Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation

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Title: Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation

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Abstract: Objective: Removal of obturating materials from the root canal during retreatment is usually accomplished by mechanical means and organic solvents to dissolve the gutta-percha. This strategy is ineffective on sealers. The aim of this research was to assess the efficacy of mechanical instrumentation aided by targeted chemical means for removal of hydraulic sealers.
Methods: A preliminary study assessed the effect of 17% EDTA and 10/20% formic acid applied for 5 or 10 minutes on dentine and for 5 minutes on Totalfill BC sealer (FKG). Microhardness and structural integrity by scanning electron microscopy and energy dispersive spectroscopy were investigated. The optimal solutions were used as adjuncts to mechanical instrumentation to remove obturations made with single cone using a standard gutta-percha and bioceramic coated gutta-percha and Totalfill BC sealer. The removal of obturation material from the root canal system of obturated teeth was evaluated using microcomputed tomography. The canal patency and reestablishment of working length were also checked.
Results: The preliminary study showed that 17% EDTA and 10% formic acid applied for 5 minutes did not damage the dentine but effected the structural integrity of the sealer. 10% formic acid used in conjunction with mechanical instrumentation was the most efficient method to remove the obturation material from the root canal, achieving over 95% removal for both gutta-percha and the bioceramic coated version and also achieve patency and reestablishment of working length.
Conclusions: The use of a targeted irrigation protocol with a chemical adjunct to the mechanical instrumentation is the best way to retreat teeth obturated with hydraulic calcium silicate sealer and gutta-percha

Cover Letter

UNIVERSITY OF BIRMINGHAM

SCHOOL OF DENTISTRY

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22nd February 2020

Christopher D. Lynch
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Dear Prof Lynch,

We would like to resubmit the manuscript entitled "**Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation**" to Journal of Dentistry for peer review.

We feel that this work investigates a valid clinical problem and thus its publication would be timely. We have used a targeted approach and use chemical adjuncts to the mechanical instrumentation. We have added the specific chemistry of the acid in the revised manuscript.

We look forward to a positive outcome. We declare no conflict of interest. This manuscript is not under consideration elsewhere and the work performed is solely of the authors mentioned.

Sincerely,

Josette Camilleri

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Response to reviewer comments

Reviewers' comments:

Reviewer #1: This interesting topic was well researched by the authors. However, they only identify their irrigant as a "weak organic acid". This is unacceptable. If you want to be vague- get a patent then publish.

Also, the discussion is weak with no comparisons to others. No comments were made on the bioceramic cones. The paper gives the impression of being rushed and not completely thought out.

Their writing needs copious simplification and clarification. Comments are attached. Please keep the subject and verb nearby, which makes it easier for non-native English speakers. Use shorter sentences, please.

Reply

I appreciate you find the withholding of information unacceptable. We had informed the editor in chief that we will divulge the name and chemistry of the solution once the manuscript has been reviewed. There is nothing to patent. It is information that is freely available to all who bother to read. The manuscript has been updated based on your comments. Thank you for your time and for your constructive comments. Discussion rewritten.

Reviewer comments

Reviewer #2:

Introduction:

The introduction is redundant, while only the last paragraph really focused on the problem to be solved. There have been some publications on this topic. Previous results of various ways to remove bioceramic sealer should be discussed in detail, advantages and drawbacks, which necessitate the present research.

M & M: What kind of acid was used? Please give brief introduction of the so-called weak organic acid about its basic physical and chemical properties.

One experiment can be added to examine the interaction between set bioceramic sealer and acid solution with simple immersion test outside root canal to test the solubility, etc. That will be more convincing than the SEM image of the root canal surface and microhardness data shown in the result.

It was said that the best concentration and application time were determined in the previous experiments. If the previous results have been published, please add reference. If not, please give brief information in the introduction part.

Statistical analysis: "The Least Significant Difference (LSD) test was used to analyse the significance of void volume between the different sealer groups ($p \leq 0.05$), whilst the t-test was used to determine the significance of heating on void volume for each individual sealer group ($p \leq 0.05$)". Void volume is not relevant to this study.

Reply

Introduction rewritten. The solutions were chosen based on their chemistry and ability to chelate calcium. In previous research organic solvents and protocols based on softening and removing gutta-percha have been employed. Thus the lack of success.

We have decided to withhold this information but have informed the editor in chief when submitting the manuscript that we will add the relevant information once the manuscript goes in review. The testing of the effect of solutions has been included. Solubility of sealers as suggested by the reviewer was not considered suitable. Solubility of sealers is measured using ISO 6876;2012. This test is difficult to adapt to test the solubility in the test solutions since it requires the replacement of water with the test solutions. When the liquid is evaporated, unlike water residue is left behind thus accounting for an increase in weight which will thus give wrong data. Boiling off an acid is hazardous. Thus, we measured the microhardness as if the microhardness is reduced that will show that the sealer is losing strength which is an important factor if it needs to be removed from the canal wall by mechanical means. We also did microstructural analysis to support our findings. We imaged both the dentine and the sealers to show the effect of the solutions on both. It is important not to damage the dentine to much in the cleaning process.

All the work undertaken is shown here including the pilot study. Text modified for clarity.

Apologies for the mistake with the statistical analysis. Now modified.

Reviewer comments

Results:
Is the change of microhardness of sealer material after acid treatment significant? P value was not given in the result.
Please use column chart instead of line chart for Fig 1.
In Fig 2b, crack can be introduced by the SEM preparation but not EDTA treatment.
Discussion: What is the mechanism of acid softening of sealer material? Only action of acid on the surface?
Any adjunct methods can be used to improve efficacy of the acid solution?

Reply

Figure 1 has been converted to a column chart as suggested. The results section has been rewritten for clarity. Indeed SEM causes crack formation but the EDTA exacerbated this as the other specimens showed no cracks. The discussion has also been rewritten. At this stage we can only suggest the use of the 3 solutions in combination to the mechanical cleaning. I do not think this was evident from the figures. It has now been modified for clarity.

*Title Page with Author Details

Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation

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Key words: Single cone root canal obturation, hydraulic root canal sealers, canal preparation, retreatment

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~~Assessment of the r~~Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation ~~technique~~

Abstract

Objective: Removal of obturating materials from the root canal during retreatment is usually accomplished by mechanical means and organic solvents to dissolve the gutta-percha. This strategy is ineffective on sealers. The aim of this research was to assess the efficacy of mechanical instrumentation aided by targeted chemical means for removal of hydraulic sealers. ~~Failure of root canal treatment results in the need to remove the obturation materials from the root canal system in order to carry out re-treatment. The gutta-percha is removed from the root canals by mechanical means, but removal of hydraulic sealers has the potential to be more problematic due to their interaction with the dentine.~~

Methods: A preliminary study assessed the effect of 17% EDTA and 10/20% formic acid applied for 5 or 10 minutes on dentine and for 5 minutes on Totalfill BC sealer (FKG). Microhardness and structural integrity by scanning electron microscopy and energy dispersive spectroscopy were investigated. The optimal solutions were used as adjuncts to mechanical instrumentation to remove obturations made with single cone using a standard gutta-percha and bioceramic coated gutta-percha and Totalfill BC sealer. The ~~The effect of three solution types (17% EDTA and 10 or 20% concentration of weak organic acid) on dentine and TotalFill BC sealer was assessed. Microhardness and microstructure of both dentine and sealer were monitored and compared to a control; the chemical which caused the most damage to the sealer but preserved the dentine was selected. r~~Removal of obturation material from the root canal system of obturated teeth was evaluated ~~for efficiency through use of~~using microcomputed tomography. ~~Human roots standardized to~~

~~14 mm were chemo-mechanically prepared and obturated with gutta-percha or bioceramic coated gutta-percha and TotalFill BC sealer using a single cone obturation technique. After the sealer had set, the obturating material was removed by mechanical means using rotary instrumentation or by a combination of mechanical and chemical means using either 17% EDTA or 10% weak organic acid, followed by sodium hypochlorite and water irrigation.~~ The canal patency and reestablishment of working length were~~as~~ also checked.

Results: The preliminary study showed that 17% EDTA and ~~The results of the study show that the~~ 10% ~~organic~~ formic acid applied for 5 minutes did not damage the dentine but effected the structural integrity of the sealer. 10% formic acid used in conjunction with ~~accompanied by~~ mechanical instrumentation was the most efficient method to remove the obturation material from the root canal, achieving over 95% removal for both gutta-percha and the bioceramic coated version and also achieve patency and reestablishment of working length. ~~This solution did not damage the root canal dentine.~~

Conclusions: The use of a targeted irrigation protocol with a chemical adjunct to the mechanical instrumentation is the best way to retreat teeth obturated with hydraulic calcium silicate sealer and gutta-percha~~Removal of obturating material composed of hydraulic cement from the root canal was fast and efficient when using a combination of mechanical removal accompanied by a 5-minute application of an organic acid.~~

1. Introduction

When root canal therapy fails, the materials used to obturate the root canal need to be removed so the root dentine can be cleaned and disinfected. Most of the obturating material is gutta-percha that can be removed mechanically and also by the use of organic

solvents. The mechanical removal of obturating materials can lead to overcutting or the dentine thus ideally chemical means that dissolve the obturating material without destroying the dentine need to be employed. ~~Root canal treatment (RCT) includes the mechanical and chemical disinfection of the root canal system followed by obturation to seal the communication between the root canal and the peri-radicular environment. This seal is a vital factor for successful outcome of primary root canal treatment (1); the obturation should be well condensed and have no voids; which is achieved by effective use of both root canal sealer and obturation material.~~

Gutta-percha is dissolved well by organic solvents (1-3), but the sealers are more resistant to chemical dissolution (4). Hydraulic calcium silicate-based root canal sealers interact chemically with dentine (5) and are mostly used with single cone obturation technique (6).

~~Sealer degradation can cause the sealer-dentine or the sealer-gutta-percha interfaces to breakdown, allowing microbial contamination and treatment failure (2). However, if the primary RCT fails, subsequent mechanical and chemical disinfection must be carried out to remove all the obturation materials from the root canal system to allow for retreatment. The secondary RCT must then be carried out to a higher standard, to minimise risk of re-infection (3). This process can be challenging if the root canal sealer used is not soluble, as removing it from the canal walls becomes more challenging, thus affecting the outcome.~~

~~Hydraulic tricalcium silicate-based sealers have become increasingly popular due to their hydrophilic nature, where moisture is an advantage to their setting process, not a detriment like conventional sealers (4). These sealers also bond well to the root canal wall due to~~

~~mineral exchange at the interface (5). A recent survey (6) on the use of hydraulic sealers amongst endodontic professionals and general practitioners shows that 51.70% were using these materials due to their belief of the improved properties. The single-cone technique was the most employed method of obturation (63.3%), which was preferred mostly by the general practitioners, as endodontic specialists utilised more of the thermoplasticised obturation techniques. The single-cone obturation technique with hydraulic sealers shows an overall success rate of 90.9% with lesions <5 mm in diameter having a significantly higher success rate than lesions >5 mm in diameter. Although sealer extrusion was observed in 47.4% of the cases, this did not have any significant effect on the treatment outcome (7).~~

~~The use of hydraulic sealers with a single-cone obturation technique~~This means that the percentage of sealer is larger in a root canal, compared to other techniques.~~results in higher volumes of sealer being present in the root canal~~. ~~It is suggested that their performance can be optimised by using bioceramic coated gutta-percha cones which have a layer of tricalcium silicate and radiopacifier on their surface, thus enhancing the sealer bond to the core point, and therefore eliminating many, if not all, potential gaps between the core and sealer (4).~~ These factors ~~all provide a good quality obturation, and work to enhance the outcome of the initial treatment, however, they~~ make the retreatment ~~protocol tougher as it is~~ more difficult as it will be challenging to completely remove these materials from the canal walls.

The ability to remove all remnants of obturating material by various means has been investigated. ~~In a recent survey 55.6% considered that hydraulic cement use may influence their ability to re-establish apical patency during retreatment (6). In fact, r~~Re-establishing

patency ~~is~~was shown to be ~~indeed~~ challenging (7, 8), the working length was not regained (9) and a lot of obturating material was left behind after retreatment ~~particularly when using hydraulic cements~~ (10, 11). Removal of these sealers takes more time ~~as well~~ (11, 12) and c~~. C~~omplex anatomy led to more sealer being left behind (13).

~~The suggested r~~Removal of sealer has been ~~using~~ mechanical ~~means~~ (8-14) which can be augmented by the use of chloroform (8, 9), heat (9) and supplementary irrigation ~~techniques~~ (15). ~~The r~~Rotary instrumentation may increase the canal size and lead to unnecessary ~~destruction~~ removal of dentine. ~~Furthermore, all the techniques investigated so far have aimed at enhancing the gutta-percha removal such as chloroform and heat (8, 9). None of the~~No technique~~s~~ ha~~s~~ve addressed the chemical removal of the hydraulic sealer without damaging ~~the~~ dentine. The aim of this research was to assess the efficacy of the removal of the obturation materials; ~~the~~ gutta-percha and ~~, as well as the~~ hydraulic sealer~~,~~ from the canal walls using mechanical instrumentation, with and without chemical aids. Micro Computed Tomography (MicroCT) was used to quantify the remaining materials on the canal walls.

~~2.~~ Materials and methods

~~2.~~

~~Assessment of the volume of sealer left on the walls after chemo-mechanical removal was assessed by microcomputed tomography. 17% EDTA and 2 concentrations of a weak organic acid were proposed to aid the chemo-mechanical removal of sealer. The effect of the cleaning solutions of the both sealer and dentine was also determined and the best concentration of the weak organic acid was determined.~~

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2.1 ~~Effect of root canal cleaning regime on dentine properties~~Preliminary study to establish the retreatment protocol

For effective chemical cleansing and dissolution of the hydraulic sealer 3 solutions were proposed. These included:

- 17% ethylene diamine tetracetic acid (Cerkamed, Stalowa Wola, Poland)
- 10% formic acid (Sigma Aldrich, Gillingham, UK)
- 20% formic acid (Sigma Aldrich, Gillingham, UK)

All three solutions were employed for either 5 or 10 minutes dentine discs and for 5 minutes on 10 x 2 mm TotalFill BC sealer FKG Dentaire, La Chaux-de-Fonds, Switzerland) discs. ~~To determine the appropriate concentration of the weak organic acid and the effect of the solutions on the material and dentine properties, the microhardness and chemistry of the materials and dentine was assessed. Cylindrical specimens 10 mm in diameter and 2 mm high of TotalFill BC sealer (FKG Dentaire, La Chaux-de-Fonds, Switzerland) were prepared and allowed to set in moist environment. Transverse sections of root dentine were also prepared and polished. Both dentine and sealer were subjected to a 5 or 10 minute immersion in 17% EDTA, and 10 or 20% dilution of the weak organic acid.~~ The change in microhardness was ~~then~~ measured ~~using~~ by determining the Vickers hardness number ~~test~~ (n = 6). Furthermore, scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were performed to assess the microstructural changes caused by the solutions on both dentine and TotalFill BC sealer (n = 3).

2.2 ~~Tooth preparation~~ Assessment of efficacy of removal of obturation material from the root canal

Tooth preparation

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Thirty-six single rooted teeth extracted for orthodontic and periodontal reasons were selected ~~by a single operator~~. Ethical approval (14/EM/1128) was sought from the Birmingham Dental Hospital and Community Health Care to use human teeth. A long cone periapical radiograph was taken to verify that one singular straight canal was present. Teeth with curvature greater than 10° were discarded. The teeth were then de-coronated using a diamond disc and the total root length ~~of each~~ was adjusted to 14 mm. For each tooth, patency was achieved using an ISO size 10 stainless steel F~~f~~lexofile (Dentsply, Tulsa OK). The root canals were subsequently cleaned and shaped using rotary instruments (ProTaper Gold; PTG, Dentsply, Tulsa, OK) following the Protaper Technique (16). ~~Throughout the preparation, c~~Copious irrigation ~~of~~ with 2% sodium hypochlorite (NaOCl) was used following each instrument~~ation~~, ending with a final rinse of 17% ~~Ethylene-Diamine-Tetra-Acetic acid (~~EDTA~~)~~ followed by saline. The following irrigation protocol was used:

1. 2% NaOCl 6 mL/1 minute – after every instrument
2. 17% EDTA: 5 mL/3 minutes – at the end once preparation is complete
3. ~~Distilled water~~Saline: 5 mL of the final irrigant administered 1 mm from working length over 3 minutes.

The teeth were divided into two groups. Half were obturated using ~~For obturation, a~~ the single cone technique ~~using~~ with TotalFill BC sealer (FKG) and a single standard master cone of Gutta-Percha (GP) the same size as the master apical file. The other group was obturated in a similar way but the ~~, which was either~~ standard GP ~~or~~ was replaced with a bioceramic coated GP (FKG) ~~was used~~. ~~Following preparation using the protocol outlined above, the thirty-six teeth were divided into two groups. Group 1 was obturated using a standard GP master cone and TotalFill BC sealer, whereas Group 2 was obturated using a coated gutta-~~

~~percha cone and TotalFill BC sealer.~~ The obturated roots were then restored with flowable composite to block off the coronal access, and provide a coronal seal. The roots were then stored in Hank's balanced salt solution at 37°C for 2 weeks to allow the sealer to set.

~~2.3~~ Retreatment protocol

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The ~~thirty-six teeth~~two groups of obturated roots were then further divided into 3 sub-groups. One group was ~~cleaned~~ retreated only by mechanical instrumentation using ProTaper Gold finisher file that was the same size as the master apical file. The choice of this file ~~and GP to~~ avoided widening the canal and ~~also~~ unnecessary removal of dentine ~~from the canal walls~~. The other two groups were cleaned by mechanical cleaning accompanied by either EDTA or ~~the weak organic acid~~formic acid. The concentration of the formic acid and the immersion time was determined in Section 2.1. ~~(the best concentration and application time determined in the previous experiments).~~ The ~~final cleaning efficiency and~~ patency and the reestablishment of working length were~~as~~ then evaluated using MicroCT. ~~Reestablishment of working length was also checked.~~

~~2.4~~ MicroCT Scanning Protocol

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The roots were scanned with a microcomputer tomography machine (SkyScan 1172, Bruker, Coventry, UK) at three stages~~;~~ following root canal preparation, post obturation and ~~a final scan~~ after the root filling material had been removed ~~using the selected retreatment protocol~~. The scans were taken using 70 kV and 140 µA, at a pixel size of 11 µm (medium camera pixels setting was used). The X-rays were filtered with a 0.5 mm aluminium filter. Acquisition settings included a rotation step of 0.45°, frame averaging of 4, and random movement of 10. Flat field correction was updated ~~on the day,~~ prior to scanning ~~anything,~~ to

provide a uniform background for the scans. ~~Once scanned, the i~~Images were reconstructed using NRecon software using ring correction of 9, ~~and~~ beam hardening correction of 20% and an attenuation coefficient range of 0.004 to 0.17. For volume analysis, the reconstructed image dataset was opened in CTAn software and the volume of the obturation and ~~subsequent~~ remaining sealer on the canal walls was quantified using the programs binary selection view and region of interest feature. Binary selection was used to highlight the obturation material in white, ~~this was compared~~ to the ~~original~~ post-obturation image to ~~make~~ ensure that the material highlighted in white was representative. Once done, region of interest function was used to select the canal from the top to the bottom of the canal length and measure~~.~~ ~~This allowed the measurement of t~~the total volume of ~~the~~ obturation~~,~~ and the volume of sealer remaining on the canal walls. The percentage removal of the sealer was calculated.~~and hence the efficiency of each retreatment irrigant used was then found.~~

~~2.5~~*2.3 Statistical analysis*

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The statistical analysis was performed using Predictive Analytics Software (PASW) version 18. One-way ANOVA was used to determine whether ~~there were~~ significant differences were detected ~~between~~ among the data sets. The data was tested to ensure it was normally distributed ~~and then with~~ analysis of variance with P = 0.05 and the turkey post hoc test was used. ~~The Least Significant Difference (LSD) test was used to analyse the significance of void volume between the different sealer groups ($p \leq 0.05$), whilst the t-test was used to determine the significance of heating on void volume for each individual sealer group ($p \leq 0.05$).~~

~~3.~~3 Results

3.1 Preliminary study to establish the retreatment protocol ~~Establishing the Protocol for Re-Treatment~~

The results of the microhardness testing on both the sealer and dentine are shown in Figure 1. The dentine was adversely affected by 5 and 10 minute rinsing with 20% formic acid ($P = 0.016$ and $P < 0.0001$ respectively). Thus a 5-minute rinse of 1~~1~~0% ~~weak organic~~formic acid was selected to avoid structural changes to the dentine. ~~The sealer microhardness followed the same pattern to that of the dentine.~~ A 5-minute exposure to EDTA enhanced the sealer and dentine microhardness. ~~The canal cleaning protocol chosen to remove the sealer remnants from the root canal was thus selected and both 10% weak organic acid and the 17% EDTA were left in the canal for 5 minutes after which they were then flushed with NaOCl 3mL for 1 minute and distilled water 3mL for another minute. This chemical protocol accompanied the mechanical preparation of the root canal with rotary instrumentation.~~

The SEM images of the ~~surface of~~ dentine ~~samples~~ surfaces in Figure 2a ~~show the difference in how the irrigation affects the tooth. The~~illustrate how the EDTA removed the smear layer exposing the dentinal tubules. The formic acidic ~~solution~~ left the smear layer intact but the higher concentration resulted in dentine degradation.

The ~~images of the~~ sealer exposed to different solutions are shown in Figure 2b. The EDTA caused cracks ~~formation~~ to form and ~~some minor microstructural changes~~the surface carbonation evident with the control group was not present after soaking in EDTA. ~~The use of the acid at~~At both concentrations the acid dissolved the sealer and ~~led to the formation of~~ pores were observed. The calcium to silicon ratio~~s worked out from the~~from EDS analysis

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~~showed a lower calcium level~~ was lower when the formic acid was used compared to EDTA ~~and the~~or control.

For retreatment, based on the preliminary analysis, the clinical protocol to be adopted included 10% formic acid or 17% EDTA left in the canal for 5 minutes followed by flushing with 3 mL NaOCl for 1 minute and 3 mL saline for another minute. Both irrigating solutions were used as adjuncts to mechanical removal.

3.2 Assessment of efficacy of removal of obturation material from the root canal~~Assessment of Efficiency of Sealer Removal~~

The percentage material removed~~al~~ from the root canal using the three ~~cleaning protocols~~methods ~~-~~ is shown in Figure 3a. All the method~~sologies~~ used to clean the root canal had a similar efficacy (P = 0.141) but the 10% formic acid used for 5 mins removed the most amount of the obturation material both with standard and bioceramic coated gutta-percha ~~in both groups~~ (P > 0.05). The EDTA used in combination with mechanical cleaning was not very effective in removal of the conventional gutta-percha cone and sealer but successfully removed most of the bioceramic coated gutta-percha. ~~.~~ ~~The~~ ~~images of the~~ pattern of removal of obturating material from the root canal is shown in Figure 3b and the apical patency in Figure 3c. All methods ~~techniques employed managed to~~ remove~~d~~ all the obturating material at the apex ~~and~~ achieving apical patency~~-~~ and re-establishing the ~~was achieved~~w~~.~~ ~~W~~orking length~~.~~ ~~was re-established in all cases.~~

~~4.~~4 Discussion

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This research investigates a premixed calcium silicate-based sealer that is used in single cone obturation technique. The hydraulic sealers ~~When endodontic treatment fails, there is a need to remove all previous obturation material and hence clean out the canal system, shape it and then allow for a fresh obturation to be performed to provide the tooth with a new hermetic seal to prevent reinfection, apical pathology and hence re-failure of the treatment (3, 8). The newer hydraulic sealers such as the one used in this study – TotalFill BC (FKG)~~ have specific properties which make removal difficult with conventional methods (8-15). The mechanical instrumentation increases the risk of enlarging the canal and creating procedural errors. In the current study the last finisher file that was used to ~~finish~~ prepare the canal was used for retreatment and gutta-percha removal to ensure no further dentine preparation. The use of adjuncts to mechanical instrumentation would aid the complete removal of the obturation material. The use of organic solvents has been shown to be ineffective to remove root canal sealers (4). Thus, a targeted approach based on the knowledge of sealer chemistry was used. EDTA is a calcium chelator and formic acid has been shown to dissolve Portland cement used in the construction industry (17). The primary factor controlling the rate of dissolution is the solubility of the calcium salt of the acid and not the acid strength (17). The calcium formate formed by reaction of calcium silicate and formic acid is readily soluble. Formic acid is also used to demineralize dentine and is usually used at 10% concentration (18, 19). Thus, a preliminary study to determine the optimal concentration and immersion times was set up.

The previous methods reported the use of heat (9), mechanical means (9-11, 13, 14), organic solvents (8, 9) and irrigant activation methods (15). None of these methods targeted

the sealer chemistry which seemed to be a crucial factor as indicated in the current research. Heat (9) and organic solvents (8, 9) are suitable to remove gutta-percha (1-3) but have already been shown to be unsuitable to remove sealers (4). Irrigant activation methods (15) can only enhance the irrigation used but are not suitable to effectively remove all sealers form the root canal. The 20% formic acid was too aggressive as indicated by the reduction in microhardess of the dentine. The 10% formic acid used for 5 minutes provided a suitable method to dissolve the sealer but leave the dentine intact. Although the EDTA is a calcium chelator, it was only effective in removing the smear layer (17) and this resulted in an increase in microhardness rather than degradation. The 5 minute contact is also reasonably acceptable clinically. ~~The use of the weak organic acid is being suggested as the hydraulic cement is susceptible to acid degradation. In fact, this was shown clearly in the scanning electron micrographs of the sealer in contact with the acid where pitting and degradation was evident. Removal with EDTA was also attempted since the sealer is calcium based and EDTA is a calcium chelator. The EDTA was effective in removal of smear layer on the dentine (17) but did not deteriorate the cement physical and chemical properties. Irrigation time was also a factor to consider, clinically during treatment, a reasonable irrigation time would have to be chosen, 5 minutes in contact with the solution to dissolve the sealer was reasonable.~~

Micro-CT scanning was effectively ~~-~~used ~~to both~~ quantitatively and qualitatively to assess the pre-obturation canal preparation, the obturation and then the quantity of sealer removed. The latter two were ~~then~~ used to quantify the ~~percentage of the obturation and~~

~~hence the~~ sealer that was removed with each irrigation protocol used in combination with mechanical removal. ~~Studies have used m~~Micro-CT ~~in this way previously due to its~~is non-destructive and can be repeated on the same sample as reported previously ~~nature and also due to its ability to perform multiple scans on the same sample to assess the changes following different stages of treatment~~ (8-10, 12-15, ~~18~~21). ~~). In this study, the total obturation volume and remaining debris in the canal was quantified, and a percentage removal was calculated.~~ All the methods used removed most of the obturation material with no risk of further dentine destruction. Enlargement of the canal (11) still does not allow the complete removal of the sealer. ~~The results from this study are in agreement with the concept that m~~Mechanical cleaning alone is not enough and ~~.~~ EDTA was not very effective in removing the standard gutta-percha cones.

Re-establishing working length and patency ~~does~~ improves periapical healing rates significantly (8, 9) so this is an important ~~factor to achieve~~characteristic for retreatment protocol. ~~Qualitatively, the samples, once set did have an apical seal which restricted patency while removing the sealant and GP, which was a required feature of a good obturation (1, 8) and during the retreatment procedure, patency and working length of all samples in this study were re-established possibly due to the combination of the irrigation dissolving the sealer and the mechanical instrumentation of the hand files.~~

Conclusions

A chemo-mechanical method for removal of hydraulic sealer used in single cone obturation technique is ~~being~~ proposed. More than 95% of obturating material was removed without

damaging the dentine by using 10% ~~solution of an organic~~formic acid ~~together~~ with mechanical preparation.

Declaration

The authors declare no conflict of interest.

References

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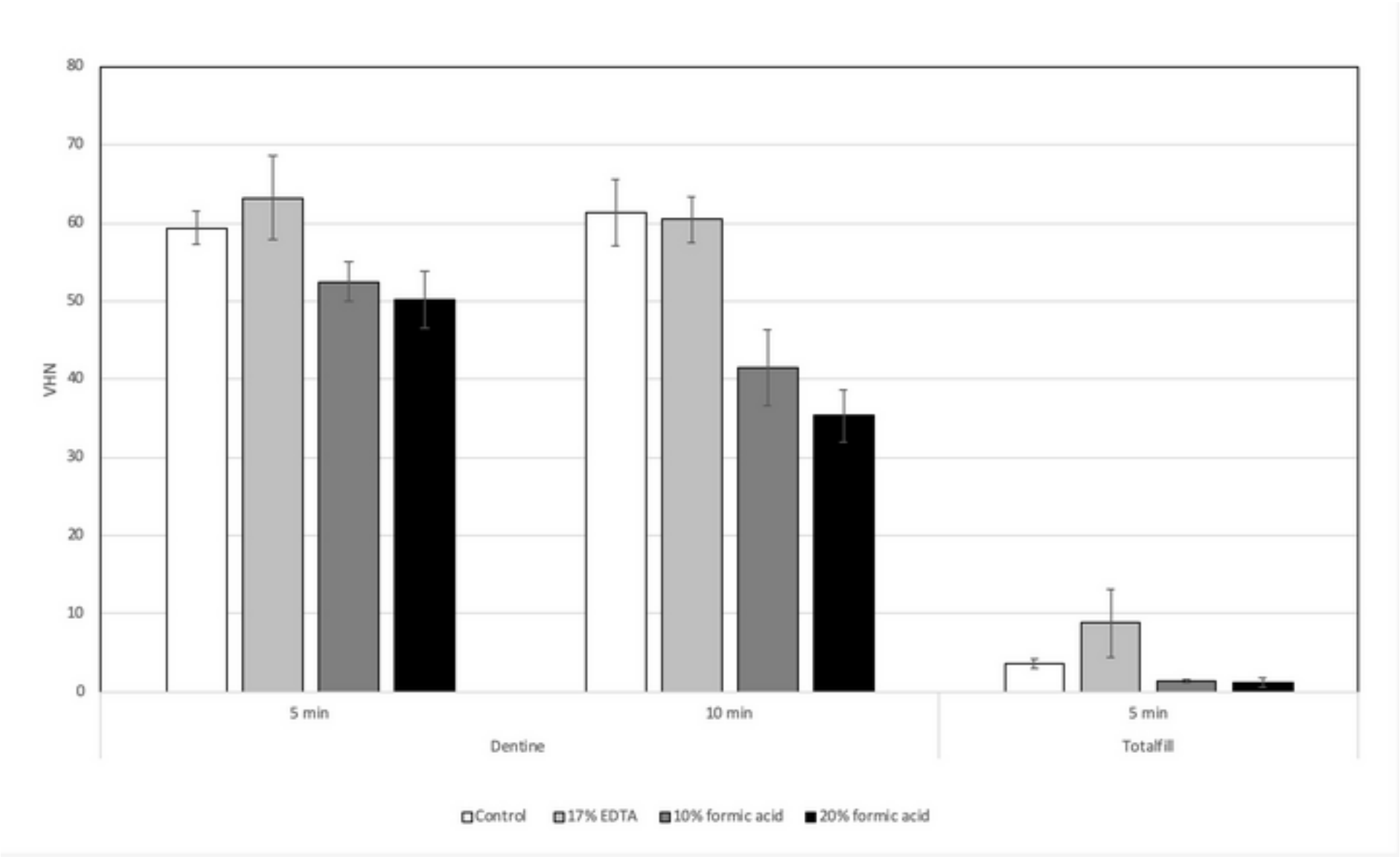
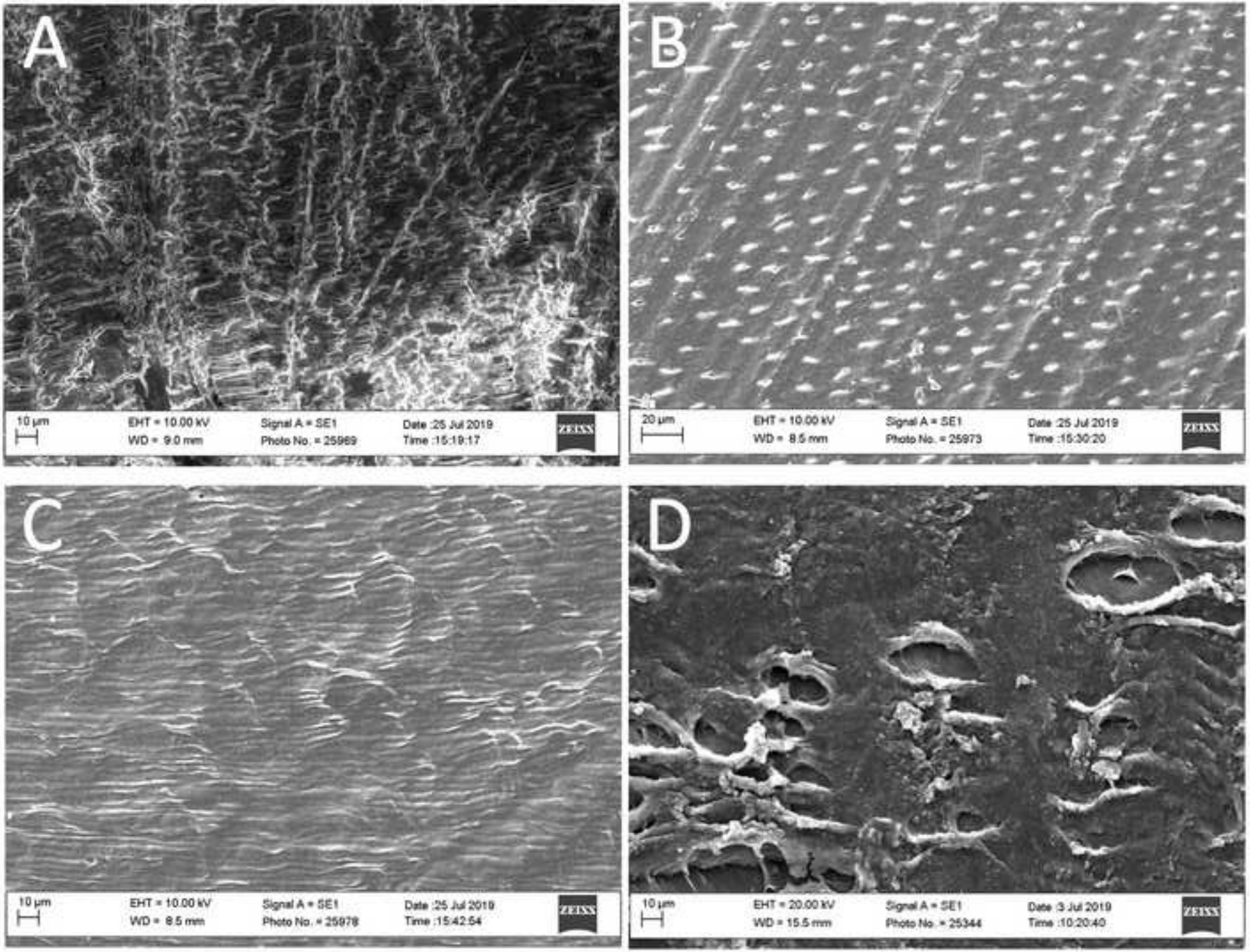
List of figures

Figure 3c: Microcomputed tomography images of the apical portion of roots after removal of obturating material using (A) mechanical means; (B) mechanical removal with 17% EDTA; (C) mechanical removal and 10% ~~weak organic~~formic acid showing the apical patency.

Figure


80
70
60
50
40
30
20
10
0
VHN
5 min
10 min
5 min
Dentine
Totalfill
Control
17% EDTA
10% formic acid
20% formic acid

Figure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WD = 9.0 mm
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20 µm
EHT = 10.00 kV
WD = 8.5 mm
Signal A = SE1
Photo No. = 25973
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Time :15:30:20
C
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EHT = 10.00 kV
WD = 8.5 mm
Signal A = SE1
Photo No. = 25978
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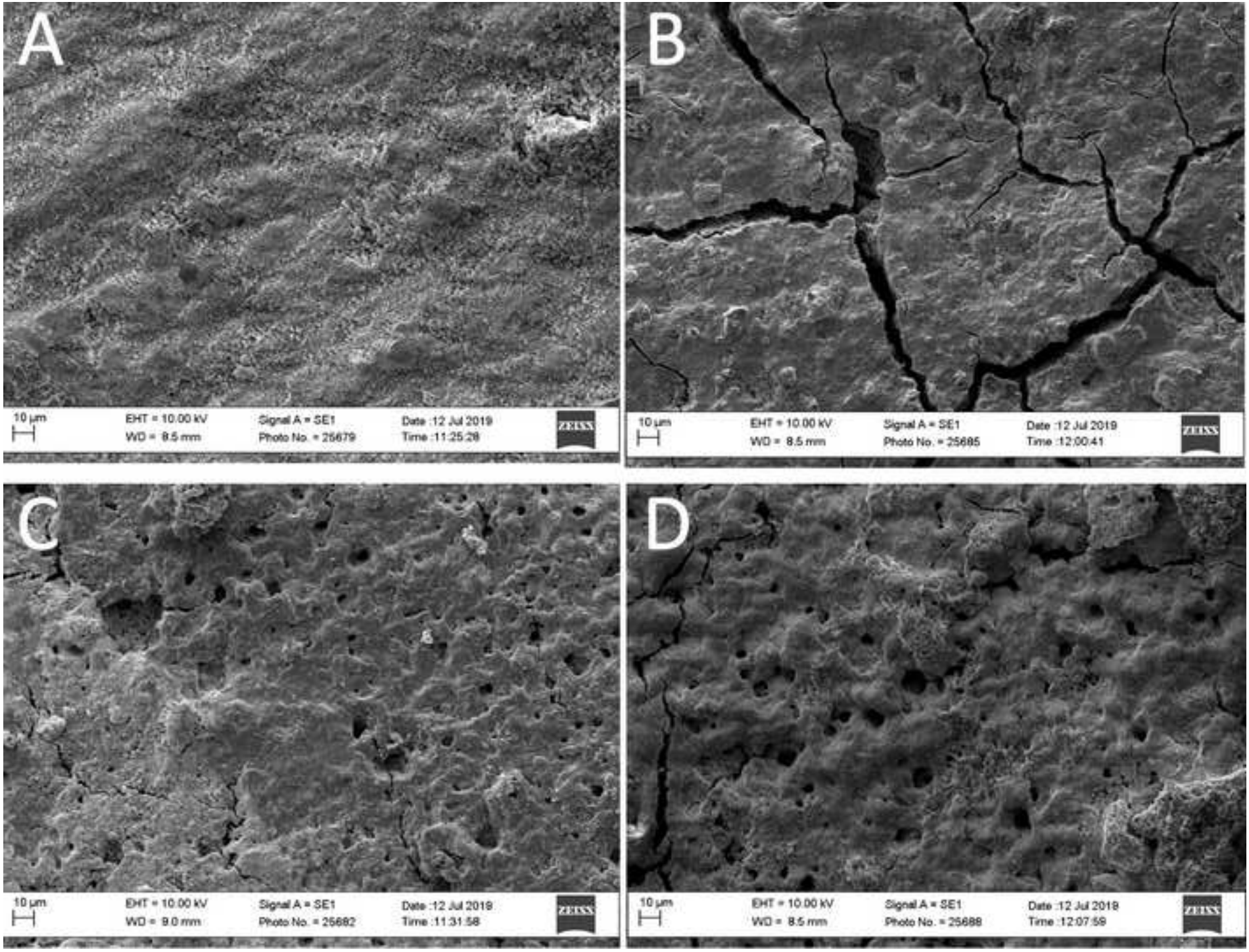
Figure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WD = 8.5 mm
Signal A = SE1
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Time :12:00:41
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WD = 9.0 mm
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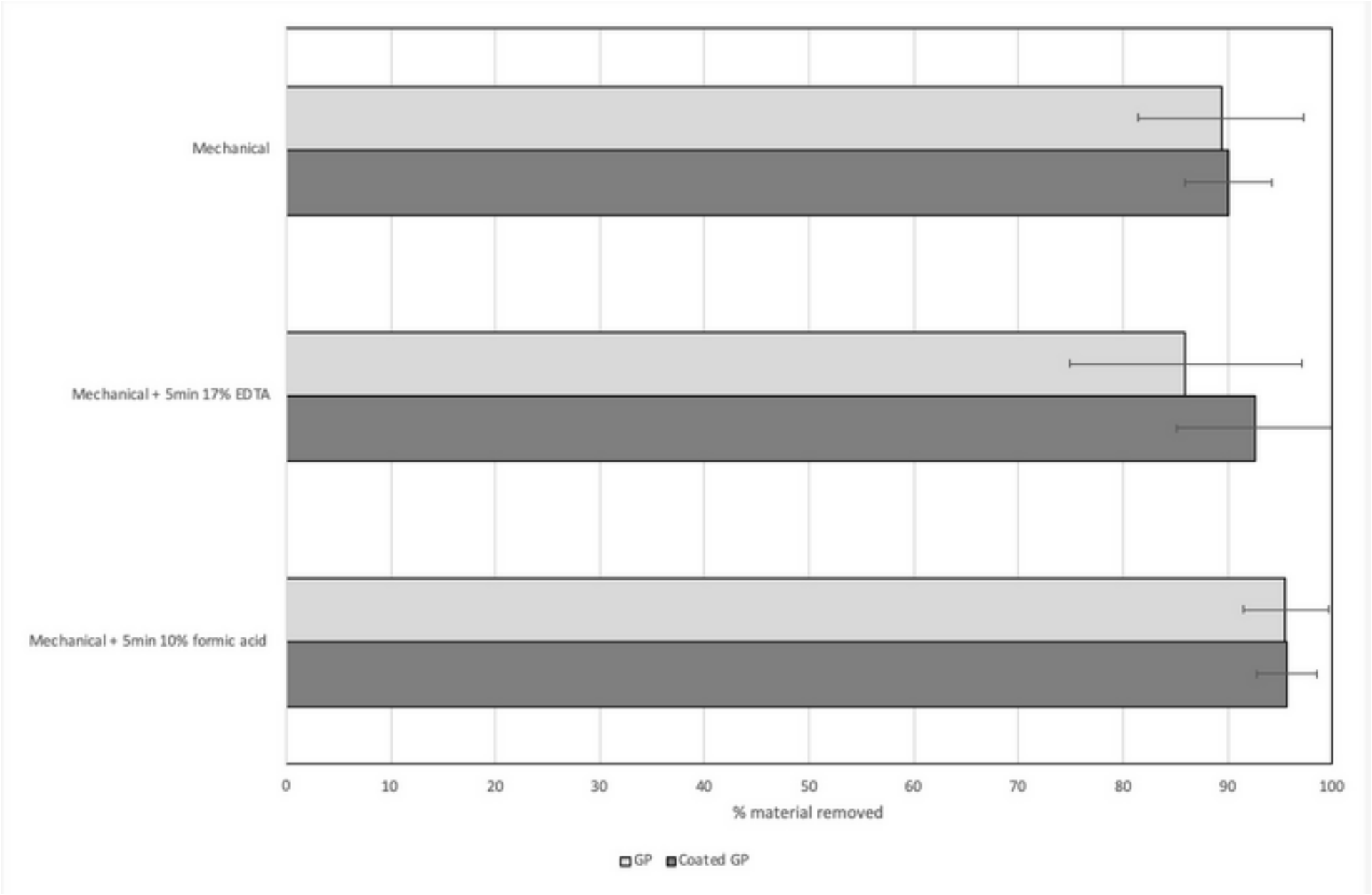
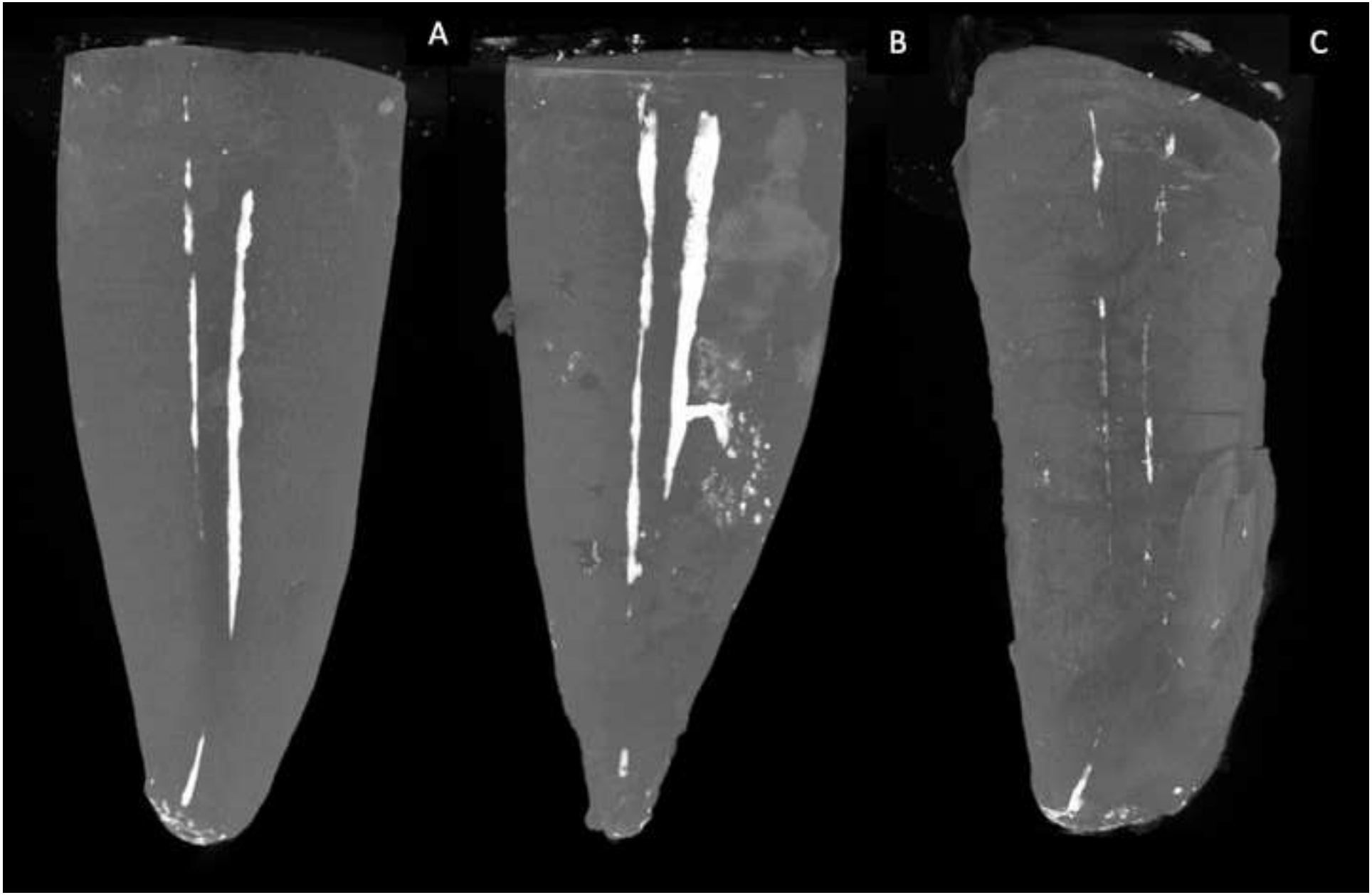
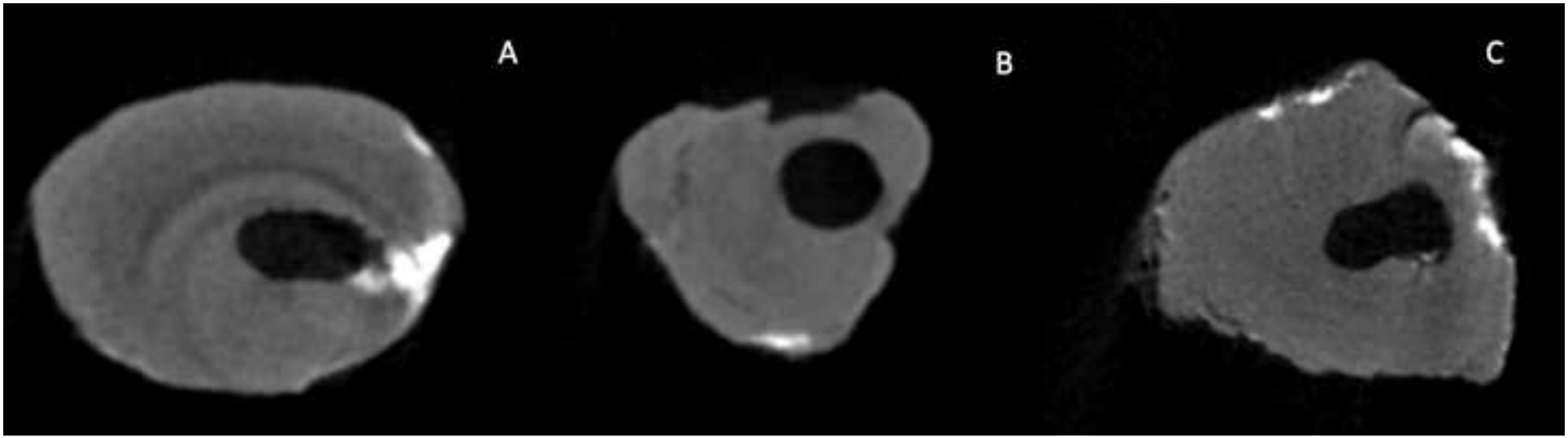
Figure


Mechanical
Mechanical + 5min 17% EDTA
Mechanical + 5min 10% formic acid
0
10
20
30
40
50
60
70
80
90
100
% material removed
GP
Coated GP

Figure



Figure



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***Permission Note**

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22nd February 2020

Dear Prof Lynch,

We confirm that this paper has been submitted solely to Journal of Dentistry and it is not concurrently under consideration for publication in another journal. The submitted work including images, are original.

Mohamed Garrib

Josette Camilleri

Conflict of interest statement

The authors declare no conflict of interest

Credit author statement

Mohammed Garrib: Investigation; Formal analysis; Methodology; Writing - original draft;

Josette Camilleri: Conceptualization; Formal analysis; Methodology; Supervision; Validation; Writing - review & editing.