

# Sealing ability of gutta-percha/Nano HA versus Resilon/Epiphany after 20 months using an electrochemical model – an *in vitro* study

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## Abstract

**Aim:** To evaluate the sealing ability of gutta-percha-nano-HA and Resilon-Epiphany by electrochemical method and micro-computed tomography (CT) scan at 48 h and 20 months using three different obturation techniques (cold lateral condensation technique, warm vertical condensation - System B, and warm vertical condensation with vibration - Down-Pak). **Methods:** 150 human mandibular single-rooted premolars were prepared and randomly allocated into 6 groups of 25 specimens each, and filled with either gutta-percha-nano HA or Resilon-Epiphany with the three different obturation techniques (cold lateral, warm vertical - System B, and warm vertical with vibration - Down-Pak). Electrochemical microleakage method was used to measure the microleakage after 48 h and after 20 months, and a micro-CT Scanner 1072 was used to evaluate the quality of obturation after 48 h. **Results:** Group 6 (Resilon-epiphany/Down-Pak technique) had the highest microleakage value, followed by Group 2 (Gutta-percha-nano HA/System B technique), Group 4 (Resilon-Epiphany/Lateral condensation technique), Group 3 (Gutta-percha-nano HA/Down-Pak technique), Group 5 (Resilon-Epiphany/System B technique), and Group 1 (Gutta-percha-nano HA/Lateral condensation technique) with the values of 4.69 (6.06) KÙ, 3.88 (2.92) KÙ, 3.77 (3.75) KÙ, 3.38 (3.92) KÙ, 2.64 (2.90) KÙ, and 2.44 (4.34) KÙ, respectively. No significant difference in the quantity of leakage was observed for each root in each group between the two tested filling materials and their sealers ( $p=0.143$ ). Micro CT scan investigations revealed more micro-voids in the Resilon-Epiphany Group obturated with Down-Pak technique. **Conclusions:** Nano-hydroxyapatite sealer with gutta-percha filling material provided a reasonable seal compared with Epiphany sealer and Resilon filling material.

**Keywords:** Resilon-epiphany, gutta-percha-Nano HA sealer, root canal filling, electrochemical test, micro-CT scan.

Received for publication: May 18, 2012

Accepted: September 13, 2012

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## Introduction

Obturation of the root canal is the final step of the endodontic treatment. The success of this step depends mainly on the root filling materials and techniques, which should have the ability to eliminate leakage from the oral cavity and periodontal tissues after cleaning and shaping. A number of obturation

techniques have been used over the years<sup>1-7</sup>. It is believed that the excellence of obturation seal is primarily related to the cohesive and adhesive interaction of filling materials to the root canal walls<sup>8-10</sup>. Recently, a new sealer has been introduced by the School of Dental Sciences, Universiti Sains Malaysia, Malaysia, known as Nano-Hydroxyapatite (Nano-HA) sealer<sup>11</sup>. The nano HA crystals, which range from 40-60 nm in size, are synthesized by wet chemical method using calcium hydroxide [Ca(OH)<sub>2</sub>] and phosphoric acid (H<sub>3</sub>PO<sub>3</sub>) as Ca and P precursors, respectively. The sealer is composed of nano-hydroxyapatite as a filler, bismuth (III) oxide as a radiopacity component and hexamethylenetetramine as a polymerization activator, and the liquid is Bisphenol A diglycidyl ether<sup>12</sup>. Previous investigations have shown no significant difference between the sealing ability of nano HA compared with AH 26, using dye penetration technique<sup>11-12</sup>.

On the basis of the in-vitro and in-vivo data available, several leakage studies have been conducted comparing Resilon-Epiphany with gutta-percha and conventional sealers. Onay et al.<sup>13</sup> (2009) using the fluid filtration method and Kaya et al.<sup>14</sup> (2007) using glucose penetration method have shown that the quality of apical seal achieved with gutta-percha and AH Plus or AH26 in combination is similar to that of Epiphany-Resilon. However, other investigators have shown, using the same techniques, that the better quality of apical seal is achieved with Epiphany-Resilon combination due to superiority of this bonding system<sup>14-16</sup>. As the bonding systems have improved, so has the resistance to bacterial and fluid penetration of the materials. However, no study has yet been conducted to evaluate the sealing ability of different materials and obturation techniques using electrochemical and micro-computed tomography (CT) scan. Therefore, the aims of this study were to evaluate the sealing ability of gutta-percha-nano-HA and Resilon-Epiphany by electrochemical method and micro-CT scan after 48 h and 20 months, using three different obturation techniques (cold lateral condensation technique, warm vertical condensation - System B, and warm vertical condensation with vibration - Down-Pak).

## Material and methods

### Selection of teeth

Extracted human single-rooted mandibular premolars were obtained and stored at room temperature in sealed vials containing saline immediately after extraction. The teeth were examined under digital stereomicroscope (Motic Digital Microscope, LTD, France) to discard those with any pre-existing root fractures. A sample of 150 teeth were selected and sectioned at the cemento-enamel junction using a diamond disc at a high-speed handpiece under continuous water spray coolant, according to the following criteria:

- The same root curvature; between 0°-5° (using Schneider technique)
- Diameter of apical foramen equals to K-file size 15
- Dentin thickness standardized between 2.5-3.5 mm from

the apical foramen to the cervical orifice.

Preoperative periapical digital radiographs from buccolingual and mesiodistal directions were taken to ensure that root samples had normal canal shape and enough thickness of dentinal walls. Then the samples were randomly divided into 6 groups of 25 roots and kept in separate plastic containers. Each sample was given a unique number.

### Sample preparation

Working length for each root canal was established using a size 15 K Flex file (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA). File was placed and advanced into the root canal until its tip was visualized at the apical foramen. The working length was set at 1 mm shorter of the apical foramen and it was equal to 15 mm. All canals were instrumented with Pro file Ni-Ti (Dentsply-Maillefer, Ballaigues, Switzerland) rotary instruments up to a size 35 master apical file following manufacturer's instructions and using crown down pressureless technique.

During preparation and between each profile file, the canals were irrigated with 2 mL of 5.25% NaOCL (Farmácia Amazon, São Carlos, SP, Brazil). After instrumentation, the canals were rinsed initially with 5 mL of 17% EDTA to remove the smear layer and followed by 5 mL of distilled water to remove any residues of NaOCL.

### Sample obturation

Group 1 (n=25): The prepared root canals were obturated with gutta-percha and Nano-HA endodontic sealer using cold lateral condensation technique. A master cone size 35 Profile was fitted to the working length and presence of tug-back was confirmed. Nano-HA sealer was mixed according to manufacturer's instructions to a creamy consistency and applied into the canals using lentulo spiral (Dentsply-Caulk, Milford, DE, USA) which was inserted within 2 mm short of the working length. After placement and condensation of the master cone at the appropriate working length, accessory cones were placed and condensed using a finger spreader (Miltex, Inc., York, PA, USA). The excess of gutta-percha was removed with a heated instrument and condensed vertically with plugger (Nordent, USA) to the level of the canal orifice.

Group 2 (n=25): Similar to group 1, the prepared root canals were obturated with gutta-percha and Nano-HA endodontic sealer using vertical condensation technique System B (Analytic, Sybron Dental Specialties, Orange, CA, USA).

Group 3 (n=25): Similar to group 1, the prepared root canals were obturated with gutta-percha and Nano-HA endodontic sealer using vertical and vibration condensation technique Down-Pak (EI-Endo, Hu-Friedy, Chicago, IL, USA).

Group 4 (n=25): The prepared root canals were obturated with Resilon/Epiphany self-etching primer and Epiphany sealer (Pentron Clinical Technologies LLC, Wallingford, CT, USA). First, the primer was inserted into the root canals and excess was removed with paper point (Dentsply Tulsa Dental Specialties). Subsequently, Epiphany

sealer was mixed according to the manufacturer's instructions and inserted into the root canals with a lentulo spiral. Resilon master cone size 35 was placed into the root canal. Following the application of the sealer, root canal filling was completed by inserting accessory cones dipped in Epiphany sealer and laterally condensing with a finger spreader. Excess Resilon cones were removed with a heated instrument and condensed vertically with a plugger to the level of the canal orifice.

**Group 5 (n=25):** Similar to group 4, the prepared root canals were obturated with Resilon and Epiphany using vertical condensation technique System B (Analytic, Sybron Dental Specialties, CA, USA).

**Group 6 (n=25):** Similar to group 4, the prepared root canals were obturated with Resilon and Epiphany using vertical and vibration condensation technique Down-Pak (EI-Endo, Hu-Friedy).

Postoperative radiographs were taken to ensure complete and void-free obturation. All the samples were wrapped in wet piece of gauze to assure 100% humidity and were stored individually in screw-capped glass vials in an incubator at 37°C for 48 h and 20 months.

### III) Quantitative microleakage measurement

Apical leakage of the obturated root canals was assessed by ac-impedance technique at 48 h and 20 months. A PVC-insulated copper wire with a 5.0mm bared end was inserted coronally into the obturated canal of each root and sealed in position with sticky wax. Thereafter, the coronal 2/3 of the external roots surfaces as well as the root/wire junctions were sealed with three layers of nail varnish. Each root was immersed in an electrolyte solution (0.9% sodium chloride solution) at room temperature. Then the ac-current was applied between the electrodes.

The current flow, denoting onset of leakage, was measured by IR drop across a 10-Ohm resistor placed in series with the electrodes and power source. Then after 20 months, 2 samples from each group were securely placed and fixed into the sample holder of the SkyScan micro-CT scanner 1072 (Micro Photonics Inc., Allentown, PA, USA) at 100 kV and source current at 120 MA mps, beam hardening set to 10. Each sample was placed for roughly 2 h to produce 1000 projections in TIFF. These images were then converted to tomograms (cross sections) saved in BMP, using NRecon (version 1.4.3; SkyScan). Next, image were examined for microleakage using image analysis programs provided by SkyScan (T-view and CT-an) using ANT for the 3D reconstruction for creating the 3D model.

## Results

The results from this study seem to suggest that, Group 6 had the highest microleakage value, followed by Group 2, Group 4, Group 3, Group 5, and Group 1 with the values of 4.69 (6.06) KÙ, 3.88 (2.92) KÙ, 3.77 (3.75) KÙ, 3.38 (3.92) KÙ, 2.64 (2.90) KÙ, and 2.44 (4.34) KÙ, respectively. No statistically significant difference in the quantity of leakage

was observed for each root in each group between the two tested filling materials and their sealers ( $p=0.143$ ), as shown in Table 1.

Down-Pak technique group exhibited the highest

**Table 1.** Comparison of microleakage values among the groups after 20 months of evaluation (Kruskal-Wallis test).

Groups	N	Median (IQR) KÙ	p value
Group 1	25	2.44 (4.34)	0.143
Group 2	25	3.88 (2.92)	
Group 3	25	3.38 (3.92)	
Group 4	25	3.77 (3.75)	
Group 5	25	2.64 (2.90)	
Group 6	25	4.69 (6.06)	

Group 1= Gutta-percha-nano HA/Lateral condensation technique; Group 2= Gutta-percha-nano HA/System B technique; Group 3= Gutta-percha-nano HA/Down-Pak technique; Group 4= Resilon-epiphany/Lateral condensation technique; Group 5= Resilon-epiphany/System B technique; Group 6= Resilon-epiphany/Down-Pak technique.

microleakage value ( $4.33 \pm 2.61$ ) KÙ, followed by the lateral condensation technique group ( $3.96 \pm 2.36$ ) KÙ, and System B group ( $3.60 \pm 1.88$ ) KÙ. In addition, no significant difference was observed among all experimental groups based on the obturation technique used ( $p=0.296$ ), as shown in Table 2.

The results of comparison between microleakage

**Table 2.** Comparison of microleakage values (one-way ANOVA test).

Technique	n	Mean(SD) KÙ	p value
Lateral Condensation	50	3.96 (2.36)	.296
System B	50	3.60 (1.88)	
Down-Pak	50	4.33 (2.61)	

measured at 48 h and 20 months was not statistically significant ( $p<0.05$ ) as shown in Figure 1.

Figure 2 (A, B and C) depict Micro-computed tomography reconstructions of root canal geometry of samples obturated with Nano-HA and Gp using cold lateral condensation technique, System B and Down-Pak technique. While, Micro-computed tomography reconstructions of root canal geometry of samples obturated with Resilon-Epiphany system using the same three obturation techniques are shown in Figure 3 (A, B and C). Micro CT scan investigations showed micro-voids were observed more in the Resilon-Epiphany Group obturated with the Down-Pak technique.

## Discussion

The results of the present study indicate that there was no significant difference in the sealing ability between the two tested filling materials and their sealers ( $p=0.143$ ) using the three obturation techniques. This may be due to Nano-HA epoxy resin, which has been shown to have a reasonable sealing ability<sup>12,17-18</sup>. In addition, it was reported that Nano-HA sealer was not affected by heat application during

continuous waves of condensation technique<sup>19-20</sup> unlike the Resilon material. The present experimental work indicated that Resilon-Epiphany root canal fillings when compared with gutta-percha/Nano-HA have no significant difference in the sealing ability when the whole length of root canal was filled with lateral, vertical and vibration techniques measured after 20 months of obturation. However, Shemesh et al.<sup>21</sup> (2008) found that, the apical 4 mm of Resilon-Epiphany root canal fillings allowed more glucose penetration than gutta-percha. Shipper et al.<sup>16</sup> (2004) detected faster bacterial leakage in gutta-percha and AH26 filling when

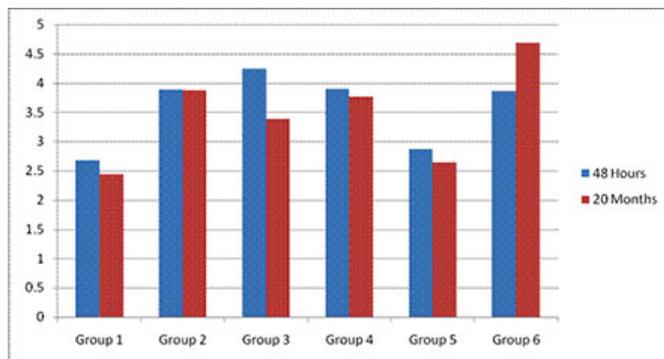


Fig. 1. Mean of Microleakage of each group after 48 h and 20 months.

compared with Resilon/Epiphany during a period of 31 days for the entire root length.

This study showed that there is no significant difference in the sealing ability of Resilon-Epiphany using 3 three different obturation techniques compared with gutta-percha-nanoHA within 48 h. However, after 20 months, sealer dissolution resulted in gap formation between root dentin and root filling material. According to De Munck et al.<sup>22</sup> (2005), after 3 months, all dentin adhesives under investigation exhibited mechanical and morphological evidence of degradation. As the bond degrades, interfacial microleakage increases, which resembles the in-vivo ageing effect. In the present study, the ageing effect through storage of the specimens over 20 months possibly evokes the current limitations of dentin bonding in the root canal system. In addition, the polymerization shrinkage of the Epiphany sealer and water sorption and solubility play an important role concerning the increased microleakage in the long term<sup>23</sup>. The solubility values reported by Versiani et al.<sup>24</sup> (2006) were 3.41% for Epiphany and 0.21% for AH Plus. Bonding is further compromised in sclerotic dentin, which is found more often in the apical area of permanent teeth<sup>25</sup>.

Electrochemical leakage tests offer advantages in terms of speed, accuracy and efficiency, as well as, its ability to

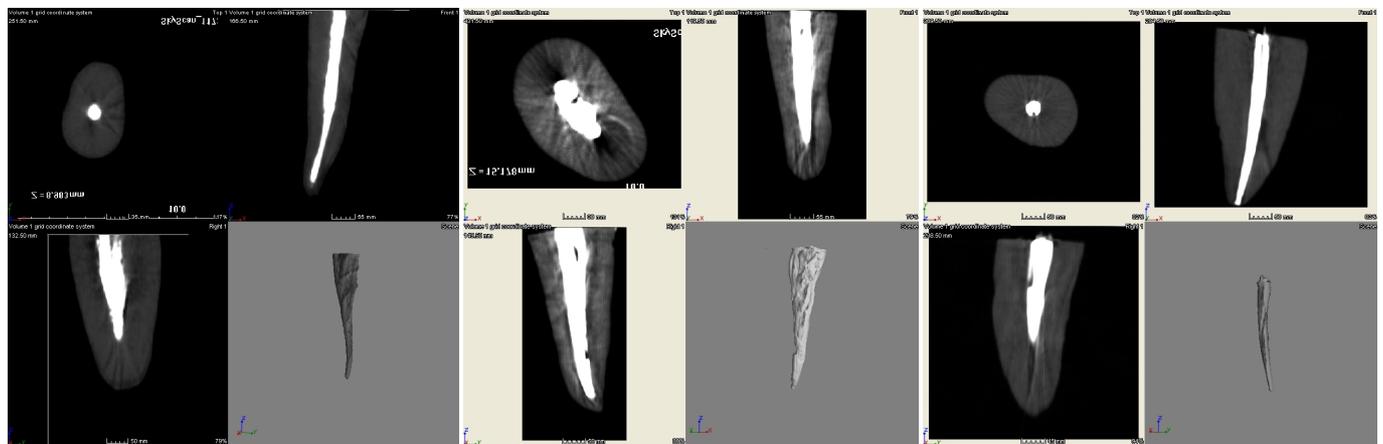


Fig. 2. Micro-computed tomography reconstructions of root canal geometry of samples filled with Nano-HA and Gp using cold lateral condensation technique (A), System B (B) and Down-Pak technique (C).



Fig. 3. Micro-computed tomography reconstructions of root canal geometry of samples filled with Resilon-Epiphany system using cold lateral condensation technique (A), System B (B) and Down-Pak technique (C).

perform longitudinal studies. The results of the present study corroborate those of previous investigations, even though, those studies used different methods for measuring microleakage, such as dye penetration<sup>26</sup>, bacterial leakage<sup>27</sup>, fluid filtration method<sup>17</sup> and glucose penetration method<sup>21</sup>. All of these studies were short-term experiments of no more than 3 months, except for one study with duration of 16 months<sup>25</sup>. The present study had duration of 20 months. Micro-CT scan investigation of samples showed more micro-voids for Resilon groups obturated by Down-Pak technique than in gutta-percha groups. Sealing ability of both Nano-HA sealer and Epiphany sealer was similar by using three techniques: cold lateral, heat applied by System B or heat and vibration applied by Down Pak pressure. As it can be seen clearly in micro-CT reconstructions in Figures 1 and 2, none of the techniques was superior in creating a hermetic seal of root canals. Generally, the micro CT scan view of root obturated with Resilon-Epiphany Down-Pak System showed poor adaptation of Resilon-Epiphany with a massive amount of voids, which coincide with the result of microleakage using electrochemical test. The electrochemical test showed that this group has the highest amount of leakage, 4.69 KÛ. However, statistically it cannot be compared between the two tests because the sample size in the micro-CT scan analysis was too small, and the machine is very expensive and time consuming test.

Nano-hydroxyapatite sealer with gutta-percha filling material provides a reasonable seal as compared to epiphany sealer and Resilon filling material. As such, it could be used as an alternative to the commercial available sealer whereas techniques and duration have no effect on the results. None of the filling materials and techniques used in the present study provided a complete tight seal in a three-dimensional manner.

Further *in vitro* studies should be conducted to evaluate the effect of increasing temperature and vibration of Down-Pak obturation technique on adhesion, dimensional stability and setting time properties of the tested obturation materials (Nano-HA with gutta-percha or Epiphany with Resilon).

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