# Shear bond strength of endodontic sealers to dentin with and without smear layer and gutta percha (An in vitro study)

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### ABSTRACT

Background: The bond strength of root canal sealers to dentin and gutta-percha seems to be an important property for maintaining the stability of root canal filling, which potentially influences both leakage and root strength. The objective of this, in vitro, study was to evaluate the shear bond strength of three different endodontic sealers (Gutta-Flow, AH Plus, Apexit Plus) to dentin, in the presence and absence of the smear layer and gutta percha.

Material and Methods: After slicing off the occlusal 2mm of 60 extracted human maxillary premolar teeth, the exposed dentin served as the tested surfaces; the teeth were fixed with cold cure acrylic, and were divided into two groups according to the smear layer presence, group A without smear layer, when dentin surfaces were irrigated with EDTA 17% followed by distilled water then subdivided into 3 subgroups according to the type of sealer used; group B when dentin surfaces were washed with distilled water only, then subdivided into 3 subgroups. Thirty samples of gutta-percha were prepared and named as group C which was subdivided into 3 subgroups. Five mm long section of polyethylene tubes were placed on the dentin or gutta percha surfaces and filled with freshly mixed sealer. After one week, all the samples were tested for shear bond strength by the Instron Universal Testing Machine at a cross head speed of 0.5 mm/min. The data was calculated in MPa and was statistically analyzed

Result: There was a highly significant difference in the shear bond strength (P < 0.05) in comparison among the tested groups, GuttaFlow showed non-significant difference in bond strength to dentin with and without smear layer, while AH Plus and Apexit Plus showed a high significant difference.

Conclusions: AHPlus showed the highest shear bond strength in all the tested samples, while GuttaFlow was the least. Additionally, AH Plus and Apexit Plus shear bond strengths were affected by the smear layer removal, while GuttaFlow was not.

Key words: Shear bond strength, GuttaFlow, Endodontic sealers. (J Bagh Coll Dentistry 2014; 26(4):86-89).

الخلاصة

أن لقوة الرابطة لسدادات قناة الجذرالى عاج السن و Gutta-percha خاصية مهمة للحفاظ على ثبات حشوة قناة الجذر الذي بدوره يؤثر على تسرب و قوة الجذر. كان الغرض من هذه الدراسة تقييم قوة الترابط القصي بين ثلاثة انواع من السدادات اللبية المختلفة (AH Plus, Gutta-FlowApexit Plus) و عاج السن بوجود اوعدم وجود طبقة الـ Smear layer و الـ Gutta-percha. بعد قطع2ملميتر من تاج 60 سن من أسنان الإنسان(الضواحك العليا) . تم تثبيت الأسنان في مادة الاكريليك ، و قسمت الى مجموعتين وفقا لوجود طبقة الـsmear layer.

المجموعة (أ) بدون Smear layer على سطح عاج السن مع غسل بمادة EDTA 17% يليه الماء المقطر وقسمت إلى 3مجموعات فرعية وفقا لنوع السدادة المستعملة (-AP) D, AH-D, GF-D.

المجموعة (ب) غسل سطح عاج السن بالماء المقطر فقط ثم قسمت إلى 3 مجموعات فرعي)(APD-S, AHD-S, GFD-S .

ثلاثونعينة من (Gutta-percha) أعدت لتكون المجموعة (ج)وقسمت إلى 3 مجموعات فرعية (AP-G, AH-G, GF-G).

استعمل انبوب بطول 5 ملم من البولي اثيلين بسداده مخلوطة حديثًا ووضع على سطح عاج السن أو الـ Gutta-percha .اختبار قوة الرابط القصي تم بعد اسبوع .

تم حساب البيانات بوحدة الميغا باسكال و تم تحليلها إحصائيًا . أظهرت النتائج فرقا معنويا كبيرا .Gutta-Flow سجل فرق معنوي غير ملحوظ في قوّة الترابط مع عاج السن بوجود او عدم وجود الـ Smear layer بينما AP Pus و Apexit Plusسجلوا فرقا معنويا عاليا.

الاستنتاج النهائي : AH Plus اظهر اعلى قوة ربط قصي في جميع العينات بينما كان Gutta-Flow الاقل بينهم . وقوة الربط القصي للـ AH Plus و Apexit plus تأثرت بازالة طبقة Smear layer الى الاحسن اما Gutta-Flow فلم تتأثر .

# INTRODUCTION

Endodontic sealers are the essential components of root-filling materials used to fill the voids and gaps between the main root-filling material and root dentin. Good adhesion to tooth material within the root canal is one of the ideal properties of a sealer cement which potentially influences both leakage and root strength <sup>(1)</sup>.

The adhesion of root canal filling to the dentinal walls is advantageous for two main reasons. In the static situation, it should eliminate any space that allows percolation of fluids between the obturating material and the dentin wall. In the dynamic situation, it is needed to resist dislodgement of the filling during subsequent manipulations <sup>(2)</sup>.

The smear layer as it relates to the root canal system is the layer of debris on the root canal wall and has been shown to be packed into the dentinal tubules. Various methods have been used to remove the smear layer. Conflict in guide as has been obtained regarding the significance of the presence or the removal of the smear layer <sup>(3)</sup>.

Some studies concluded that removal of the smear layer prior to filling the root canal system may enhance the ability of filling material to enter the dentinal tubules. This increases the adhesive strength of sealer to dentin; others concluded that removal of the smear layer may impair sealer adhesion to dentin <sup>(4,5)</sup>.

Different types of sealer have been introduced to endodontics. Epoxy resin-type sealers have been used for many years. They showed higher bond strength to dentin than zinc oxide eugenol types and calcium hydroxide-based sealer <sup>(6)</sup>. GuttaFlow®2 sealer is an alternative root filling

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material introduced into the endodontic practice. GuttaFlow®2 is a cold flowable filling system for root canals, combining sealer and gutta-percha in one product.

The sealers used in this study were Gutta-Flow®2 sealer (Coltène/Whaledent, Germany), AH Plus (DeTrey Dentsply, Germany), Apexit Plus (IvoclarVivadent, Liechtenstien).

The purpose of this study was to evaluate the shear bond strength of GuttaFlow (Silicon based sealer), AH Plus (Epoxy resin based sealer), Apexit Plus (Calcium hydroxide based sealer) to dentin before and after removal of smear layer and smear layer and gutta percha.

### **MATERIALS AND METHODS**

In this study, 60 extracted, non-carious, human, maxillary, premolars teeth were collected from the Orthodontic department, College of Dentistry, University of Baghdad. Standardized preparations of flat dentin surfaces were obtained. By the use of a digital caliper, 2 mm from the occlusal surface of the teeth, were sectioned by the use of a diamond disc in a straight handpiece with a water coolant. The exposed dentin surfaces were inspected with a stereomicroscope to ensure that no enamel remained <sup>(7)</sup>. A custom-made two L-shaped brass molds were set at 24-20-16mm dimensions were used to construct the acrylic blocks, the section of tooth which included the root was embedded in the acrylic within the mold in a direction that standardized for all the samples with the use of dental surveyor. The two parts of mold were separated after the completion of the polymerization process.

For dentin sample with smear layer, the dentin surface was washed by distilled water only.<sup>(8)</sup>For dentin sample without smear layer, the smear layer was removed by irrigation of the dentin surface with 1ml of EDTA 17% for 1 minute followed by 3ml of distilled water for 1 minute <sup>(9, 10)</sup>.

Thermostat controlled  $(45\pm3^{\circ}C)$  water bath was used for softening the standardized cones of gutta percha ISO size 140 <sup>(11)</sup>. Then they were compacted into cupper rings of 10mm in diameter and 4mm high; the same mold that was used for dentin sample had been used to construct the acrylic blocks for gutta percha. Polyethylene tubes were carefully placed with one open side contacting the dentin or gutta-percha, perpendicular to its surface then filled with the freshly mixed sealer <sup>(12)</sup>.

A custom made device consisted of a metal board with a fixed handle to hold the sample was used, also there was a handle supporting 400g weight for standardization of sealer weight application.

All sealer cylinders were allowed to bench set for 2 hours and stored at 100% humidity at 37° C for 1 week <sup>(13)</sup>. All the specimens were loaded until failure by the Instron Universal Testing Machine at across head speed of 0.5 mm/min, the load was parallel to the dentinal surface of the tooth, or gutta-percha surfaces and perpendicular to the long axis of sealer cylinder. The force was recorded in Newton divided by the surface area to obtain the shear bond in Mpa.

The ninety samples were divided into three groupsand each groupwas subdivided into 3 subgroups according to the type of the sealer used: Gutta-Flow, AH Plus, Apexit Plus;10 specimens for each sealer.

**Group A:** 30 dentin samples without smear layer (GF-D, AH-D, Ap-D).

**Group B:** 30 dentin samples with smear layer (GFD-S, AHD-S, ApD-S).

**Group C:** 30 gutta-percha samples (GF-G, AH-G, Ap-G).

## RESULTS

Figure 1 shows the mean shear bond strength, in MPa, of the tested sealers to dentin with and without smear layer and gutta-percha.



Figure 1: The mean shear bond strength (MPa).

The descriptive statistic results of shear bond strength, in MPa, between the tested sealers and dentin without smear layer are seen in Table 1.

Table 1: Descriptive statistic results of shearbond strength, in MPa, between the sealersand dentin without smear layer.

Groups	Ν	Mean	S.D.	Min.	Max.
GF-D	10	0.18	0.07	0.15	0.31
AH-D	10	1.30	0.21	0.95	1.59
Ap-D	10	0.42	0.11	0.31	0.63
SD – standard deviation					

S.D.= standard deviation Min.=minimum Max.=maximum Statistical analysis of the data by using the analysis of variance (ANOVA) was done. There was a highly significant difference in shear bond strength (P < 0.05) in comparison among all tested groups. To compare between groups, independent sample t-test was performed and the results are shown in Table 2.

Comparison	t-test	<b>P-value</b>		
GF-D vs. AH-D	-16.01	0.000 (HS)***		
GF-D vs. Ap-D	-5.96	0.000 (HS)		
AH-D vs. Ap-D	11.76	0.000 (HS)		
***IIC. highly Cignificant				

\*\*\*HS: highly Significant.

The descriptive statistic results of shear bond strength, in MPa, between the tested sealers and dentin with smear layer are compiled in Table 3.

#### Table 3: Descriptive statistic results of shear bond strength, in MPa, between the tested sealers and dentin with smear layer

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Groups	Ν	Mean	S.D.	Min.	Max.
GFD-S	10	0.17	0.05	0.15	0.31
AHD-S	10	0.31	0.11	0.15	0.47
ApD-S	10	0.20	0.08	0.15	0.31

The analysis of variance (ANOVA) showed a highly significant difference in shear bond strength (P < 0.05). Table 4 shows the Independent sample t-test results.

 Table 4: Independent sample t-test results

Compared groups	t-test	p-value
GFD-S vs. AHD-S	-3.86	0.001 (HS)***
GFD-S vs. ApD-S	-1.10	0.288 (NS)*
AHD-S vs. ApD-S	2.69	0.015 (S)**
NS: Not Significant		

\*NS: Not Significant. \*\*S: Significant.

\*\*\*HS: highly Significant.

The descriptive statistic results of shear bond strength, in MPa, between the tested sealers and gutta-percha are seen in Table 5; while Table 6 showsthe Independent sample ttest results comparing the paired groups regarding shear bond strength between the tested sealer and gutta percha.

Table 5: Descriptive statistic results of shear bond strength, in MPa, between the tested sealers and gutta-percha

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Groups	Ν	Mean	S.D.	Min.	Max.
GF-G	10	0.25	0.08	0.15	0.31
AH-G	10	0.73	0.17	0.47	0.95
Ap-G	10	0.28	0.13	0.15	0.47

<b>Fable 6: Independent sample t-test results</b>					
Compared groups	t-test	P-value			
GF-G vs. AH-G	-7.95	0.000 (HS)***			
GF-G vs. Ap-G	-0.67	0.511 (NS)*			
AH-G vs. Ap-G	6.64	0.000 (HS)			

\*NS: Not Significant.

\*\*\*HS: highly Significant.

Finally, paired groups were compared in order to find the effect of the removal of the smear layer on the shear bond strength, of the different sealers used, to both dentin and gutta percha.

Sealer type	Compared groups	t-test	Sig.
Cutto	GF-D vs. GFD-S	0.60	(NS)*
Flow	GF-D vs. GF-G	-1.90	(NS)
	GFD-S vs. GF-G	-2.61	(S)**
AH Plus	AH-D vs. AHD-S	13.29	(HS)***
	AH-D vs. AH-G	6.70	(HS)
	AHD-S vs. AH-G	-6.50	(HS)
Apexit Plus	Ap-D vs. ApD-S	5.33	(HS)
	Ap-D vs. Ap-G	2.74	(S)
	ApD-S vs. Ap-G	-1.71	(NS)

 Table 7: t-test to compare each two groups

\*NS: Not Significant.

\*\*S: Significant.

\*\*\*HS: highly Significant.

#### DISCUSSION

Shear bond test was used, in this study, because it is easier to be performed and allowed testing gutta percha and dentin specimens in a similar manner. Also it provided homogenous results with considerably low variation of bond strength  $^{(11,12)}$ .

The adhesion of endodontic sealers to the coronal dentin was used rather than root dentin, because root dentin is not uniform and the surface of the canal walls may differ widely. Also there is a gradual decrease in the number of dentinal tubules from coronal to apical part of dentin, this agreed with Kandaswamy et al. <sup>(2)</sup>.

Ethylene diamine tetra acetic acid (EDTA) 17% solution was used, in this study, because from the shear bond aspect, EDTA was a good irrigant to be used as a final rinse for smear layer removal <sup>(2)</sup>.

AH plus showed a superior dentin bond strength than Apexit plus, with and without smear layer; agreed with Eldeniz et al. <sup>(7)</sup>; Gopi-krishna et al. <sup>(14)</sup>; this may be due to its ability to react with any exposed amino groups in collagen to form covalent bonds. AH Plus has a very low shrinkage rate while setting and its long-term dimensional stability. Gutta-Flow showed the least bond strength to dentin, this result is in

agreement with Saleh et al.<sup>(4)</sup> and Coba-nkara et al. <sup>(15)</sup>. This may be due to the poor wetting of GuttaFlow on the dentin surface because of the presence of silicon, which possibly produces high surface tension forces, making the spreading of these materials more difficult.AH plus scored the highest shear bond strength to gutta-percha, due to the presence of Bisphenol A Epoxy resin in its formulation that bond chemically with gutta percha agreed with Mamdooh <sup>(16)</sup> and disagreed with Stoll et al. <sup>(17)</sup>; while the setting reaction of Apexit plus form an amorphous calcium disalicylate, which does not bond to gutta-percha. Finally to evaluate the effect of smear layer removal on each sealer, it was found that AH plus and Apexit plus were highly affected by the removal of smear layer, this finding agreed with Gopikrishna et al. <sup>(14)</sup>. While GuttaFlow bond strength was not affected by the removal of the smear layer because EDTA may significantly decreases the wetting ability of dentinal wall. Therefore; a suitable dentin substrate could be provided for the adhesion of materials with hydrophobic nature as AH plus.Furthermore, the removal of the smear layer allowed the extension of the sealer tags into the opened dentinal tubules, creating an efficient microretention<sup>(5)</sup>.

The conclusions that can be drawn from this study are:

- 1. AH Plus showed the highest shear bond strength in all the tested samples.
- 2. Gutta-Flow scored the least shear bond strength.
- 3. AH Plus and Apexit Plus shear bond strengths to dentin were affected by the smear layer removal, while Gutta-Flow was not.

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