

# Efficacy of various cleansing techniques on dentin wettability and its influence on shear bond strength of a resin luting agent

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**PURPOSE.** To evaluate the shear bond strength of resin luting agent to dentin surfaces cleansed with different agents like pumice, ultrasonic scaler with chlorhexidine gluconate, EDTA and the influence of these cleansing methods on wetting properties of the dentin by Axisymmetric drop Shape Analysis - Contact Diameter technique (ADSA-CD). **MATERIALS AND METHODS.** Forty coronal portions of human third molar were prepared until dentin was exposed. Specimens were divided into two groups: Group A and Group B. Provisional restorations made with autopolymerizing resin were luted to dentin surface with zinc oxide eugenol in Group A and with freegenol cement in Group B. All specimens were stored in distilled water at room temperature for 24 hrs and provisional cements were mechanically removed with explorer and rinsed with water and cleansed using various methods (Control-air-water spray, Pumice prophylaxis, Ultrasonic scaler with 0.2% Chlorhexidine gluconate, 17% EDTA). Contact angle measurements were performed to assess wettability of various cleansing agents using the ADSA-CD technique. Bond strength of a resin luting agent bonded to the cleansed surface was assessed using Instron testing machine and the mode of failure noted. SEM was done to assess the surface cleanliness. Data were statistically analyzed by one-way analysis of variance with Tukey HSD tests ( $\alpha=.05$ ). **RESULTS.** Specimens treated with EDTA showed the highest shear bond strength and the lowest contact angle for both groups. SEM showed that EDTA was the most effective solution to remove the smear layer. Also, mode of failure seen was predominantly cohesive for both EDTA and pumice prophylaxis. **CONCLUSION.** EDTA was the most effective dentin cleansing agent among the compared groups. [J Adv Prosthodont 2012;4:139-45]

**KEY WORDS:** EDTA; Dental bonding; Microscopy; Electron; Scanning; Wettability; Zinc-oxide Eugenol cement; Shear strength

## INTRODUCTION

Residual provisional cements and debris on prepared abutment teeth may negatively influence the performance of the definitive luting agent.<sup>1</sup> Apart from the choice of restorative materials, clinical outcome may be influenced by factors such as tooth preparation, preparation coarseness, type of luting agent, fit of restoration, type of provisional cement and also cleansing techniques used to remove the remnants of provisional cements.

Indirect restorations usually require temporization for protection of the pulp and to restore the patients esthetic and functional needs. Terata<sup>2</sup> showed that both residual zinc oxide eugenol and non-eugenol containing temporary cements reduced the tensile bond strength of resin luting agents. For this reason, it is imperative to remove any remnants of the provisional luting agent.

Grasso *et al.*<sup>3</sup> showed that pumice cleansing was known to be more effective than other cleansing techniques such as explorer/air-water technique or with 0.12% chlorhexidine gluconate.

Hülsmann *et al.*<sup>4</sup> showed that 17% EDTA solution has a good cleaning effect on the root canal walls and removes the smear layer by dissolving the inorganic components. This ensures better penetration of resin and subsequently increases the shear bond strength values.

Adhesion involves intimately joining two materials and the contact may be physical or chemical. For this reason, the resin must wet the dentinal surface to produce sound adhesion. The manner in which liquid spreads on a surface expresses the wettability of the surface. High wettability provides intimate contact and enhanced adhesion.<sup>5</sup>

Contamination of provisional cement on dentin surface

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may interfere with the spreading and penetration of resin through the dentinal tubules.<sup>6</sup> To increase the bond strength of resin, acids have been used to demineralize the dentin surface and to remove the subsequent debris and remnants of provisional cements present in it.

This *in vitro* study was aimed to evaluate 1) the shear bond strength of resin luting agent to dentin surfaces cleansed with different agents like pumice, ultrasonic scaler with chlorhexidine gluconate, EDTA. In addition mode of failure was observed under scanning electron microscope, 2) the influence of these cleansing methods on wetting properties of the dentin by Axisymmetric drop Shape Analysis - Contact Diameter technique (ADSA-CD), and 3) cleanliness of resulting dentin surfaces under a field emission scanning electron microscope.

## MATERIALS AND METHODS

### Specimen preparation

Freshly extracted 40 unrestored, caries free third molars were cleaned and the coronal portion was separated mesiodistally at the central fossa with a water cooled diamond coated disc. The resultant 80 specimens were then mounted with the buccal or lingual surfaces facing upward with autopolymerizing resin (Denture base polymer resin, DPI - RR Cold cure, New Delhi, India) (Fig. 1).

The buccal and lingual surfaces were prepared with a standard-grit diamond rotary cutting instrument until the dentin surface was reached and preparation was finished with a fine-grit diamond instrument. To simulate the provisional restoration, discs (3 mm × 1 mm) were made with autopolymerizing resin (DPI, Self cure tooth moulding powder, New Delhi, India) (Fig. 2) and luted with one of the provisional cements :

- Group A - Zinc oxide eugenol (Dental products of India Ltd, New Delhi, India)
- Group B - Non eugenol cement (lot no: 409274, Rely X Temp NE, 3M ESPE, Germany).

All specimens were stored in distilled water at room temperature for 24 hours. After that, provisional cements were mechanically removed with explorer until the dentin surface appeared macroscopically clean and then specimens were divided into 4 subgroups depending upon the cleansing methods used to clean the dentin surface

- Subgroup I → Control group → Air water spray
- Subgroup II → Cleaned with pumice prophylaxis
- Subgroup III → Cleaned with ultrasonic scaler using 0.2% chlorhexidine gluconate as irrigant
- Subgroup IV → Scrubbed with cotton pellet soaked in 17% EDTA (lot no:110224 Glyde, Dentsply, USA) for 15 seconds

After this, the cleansed specimens were equally divided into three groups and subjected to shear bond test, contact angle test and SEM respectively.

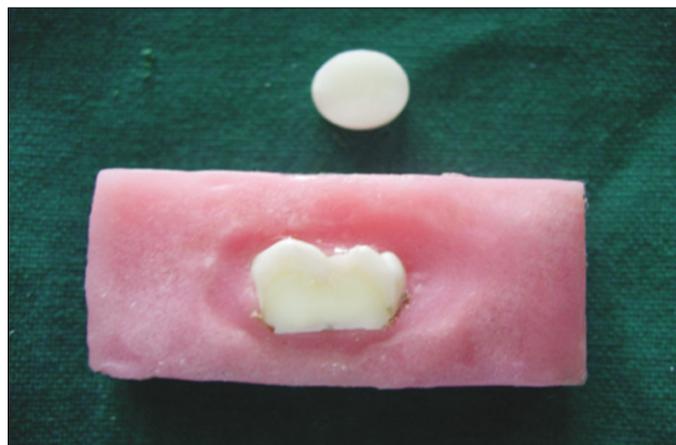
### Shear bond strength

To test the bond strength of composites bonded on to the cleansed dentin surface the following procedures were carried on:

A composite material (Esthet X Micromatrix restorative Dentsply, L.D. Caulk Division, Milford, DE, USA) was placed into a plastic transparent mould of 3 mm diameter and 1mm height and cured for 20 seconds. Dentin surfaces were etched, dentin adhesive and dentin bonding agent were applied according to manufacturer's instructions. Resin luting agent (lot no: P2 1188, Variolink II, Dual curing luting composite system, Ivoclar Vivadent, Liechtenstein) was placed in between composite and dentin surface. Each specimen was polymer-



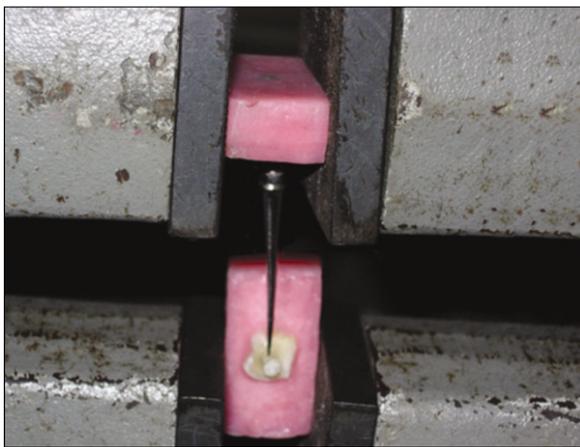
**Fig. 1.** Dentin exposed coronal portion of tooth mounted in autopolymerizing resin block.



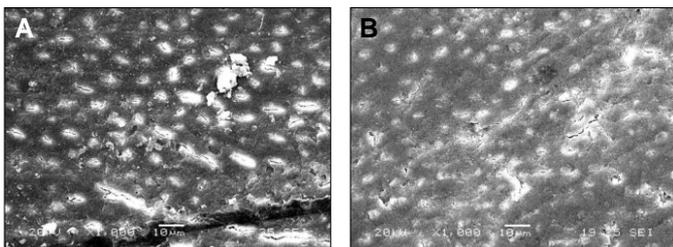
**Fig. 2.** Prepared specimen and provisional disc (3 mm × 1 mm) to be luted.

ized for 40 seconds at a distance of 1mm using visible light polymerizing unit (Blue phase C5, Ivoclar Vivadent, Liechtenstein) and specimens were stored in distilled water at room temperature for 24 hours.

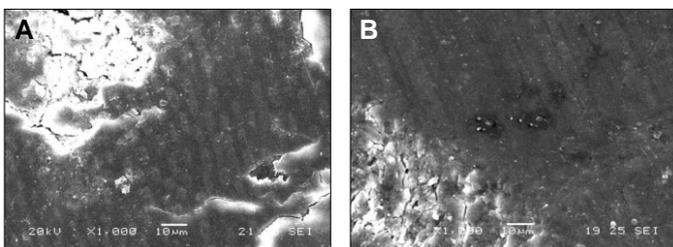
The shear bond strength was measured by an Instron universal testing machine (Fig. 3). In addition, the type of failure was observed by scanning electron microscope at a magnification of  $1000\times$ . The nature of failure was noted as adhesive, cohesive or mixed (Figs. 4-7).



**Fig. 3.** Specimen sheared under Instron universal testing machine.



**Fig. 4.** A: SEM Observation- Mode of failure - Control group (Group A- adhesive failure), B: SEM Observation- Mode of failure - Control group (Group B- adhesive failure).

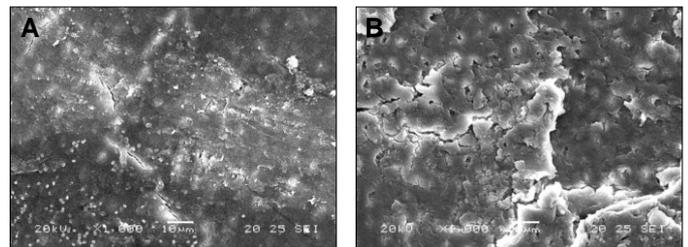


**Fig. 5.** A: SEM Observation- Mode of failure - Pumice prophylaxis (Group A- cohesive failure), B: SEM Observation- Mode of failure - Pumice prophylaxis (Group B- cohesive failure).

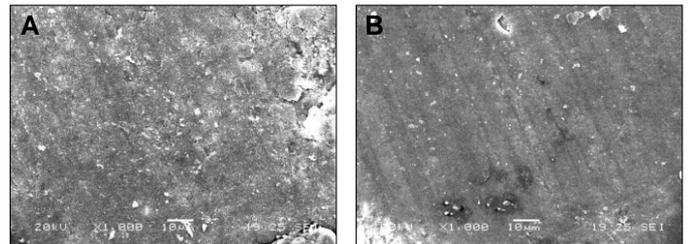
### Contact angle test

For contact angle measurements, the dentin surfaces that were contaminated with provisional cement and cleansed with different techniques were wetted with water and dentin wetting was analyzed by measuring the contact angle of water.

For contact angle measurement, the ADSA-CD technique was used. This technique permits measurement of the contact angle of sessile drops when they have a flat profile. One  $10\ \mu\text{L}$  drop of "Deionized" water was placed on the cleansed dentin surface of the prepared specimen (Fig. 8) and the contact angle was measured. The drop image was captured with a micro video



**Fig. 6.** A: SEM Observation- Mode of failure - Ultrasonic scaling (Group A- mixed failure), B: SEM Observation- Mode of failure - Ultrasonic scaling (Group B- mixed failure).



**Fig. 7.** A: SEM Observation- Mode of failure - EDTA (Group A- cohesive failure), B: SEM Observation- Mode of failure - EDTA (Group B- cohesive failure).



**Fig. 8.** "Deionized" water placed using pipette.



**Fig. 9.** Contact angle values as seen in the computer.

system when the drop was in equilibrium. The video signal was transmitted to a computer that provided the contact angle values (Fig. 9).

The mean, standard deviation and test of significance of mean values between the groups were studied.

One-way ANOVA was used to calculate the *P* value. Multiple range Tukey HSD procedure was employed to identify the significant groups at 5% level and pair t-test was used to compare the shear bond strength and contact angle values among the groups.

**SEM observation**

To evaluate the effect of the cleansing technique on the dentin surface, specimens were prepared as already mentioned. All specimens were allowed to dry overnight and were then gold-sputtered and examined under scanning electron microscope at 15 KV. The SEM photomicrographs were observed with 1000 × magnification (Figs. 10-13).

**RESULTS**

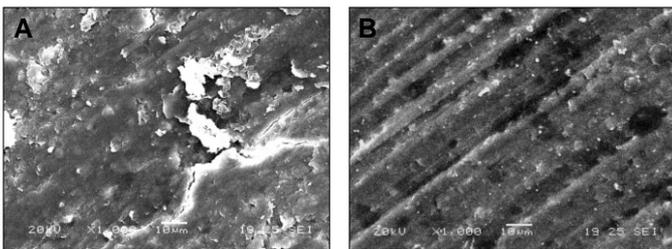
In Group A, the mean shear bond strength value of Group IV was significantly higher than the mean shear bond strength value of all other groups (Table 1). In Group B, the mean shear bond strength value of Group IV was significantly higher than the mean shear bond strength value of Group II followed by Group III and Group I (Table 2).

Table 3 shows the comparison of shear bond strength values among the groups.

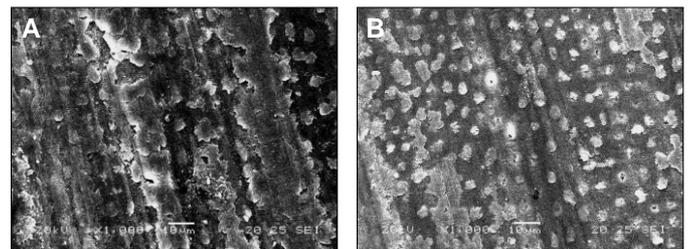
The failure types for Group A is presented in Table 4 and for Group B in Table 5.

In Group A, the mean contact angle of Group IV was significantly lower than the Group I and Group III (Table 6). In Group B, the mean contact angle value of Group IV was significantly lower than the mean contact angle of Group II followed by Group III and Group I (Table 7).

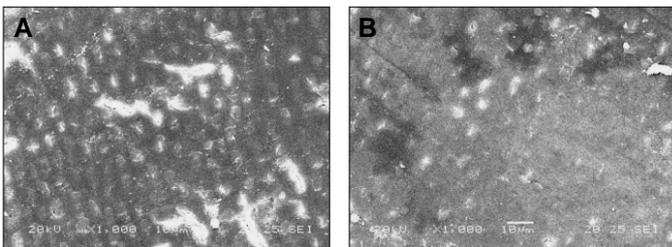
Table 8 shows the comparison of contact angle values among the groups.



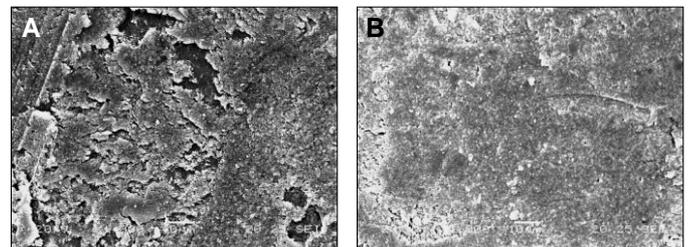
**Fig. 10.** A: SEM Observation- Surface Cleanliness - Control group (Group A), B: SEM Observation- Surface Cleanliness - Control group (Group B).



**Fig. 12.** A: SEM Observation- Surface Cleanliness - Ultrasonic scaling (Group A), B: SEM Observation- Surface Cleanliness - Ultrasonic scaling (Group B).



**Fig. 11.** A: SEM Observation- Surface Cleanliness - Pumice prophylaxis (Group A), B: SEM Observation- Surface Cleanliness - Pumice prophylaxis (Group B).



**Fig. 13.** A: SEM Observation- Surface Cleanliness - 17% EDTA (Group A), B: SEM Observation- Surface Cleanliness - 17% EDTA (Group B).

**Table 1.** Shear bond strength (MPa) of zinc oxide eugenol group (Group A)

Group	Mean $\pm$ SD	P value	Significance group at 5% level
I	1.898 $\pm$ 0.459	.001	IV vs I, II, III
II	2.861 $\pm$ 0.192		
III	2.602 $\pm$ 0.101		
IV	3.260 $\pm$ 0.435		

**Table 2.** Shear bond strength (MPa) of freegenol group (Group B)

Group	Mean $\pm$ SD	P value	Significance group at 5% level
I	4.309 $\pm$ 0.172	.001	IV vs I, II, III
II	4.774 $\pm$ 0.094		
III	4.291 $\pm$ 0.282		
IV	5.288 $\pm$ 0.377		

**Table 3.** Pair t-test among groups

Subgroup	Group A Mean	Group B Mean	Significance
I	1.898	4.308	0.001
II	2.861	4.774	0.002
III	2.602	4.291	0.001
IV	3.260	5.288	0.001

**Table 4.** Failure types for Group A (zinc oxide eugenol)

Subgroup	AD (%)	CO (%)	MI (%)
I	35 (70%)	5 (10%)	10 (20%)
II	-	40 (80%)	10 (20%)
III	10 (20%)	10 (20%)	30 (60%)
IV	10 (20%)	35 (70%)	5 (10%)

The SEM observation of the specimen showed that dentinal tubules were completely covered by the smear layer and the remnants of the provisional cements on the dentin surface. Widespread remnants of the provisional cements were seen on the micrograph of the untreated dentin surface. The use of pumice prophylaxis for both zinc oxide eugenol and freegenol groups produced a smoother and cleaner surface than for other groups, despite formation of large remnant particles. The remnants smeared to the surface were more evident on the dentin cleansed with the ultrasonic scaler with 0.2% chlorhexidine gluconate irrigant (Figs. 10-13).

## DISCUSSION

The adverse effects of residual eugenol from the provisional cements on the bonding characteristics of subsequent restorations have been well documented.<sup>7-10</sup> Several investigators<sup>2,3</sup> have hypothesized that different cleaning techniques for the

**Table 5.** Failure types for Group B (Freegenol)

Subgroup	AD (%)	CO (%)	MI (%)
I	38 (76%)	3 (6%)	9 (18%)
II	5 (10%)	39 (78%)	6 (12%)
III	5 (10%)	5 (10%)	40 (80%)
IV	5 (10%)	40 (80%)	5 (10%)

**Table 6.** Contact angle values for zinc-oxide eugenol (Group A)

Group	Mean $\pm$ SD	P value	Significance group at 5% level
I	69.041 $\pm$ 0.412	.001	IV vs I, III
II	63.150 $\pm$ 0.845		
III	67.389 $\pm$ 1.05		
IV	62.609 $\pm$ 0.635		

**Table 7.** Contact angle values for freegenol group (Group B)

Group	Mean $\pm$ SD	P value	Significance group at 5% level
I	68.459 $\pm$ 0.599	.001	I vs II, III, IV
II	64.271 $\pm$ 0.083		II vs I, III, IV
III	65.786 $\pm$ 0.639		III vs I, II, IV
IV	62.686 $\pm$ 0.736		IV vs I, II, III

**Table 8.** Pair t-test contact angle

Subgroup	Group A Mean	Group B Mean	Significance
I	69.041	68.459	0.213
II	63.115	64.271	0.047
III	67.389	65.786	0.001
IV	62.609	62.686	0.879

removal of the provisional cement remnants from the dentin, like air-water spray, pumice prophylaxis, and use of cleansing agents affect the bond strength of resin luting agent to the dentin and the water contact angle of the dentin surface.

After tooth preparation, the dentin is covered with a smear layer that is composed primarily of cut, mineralized collagen fibrils. When Eugenol containing, provisional cement is applied on the smear layer, eugenol leaches into and through the smear layer to the dentin tubules, contaminating the dentin surface.<sup>11</sup>

Various techniques of removing the smear layer have been used in literature.<sup>12-14</sup> In comparison, EDTA and pumice prophylaxis have been found to be more effective in cleaning the remaining dentinal surface of residual debris and the smear layer.<sup>3,12,15,16</sup> Cameron<sup>17</sup> claimed that cleanliness is achieved following ultrasonically agitated irrigation with distilled water.

Dentin bonding is achieved by resin penetration into dentinal tubules, and into interfibrillar spaces. Higher shear bond

strengths have been achieved with dual cure cements rather than the chemical cure cements<sup>18</sup> and Lafuente *et al.*<sup>19</sup> showed that dual cements had higher bond strength to dentin than to enamel. The rationale of using this material is to have a material with extended working time and capable of reaching a high degree of conversion to have a better bond.

Hülsmann *et al.*<sup>4</sup> have reported that 17% EDTA has a good cleansing effect on dentin and removes the smear layer by dissolving the inorganic compounds. The shear bond strength achieved with pumice prophylaxis was found to be higher than ultrasonic cleansing and air-water spray (control), which is in accordance with the studies by Grasso *et al.*<sup>3</sup> Ultrasonic scaling with 0.2% chlorhexidine gluconate has showed lower shear bond strength compared to other cleansing regimes which is in accordance with the study by Abott *et al.*<sup>15</sup>

The shear bond strength of the freegenol group (Group B) in our study also shows that 17% EDTA has better shear bond strength values compared to pumice and ultrasonic scaler with 0.2% chlorhexidine gluconate. Here in this group EDTA has good efficiency in removing the remnants of provisional cement and the smear layer covering the dentinal tubules compared to eugenol group (Table 3). Specimens treated with freegenol cement have beneficial effects on the shear bond strength values compared to those treated with zinc oxide eugenol cement which is in accordance with the studies conducted by various investigators.<sup>7-9,20</sup>

Acid etching is a procedure that changes substrate conditions because it eliminates the dentin mineral content and exposes the collagen layer.<sup>7</sup> On the etched substrate, adhesive resins penetrate and forms the hybrid layer.<sup>21</sup> Acid etching dissolves the residual eugenol, along with the microscopic remnants.<sup>7</sup> Also conditioning of dentin surface with 37% phosphoric acid removes the smear layer and smear plugs and opens the dentinal tubules, in addition to demineralizing the intertubular and peritubular dentin.

In this present study, although all specimens were etched with 37% phosphoric acid, and adhesive resins were used, shear bond strengths showed significant differences. It was concluded that the cleansing techniques had considerable effect on the bond strength of the resin luting agent.

The modes of failures for specimens were of 3 types: adhesive, cohesive and mixed. Mixed and cohesive fractures were the most common types of failure for all groups (Tables 4, 5). However, the control specimens of both the groups showed adhesive failures. EDTA and Pumice shows cohesive failure for both zinc oxide eugenol and freegenol cements (Figs. 4-7).

In our study, the lowest contact angle were obtained with 17% EDTA for both zinc-oxide eugenol and freegenol groups, which indicates that EDTA is more effective at removal of the remnant of the provisional cement and the smear layer, so EDTA creates appropriate physical and chemical interactions between the resin cement and dentin.

SEM observation shows that 17% EDTA was the most effective solution to remove the smear layer. Also EDTA leaves the surface rough for better resin penetration (Figs. 10-13).

Kielbassa *et al.*<sup>11</sup> showed that diffusion of eugenol may be more than 200  $\mu\text{m}$  in depth, so that acid etching may not eliminate the effects of eugenol. It is interesting to note from this study (Table 8). that negative effects of eugenol on dentin adhesion are not based on the wetting process but on adhesion test results.<sup>5</sup>

The result of this present study suggests that the shear bond strength and wettability of dentin were affected not only by the acid etching and or dentin primers but also dentin cleansing techniques.

In this study, EDTA has been used as an effective dentinal cleansing agent even in the presence of eugenol in an *in vitro* situation. An effectiveness of EDTA with different luting agents requires special consideration. The contact angle measurement, in an *in vivo* situation, would consider various factors like thermal and mechanical changes, oral fluid contaminations, organic and inorganic residues, etc. Further, a three dimensional model of the bonding surfaces would better reflect the failures which occur as a combination of various compressive, tensile and shearing forces.

Future advancements in research methodologies including the finite element method for a 3D model and a digitized ADSA-CD technique could be incorporated to achieve higher levels of accuracy and relevance.

## CONCLUSION

Within the limitation of this study, it was concluded that

1. Significantly different shear bond strength were found with the different cleansing techniques evaluated.
2. Specimens treated with EDTA showed the highest shear bond strength for both zinc oxide eugenol and freegenol groups. Higher shear bond strength values were also obtained for pumice prophylaxis when compared to control group and ultrasonic scaler group.
3. EDTA showed the lowest contact angle for both the groups.
4. SEM showed that EDTA was the most effective solution to remove the smear layer, and pumice prophylaxis leaves large remnant particles. Also, mode of failure seen was predominantly cohesive for both EDTA and pumice prophylaxis.

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# Cortical and cancellous bone thickness on the anterior region of alveolar bone in Korean: a study of dentate human cadavers

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**PURPOSE.** The cortical bone thickness on the anterior region is important for achieving implant stability. The purpose of this study was to examine the thickness of the cortical and cancellous bones on the anterior region of the maxilla and mandible. **MATERIALS AND METHODS.** Twenty-five cadaver heads were used (16 male and 9 female; mean death age, 56.7 years). After the long axis of alveolar process was set up, it was measured in 5 levels starting from 2 mm below the cemento-enamel junction (L1) at intervals of 3 mm. All data was analysed statistically by one-way ANOVA at the .05 significance level. **RESULTS.** The cortical bone thickness according to measurement levels in both the labial and lingual sides increased from L1 to L5, and the lingual side below L3 was significantly thicker than the labial side on the maxilla and mandible. In particular, the labial cortical bone thickness in the maxilla was the thinnest compared to the other regions. The cancellous bone thickness according to measurement levels increased from L1 to L5 on the maxilla, and on the mandible it was the thinnest at the middle level of the root. **CONCLUSION.** For implant placement on the anterior region, a careful evaluation and full knowledge on the thickness of the cortical and cancellous bone are necessary, therefore, these results may provide an anatomic guideline to clinicians. [J Adv Prosthodont 2012;4:146-52]

**KEY WORDS:** Cortical bone thickness; Cancellous bone thickness; Anterior region; Implant placement

## INTRODUCTION

The anterior teeth are an important factor in dental and facial esthetics.<sup>1</sup> At this time, in the anterior region they are difficult for both stabilization of the implant fixture and aesthetics of the restoration because they have narrower alveolar ridge and thinner cortical bone than in the posterior region.<sup>2,3</sup> In addition, the thickness of the cortical bone has a larger influence on the initial stabilization than the length of the implant fixture in an edentulous region. Therefore, information on the cortical and cancellous bone thickness in the labial and lingual sides, especially the anterior part, is the key to successful dental implantation.<sup>4</sup>

Many researchers have evaluated the thickness of labial cortical bone using various methods in the anterior region. Above all, computed tomography (CT) is used widely to measure the

alveolar bone for preoperative evaluation before dental prosthetic and orthodontic treatment because it can measure a large number of the samples and various age groups. Using a CT, Flanagan<sup>5</sup> compared the thickness of the labial and lingual cortical bone on the edentulism, and Swasty *et al.*<sup>6</sup> measured the thickness of the mandibular alveolar bone in various age groups. In addition, Deguchi *et al.*<sup>7</sup> and Lim *et al.*<sup>8</sup> evaluated the thickness of the buccal and lingual cortical bone on the posterior region using CT for the implantation of mini-screws..

Recently, micro-CT is often used for measuring the bone structures because it is convenient, noninvasive tool and has the higher resolution compared to conventional CT image.<sup>9</sup> However, until now, most of these researches have been done by using the radiographic methods such as CT. Despite the limited number of the cadaver samples, few studies by direct manual measurement have evaluated the thickness of cortical and can-

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