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# The Effect of Smear Layer on the Push-out Bond Strength of Silicone-based Root Canal Sealers

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## Abstract

**Aims:** The study aimed to evaluate the effect of smear layer on the push-out bond strength of silicone-based root canal sealers. **Materials and methods**: Sixty extracted, single-rooted, sound human mandibular first premolar teeth were selected for this study. The Crown portion of each tooth was decoronated to the level of cement-enamel junction to standardize the root length to 16 mm. The working length was measured by subtracting 1mm from the visually determined canal length and the canals were instrumented using ProTaper universal rotary system up to size F3. The canals were divided into two groups according to the irrigating solutions used. Group.1 was rinsed with 2.5% sodium hypochlorite followed by 17% EDTA to ensure complete smear layer removal. Group.2 was rinsed using 0.9% normal saline to keep the smear layer. Then the samples were subdivided into three subgroups according to the sealer used with gutta-percha. These include AH Plus, GuttaFlow 2, and GuttaFlow Bioseal groups. The push-out bond strength test was performed using the universal testing machine and the data were analyzed using independent sample T-test at ($p \leq 0.05$) to compare the results of each sealer group in the presence and absence of the smear layer. **Results:** For all sealers' groups the results has shown a statistically significant difference in the bond strength at ($p \leq 0.05$) between group 1 (without smear layer) and group 2 (with smear layer). The smear layer removal produced higher bond strength in AH Plus sealer, whereas the bond strength of both GuttaFlow 2 and GuttaFlow Bioseal was decreased when the smear layer was removed. **Conclusion:** Smear layer removal has a positive effect on the bond strength value of AH Plus sealer. The bond strength of GuttaFlow 2 and GuttaFlow Bioseal was not improved following smear layer removal.

## الخلاصة

**الاهداف**: تهدف الدراسة الى تقيم تأثير بقاء أو إزالة الطبقة اللطاخة على قوة الربط التخرجي لإثنين من السمنت اللبي فئة السيليكون وهما: (Gutta Flow2 و GuttaFlow Bioseal) مع استخدام السمنت اللبي (AH Plus) كمادة مرجعية. **المواد والطرائق العمل**: أستخدم ستون سناً بشرياً سليماً مقلوعاً من فئة الجذر الواحد المستقيم (الضاحك الأول)، وقد تم قطع تيجان هذه الأسنان عند مستوى الملتقى المينائي الملاطي كي يبلغ طول الجذر ١٦ملم. بعد ذلك تم قياس طول العمل للقنوات اللبية وتحضيرها بإستخدام أنظمة تيتانيوم-النيكل الدوّارة(protaper) الى حجم F3 قسمت العينات الى مجموعتين رئيسيتين حسب نوع السوائل الإروائية المستخدمة في غسل قنوات الجذور: المجموعة الأولى: أستخدم فيها محلول هيبوكلوريت الصوديوم بتركيز ٢,٥٪ ومحلول (إي دي تي أي) بتركيز ١٧٪. المجموعة الثانية: أستخدم فيها محلول كلوريد الصوديوم بتركيز ٠,٩٪. ملئت القنوات بإستخدام Gutta-percha والسمنت اللبي (GuttaFlow 2, GuttaFlow Bioseal, AH Plus) بعد اجراء فحص قوة الربط التخرجي للعينات تم تحليل البيانات إحصائياً عن طريق إختبار التحليل الأحادي الإتجاه للتباين واختبارات دنكان. **النتائج**: أظهر التحليل الإحصائي للنتائج وجود فروق معنوية لقوة الربط التخرجي عند مستوى (p≤٠,٠٥) لكل مجموعة من مجاميع السمنت اللبي الثلاثة. أدت إزالة الطبقة اللطاخة الى زيادة قوة الربط التخرجي للسمنت اللبي نوع ((AH Plus في حين ان قوة الربط تناقصت في حالة السمنت اللبي نوع (GuttaFlow 2 و GuttaFlow Bioseal) بعد ازالة الطبقة اللطاخة. **الإستنتاجات**: من النتائج اعلاه نستنتج أن إزالة الطبقة اللطاخة ذو تأثير إيجابي على قيمة قوة الربط التخرجي للسمنت اللبي ((AH Plus لكن إزالة هذه الطبقة لم تحسن من قيمة قوة الربط التخرجي في حالة السمنت اللبي نوع GuttaFlow 2) و GuttaFlow Bioseal).

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## INTRUDUCTION

The main goal of root canal treatment is to eliminate all vital and necrotic tissues from the root canal [(1)]. During root canal therapy a layer of material is formed with all kinds of cutting instruments. This layer consisted of organic and inorganic substances that cover the prepared root canal walls. It appears as an amorphous granular and irregular which was named as Smear layer [(2)(3)]. It may have some adverse effects regarding preventing irrigants and sealants from penetrating the dentinal tubules [(4)]. The smear layer was shown to be composed of particles ranging from less than 0.5–15 µm in size [(5)]. Removal of the smear layer may permit for more cleaning of the walls of the root canals and allow for better adaptation of the root canal sealers that enter the dentinal tubules. Nevertheless, some studies decline the effect of smear layer removal on sealer bond strength [(3)].

The invention of silicone-based root canal sealers was in an attempt to improve the properties of root canal sealers involving physical, chemical and biological leading to a series of material generations [(6)]. GuttaFlow 2 (Coltene/Whaldent, Switzerland) was available in 2012 as an advancement of the previous GuttaFlow material, having the same excellent properties but with a stiffer consistency and changes in the form of the silver particles used. GuttaFlow 2 is composed of a mixture of gutta-percha powder with particle size smaller than 30 µm, poly-dimethyl-siloxane, zirconium dioxide, platinum catalyst and a preservative of microsilver particles. This cold flowable system combines both gutta-percha and sealer, having a solubility close to zero [(7-10)]. GuttaFlow2 also does not shrink, instead, it expands slightly by about 0.2%. Also it adheres considerably to gutta-percha points and to dentin walls [(11)].

GuttaFlow Bioseal (Coltene/ Whaldent, Switzerland) has been produced in late 2015. The main difference of this sealer than the other silicone-based sealers is in its composition as it has a bioactive ceramic glass in addition to gutta-percha, polydimethylsiloxane, zirconium oxide and platinum [(12)]. GuttaFlow Bioseal has a nanosilver component instead of microsilver. These bioactive components are capable to form hydroxyapatite crystals when they contact tissue fluids, so that they could result in stimulation of tissue regeneration and healing [(13)(14)]. Regarding working and setting times, they are shorter than that of GuttaFlow 2 [(15)]. It has a minimal solubility and a good alkalinizing ability with pH (8-9) that prevents the activity of osteoclasts and stimulates alkaline phosphatase enzyme, which in turn helps in the reformation of periapical bone. The flow of this material is slightly lesser than that of GuttaFlow 2 [(16)].

The sealer bond strength to the root canal dentin wall is a very desirable property because it helps keep the integrity of the sealer-dentin interface without disruption in the long term [(17)]. Recent theories of dentin bonding mechanisms include either smear layer modification and direct bonding to it, or smear layer removal and bonding to the tooth structure [(18)(2)].

This study aimed to assess the effect of smear removal on the bond strength of silicone-based sealers.

## MATERIALS AND METHODS

Sixty single-rooted, sound human mandibular first premolar teeth with completely formed apexes extracted for orthodontic purposes were selected for this study. The teeth were radiographed to exclude any signs of internal or external resorption, fractured or cracked teeth, endodonticaly treated teeth, and teeth with developmental defects, calcified canals from the study. The surface of each root was cleaned from any soft tissue remnants with scaling instruments, the teeth were disinfected using 5.25% sodium hypochlorite NaOCl solution for 30 minutes. Then, they were carefully rinsed under running tap water and kept in sterile distilled water at room temperature to avoid dehydration till further use. The crown portion of each tooth was decoronated to the level of cement-enamel junction using 0.2 mm thick diamond disk with a high-speed handpiece under copious water cooling to standardize the root length to 16 mm. Pulpal tissue was removed using a barbed broach. The apical patency was ensured using No. 10 K file. Then, No. 15 K file was inserted inside the canal and advanced gently using a reciprocating back and forth motion until the tip of the file was seen at the apical foramen. This distance was measured by endodontic ruler. The working length was established by subtracting 1mm from the visually determined canal length. Before instrumentation, the samples were divided into two groups (n=30) according to the irrigating solution to be used. Group.1 has thirty canals were rinsed using freshly prepared 3ml of 2.5% sodium hypochlorite between each instrument change. Group.2 has thirty samples rinsed with 3ml of 0.9% normal saline between each instrument change. The samples were subjected to the same crown down instrumentation procedure through enlargement of the root canals using Nickel-Titanium ProTaper universal rotary system instruments up to size F3. The speed of rotation was maintained at 300 rpm (revolutions per minute) and torque 3.0 Ncm (Newton centimeter). After completing the canal instrumentation, the canals in group.1 were washed with 3ml of distilled water followed by 5ml of 17% EDTA solution for 1 minute to ensure complete smear layer removal. After that, the canals in group 1 were rinsed with additional 5ml of sodium hypochlorite for 1 minute to stop the action of EDTA.

Finally, the samples in both groups (1and 2) were flushed with 5ml of distilled water to remove the remnants of the irrigating solutions from the canals. After that, all canals were dried carefully with F3 paper points to be ready for obturation. The canals of each group were subdivided randomly into three subgroups (n=10) according to the root canal sealer to be used. These are A) AH Plus, B) GuttaFlow 2 and C) Gutta Flow Bioseal subgroups. The sealer in each group was handled and applied according to the manufacturer's instructions and the root canal obturation were completed with size F3 gutta-percha single cone. When the obturation procedure was completed, the apical and coronal portions of the roots were sealed with soft wax. Then, samples of all groups were kept moist by wrapping them in a saline moistened gauze in a closed container, and were incubated for 7 days at 37 C° with 100% humidity in an incubator to ensure complete setting of the sealer.

## Sample sectioning

The roots were vertically placed and centered in clear cold cured acrylic resin custom-made molds. Sectioning of each root was carried out in a horizontal plane perpendicular to the long axis of the main canal using a circular water cooled diamond disk to obtain 2 mm thick disks (apical, middle and coronal root sections respectively). The exact thickness of each disk was measured using a digital caliper. Each segment was marked on its apical side with a marker to make sure that the load is applied in an apical-coronal direction. Both coronal and apical surfaces were carefully examined to select a circular root section with a uniform sealer layer in order to ensure a uniform distribution of the force during push-out test and getting accurate measurements.

## Push-out Bond Strength Test Evaluation

Each sample was carefully positioned on a metal base with a central hole with the apical surface facing the plunger of a universal testing machine (Gester; China) as shown in (figure.1). The center of the tested specimen was aligned over the hole so that the filling material can fall freely through once the bond between the dentin wall and the test material was broken. A vertical load was applied by a cylindrical stainless steel plunger especially designed for this study in an apical to the coronal direction to avoid any constriction interference resulted from the root canal taper. The diameter of each canal was measured in both apical and coronal aspects using a stereomicroscope (Optica, Italy) at 40x magnification. Three different sizes of plungers were utilized according to the measured diameters of the samples, these include (0.7mm for coronal third, 0.5mm for middle and 0.3mm for apical third). The plunger was positioned so that it only contacts the root canal filling to displace it downward and not to touch the dentinal walls to avoid misreading. The test was performed at a cross-head speed of 1 mm/min until the occurrence of bond failure and the highest force value at the time of debonding was recorded.

The bond strength was measured in megapascals (MPa) and calculated by dividing the maximum force (F) measured in newton (N) over surface area (A):

$$\text{Bond Strength (MPa)} = \frac{\text{debonding force (N)}}{\text{Surface area (mm}^2)}$$

The surface area was measured by the following formula:

$$A(mm^2) = \pi(r_1 + r_2)\sqrt{[(r_1 - r_2)^2 + h^2]}$$

Where $\pi$ is the constant 3.14, $r_1$ is the coronal radius, $r_2$ is the apical radius, h is the thickness of the section in mm.

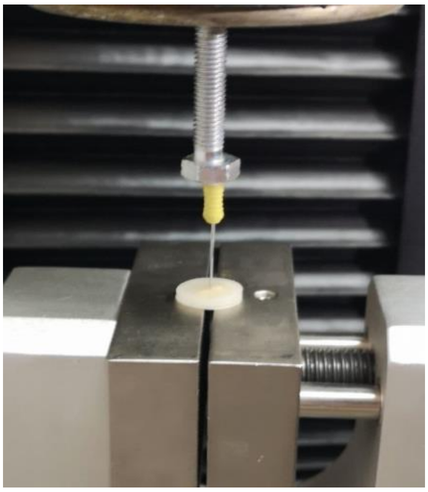

**Figure (1):** The sample under load of the universal testing machine.

The statistical analysis was performed using SPSS software and the data were analyzed using independent sample T-test at ($p \leq 0.05$) to compare the mean bond strength values for each sealer group in the presence or absence of the smear layer.

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## RESULTS

For AH Plus sealer group, there was a statistically significant difference at ($p \leq 0.05$) between groups A1 and A2 as shown in (Table.1). Group A1 (without smear layer) showed higher bond strength mean value than group A2 (with smear layer). The smear layer removal has improved the bond strength of AH Plus sealer.

**Table (1):** Independent sample T-Test of push-out bond strength for AH Plus sealer of both groups A1 and A2.

| AH Plus sealer Groups | N | Mean | Std. Deviation | Std. Error Mean | t-value | Sig.* |
|---|---|---|---|---|---|---|
| Group A1 (without smear) | 30 | 3.605 | 1.198 | 0.218 | 4.086 | 0.00 |
| Group A2 (with smear layer) | 30 | 2.392 | 1.098 | 0.2 | | |

N: Number of samples, Std. Deviation: standard deviation, Std Error Mean: standard error mean, Sig.: high Significance (P-value≤0.05).

In both GuttaFlow 2 and GuttaFlow Bioseal groups there was a statistically significant difference at (p≤0.05) when comparing the mean bond strength in the presence and absence of the smear layer (Tables 2-3). The bond strength of both sealers was not improved by smear layer removal.

**Table (2):** Independent Sample T-Test of push-out bond strength for GuttaFlow2 sealer of both groups B1 and B2.

| GuttaFlow 2 sealer Groups | N | Mean | Std. Deviation | Std. Error Mean | t-value | Sig.* |
|---|---|---|---|---|---|---|
| Group B1 (without smear layer) | 30 | 0.822 | 0.490 | 0.089 | | |
| Group B2 (with smear layer) | 30 | 1.336 | 0.480 | 0.087 | 4.10 | 0.00 |

N: Number of samples, Std. Deviation: standard deviation, Std Error Mean: standard error mean, Sig.: high Significance (P-value≤0.05).

**Table (3):** Independent sample T-Test of push-out bond strength for GuttaFlow Bioseal sealer of both groups C1 and C2.

| GuttaFlow Bioseal Groups of irrigation | N | Mean | Std. Deviation | Std. Error Mean | t-value | Sig.* |
|---|---|---|---|---|---|---|
| Group C1 (without smear layer) | 30 | 1.833 | 0.793 | 0.144 | | |
| Group C2 (with smear layer) | 30 | 2.275 | 0.850 | 0.155 | 2.08 | 0.042 |

N: Number of samples, Std. Deviation: standard deviation, Std Error Mean: standard error mean, Sig.: Significance (P-value≤0.05).

## DISCUSSION

Treating the surface of root canal dentin with different endodontic irrigants during chemomechanical preparation can induce some alterations in its structure and chemical composition and changing its solubility and permeability. This affects the bond strength of endodontic sealers to root canal dentin [(19)]. Many authors have suggested the successive use of organic and inorganic solutions as endodontic irrigants because no single solvent has proved to be able to remove the smear layer alone [(2)]. In the present study, irrigation of group 1 was done using (3ml) of (2.5%) sodium hypochlorite (NaOCl) since it is the simplest available endodontic irrigant having an organic tissue dissolving property. Inorganic component of smear layer was removed using (5ml) of (17%) EDTA remained in the canal for 1 minute since it may cause peritubular and intertubular erosion of the dentin if it is applied for more than this time [(20)]. The combined use of these two irrigants represents the most commonly used protocol in clinical practice. In this study, three different sealers were tested, therefore the use of gutta-percha as a main core material was to be considered as a constant, besides to firmly simulate the clinical conditions [(21)]. A single master cone of gutta-percha was placed with a sealer to provide a final compact mass with no spaces [(22)]. The push-out bond strength test was conducted in the current in-vitro study as it is one of the most reliable and reproducible techniques [(23)]. It can evaluate the root canal sealer materials even with low values of bond strength [(24)]. The results of this study have shown that the smear layer removal obviously increased the bond strength of AH Plus sealer. This finding is in agreement with that of Donnermeyer *et al.,* (2019) [(25)]; and Jain *et al.,* (2019) [(19)]. This might be related to the fact that smear removal allows intimate contact between resin sealer and dentinal surface creating an effective micro-retention due to sufficient penetration of resin into the dentinal tubules. Moreover, the inherent volumetric expansion property of AH Plus sealer that can form a covalent bond between open epoxide ring of epoxy resin sealer and the exposed amino-groups of radicular dentin. The complete exposure of the amino-groups following smear removal can increase the number of the covalent bonds leading to a more potent link of AH Plus to radicular dentin[(25)(19)].

In accordance with the data of the present study, the push-out bond strength of both GuttaFlow 2 and GuttaFlow Bioseal has a lesser value when the smear layer was removed compared with the group in which smear layer is preserved. This agrees with a previous study of Upadhyay *et al.,* (2018) [(11)]. The chelating solutions not only eliminates the smear layer, but it can demineralize the intertubular and peritubular dentin leading to collagen fibers exposure and patent dentinal tubules with increased surface roughness thus it decreases the surface free energy of radicular dentin [(26)(27)]. The resultant coarse hydrophobic surface of the dentin makes the wetting of silicone-based sealers to the radicular dentin poorer. In addition, the silicone presents in GuttaFlow sealers can produce greater forces of surface tension which makes the material flow more difficult into the rough surface [28] resulting in lower bond strength. On the other hand, according to Nagas *et al.*, (2012)[29] the calcium silicate containing material needs moisture during setting to get high strength and resist forces of dislodgment. Once GuttaFlow Bioseal contacts with the tissue fluids, the bioactive material yields calcium silicate that will form a physical bond with the dentinal surface through formation of hydroxyapatite interface deposits [30][6]. After release of such ions, tag like structures will extend into the dentin. This will significantly improve sealer's adhesion and hence the push out bond strength [31]. So that, the maintenance of the smear layer leads to increase of dentinal wall moisture condition which could have a positive influence on the adhesion of GuttaFlow Bioseal material.

## CONCLUSION

According to the types of the sealer used in this study and smear layer treatment condition, the push out bond strengths was influenced by sealer types and by existence or absence of the smear layer.

### Conflicts of interest

There are no Conflicts of interest.

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