# THE INTERFACE OF GLASS IONOMER SEALER AND CONDITIONED ELEPHANT TUSK DENTINE: A SCANNING ELECTRON MICROSCOPY STUDY

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#### ABSTRACT

The purpose of this in vitro study was to characterize the interface of glass ionomer sealer and dentine conditioned with common endodontic irrigants through scanning electron microscope.

The interface between Ketac-Cem radiopaque glass ionomer sealer and elephant tusk dentine conditioned with distilled water, citric acid and phosphoric acid was investigated. Cylinder of glass ionomer sealer was formed on the conditioned dentine surface and allowed to set for 60 minutes. The specimens were first shear tested to failure to determine their adhesive bond strength. Three debonded dentine specimens from each group were then processed for scanning electron microscopic study. The test failed surface of the strongest, intermediate and weakest bond strength specimens were examined. The specimens were cross sectioned and the interface was examined. The microscopic detail of the debonded interfaces between glass ionomer sealer and dentine was assessed in this study. Failures in all of the specimens were found to be mainly cohesive in nature.

**Key Words:** *Ketac-Cem radiopaque sealer, Elephant tusk dentine, Interface, Smear layer, Scanning electron microscope.* 

#### INTRODUCTION

Infection of the root canal system can lead to periradicular lesion. Such infection can occur as a result of interaction between the bacteria, bacterial products and host tissues.<sup>1</sup>The objective of a root canal treatment is to protect the periradicular tissues from their effects.<sup>1</sup>This can be done by filling the root canal space to remove the existing micro-organisms and also

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to prevent their recolonization. However, conventional root canal procedures probably do not eliminate all intra-canal micro-organisms and some part of the root canal may be infected at the time of root canal filling.<sup>2</sup> Therefore, a root filling material must have a continuous antimicrobial activity in order to remove residual microorganisms and also to resist the ingress of the new microbial agents. However, this antimicrobial activity must not compromise the biocompatibility of the material.

A desired property of a root canal sealer is to have adhesive strength both to the dentine and to the core material which is gutta purcha. The sealer should also have cohesive strength to hold the obturation together.<sup>3</sup> Between the different types of sealers available for root canal filling, the glass ionomer sealer has shown the ability to bond chemically to dentine with excellent flow <sup>4</sup>, antimicrobial activity <sup>4</sup> and biocompatibility.<sup>5,6</sup> In endodontics the role of smear layer has been the subject of significant debate since it was first discov-

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ered in 1975.<sup>7</sup> It was found that smear layer prevents the penetration of root canal sealer inside the dentinal tubules.<sup>8</sup> Thus the removal of smear layer has been recommended to improve the obturation seal <sup>9</sup> and reduce the coronal leakage.<sup>10, 11</sup>

The scanning electron microscope uses a focused beam of high energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the specimen including external morphology, chemical composition, crystalline structure and orientation of materials making up the specimen. The specimens must be solid and they must fit into the microscope chamber.<sup>12</sup>

The objective of this scanning electron microscopic study was to characterize the nature of failure of glass ionomer sealer adhesion to dentine and to examine the interface between the glass ionomer sealer and dentine conditioned with the endodontic irrigant.

## METHODOLOGY

In this study the elephant tusk dentine was prepared in the form of discs of diameter 2.4mm thickness with a hole of 1.6 mm in the centre. The hole was made with the help of 1.6mm smooth drill bit being held in a bench lathe machine. The fragment of elephant tusk is referred as ivory and is composed of dentine. The elephant dentine was chosen because it is not only micro structurally similar to human dentine but also the tubule density and mineral content appear to be similar to human dentine. However, there are some differences between the elephant human dentine like in elephant dentine the tubules are more elliptical in shape and the peritubular cuff is small or non-existent as compared to human dentine. In the present study Ketac-Cem radiopaque glass ionomer sealer is used. Its liquid is clear and had a low viscosity. The powder is white and its coarse particles were distinguished during spatulation.

Immediately before experimentation, the smear layer was produced on the dentinal surface with #600 silicon carbide paper and barbed broaches under water irrigation. The specimens were then randomly assigned into three groups of ten specimens each (Group 1, 2 and 3). Specimens of group 1 were cleaned and irrigated with distilled water and was placed as a control group; Specimens of group 2 were cleaned and irrigated with 6% citric acid; Specimens of group 3 were cleaned and irrigated with 35 % phosphoric acid. Group 2 and 3 specimens were placed in experimental groups. Each conditioning medium was directly syringed onto the dentine for over a period of 20 seconds. Paper point was then used to dry the dentine. After the sealer was mixed following the manufacturer instruction, standardized cylinders of the sealer were formed on the dentine surfaces. The sealer was allowed to set and appropriate method and time was given to ensure complete setting of the sealer before being tested for the shear strength. According to manufacturer instructions approximately 60 minutes were given to the sealer to set. The specimens were placed in distilled water to avoid dehydration. Immediately before testing it for the shear strength the specimens were then carefully removed from the distilled water and shear tested to failure in an Instron Model 5584 Universal testing machine. The shear bond strength value was determined in megapascals (MPa). The mean and standard values were calculated for each group of 10 specimens. One factor ANOVA test was used to compare the shear bond strength of the glass ionomer sealer to the three conditioning protocols.

The debonded dentine specimens were separated and three specimens from each group were selected for scanning electron microscopic examination. One specimen from each group that demonstrated the strongest bond, the other with the intermediate bond and third specimen with the weakest bond was taken and each specimen was cross sectioned into two equal parts with a lath cut machine and the interface was examined.

Each specimen was then carefully mounted onto a metal disc. The specimens were attached to it through a double tape. Each of specimens was then sputtercoated with gold in Agar auto sputter coater coating unit. The specimens were then observed under a scanning electron microscope (Quanta 3D FEG) at an operating voltage of 20 KV at high vacuum and photographed at a magnification of x100, x400 and x1000. The debonded surfaces were examined to indicate the mode of failure: adhesive (at the dentine/sealer interface), cohesive (within the sealer) or a combination of the two.

#### RESULTS

The shear bond strength of Ketac-Cem sealer was measured for various elephant dentine samples being treated with common endodontic irrigants before cross sectioning for the scanning electron microscope ex-

TABLE 1: MEAN SHEAR BOND STRENGTH VALUES OF DIFFERENT GROUPS   Bond Strength (MPa/Conditioning Media)						
Sealer	Dona Streng	P-Value				
Ketac-Cem Sealer	Distilled Water (Group 1)	Citric Acid (Group 2)	Phosphoric Acid (Group 3)			
Means (MPa±SD Values	0.431 <u>+</u> 0.271	1.072 <u>+</u> 0.267	$1.130 \pm 0.318$	<0.0001		

TABLE 2: SUMMARY OF ONE WAY ANOVA TEST

Groups	Group 1 N=10	Group 2 N=10	Group 3 N=10
Mean (MPa)	0.4310	1.0720	1.130
SD Value	0.271	0.267	0.318
Maximum Value	0.97	1.40	1.50
Minimum Value	0.15	0.60	0.50
95% Confidence Interval for mean	0.26	0.90	0.96

amination. The mean shear bond strength values are given in Table 1.

The results showed that shear bond strength of group 3 was significantly (P < 0.0001) higher than that of group 1. The difference between group 2 and group 3 was not significant. In the groups with the smear layer removed bond strength ranged from 0.267-1.130 MPa. The mean bond strengths for samples treated with phosphoric acid (1.13MPa) and citric acid (1.07 MPa) were similar and showed a significant difference from those treated with water (0.43 MPa). The results of ANOVA test has been summarized in Table 2.

Scanning electron microscopic images of specimens in all three groups showed crack formation either within the dentine or the glass ionomer surface. The cracks may have occurred due to either dehydration of the specimen or may have occurred during the shear force testing.

In control group 1 the dentine specimen showed the retained smear layer with smear layer plugs formed in the orifice of some of the dentinal tubules. The specimens also showed small amount of glass ionomer attached to the dentine surface with visible cracks (Figure 1).

In the experimental group 2 the specimen showed sufficient amount of glass ionomer sealer being attached to the dentine surface but cracks were visible on the dentine surface as well as within the sealer material (Figure 2).

In the experimental group 3 the specimen showed adequate amount of glass ionomer sealer being

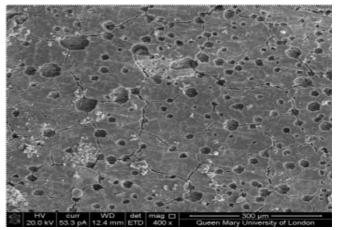


Fig 1: Scanning electron microscope images of the control group 1 specimen treated with distilled water seen at magnification of x400

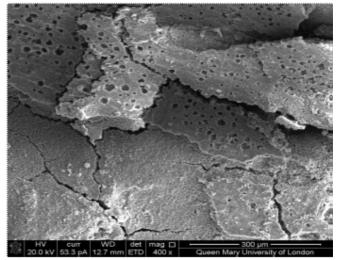


Fig 2: Scanning electron microscope images of the experimental group 2 specimen treated with 6% citric acid seen at magnification of x400

attached to the dentine surface with minimum visibility of the underlying dentine surface but few cracks were also visible on the surface within the sealer (Figure 3).

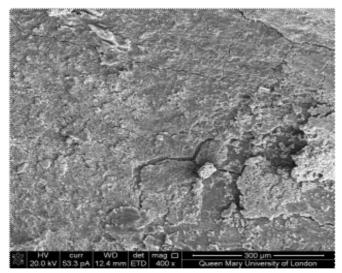


Fig 3: Scanning electron microscope images of the experimental group 3 specimen treated with 35% phosphoric acid seen at magnification of x400

Examination with scanning electron microscope showed that the sealer penetration into the dentinal tubules is obstructed due to the presence of smear layer. The successful application of 6% citric acid and 35% phosphoric acid had removed the smear layer and allowed the sealer to penetrate the dentinal tubules.

When surface characteristics was examined, no significant difference was noted among the specimens treated with phosphoric or citric acid except for the patches of an amorphous substance that was seen on the dentine surface below the glass ionomer sealer.

The results revealed that all of the shear tested failures were principally cohesive in nature i.e. within the sealer material and this indicates a good adhesion between the sealer and the dentine surface.

# DISCUSSION

Using scanning electron microscopy, various studies on adhesion of endodontic sealers have concluded that micromechanical retention by penetration of sealer tags inside the tubules may not be an important factor affecting the adhesion of root canal sealers.<sup>13</sup> Actually the adhesion of endodontic sealer to root dentine is a complex process with a different mechanism for various sealers and different dentine surfaces. Glass ionomer sealer binds through physical and chemical interaction with the dental tissue. The physical interaction mainly consists of micromechanical interlocking between the material and the tooth surface irregularities. <sup>14</sup> The chemical interaction occurs by binding of glass ionomer cement to hydroxyapatite and collagen in which the polyacrylate ions irreversibly displaces the phosphate ions from the hydroxyapatite surface and forms hydrogen bonds between free carboxylic and amino groups of the collagen and carboxylic groups of glass ionomer cement. <sup>15</sup>

In the current study, the scanning electron microscopic examination of the debonded surfaces of the dentine specimens revealed the failure to be principally cohesive in nature. This indicates that good adhesion has occurred between the sealer and the dentine surface. When surface characteristics was examined, no significant difference was noted among the specimens treated with 35% phosphoric or 6% citric acid except for the patches of an amorphous substance that was seen on the dentine surface below the glass ionomer sealer. The results of this study correlates with the work done by Lalh *et al* who found that failure in all of the dentine specimens were mainly cohesive in nature. <sup>16</sup>

When the root canals are instrumented an endodontic smear layer is formed on the surface of dentinal walls. It was also observed by Kousav *et al*<sup>8</sup> that smear layer obstructs the penetration of sealer tags into the dentinal tubules. The present study confirms these findings, as scanning electron microscopic pictures revealed that sealer was more penetrated and covered most of the dentine surface which were conditioned with citric acid or phosphoric acid as compare to that of dentine surfaces being treated with water.

In scanning electron microscopy at low magnification level the smear layer has a typical amorphous structure and has a globular pattern at a higher magnification. <sup>15</sup> It has also been proposed that the bond that develops basically between the material and the smear layer and not with the underlying mineralized dentine matrix. <sup>17</sup> Pre-treatment of dentine with phosphoric acid or citric acid was found to be more effective than EDTA in removing the smear layer. <sup>18, 19</sup>

The point that the failure of the shear tested specimens was seen in the form of cracks mainly through the sealer rather than at the dentine-sealer interface showed that adhesion of the glass ionomer sealer to dentine is stronger than the sealer itself. Clinically this fact suggests that there may be a reduce chance of troubling the seal of the root canal after it has set as compare to those materials which has less adhesion to dentine. However, this possibility is speculative and further investigation is required before we can make a definitive statement.

### CONCLUSION

Within the limitation of this study the following conclusions were made:

- The glass ionomer sealer showed good adhesion to the elephant tusk dentine.
- The observed bonding failure was mainly cohesive in nature.

#### REFERENCES

- 1 Nair PNR, Sjogren U, Krey G, Kahnberg KE, Sundqvist G. Intraradicular bacteria and fungi in root filled, asymptomatic human teeth with therapy resistant periapical lesions: a long term light and electron microscopic follow-up study. J Endod 1990; 16: 580-88.
- 2 Sjogren U, Figdor D, Persson S, Sundqvist G, Influence of infection at the time of root filling on the outcome of endodontic treatment of teeth with apical periodontitis. Int Endod J 1997; 30: 297-306.
- 3 Spångberg LSW. Instruments, materials and devices. In: Cohen S, Burns RC, eds. Pathways of the pulp. 7th ed. St. Louis: Mosby, 1998: 510.
- 4 Shalhav M, Fuss Z, Weiss El. In vitro antibacterial activity of glass ionomer endodontic sealer. J Endod 1997; 23: 616-19.
- 5 Kolokuris I, Beltes P, Economides N, Vlemmas I. Experimental study of the biocompatibility of a new glass-ionomer root canal sealer (Ketac-Endo). J Endod 1996; 22: 395-98.
- 6 Hajmiragha H, Nokar S, Alikhasi M, Nikzad S, Dorriz H. Solubility of three luting cements in dynamic artificial saliva. Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran 2008; 5(3): 95-98.

- 7 McComb D, Smith DC. A preliminary scanning electron microscopic study of root canals after endodontic procedures. J Endod 1975; 1: 238-42.
- 8 Kouvas V, Liolios E, Vassiliadis L, Parissis-Messimeris S, Boutsioukis A. Influence of smear layer on depth of penetration of three endodontic sealers: an SEM study. Endod Dent Traumatol 1998; 14: 191-95.
- 9 Cergneux M, Ciucchi B, Dietschi JM, Holz J. The influence of the smear layer on the sealing ability of canal obturation. Int Endod J 1987; 20: 228-32.
- 10 Saunders WP, Saunders EM. The effect of smear layer upon the coronal leakage of gutta-percha fillings and a glass ionomer sealer. Int Endod J 1992; 25: 245-49.
- 11 Violich DR, Chandler NP. The smear layer in endodontics a review. Int Endod J 2010; 43: 2-15.
- 12 Susan Swapp. An article 'Scanning electron microscopy'. Integrating research and education: Geochemical instrumentation and analysis: Science education research centre 2012.
- 13 El-Askary FS, Nassif MS, Fawzy AS. Shear bond strength of glass-ionomer adhesive to dentin: effect of smear layer thickness and different dentin conditioners. J Adhes Dent. 2008 Dec; 10(6): 471-79.
- 14 Shen C, Anusavice KJ. Philip's science of dental materials. 10th ed. Philadelphia: W. B. Saunders Co 1996: 535-42.
- 15 Akinmade AO, Nicholson JW. Review: Glass-ionomer cements as adhesives. Part I. Fundamental aspects and their clinical relevance. J Mater Sci 1993; 4: 95-101.
- 16 Manjinder S. Lalh, Keith C, Calvin D. Torneck and Shimon Friedman. Scanning electron microscopic study of the interface of glass ionomer cement sealers and conditioned bovine dentin. J Endod 1999; 25(11): 743-46.
- 17 Pashley DH, Tao L, Boyd L, King GE, Horner JA. Scanning electron microscopy of the substructure of smear layers in human dentine. Arch Oral Biol 1988; 33: 265-70.
- 18 Timpawat S, Harnirattisai C, Senawongs P. Adhesion of a glass ionomer root canal sealer to the root canal wall. J Endod 2001; 27: 168-71.
- 19 Haragushiku GA, Sousa-Neto MD, Silva-Sousa YT, Alfredo E, Silva SC, Silva RG. Adhesion of endodontic sealers to human root dentine submitted to different surface treatments. Photo medicine and Laser Surgery 2010; 28(3): 405-10.