Gilson, Middleton, WI, USA) (Figure 13). The contact angle was recorded using a PGX goniometer (Fibro Systems AB; Stockholm, Sweden) (Figure 14) immediately after the sealer detachment from the positive displacement pipette in static mode at 40 seconds. The contact angle measurement was performed in triplicate on each dentin disc. The average of these measurements represented the contact angle value for each dentin disc. All contact angle measurements were taken at room temperature.

ENERGY DISPERSIVE X-RAY MEASUREMENT

An additional 18 dentin discs were prepared, randomly divided into 6 groups, treated as described previously and used for energy-dispersive x-ray (EDX) analyses to observe the chemical changes in surface dentin after various treatments (n = 3). After treatment, each sample was dried for 48 hours, and the weight percentages of calcium (Ca), phosphorus (P), carbon (C) and nitrogen (N) were measured from treated dentin surfaces using scanning electron microscopy (JEOL 7800F; JEOL, Peabody, MA) (Figure 15) equipped with EDX spectroscopy (Octane Super Detector; EDAX, Mahwah, NJ). The samples were not sputter-coated to prevent error in identification of all selected elements. The EDX system was operated at 15 kV accelerating voltage and X1000 magnification. EDX analyses were performed on five randomly selected spots on four corners and the center of each treated surface. The relative contributions of the four measured elements were then automatically normalized to a total of 100 percent.

SCANNING ELECTRON MICROSCOPE

After EDX analyses, the same 18 samples were prepared for SEM analysis to detect any remaining $Ca(OH)_2$ after treatment and to observe morphological changes in the treated dentin samples. Each sample was air-dried with a low-vacuum-pressure desiccator (Bel-Art, Wayne, NJ, USA) containing silica gel crystals for 48 hours. The samples were sputter coated for 70 seconds with gold/palladium using a sputter coater (POLARON Sputter Coating System, Energy Beam Sciences, Ageam). Three SEM images were taken for each sample at X1000 magnification from the treated side of the dentin samples; two images from the edge of the sample, and one image at the center of the sample. (JEOL 7800F, JEOL, Peabody, MA, USA). Additional X7000 magnification images were also taken at the center of each sample.

The evaluation of cleanliness was performed based on Alturaiki et al.[76] whose procedure was used to evaluate according to the following 5-grade scale:

Score 1: 80% to 100% removal of $Ca(OH)_2$ (total cleanliness).

Score 2: 60% to 80% removal of $Ca(OH)_2$ (significant cleanliness).

Score 3: 40% to 60% removal of $Ca(OH)_2$ (partial cleanliness).

Score 4: 20% to 40% removal of $Ca(OH)_2$ (light cleanliness).

Score 5: 0% to 20% removal of $Ca(OH)_2$ (no cleanliness).

SAMPLE SIZE

The standard deviation within-groups was estimated to be 6°. With a sample size of 13 samples per group the study had 80-percent to detect a difference of 7° between any two groups, assuming two-sided tests with each conducted at a 5-percent significance level.

STATISTICAL METHODS

Three-way ANOVA was used to test the effects of $Ca(OH)_2$ application, duration of $Ca(OH)_2$ application and sealer type on contact angle between root canal sealers and dentin. Pair-wise comparisons between groups were conducted using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent.

Two-way ANOVA was used to test the effects of group and duration of application on chemical composition. Pair-wise comparisons between groups were conducted using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent. Cochran-Mantel-Haenszel tests were used to test the effects of group and duration of application on degree of removal of calcium hydroxide particles.

RESULTS

CONTACT ANGLE

Means (M) and respective standard deviations (SD) for contact angle measurements are presented in Table III and Figure 16. For Tubli-Seal groups (G1-G6), G4 had the highest mean of contact angles at 104.9 ± 1.9°, whereas G5 presented the lowest mean of contact angles at 85.4 ± 15.1°. Among BC sealer groups (G7-G12), G10 had the highest mean of contact angles at 145.4 ± 1.3°, while G11 demonstrated the lowest mean of contact angles at 130.2 ± 2.6°. Three-way interaction among the three factors was significant; however, the comparisons were examined to determine when comparisons could be generalized across other effects (Table IV). Effect of sealer, Tubli-Seal (G1-G6) had significantly lower contact angles than BC sealer (G7-G12) (Figure 17 and 18).

Effect of duration is shown in Figure 19 and Figure 20. For Tubli-seal at two weeks (G1-G3) and four weeks (G4-G6) duration, G1 had significantly lower contact angle than G4 whereas G2 showed significantly higher contact angle than G5. No significant difference was found between G3 and G6 (Figure 19). For BC sealer at two weeks (G7-G9) and four weeks (G10-12) duration, no significant difference was found between G7 and G10. However, G8 had significantly higher contact angle than G11, whereas, G9 had significantly lower contact angle than G12 (Figure 20).

Comparisons of sample groups treated with $Ca(OH)_2$ at the same duration are shown in Figure 21 and Figure 22. For Tubli-Seal at two weeks duration, G2 showed significantly lower contact angles than G3. A similar trend was also observed at four weeks duration, G5 had significantly lower contact angles than G6 (Figure 21). However, with BC sealer, at two weeks duration G8 had significantly higher contact angles than G9, whereas at four weeks, G11 demonstrated lower contact angles than G12 (Figure 22).

A significant effect on remaining $Ca(OH)_2$ after water irrigation when compared with the control is shown in Figure 23 and Figure 24. At four weeks treatment duration with both sealers, the control groups (G4 and G10) showed significantly higher contact angles than $Ca(OH)_2$ with sterile water irrigation (G5 and G10). However, at two weeks treatment duration, there was no significant difference for both Tubli-Seal (G1 vs G2) and BC sealer (G7 vs G8).

For Tubli-Seal, no significant effect on contact angle was found when $Ca(OH)_2$ was removed with chemical irrigation when compared with the control at two weeks duration (G1 vs G3) and four weeks duration (G4 vs G6) (Figure 25). A similar trend was found with BC sealer at four weeks duration (G10 vs G12). However, G7 had significantly higher contact angles then G9 at four weeks duration ($p < 0.05$) (Figure 26).

ENERGY DISPERSIVE X-RAY MEASUREMENT

Means and respective standard deviations for weight percentages of calcium (Ca), phosphorus (P), carbon (C), and nitrogen (N) measurement are presented in Table V and Figure 27. The two-way interaction between group and duration was significant for N; however, the comparisons were examined to determine when comparisons could be generalized across other effects (Table VI). At both durations, $Ca(OH)_2$ with water irrigation $Ca(OH)_2$ had significantly lower C and N and higher P and Ca than $Ca(OH)_2$ with chemical treatment and no $Ca(OH)_2$ with chemical irrigation (Figure 28). For both durations, $Ca(OH)_2$ with chemical treatment had significantly lower C than no $Ca(OH)_2$ with chemical irrigation but was not different for N, P, or Ca. Within the same treatment groups, two week treatment had significantly lower P-values than four week treatment (Figure 29). The two week duration showed significantly higher N than four week treatment, but no significant difference for no $Ca(OH)_2$ with chemical irrigation or $Ca(OH)_2$ with water. Two and four week treatments were not significantly different for C or Ca.

SEM ANALYSIS

Cochran-Mantel-Haenszel tests were used to test the effects of group and duration of application on degree of removal of calcium hydroxide particles. Cleanliness scores are presented in Table VII. Duration had no effect on degree of removal of calcium hydroxide particles. Irrigation solution appeared to have significant effect on cleanliness score ($p = 0.0007$). Water irrigation solution showed higher remaining $Ca(OH)_2$ than irrigated with chemical solution. $Ca(OH)_2$ with chemical irrigation demonstrated no $Ca(OH)_2$ remaining after irrigation, similar to the surface of the control group.

TABLES AND FIGURES

TABLE I

Description of experimental groups

Sealer	Treatment time (week)	Treatment	Group
Tubli-Seal	2 weeks	No $Ca(OH)_2$ + chemical irrigation	G1
		$Ca(OH)$ + water irrigation	G2
		$Ca(OH)_2$ + chemical irrigation	G3
	4 weeks	No $Ca(OH)_2$ + chemical irrigation	G4
		$Ca(OH)$ + water irrigation	G5
		$Ca(OH)_2$ + chemical irrigation	G6
BC Sealer	2 weeks	No $Ca(OH)_2$ + chemical irrigation	G7
		$Ca(OH)$ + water irrigation	G8
		$Ca(OH)_2$ + chemical irrigation	G9
	4 weeks	No $Ca(OH)_2$ + chemical irrigation	G10
		$Ca(OH)$ + water irrigation	G11
		$Ca(OH)_2$ + chemical irrigation	G12

TABLE II

Description of root canal sealer used in this study

Root Canal Sealer	Brand Name	Manufacturer	Mixing	Composition
Zinc oxide eugenol sealer	Tubli-seal Xpress	SybronEndo, Orange, CA, USA	Automix	Zinc oxide, Oloe resin, Bismuth trioxide, Thymol iodide, Eugenol, Polymerized resin, Annidalin
Calcium-Silicate-Phosphate-based bioceramic sealer	BC Sealer	Brasseler, GA, USA	Premix	Tricalcium silicate, Dicalcium silicate, Calcium phosphates, Colloidal silica, Calcium hydroxide, and Zirconium oxide

TABLE III

Contact angle means and SD of dentin specimens*

Group	Mean Contact Angle	SD	Min	Max
G1 ^{a}	94.6	11.1	70.3	109.9
G2 ^{b}	92.6	7.6	81.4	108.8
G3 ^{c}	98.9	6.6	89.6	113
G4 ^{d}	104.9	6.7	93.8	119.2
G5 ^{e}	85.4	15.1	57.3	104.5
G6 e	99.2	9.2	83.8	114.2
G7 ^{f}	141.4	4.1	132.4	148.1
G8 ^{f}	137.1	6.2	123.2	145.8
G9 ^{g}	130.2	9.4	114.9	151.2
G10 ^{f}	145.4	4.8	138.7	154.7
G11 ^{h}	128.8	8.1	114.6	142.0
G12 ^{i}	142.6	8.2	129.6	154.4

*Superscript lowercase letters $^{(a\text{-}i)}$ indicate statistically significant values ($p < 0.05$) between groups.

TABLE IV

Three-way ANOVA between contact angle and duration, sealer type, and treatment type

Effect	F Value	p-value
Duration	1.93	0.1670
Sealer type	922.98	<0.0001
Duration*Sealer type	0.31	0.5796
Treatment	20.44	<0.0001
Duration*Treatment	12.37	<0.0001
Sealer type*Treatment	2.45	0.0895
Duration*Sealer type*Treatment	3.97	0.0210

TABLE V

Energy dispersive x-ray measurement of carbon (C), nitrogen (N), phosphorus (P), and calcium (Ca)*

Treatment	Duration	% C (SD)	% N (SD)	% P (SD)	% Ca (SD)
Chemical (Control)	2 weeks	39.3 (0.3) ^{a}	59.4 (0.2) ^{a}	0.6 (0.1) ^{a}	0.8 (0.1) ^{a}
	4 weeks	40.5 (1.1) ^{a}	57.2 (0.2) ^{a}	1.1 (0.4) ^{c}	1.3 (0.5) ^{a}
$Ca(OH)_2$ + Water	2 weeks	11.2 (0.7) ^{b}	18.6 (2.4) ^{b}	16.6 (1.2) ^{b}	53.6 (2.9) ^{b}
	4 weeks	10.9 (0.4) ^{b}	19.2 (0.5) ^{b}	18.1 (0.2) ^{d}	51.8 (0.8) ^{b}
$Ca(OH)_2$ + Chemical	2 weeks	34.2 (0.4) ^{c}	61.4 (2.2) ^{a}	1.4 (0.1) ^{a}	1.3 (0.1) ^{a}
	4 weeks	36.1 (4.7) ^{c}	53.2 (1.0) ^{c}	2.9 (0.8) ^{c}	4.0 (1.0) ^{a}

*Superscript lowercase letters $^{(a\text{-}c)}$ indicate statistically significant values ($p < 0.05$) between groups of the same element.

TABLE VI

Two-way ANOVA between percentages of element and duration, and treatment type

Element	Effect	F Value	p-value
C	Duration	0.34	0.5718
	Treatment Type	119.46	<0.0001
	Duration*Treatment type	0.18	0.8385
N	Duration	8.04	0.0150
	Treatment Type	505.28	<0.0001
	Duration*Treatment type	5.15	0.0242
P	Duration	5.62	0.0354
	Treatment Type	468.17	<0.0001
	Duration*Treatment type	0.52	0.6098
Ca	Duration	0.24	0.6321
	Treatment Type	1026.86	<0.0001
	Duration*Treatment type	1.50	0.2623

TABLE VII

Cleanliness score of the dentin specimens*

Group	Weeks	Score				
		1	2	3	4	5
No $Ca(OH)_2$ + Chemical Only	2	3	0	0	0	0
	4	3	0	0	0	0
$Ca(OH)_2$ + Water	2	0	0	2	1	0
	4	0	0	1	1	1
$Ca(OH)_2$ + Chemical	2	3	0	0	0	0
	4	3	0	0	0	0

*Cleanliness score based on Alturaiki et al.:[76]

Score 1: 80% to 100% removal of $Ca(OH)_2$ (total cleanliness).
Score 2: 60% to 80% removal of $Ca(OH)_2$ (great cleanliness).
Score 3: 40% to 60% removal of $Ca(OH)_2$ (partial cleanliness).
Score 4: 20% to 40% removal of $Ca(OH)_2$ (light cleanliness).
Score 5: 0% to 20% removal of $Ca(OH)_2$ (no cleanliness).

FIGURE 1. Illustration of specimen cross-sectioned; reprinted with permission.[72]

FIGURE 2. IsoMet (Buehler) used for cross-sectioning crowns.

FIGURE 3. RotoForce-4 (Struers) used for polishing specimens.

FIGURE 4. Specimens with pulpal side polished.

FIGURE 5. Seventeen-percent EDTA used for removing smear layer.

(A)

(B)

FIGURE 6. Specimens stored with wet cotton in 2-mL conical sample cups either with (A) or without (B) $Ca(OH)_2$.

FIGURE 7. $Ca(OH)_2$ UltraCal XS (Ultradent).

FIGURE 8. Study design diagram.

FIGURE 9. Specimens stored in container lined with wet paper to ensure 100% humidity.

FIGURE 10. Incubator, used for incubating specimens at 37°C with approximately 100% humidity.

FIGURE 11. Tubli-Seal Xpress (SybronEndo).

FIGURE 12. BC Sealer (Brasseler).

FIGURE 13. Positive displacement pipette (Microman, Gilson) with built-in piston capillary tip (CP10, Gilson).

(A)

(B)

FIGURE 14. Pocket Goniometer (A) with silicone supporting CP10 capillary tip (B).

FIGURE 15. A JEOL SEM (JSM-6390) used for surface micro-morphological evaluation.

FIGURE 16. Mean of contact angles between root canal sealers and treated dentin surfaces, by sealer type, treatment type, and treated duration. Different lowercase letters (a-h) indicate statistically significant values (P < .05) between groups.

FIGURE 17. Mean of contact angles between root canal sealers and treated dentin surfaces for 2 week treatment, by treatment type and sealer type.

FIGURE 18. Mean of contact angles between root canal sealers and treated dentin surfaces for 4-week treatment, by treatment type and sealer type.

FIGURE 19. Mean of contact angles between Tubli-Seal and treated dentin surfaces by treatment type and treatment duration.

FIGURE 20. Mean of contact angles between BC sealers and treated dentin surfaces by treatment type and treatment duration.
For $Ca(OH)_2$ + Water, $p = 0.0014$.
For $Ca(OH)_2$ + Chemical, $p < 0.003$.

FIGURE 21. Mean of contact angles between root canal sealers and treated dentin surfaces for Tubli-Seal by irrigation and duration. $P < 0.0001$ for values at 2 weeks and 4 weeks.

FIGURE 22. Mean of contact angles between root canal sealers and treated dentin surfaces for BC Sealer by irrigation and duration.

FIGURE 23. Mean of contact angles between root canal sealers and treated dentin surfaces for Tubli-Seal by chemical and duration.

FIGURE 24. Mean of contact angles between root canal sealers and treated dentin surfaces for BC Sealer by chemical and duration.

FIGURE 25. Mean of contact angles between root canal sealers and treated dentin surfaces for Tubli-Seal, by presence of Ca and duration.

FIGURE 26. Mean of contact angles between root canal sealers and treated dentin surfaces for BC Sealer, by presence of Ca and duration.

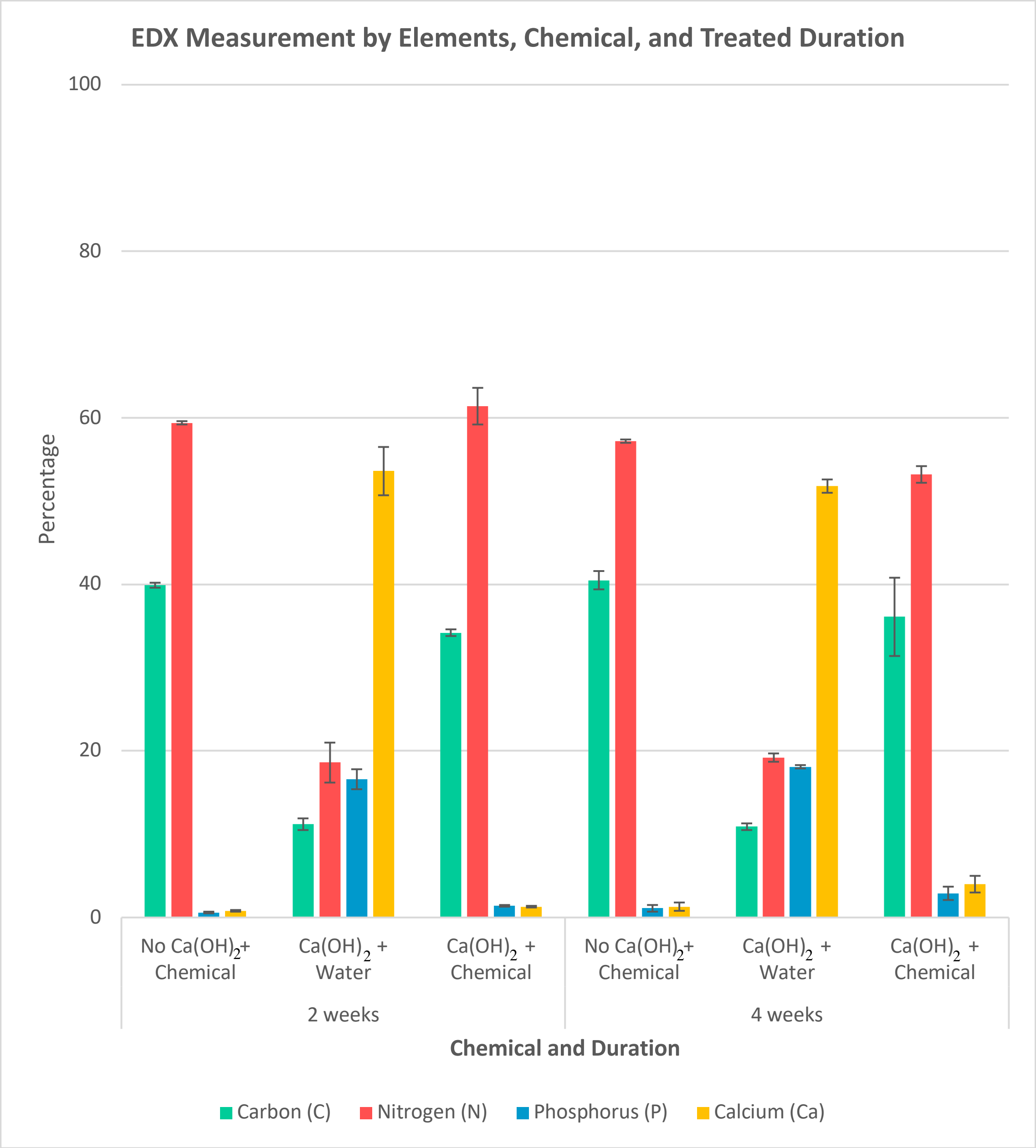

FIGURE 27. Means ± SD of carbon, nitrogen, phosphorus, and calcium measured on the surface of treated dentin specimens with EDX.

FIGURE 28. Means ± SD EDX percentage of each element by treatment.

FIGURE 29. Means ± SD EDX percentage of each element by duration. P-values of significance: Nitrogen, p = 0.0014; Phosphorus, p = 0.0354.

FIGURE 30. Illustration of $Ca(OH)_2$ treated specimen after water irrigation. Notice remaining $Ca(OH)_2$ on the surface.

(A)

(B)

FIGURE 31. Representative SEM micrographs of specimen without $Ca(OH)_2$ treatment at 2 weeks after chemical irrigation at X1000 (A) and X7000 (B) magnification.

(A) **(B)**

FIGURE 32. Representative SEM micrographs of specimen without $Ca(OH)_2$ treatment at 2 weeks after chemical irrigation at X1000 (A) and X7000 (B) magnification.

(A) **(B)**

FIGURE 33. Representative SEM micrographs of $Ca(OH)_2$ treated specimen at 2 weeks after chemical irrigation at X1000 (A) and X7000 (B) magnification.

(A) **(B)**

FIGURE 34. Representative SEM micrographs of $Ca(OH)_2$ treated specimen at 4 weeks.

(A)

(B)

FIGURE 35. Representative SEM micrographs of $Ca(OH)_2$ treated specimen at 2 weeks.

(A) **(B)**

FIGURE 36. Representative SEM micrographs of $Ca(OH)_2$ treated specimen at 4 weeks after water irrigation at X1000 (A) and X7000 (B) magnification.

(A)

(B)

(C)

FIGURE 37. Representative $Ca(OH)_2$ treated specimen followed by chemical irrigation and tested with Tubli-seal at top view (A), horizontal view (B), and PGX goniometer view.

(A)

(B)

(C)

FIGURE 38. Representative $Ca(OH)_2$ treated specimen followed by water irrigation and tested with Tubli-seal at top view (A), horizontal view (B), and PGX goniometer view (C).

(A)

(B)

(C)

FIGURE 39. Representative $Ca(OH)_2$ treated specimen followed by chemical irrigation and tested with BC sealer at top view (A), horizontal view (B), and PGX goniometer view (C).

(A)

(B)

(C)

FIGURE 40. Representative $Ca(OH)_2$ treated specimen followed by water irrigation and tested with BC sealer at top view (A), horizontal view (B), and PGX goniometer view (C).

DISCUSSION

In endodontics, the most commonly used irrigation solution is NaOCl, because it demonstrates properties of bactericidal cytotoxicity, dissolution of organic material, and minor lubrication. However, NaOCl has no effect on the smear layer, thus clinicians commonly use EDTA in adjunct with NaOCl. According to a previous study by Kenee et al., no significant difference in remaining $Ca(OH)_2$ was found between using NaOCl alone and the combined NaOCl/EDTA irrigation.[77] However, in this study, the combination of NaOCl and EDTA was used to mimic the clinical situation.

As a method for dispersing sealer, in a previous study, Ballal et al. reported using a micropipette manually maneuvered through a tiny hole with a controlled volume of 0.1 mL.[69] Alternatively, Bohn et al. used an endodontic spreader to deliver a droplet of sealer with 1-mm diameter.[68] Others reported using a nozzle to dispense sealer onto the dentin samples.[49] Without a standardized dispersing device, the amount of sealer dispersed to form the droplet may vary within the study and may cause inaccurate measurements. In this study, a positive displacement pipette was used to ensure consistent sealer volume on the samples as well as a customized putty index for a consistent ejecting angle onto the specimens. Positive displacement pipettes are recommended to be used for dense and viscous fluids.[78] A higher precision in volume occurs with positive displacement pipettes when compared with forward and reverse pipettes.

Kenee et al. reported that irrigation alone showed significantly less ability to remove $Ca(OH)_2$ on unpolished root canal surfaces when compared with irrigation with mechanical assistance.[77] However, agitated irrigation or ultrasonic irrigation was not employed in the current study, as specimen polished surfaces were to be preserved. In this study, all specimens were polished to reduce the influence of the roughness on the surface energy of the samples, and in turn, reduce its influence on the contact angle. Adding surface roughness enhances the wettability caused by the chemistry of the surface. If the surface is hydrophobic, it will become even more hydrophobic. In the present study, chemical irrigation successfully removed calcium particles from the dentin surface. No remaining calcium particles were observed on the tooth surface (Figure 31 to Figure 34). EDX measurement also confirmed similar element percentages on the dentin surface between the control group and the treated groups with chemical irrigation. However, cleanliness after irrigation may be different in clinical situations as root canals are irregular and unpolished which may lead to an increase in $Ca(OH)_2$ remnants. Furthermore, in conventional irrigation, fewer calcium particles in the coronal and middle third were reported when compared with the apical third of the root canal.[76]

To simulate the clinical situation, $Ca(OH)_2$ was left on the sample for 2 weeks and 4 weeks before irrigation. Sterile water irrigation was used to observe the effect of remaining $Ca(OH)_2$ on contact angle of two sealers. Chemical irrigation without $Ca(OH)_2$ was used as a control group to rule out the effect of remaining $Ca(OH)_2$ after irrigation. According to this study, chemical irrigation removes $Ca(OH)_2$ better than water. Remaining calcium was observed with the naked eye when water irrigation was used (Figure 30). Using SEM, calcium particles were not observed when using chemical irrigation regardless of treatment time (Figure 31 to Figure 34), whereas calcium particles were observed after water irrigation (Figure 35, Figure 36). EDX measurement also showed an increase in the calcium and phosphate elements. This may indicate that particles shown on SEM were calcium phosphate particles. With remnants of calcium in the water irrigation, wettability of both sealers increased when compared to groups without remaining calcium, except for BC sealer at 2 weeks (Figure 37 through Figure 40). Samples with water irrigation with $Ca(OH)_2$ treated for 4 weeks had better wettability with both sealers when compared with 2 weeks duration. This coincided with more calcium particles remaining on the SEM image of specimens treated with $Ca(OH)_2$ in the 4 week group (Figure 35, Figure 36). In the present study, remaining calcium particles demonstrated a decrease in contact angle of sealers. However, a previous study had reported that by removing the smear layer and exposing dentin tubules, improved sealer wettability resulted.[79]

Type of sealer also played a significant role; Tubli-seal had a lower contact angle on the dentin surface when compared with BC sealer regardless of treatment type. This result is in contradiction with Zhang et al., who reported low contact angles. However, they measured the contact angle of sterile water on the BC sealer, which represents wettability of water with sealer, not sealer to dentin.[80] The current study measured the wettability of sealer on dentin, which represents more accurate behavior of the sealer to dentin. This could indicate that wettability between water and sealers do not have the same characteristic as wettability between dentin and sealers. No literature, up to this date, has described the wettability behavior of BC sealer on dentin. The authors believe that the higher contact angle from BC sealer may result from higher intermolecular attraction and surface tension than Tubli-Seal. The greater intermolecular attraction leads to an increase in solid-liquid interfacial energy (γ_{SL}) in Young's equation, which results in higher contact angles. Liquid surface tension can be altered by using surfactants and heat. Furthermore, surface tension is responsible for liquid droplet formation, as surface tension increases, the ability of withstanding external forces, such as gravity, also increase. One limitation of this study was that no load was applied to the sealer. BC sealer and Tubli-seal may behave differently when pressure is applied during root canal obturation. In the study by Zhou et al., BC sealer demonstrated better flow than GuttaFlow, AH Plus, and Thermaseal, but less flow than MTA Fillapex.[81] The current study also used air-dry as the standardized method of eliminating water droplets from the specimen that could create an overly dry surface, given that the BC sealer requires moisture to initiate setting. Paper points could have been used to simulate the clinical situation. However, air-dry was used to eliminate potential disturbance to specimen surfaces and $Ca(OH)_2$ remnants. Drying with alcohol also was not utilized in this study as it may also dehydrate and disturb the $Ca(OH)_2$ remnant.

The result of the no-$Ca(OH)_2$ treated group was similar to a previous study. Tummala et al. reported an average contact angle of 89.9° with zinc oxide eugenol sealer after treating dentin with 10 ml, 17-percent EDTA for 1 min followed by 10 mL of 3-percent NaOCl solution for 1 min.[49] The current study measured contact angles of 94.6° at two weeks and 104.9° at four weeks duration. The difference in result may be from the different NaOCl concentrations, the order of NaOCl/EDTA irrigation, or variations in the amount of sealer dispensed using the positive displacement pipette. As reported by Ballal et al., the use of EDTA for final irrigation on dentin demonstrated a decrease in wettability when compared with NaOCl as the final irrigation.[69] Ballal et al. explained that by using EDTA, the smear layer was removed and resulted in increasing surface roughness of the dentin. Attal et al. also reported that EDTA should have caused poorer wettability. They explained that EDTA has a mild etching ability causing an exposure of hydrophobic fibers.

Even though, the present study showed a positive effect of remaining $Ca(OH)_2$ with contact angle between BC sealer and dentin, multiple factors involving endodontic success beside wettability had been described.[39] Careful evaluation is needed as negative effects of remaining $Ca(OH)_2$ have been shown. According to previous studies, $Ca(OH)_2$ remnants on root canals is undesirable clinically as it leads to decreased sealer bond strength and increased risk of apical leakage of gutta-percha obturation when zine oxide eugenol sealer is used.[82,83] Ghabrai et al. al reported a negative effect of $Ca(OH)_2$ remnants on push out bond strength with AH-26 and BC sealer. $Ca(OH)_2$ was shown to prevent sealer from penetrating into the dentinal tubules.[84] Moreover, $Ca(OH)_2$ remnants also had a negative effect of bond strength of resin cement, even after phosphoric acid irrigation.[85]

SUMMARY AND CONCLUSION

Within the limitations of this *in-vitr*o study, the following conclusions were drawn:

1. Tubli-Seal has better wettability on dentin than BC sealer.
2. Interaction between sealer, treatment type and duration was found to affect contact angle; however, careful evaluation is needed to generalize across other effects.
3. Interaction between treatment type and duration was found to affect surface element composition; however, careful evaluation is needed to generalize across other effects.
4. Two-minute irrigation with 6.0-percent NaOCl and 17-percent EDTA can remove calcium hydroxide from polished dentin surfaces.

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ABSTRACT

EVALUATION OF CONTACT ANGLE BETWEEN ROOT CANAL SEALERS AND DENTIN TREATED WITH CALCIUM HYDROXIDE AND IRRIGATION SOLUTIONS

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Background: Numerous studies have reported the effect of long-term use of calcium hydroxide $Ca(OH)_2$ to dentin. Nevertheless, there is little information available about the effect of $Ca(OH)_2$ on wettability to the dentin. Objective: To investigate the effect of $Ca(OH)_2$ application on dentin for two and four weeks on the wettability of two root canal sealers.

Methods: Polished caries-free human dentin discs (n = 156) were allocated into 12 groups; G1 and G3 had two weeks' treatment, G4 and G6, four weeks treatment. G1 and

G4 were treated with sterile water. G2, G3, G5 and G6 were treated with $Ca(OH)_2$. G1, G3, G4, and G6 were irrigated with 6.0-percent NaOCl and 17-percent EDTA while G2, and G5 were irrigated with sterile water. Then, contact angles between Tubli-Seal and the treated dentin surfaces were measured. G7 and G12 were treated in the same fashion but were treated with BC sealer. Surface morphology evaluation of G1 and G6 was carried out by scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX). Statistics were performed using three-way ANOVA and pair-wise comparisons between groups ($\alpha = 0.05$).

Results: Tubli-Seal (G1 through G6) had significantly smaller values for contact angles than BC sealer (G7 through G12) ($p < 0.05$). For the Tubli-Seal groups (G1 through G6), G4 had the highest mean of contact angles at 104.9 ± 1.9°, whereas G5 presented the lowest mean of contact angles at 85.4 ± 15.1. For the BC sealer groups (G7 through G12), G10 had the highest mean of contact angles at 145.4 ± 1.3°, while G11 demonstrated the lowest mean of contact angles at 130.2 ± 2.6°. Groups with $Ca(OH)_2$ treatment with water irrigation (G2, 5, 11) had significantly lower contact angle than groups with $Ca(OH)_2$ with chemical irrigation (G3, 6, 12) ($p < 0.05$), except G8, 9. According to SEM and EDX, water irrigation solution showed higher remaining $Ca(OH)_2$ than irrigation with the chemical solution while $Ca(OH)_2$ with chemical irrigation

demonstrated no $Ca(OH)_2$ remaining after irrigation, similar to the surface of the control group.

Conclusion: Within the limitations of this study, Tubli-seal has better wettability on dentin than BC sealer. Remaining calcium hydroxide demonstrated a trend toward decreased contact angle between dentin and root canal sealers. Moreover, two-minute irrigation with 6-percent NaOCl and 17-percent EDTA can remove calcium hydroxide from polished dentin surfaces.

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