An evaluation of the bond strength of various adhesive root canal filling materials

Thesis

Submitted to the Faculty of Dental Medicine - Al-Azhar University
in Partial Fulfillment of the Requirements for
Master Degree in Endodontics

By

Amr Abdelwahab Abdelhamed Bayoumi

B.D.S (Al-Azhar University 2004G),

Demonstrator of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University (Cairo-Boys).

Endodontic Department,

Faculty of Dental Medicine,

Al-Azhar University (Cairo-Boys)

2014G - 1435H

Supervisors

Dr. Taher Medhat Islam

Associate Professor and Head of Endodontic Department,

Faculty of Dental Medicine,

Al-Azhar University,

(Cairo-Boys)

Dr. Ashraf Samir Mahmoud Refai

Lecturer of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University,

(Cairo-Boys)

Dr. Motaz-Bellah A. Al-Khawas

Lecturer of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University,
(Cairo-Boys)

I would like to dedicate this work to my parents & my dear wife for their endless love, support & encouragement so this work could be undertaken

Acknowledgment

I would like to thank, first and foremost ***Professor Dr. Taher Medhat Islam,*** for his insightful guidance, advice and support throughout the course of this study.

I would like to express my great appreciation to ***Dr. Ashraf Samir Refai***, for his valuable and constructive recommendations during the planning and development of this research work.

I would also like to acknowledge and extend my heartfelt gratitude to ***Dr. Moataz AlKhawas*** for his advice and assistance in keeping my progress on schedule.

I wish to thank ***Dr. Mohamed Abbas,*** Lecturer of Dental Biomaterials, Faculty of Dental Medicine, Al-Azhar University, for his contribution and valuable technical support on this work.

Finally I would like to express my deepest thanks to all members of Endodontic Department Al-Azhar University.

Table of Contents

List of Tables

List of Figures

List of abbreviations

Abbreviation	Structure, meaning, description	page
4-META	4-Methacryloloxy Ethyl Trimellitate Anhydride	2
PMMA	Poly Methyl Methacrylate-Based	2
poly(HEMA)	poly [2-Hydroxylethyl Methacrylate]	8
RDIZ	Resin-Dentin Inter-Diffusion Zones	8
NaOCl	Sodium Hypochlorite	8
EDTA	EthelyneDiamineTetraAceticacid	8
GP	Gutta percha	8
SE	self-etching	11
SEM	scanning electron microscope	13
RS	RealSeal	13
ADH	AdheSE DC	13
EXC	Excite DSC	13
FESEM	field emission scanning electron microscopy	13
TEM	transmission electron microscopy	13
MTBS	microtensile bond strength	19
FEA	finite element stress analysis	21
MTA	Mineral Trioxide Aggregate	21

Introduction

Success in endodontic treatment is mainly determined by a hermitically three dimensional obturation of root canal system after chemo-mechanical preparation. The main objective of root canal obturation is to create a fluid tight seal throughout the root canal system to eliminate coronal and apical microleakage [1].Traditionally, obliteration of the root canal system has been done using gutta percha obturating material in combination with zinc oxide based sealer. Unfortunately, this combination does not completely prevent bacterial leakage due to lack of adhesion to radicular dentin [2,3,4].

Therefore, good adhesion within the root canal system is one of the ideal properties of a sealer which potentially influences both leakage and root strength [5,6]. Adhesion of a sealer to the dentinal walls seems advantageous for two main purposes; in a static situation, it should eliminate any space that may allow percolation of fluids between the obturating material and the canal wall. While in a dynamic situation, it should resist dislodgement of the obturating material during subsequent manipulation [7,8].

There has been a continuous quest throughout the history of endodontics for a sealing material that bond to canal walls as well as to the core material to form a "monoblock" [9].This concept is theoretically related to a gap-free solid filling mass that is able to produce a real fluid-tight seal and also improve the fracture resistance of the root [10].

Two prerequisites are simultaneously required for a "monoblock" to function as a mechanically homogenous unit. First, the adhesive material used should have the ability to bond strongly and mutually to one another, as well as to the substrate that the monoblock is intended to reinforce. Second, the materials should have modulus of elasticity that is similar to that of dentin [11].

Lately, the introduction of a polybutadiene-diisocyanate-methacylate resin-coating of gutta-percha enables the gutta percha to be chemically coupled to methacrylate-based resin root canal sealers. A dual curable methacrylate-based resin sealer (EndoREZ) has been introduced that can be used in combination with gutta percha or with resin-coated gutta percha to form a "monoblock" [12].

More recently, a 4-methacryloloxyethyl trimellitate anhydride (4-META) containing polymethyl methacrylate-based (PMMA) endodontic sealer (MetaSEAL) has been introduced. MetaSEAL a self-etching, hydrophilic sealer can promote monomer diffusion into the underlying intact dentin to bond to radicular dentin as well as thermoplastic root filling materials via the creation of hybrid layers in both substrates. This allows the two materials to bond [13,14].

Several studies have investigated the adhesion of root canal filling materials to the root canal walls. Among of the methods is the push out test which gives an accurate quantitative value for each specimen [15]. Very little studies have been done to evaluate the push out bond strength of these new obturation materials.

Review of literature

Section outline:

2.1 Bonding of adhesive root canal obturating materials.

2.2 Effect of different variables on bond strength of adhesive root canal obturating materials.

Review of literature

2.1 Bonding of adhesive root canal obturating materials:

One of the main objectives of root canal treatment is the total obliteration of the entire root canal system in order to provide a good seal and to allow healing of periapical tissues. Basically, obliteration of the root canal system has been done using gutta percha obturating material in combination with zinc oxide and eugenol sealer. Unfortunately, this conventional root canal obturating technique showed limitations in the bonding ability between sealer and canal wall or between sealer and core material [16].

Over the last decade, bonding of the root canal obturating material is based on the “monoblock” concept. This concept is theoretically related to a gap-free solid filling mass that is able to produce a real fluid-tight seal and also improve the fracture resistance of the root [10].

In 2007, **Tay & Pashley** [11] classified *“Monoblocks”* that created in the root canal spaces into *primary*, *secondary*, or *tertiary* depending on the number of interfaces present between the bonding substrate and the core obturating material.

A *primary "monoblock"* has only one interface that extends circumferentially between the core material and the root canal wall such as Hydron & orthograde MTA.

Secondary "monoblocks" are those that have two circumferential interfaces, one between the sealer and dentinal canal wall while the other between the sealer and the core material such as Resilon and MetaSEAL.

Tertiary "monoblocks" are those in which an additional circumferential interface is introduced by coating the non-bondable gutta percha points with materials that render them bondable to the root canal sealers such as EndoREZ &Activ GP.

According to **Schwartz R.** [(17)], a reliable *"monoblock"* root canal filling has facing some challenges because of some factors including:

a) The heterogeneous composition of root dentin as well as its complex anatomical configuration that includes fins, extensions and isthmuses.
b) The high frequency of sclerotic dentin in the apical third of the root canal.
c) The great amount of hard tissue debris accumulated in the canal space.
d) Unfavorable geometry of long narrow root canals that adversely affect the bonding as a result of unfavorable C-

factor (ratio of bonded to unbounded surfaces) of adhesive filling.

On the other hand, there are some limitations when using dentin bonding materials such as polymerization shrinkage, deterioration of the resin bond with time, functional forces and the incompleteness of polymerization and infiltration of resin into the demineralized dentin [18].

With regard to resin infiltration into the demineralized dentin, dentin morphology in root canal was evaluated in terms of tubule density, orientation and surface area after etching. The results revealed formation of a cylindrical hybrid layer which anchors the resin tags to adjacent intertubular dentin. It was concluded that the etching technique increased the surface area of demineralized dentin available for bonding to root canal, but not all areas exhibited equal responses to etching [19].

While with regard to the effect of functional forces, the regional bond strength of two resin cements (panavia and C&B metabond) in cervical, middle and apical regions of the root canal was investigated. The results indicated that both resin cements have high bond strengths and the apical region was significantly higher compared to middle or cervical third [20].

After introducing self-priming, self-etching, and self-adhesive resin luting technologies in restorative dentistry, low viscosity methacrylate resin–based root canal sealers have since been available for endodontic use. This generation of bondable root canal sealers has been aggressively promoted with the highly desirable property of creating *"monoblock"* within the root canal space [21,22].

Up to date, ***four generations*** of methacrylate resin–based sealers have been introduced. The ***first generation*** appeared in the mid-1970s was named Hydron (Hydron Technologies, Inc., Pompano Beach, FL). It based on poly 2-hydroxylethyl methacrylate [poly (HEMA)] as a major ingredient that rendered the sealer very hydrophilic [23]. Hydron was injected into root canals to be polymerized in situ [24], often in the presence of residual moisture to form soft hydrogels that are highly permeable and leachable [25,26].

Polymerized Hydron was histologically compared with fully set AH26 and Teflon by implanting into the mandible of guinea pigs at 2 days, 1, 2, 4, 12 and 26 weeks. Results showed that none of the materials tested elicited signs of significant tissue damage, and polymerized Hydron was assessed to be as biocompatible as fully set AH26 and Teflon. Bone was formed in very close apposition to the polymerized Hydron, whereas a soft tissue capsule separates the regenerated bone from implants of AH26 and Teflon [27].

The differences in the amount of leakage of human serum albumin placed into root canals obturated with gutta percha (lateral and vertical methods) and Hydron was determined quantitatively. Leakage results obtained for teeth obturated with gutta percha by lateral and vertical methods were not significantly different. While those obturated with Hydron, showed significantly greater leakage values than when using gutta percha [(28)].

In another study Hydron was clinically and radiographically evaluated in compared with gutta percha and AH-26 root canal obturating materials. A preoperative, postoperative and follow up radiographs were taken. Hydron showed radiographic signs of failure while, gutta percha and AH-26 root canal obturating materials showed no radiographic signs of failure. On the other hand, clinical examination on the selected patients revealed no adverse signs or symptoms. It was concluded that, both of Hydron and gutta percha& AH-26 root canal obturating materials were well accepted but on the basis of radiographic assessment, success with gutta percha and AH-26 root canal obturating materials was more predictable [(24)].

From the histological view, it was concluded that, Hydron is probably not a suitable obturating material when evaluating response in the periapical tissues to Hydron and gutta percha in monkeys as the former showed severe inflammatory reactions [(29)].

Lately, the introduction of a ***second generation*** of bondable sealer named EndoREZ (Ultradent Products Inc, South Jordan, UT) that utilizes polybutadiene-diisocyanate-methacylate resin-coating of gutta percha .This resin coating enables the polyisoprene component of gutta percha to be chemically coupled to methacrylate-based resin root canal sealers [30,31,32].

The effectiveness of using passively fitting resin coated gutta percha cones in combined with EndoREZ root canal sealer was evaluated. The hydrophilic nature property of the sealer enabled creation of an extensive network of 800 to 1200 µm long sealer resin tags after removal of the smear layer. Nevertheless, interfacial gaps and silver leakage could be observed along the sealer-dentin interfaces that might be attributed predominantly to polymerization shrinkage of the sealer [33].

Also the bond strength of various obturating materials to root canal dentin was evaluated using a push out test. Root canals were instrumented and irrigated using 5.25% sodium hypochlorite (NaOCl) and a final rinse with 17% EDTA. After instrumentation, the roots were randomly divided into five single-matched cone obturation groups as follows: group 1: gutta percha with Kerr EWT; group 2: gutta percha with AH

Plus; group 3: Resilon obturating system; group 4: Activ GP obturating system; and group 5: EndoREZ obturating system. The obturated roots were cut perpendicular to the long access to create 1-mm thick slices from the apical, middle, and coronal thirds. Results of bond strength showed that group 2 (GP/AH Plus) had a significantly greater bond strength compared with all other groups. Also, groups 1 and 4 (GP/Kerr & Activ GP) had significantly higher bond strengths compared with groups 3 and 5 (Resilon & EndoREZ) [(34)].

Additionally, The microshear bond strength of three resin based sealers (EndoREZ, AH Plus, and RealSeal) to root dentin was compared and failure modes were examined under light and SEM to assess the behavior in thin and thick films. Results showed that the epoxy resin-based sealers had the highest microshear bond strength to root dentin compared with urethane dimethacrylate based sealers. While the bond strength for the thick sealer group was significantly higher than the thin sealer group and may reflect different patterns of behavior when the sealer is present as a thin layer [(35)].

In another study, the bond strength and apical sealing ability of master gutta percha points with different tapers were studied as used in conjunction with EndoREZ ,AH Plus and Ketac-Endo. Each sealer was combined with one of the following obturating techniques: 0.02-tapered gutta percha master cone using lateral condensation, 0.04-tapered gutta percha master cone using lateral condensation, or 0.06-tapered gutta percha as a single cone. The bond strength and apical sealing ability were measured with the push out test and dye penetration test, respectively. The results showed that both the taper of the gutta percha point and root canal sealers had significant effects on push out bond strength. According to the microleakage data, AH Plus exhibited significantly lower overall leakage, whereas no difference was found between master cone points. In was concluded that the use of matched-taper gutta percha points in canals prepared with tapered rotary instruments may be advocated to improve bond strength ability of root canal filling material. Furthermore, AH Plus may be preferable to EndoREZ and Ketac-Endo in terms of improved sealing and bonding quality of filling material [36].

Also the shear bond strength of three sealers (EndoREZ, Diaket, and AH Plus) was evaluated. The smear layer of the exposed dentin surfaces were removed using 17% EDTA followed by 5.25% NaOCl and the teeth were randomly divided into two groups. Group 1: was kept as control and in group 2: uniform smear layer was created using waterproof polishing papers. Results showed a significant difference among the bond strength of the sealers, smear layer, and control groups. AH

Plus sealer showed the highest bond strength in smear layer removed surfaces. Also pretreatment with EDTA/NaOCl affected bond strength of AH Plus and AH Plus had the highest bond to dentin with or without smear layer [37].

Another study tested the hypothesis that the polymerization shrinkage can break the close initial contact between the main core and the surrounding dentin even if root dentin infiltration has occurred. The selected roots were prepared and divided into 4 groups; 1 and 2: hydrophilic resin filler injected and spread, roots sectioned after setting; group 3: hydrophilic resin filler injected and spread, dental substrate dissolved after setting; group 4: hydrophobic resin sealer lentulo-spiraled and spread, roots sectioned after setting (as control). Microscopic examination of the resin-dentin interfaces of groups 1 and 2 showed the existence of resin-dentin inter-diffusion zones (RDIZ); however, the close initial contact between the main core and the surrounding dentin was often lost. In group 3, resin tag morphology was well characterized and identical morphological features were observed in the resin-dentin interfaces in group 4. Since the existence of resin tags did not exclude the existence of a gap between the main core and the adjacent RDIZ, the present results confirmed the proposed hypothesis [32].

Also the improvement of the seal of EndoREZ with an adhesive-modified technique was assessed. Instrumented single-rooted teeth were filled with: (a) Warm vertical compaction with AH Plus (control); (b) EndoREZ with master cones and passive application of accessory cones; (c) Application of Clearfil Liner Bond 2V before EndoREZ and gutta percha condensation. Leakage was assessed by fluid filtration before root resection and after 3 to 12 mm apical resections. Results showed that EndoREZ exhibited significantly higher overall leakage, while no difference was found between AH Plus and the adhesive-modified EndoREZ technique. Apical resection of EndoREZ to 12 mm exhibited more leakage than all other interactions, but was not significantly different from the same material resected to 9 mm. it was concluded that, although EndoREZ exhibited an acceptable apical seal, its coronal seal may be improved with the use of a dual-cured self-etch adhesive [38].

Another alternative approach to optimize the bond strengths of filling materials to radicular dentin was performed using an indirect dentin bonding procedure with an acrylic core material. The selected roots were prepared and obturated with EndoREZ, Epiphany, or the bonding of an acrylic point with self-etching SE bond using a direct or an indirect bonding technique. Push out bond strength results of EndoREZ and

Epiphany to radicular dentin were less than the direct bonding technique with acrylic points and the self-etching adhesive with the indirect technique. It was concluded that, the use of the indirect bonding protocol with an acrylic point to compensate for polymerization stresses appears to be a viable means for optimizing bond strengths of endodontic filling materials to radicular dentin [(6)].

On the other hand, the feasibility of creating oxygen inhibition layers on resin-coated gutta percha cones via the adjunctive application of a dual-cured dentin adhesive just before bonding was examined. Composite cylinders were bonded with EndoREZ to flat, resin-coated gutta percha disks and similar disks that were post-treated with Prime&Bond NT dual cure adhesive and stressed to failure using a modified microshear testing design. Results showed a 5-fold increase in shear strength after adhesive application, with complex interfacial failures instead of complete sealer delamination from the resin-coating. It was concluded that, in-situ dentin adhesive application appears to have merits in enhancing the coupling of resin-coated gutta percha to methacrylate sealers [(39)].

Recently, a ***third generation*** of methacrylate resin-based sealers contains a self-etching primer and dual cured resin composite root canal sealers were introduced. The use of self-etching primers reintroduced the concept of incorporating smear layers created by hand/rotary instruments along the sealer-dentin interface when acidic primer is applied to the dentin surface that penetrates through the smear layer and demineralizes the superficial dentin [40,41].Provided that these materials are sufficiently aggressive to etch through thick smear layers, the technique sensitivity of bonding to root canals might be reduced when smear layers are inadvertently retained in the apical third of instrumented canal walls [42].

In 2004, Resilon (Resilon Research LLC, Madison, CT) a dimethacrylate-containing polycaprolactone-based thermoplastic root canal obturating material combined with self-etching adhesive and a dual-cured composite resin sealer was commercially introduced [43].

Bond strength, polymerization stress and flow of two resin based root canal sealers (AH Plus and dual cure Epiphany) were assessed. Bond strength was analyzed through push out test and failure mode was examined using scanning electron microscope (SEM). Polymerization stress was monitored for 60 min in 1-mm thick discs bonded to two glass rods attached to a universal testing machine. Flow was evaluated by measuring the diameter of uncured discs of sealer after 7 minutes compression between two glass plates. Results showed that Epiphany had higher flow and polymerization stress and lower bond strength values to dentin than AH Plus. In view of these findings, it can be implied that AH Plus would provide a better seal [44].

The interfacial strengths of Resilon/Epiphany and gutta-percha/AH Plus using a push out test design was compared and failure modes were examined using SEM. Results indicated that the gutta percha group exhibited significantly higher interfacial strength than the Resilon group. Also gutta percha root slices failed exclusively along the gutta percha/sealer interface but Resilon root slices failed predominantly along the sealer/dentin interface with recognizable, fractured resin tags. It was concluded that low interfacial strengths achieved with both types of root filling challenges the concept of strengthening root filled teeth with the new endodontic material [9].

Also the interfacial adaptation of gutta percha/AH-26 (GP), Resilon points/RealSeal (RS), AdheSE DC/Multicore Flow (ADH, self-etch control), and Excite DSC/Multicore Flow (EXC, total-etch control) was compared. Specimens were analyzed with electron microscopy using three methods: (a) field emission scanning electron microscopy (FESEM) of the interface; (b) transmission electron microscopy (TEM) of the interface; and (c) FESEM of the material fitting surface. The results showed that the three adhesive materials (RS, ADH, and EXC) formed a dentin hybrid layer, which nonetheless resulted in interfacial separation. Gaps were more frequent for GP, which did not hybridize dentin. The fitting surfaces exhibited resin tags at all levels for EXC. Tags were less frequent with ADH, especially in the apical third. For RS, resin tags were rare and virtually absent from the apical half, whereas GP did not form tags. It was concluded that despite the hybridization, a tight seal of the root canal is difficult to achieve because of the complexity and the mechanical challenge of the substrate [45].

In another study the adhesion of Epiphany and AH Plus to root dentin treated with 1% NaOCl and 1% NaOCl+17% EDTA, was evaluated using the push out test. Root cylinders were prepared and randomly assigned to 3 groups, according to root dentin treatment: group 1: distilled water (control), group 2: 1% NaOCl and group 3: 1% NaOCl+17% EDTA. Each group was divided into 2 subgroups filled with either Epiphany or AH Plus. Results showed that AH Plus sealer presented greater adhesion to dentin than Epiphany, regardless of the treatment of root canal walls [46].

Additionally the interfacial strength and failure mode of root canal fillings consisting of different technique/material combinations were evaluated using push out test. Root canals were filled with gutta percha or Resilon core materials combined with AH Plus, Ketac-Endo or Epiphany using cold lateral compaction or System B with Obtura II. Fracture modes of all root slices were evaluated stereomicroscopically. Results showed that all the parameters except compaction techniques had significant interactions. Gutta-percha/Ketac-Endo/cold lateral compaction and gutta-percha/AH Plus/cold lateral compaction groups had the highest micropush out bond strength values. It was concluded that the push out bond strength of Resilon/Epiphany combinations were lower than those of gutta percha/conventional root canal sealer combinations [47].

On the other hand, the fracture resistance of teeth filled with various canal filling materials was evaluated. The root canals were shaped and divided into six groups as follows: group 1: AH-Plus/gutta percha (Cold lateral compaction), group 2: Resilon/Epiphany (Cold lateral compaction), group 3: ActiV GP obturating system, group 4: ActiV GP sealer/gutta percha (Cold lateral compaction), group 5: No instrumentation or filling, group 6: Instrumentation but no filling. After the sealers had set, the roots were embedded in acrylic molds and subjected to a compressive loading at a rate of 1 mm/ min. Results showed that the fracture values of the experimental teeth were significantly higher than those of the instrumented but unfilled group. Also Teeth in the AH Plus/lateral compaction group had higher fracture resistance compared with the ActiV GP sealer/gutta percha group.it was concluded that, systems aiming to obtain a "monoblock" system were not superior to the conventional AH Plus/gutta percha technique in terms of fracture resistance [48].

While other investigators evaluated the adhesive strength of Resilon to RealSeal using a modified microshear bond strength test. Flat Resilon surfaces with different roughness were created for bonding to the sealer and compared to a composite control. The results of composite control group exhibited shear strength 7.3 to 26.9 times higher than those of the Resilon groups. Shear strength differences among the Resilon groups of different surface roughness highlighted the contribution of micromechanical versus chemical coupling in sealer retention. Ultrastructural evidence of phase separation of polymeric components in Resilon suggested that the amount of dimethacrylate incorporated into this filled, thermoplastic composite may not yet be optimized for effective chemical coupling to methacrylate-based sealers [49].

More recently, a ***fourth generation*** of methacrylate resin based sealers has eliminated the use of separate self etching primers by incorporating acidic resin monomers in the sealers to render them self-adhesive to dentin [50]. The combination of an etchant, a primer, and a sealer into an all-in-one self-etching, self-adhesive sealer is advantageous in that it reduces the application time as well as errors that might occur during each bonding step.

A material was developed by Sun Medical in Japan and is distributed under the name MetaSEAL™ (Parkell Inc, Edgewood, NY) in the USA and as Hybrid Root Seal (J. Morita Europe GmbH, Dietzenbach, Germany) in Europe.

MetaSEAL is the first commercially available fourth generation self-adhesive dual cured sealer [51]. The inclusion of an acidic resin monomer, 4-methacryloyloxyethyl trimellitate anhydride (4-META), makes the sealer self etching, hydrophilic, and promotes monomer diffusion into the underlying intact dentin to bond to radicular dentin as well as thermoplastic root filling materials via the creation of hybrid layers in both substrates [13].

In 2009, the true self-etching potential of MetaSEAL was evaluated. Mixed MetaSEAL sealer was applied to group1: fractured radicular dentin that was devoid of smear layers, group 2: instrumented canal wall radicular dentin that was irrigated with water as the final rinse to preserve the smear layer, and group 3: instrumented canal wall radicular dentin that was irrigated with EDTA as a final rinse to remove the smear layer. Cryofractured tooth halves without sealer application were examined using SEM to identify the characteristics of the bonding substrates. The other tooth-halves were filled with sealer and examined using TEM. Results showed that MetaSEAL did not demineralize fractured radicular dentin that was devoid of smear layer and smear plugs. The self adhesive sealer was incapable of etching beyond the 1- to 2-µm-thick smear layer retained on water-irrigated instrumented dentin to demineralize the underlying radicular dentin. It was concluded that the limited self-etching potential of MetaSEAL is a clinically legitimate concern. Incomplete smear layer removal from instrumented canal wall regions that are not reached by calcium chelating agents might jeopardize its bonding and sealing performance [(52)].

Also the interfacial strengths and failure modes of new polymeric endodontic obturating systems consisting of different material combinations were compared. Single-rooted teeth were instrumented and obturated with different combinations of core and sealer as follows: group 1: RealSeal/Resilon; group 2: RealSeal/Herofill; group 3: MetaSEAL/Resilon; group 4: MetaSEAL/Herofill; group 5: MM-Seal/Resilon; group 6: MM-Seal/Herofill; group 7: (control). Failure modes after push out testing were examined using stereomicroscope and SEM. Results showed that MetaSEAL/Resilon combination had significantly greater bond strength than all the other groups while RealSeal/Resilon combination proved to have the second highest bond strength. It was concluded that push out bond strengths of (MetaSEAL and RealSeal) and (Resilon) combinations were higher than epoxy-resin–based sealer (MM-Seal) and gutta percha (Herofill) combination [53].

While the morphological characteristics in the interface between resinous sealer and radicular dentin, SEM observation and microtensile testing of MetaSEAL and Super-bondTM RC sealer (Sun Medical; Japan) were compared with Epiphany and Epiphany SE. Results of SEM micrographs of MetaSEAL and RC sealer showed the formed hybrid layer and resin tags in all the resin dentin interfaces. Although those of Epiphany groups resin tags were observed in part of resin dentin interface, the hybrid layer was not observed. Microtensile testing of MetaSEAL and RC sealer were significantly higher than those of Epiphany groups. It was considered that MetaSEAL and RC sealer had good adaptation in resin-dentin interface due to high degree of infiltration and polymerization into radicular dentin. Also, formation of hybrid layer was not observed in case of Epiphany groups because NaOCl caused strong inhibition of polymerization. These results indicated that, MetaSEAL and RC sealer could prevent coronal and apical leakages by formation of hybrid layer to radicular dentin [54].

Microscopically, the gaps and voids occurring in roots obturated using three different resin based sealers were evaluated. Single-rooted teeth were instrumented and obturated with one of the following: Epiphany with Resilon, MetaSEAL with gutta percha or AH Plus with gutta percha using single-cone technique. After storage, the teeth were horizontally sectioned and photographs were taken from the coronal, middle and apical parts using a stereomicroscope .The images were analyzed using image analysis software. Results showed that MetaSEAL/gutta percha group showed more gap or void-free interfaces, While no significant difference was found in the scores for the gap areas and the MetaSEAL showed similar interfaces with Epiphany [(55)].

While the adhesive strengths, interfacial ultrastructure, and tracer penetration of a nonetching (EndoREZ) and two self adhesive methacrylate resin based sealers (MetaSEAL and RealSeal SE) were evaluated. A modified push out testing design was used to evaluate the adhesive strength while TEM was used to examine the ultrastructure and nanoleakage within the sealer dentin interface. Results showed that, both MetaSEAL and RealSeal SE exhibited higher push out strengths than EndoREZ when EDTA was used as the active final rinse. Also all the tested sealers showed a 1 to 1.5-µm thick zone of partially demineralized dentin, with the EDTA dentin demineralization effect masking the true self etching potential of MetaSEAL and RealSeal SE. It was concluded that incomplete smear layer removal from the apical third may jeopardize the performance of self adhesive sealers should they fail to self-etch without the adjunctive use of calcium chelating agents (56).

Also the push out bond strength to radicular dentin and SEM observations of MetaSEAL sealer were executed. Single-rooted teeth were instrumented and irrigated with the combination of EDTA and NaOCl and filled with MetaSEAL, Epiphany, Epiphany SE or AH26. From the results of push out testing, the value of MetaSEAL was significantly higher than those of the other sealers. Furthermore, failure mode of MetaSEAL was almost cohesive failure within the sealer itself. The SEM micrographs of MetaSEAL showed hybrid layer and resin tags were formed with no gaps in resin-dentin interface. It was concluded that MetaSEAL had the highest push out bond strength compared with the other resinous sealers and SEM observation showed good adhesion and adaptation by formation of hybrid layer in resin-dentin interface. These results indicated that MetaSEAL could act as a complete hermetic apical seal (57).

In another study the push out strength of MetaSEAL, Epiphany and Epiphany SE to root canal dentin was evaluated.

Roots were prepared and distributed to six groups according to the filling material: group 1: Epiphany SE, group 2: Epiphany primer and sealer, group 3: Epiphany primer, sealer and resinous solvent, group 4: Clearfil DC Bond and Epiphany sealer, group 5: Clearfil, Epiphany sealer and solvent and group 6: MetaSEAL. Resilon cones were used in all groups. Results showed that MetaSEAL had greater push out strength to root canal dentin than Epiphany SE and Epiphany. Also the use of primer, solvent and adhesive system did not influence the adhesion of Epiphany [58].

In another study, microtensile bond strength (MTBS) and SEM observation in resin dentin interface of MetaSEAL were executed to evaluate the effect on bondability as a function of time after aging. Single-rooted teeth were instrumented and irrigated with EDTA/NaOCl and then filled with MetaSEAL using a single cone technique. Specimens were cut horizontally against tooth axis and sliced into 0.8mm thick after the following storage periods in water-soaked gauze at 37°C: 24h, 1week, 2weeks, or 1month. The MTBS values increased with an increase of storage periods, while that of 2weeks was not significantly different from 1month. SEM micrographs of all samples demonstrated interfaces with approximately a 1-3µm thick hybrid layer and funnel-shaped resin tags into radicular dentin. It was concluded that, storage periods significantly affected the bondability of MetaSEAL because MTBS values increased according to increase of storage periods. It was considered that the performance of MetaSEAL should be evaluated over a period of 2weeks after root filling due to slow polymerization of this methacrylate based sealer [59].

Also the push out bond strength of different obturating materials was evaluated. Single-rooted human teeth were prepared and obturated with Resilon/RealSeal, Resilon/RealSeal SE, Resilon/MetaSEAL, or gutta-percha/Kerr EWT sealer. The roots were then sectioned into 1-mm-thick slices and subjected to vertical loading to displace the obturating material toward the coronal side of the slice. Slices were examined using a stereomicroscope at 30× to determine the mode of failure. Results showed that the push out bond strengths of Resilon/MetaSEAL and gutta-percha/Kerr EWT were significantly higher than either Resilon/RealSeal or Resilon/RealSeal SE but Resilon/MetaSEAL and gutta-percha/Kerr EWT did not differ significantly [60].

Additionally, the mechanical properties of MetaSEAL were compared over time to a traditional root canal sealer based on the Grossman formula. Flexural strength and flexural modulus of MetaSEAL and U/P Grossman formula root canal sealer were evaluated by 4 point bending test. Results showed that there was an increase in both flexural strength and modulus during aging for MetaSEAL. It was concluded that, MetaSEAL had much higher flexural strength and modulus than U/P and gave values that were closer to that of dentin, which may help distribute stresses between root dentin and root canal filling material and lead to longer endodontic success [61].

In 2011, the finite element stress analysis (FEA) of primary, secondary and tertiary "monoblocks" and the effect of interfaces on stress distribution were evaluated. Seven models representing different "monoblocks" using several materials were created as follows: (a) primary "monoblock" with Mineral Trioxide Aggregate MTA; (b) secondary "monoblock" with sealer (MetaSEAL) and Resilon; (c) tertiary "monoblock" with EndoREZ ; (d) primary "monoblock" with polyethylene fiber post-core (Ribbond); (e) secondary "monoblock" with glass fiber post and resin cement; (f) tertiary "monoblock" with bondable glass fiber post; (g) tertiary "monoblock" with silane-coated ceramic post. Results showed that maximum stresses were concentrated on force application areas. Also the stresses within the models increased with the number of interfaces both for the "monoblocks" created by the sealers and those created by post-core systems. It was concluded that, stresses within roots increased with an increase in the number of the adhesive interfaces. Creation of a primary "monoblock" within the root canal can reduce the stresses that occur inside the tooth structure [(62)].

While the dislocation resistance of MetaSEAL and AH Plus sealers using either a single cone technique or warm vertical compaction was compared using the push out test. The roots were sectioned at the coronal and middle thirds to obtain thin slices, which were subjected to compressive loading to displace the set sealer/filling toward the coronal side of the slice. The results showed that the push out bond strength of AH Plus was significantly higher than MetaSEAL irrespective of filling techniques. A minimal hybrid layer was seen in radicular dentin, and resin tags were inconsistently identified from canal walls in the MetaSEAL-filled canals. The lower dislocation resistance in MetaSEAL-filled canals challenges the use of a self adhesive bonding mechanism to create continuous bonds inside root canals [(51)].

Another study was held in 2012 evaluated the influence of MetaSEAL and AH Plus on the resistance to vertical root fracture when either the matched-taper single-cone or lateral condensation technique was used. All of the roots were mounted vertically and subjected to a vertical loading force (1 mm/min). Results showed that, groups in which AH Plus and MetaSEAL were used with the matched taper single cone technique showed significantly higher fracture resistance values than the instrumented but not obturated roots. The force required to fracture the roots in the group treated with AH Plus and the lateral condensation technique was similar to that required to fracture intact roots, whereas the group treated with MetaSEAL and the lateral condensation technique revealed comparable values to the instrumented but not obturated roots. Therefore when used with the matched-taper single-cone technique, MetaSEAL and AH Plus have the potential to reinforce endodontically treated teeth (63).

2.2. Effect of different variables on bond strength of adhesive root canal obturating materials:

With regard to the effect of irrigation on the bond strength, a study examined the effects of commonly employed endodontic irrigants on Epiphany-dentin bond strength. Smear layers were created on dentin discs obtained from human molars. teeth were treated with one of the following: (a) water; (b) 2% chlorhexidine CHX; (c) 6% sodium hypochlorite (NaOCl); (d) 6% NaOCl followed by EDTA and water; or (e) 1.3% NaOCl followed by MTAD(a mixture of tetracycline isomer, an acid, and a detergent). The treated surfaces were air dried and treated with Epiphany primer. Samples were tested for shear bond strength after 7 days of storage. Results revealed that using water or chlorhexidine as an irrigant resulted in significantly lower bond strength when compared with NaOCl, NaOCl/EDTA, or NaOCl/MTAD. Also neither EDTA nor MTAD significantly improved Epiphany-dentin bond strength when compared with NaOCl used alone (64).

Also the bond strength of Resilon/Epiphany SE and gutta percha/AH26 was assessed after different irrigation protocols. Selected teeth were divided into 4 groups as follows: 5.25% NaOCl followed by 17% (EDTA) and 1.3% NaOCl followed by MTAD (groups 3 and 4). The root canals were obturated with either gutta-percha/AH26 or Resilon/Epiphany SE. Results of Gutta-percha/AH26 showed significantly higher bond strength than Resilon/Epiphany SE. It was concluded that irrigation with 5.25% NaOCl/EDTA can be a better conditioner before using gutta-percha/AH26 while the bond strength of Resilon/Epiphany SE was not different after irrigation with 5.25% NaOCl/EDTA or 1.3% NaOCl/MTAD (65).

Additionally the effect of irrigants employed for removing smear layers on the formed hybrid layer by MetaSEAL was tested. Teeth were instrumented and irrigated as follows: group 1: EDTA as initial rinse/NaOCl as active final rinse; group 2: NaOCl as initial rinse/EDTA as active final rinse. Root slices derived from the coronal, middle and apical thirds of the roots were examined using TEM after removing the gutta percha, leaving the sealer intact. Additional filled canals from the two groups were evaluated for fluid leakage. Results showed that hybrid layer was absent in group 1 and was present only when a collagen matrix was produced by EDTA demineralization (group 2). Significantly more leakage was observed in the absence of dentin hybridization [(66)].

While the effect of four different irrigants on the adhesive strength of MetaSEAL and ActiV GP was compared using a push out test. Results showed that, smear layer removal by rinsing with citric acid and sodium hypochlorite for ActiV GP showed the lowest shear bond strength values of all ActiV GP groups. While the lowest shear bond strength value was measured in the MetaSEAL group after the final drying with alcohol. Results showed that MetaSEAL had significantly higher bond strength to dentin than ActiV GP regardless of the irrigation protocol and its adhesion to dentin is not influenced by the different irrigation protocols [(67)].

In 2013, the effect of different final irrigants on the bond strength of Epiphany/Resilon and Epiphany SE/Resilon was evaluated using push out test. The root canals were prepared and the smear layer was removed using 17% EDTA, Results of push out test showed that there was no significant difference between Epiphany/Resilon and Epiphany SE/Resilon.

Considering the irrigation protocols, final irrigation with 2.5% NaOCl was associated with significantly lower bond strength of both filling materials than the other irrigants. EDTA, CHX and normal saline had similar effects on the bond strengths of filling materials. It was concluded that**,** final irrigation of the root canals with 2.5% NaOCl following application of EDTA had a negative effect on the bond strength of Epiphany and Epiphany SE obturating systems [68].

In 2009, Hashem A. et al, [69] evaluated the bond strength of ActiV GP root canal system and gutta-percha/AH plus sealer when used after final rinse with different irrigation protocols. Results indicated that, the combination of EDTA/CHX/ActiV GP showed the highest bond strength value while the significantly lowest bond strength was recorded for EDTA/ActiV GP. It was concluded that the bond strength of ActiV GP was improved by using 2% CHX in the final irrigation after 17% EDTA, whereas CHX did not enhance the effect of MTAD on the bond strength of the material. Also the bond strength of gutta percha/AH plus was adversely affected by MTAD and MTAD/CHX.

The bond strength of Epiphany and AH Plus sealers to root canal dentin was evaluated using a push out test after the use of different irrigants. The specimen groups according to the dentin surface treatment were as follows: group 1: 1% NaOCl (30 min); group 2: 1% NaOCl (30 min)/17% EDTA (5 min); group 3: 17% EDTA (30 min); group 4: 24% EDTA gel (30 min); group 5: 2% CHX gel (30 min). Results of push out test showed that AH Plus had significantly higher bond strength than Epiphany. A 1% NaOCl/17% EDTA was associated with significantly higher bond strength values than the other irrigants. A17 % EDTA, 24% EDTA gel and 2% CHX gel had intermediate values that were not significantly different from each other. A 1% NaOCl was associated with the lowest mean values. Except for 1% NaOCl, the removal of smear layer with the other irrigants increased the bond strength of AH Plus to root canal dentin. Also the use of 1% NaOCl for 30 min with 17% EDTA as final irrigant for 5 min increased the bond strength of Epiphany [70].

The effect of either a strong (MTAD) or a soft (1-hydroxyethylidene-1, 1-bisphosphonate [HEPB]) chelating solution on the bond strength of Resilon/Epiphany root fillings was assessed. Both 17% EDTA and the omission of a chelator in the irrigation protocol were used as reference treatments. Results showed that EDTA and MTAD-treated samples revealed intermediate bond strength. The lowest bond strength was found in NaOCl treated samples while the highest bond strength was reached in the HEBP-treated samples. It was concluded that the soft chelating irrigation protocol (18% HEBP) optimized the bonding quality of Resilon/Epiphany root fillings [71].

Recently, the effect of a modified self-etching primer incorporating chitosan on the bond strength to radicular dentin and its antibacterial activity were examined. A modified self-etching primer was prepared by adding chitosan solutions at 0.03%, 0.06%, 0.12% and 0.25% (*W/W*) to RealSeal self etching primer. The results of push out test showed that the modified self-etching primer incorporating chitosan showed no significant differences in the bond strength as compared with the control. It was concluded that modified self-etching primer incorporating chitosan is a promising antibacterial primer which does not adversely affect the bond strength of the RealSeal system to radicular dentin [72].

Also the antibacterial activity of $Ca(OH)_2$ combined with chitosan solutions against E. faecalis-infected root canal dentin and their effects on the bond strength of RealSeal sealer were evaluated. An experimental intracanal medicament was prepared by mixing different concentrations of chitosan solution (25%, 50%, and 100%, *W/V*) to $Ca(OH)_2$powder. Results showed that $Ca(OH)_2$ combined with different concentrations of chitosan solutions showed better antibacterial activity than Ca (OH) $_2$mixed with saline, without significantly affecting the bond strength of RealSeal sealer to radicular dentin. The findings suggest that Ca $(OH)_2$ combined with chitosan is a promising intracanal medicament and may be effective in endodontic therapy (73).

The effect of various medications on microtensile bond strength of root canal sealers to root canal dentin was assessed. The root canal dentin walls were treated with either 5% NaOCl, 3% H_2O_2, the combination of H_2O_2 and NaOCl, or 0.2% CHX for 60 s; or Ca (OH) $_2$or formocresol for 24 h. The teeth in control group were irrigated with water. The root canals were obturated using C&B Metabond. Results of microtensile bond strengths indicated that NaOCl, H_2O_2, or a combination of NaOCl and H_2O_2 treatment decreased bond strength to root canal dentin significantly. While teeth treated with chlorhexidine solution showed the highest bond strength values (74).

The effect of placement of Ca (OH) $_2$ dressings on the bond strength of Epiphany resin based sealer to root dentin was evaluated. The teeth were assigned to 3 groups according to the intracanal dressing: group 1: Ca (OH) $_2$/saline; group 2: Ca (OH) $_2$/2% CHX gel; and group 3: saline (control). After 10 days of storage in 100% humidity at 37°C, the dressings were removed and the root canals were filled with Epiphany sealer. Results of push out test showed a statistically significant decrease in bond strength when a Ca $(OH)_2$ dressing was used before root canal filling with Epiphany but even though the values were within the acceptable range found in the literature [75].

While the effect of time and concentration of ascorbate on the bond strength was evaluated. Roots were prepared and irrigated with different combination and concentrations of NaOCl and ascorbate. All roots were then filled with C&B Metabond, stored 1 day in water, and then cross-sectioned, trimmed and tested for tensile bond strength. The results demonstrated that 5.25% NaOCl irrigation produced significant reduction in resin dentin bond strength, but this can be reversed by 10% ascorbate treatment for 1 min [76] .

An evaluation of the effect of lactic acid on the shear bond strength of Epiphany sealer to root dentin was compared with other irrigating solutions. Teeth were ground wet and treated with one of the following: 1) no irrigant (control); 2) 5% H2O2; 3) 5%NaOCl; 4) 15% EDTA; 5) 10% lactic acid; or 6) 20% lactic acid. Results showed improvement in Epiphany dentin bond strength with lactic acid when compared with other irrigants. It was concluded that lactic acid performed similarly to 15% EDTA and demonstrated higher bond strength of Epiphany sealer to dentin surface [77].

On the other hand, the effect of two gutta percha solvents (chloroform versus halothane) on microtensile bond strength to root canal dentin was evaluated. The root canals were treated with water, chloroform, or halothane for 60 s. All root canals were obturated using C&B Metabond. After 24 h of storage in distilled water, serial 1-mm-thick cross-sections were cut and trimmed. The results indicated that water-treated roots had significantly higher resin-dentin bond strengths compared with chloroform or halothane treatment groups. It was concluded that gutta percha solvents have an adverse effect on bond strengths of adhesive materials to root canal dentin [78].

The effect of different light-emitting diode (LED) polymerization modes on the bond strength of a methacrylate based sealer used with Resilon or gutta percha was investigated. The roots were randomly assigned into 1 of the following groups group 1: RealSeal/Resilon and group 2: RealSeal/gutta-percha. In each group, specimens were further sub grouped according to the LED polymerization mode used to: subgroup 1: standard (20 seconds of maximum intensity) and subgroup 2: exponential (5 seconds of exponential power increase, followed by 15 seconds of maximum intensity). Results showed that the tested polymerization modes had no significant effect on the bond strength values. RealSeal/gutta percha yielded significantly greater bond strength than RealSeal/Resilon. In all groups, the bond strength values decreased significantly from coronal to apical direction. It was concluded that the exponential photo polymerization mode had no significant advantage over the standard regimen in terms of dentin bond strength [(79)].

Also the effect of different photoactivation methods on the push out bond strength and coronal microleakage of the Epiphany/ Resilon were evaluated. Roots were prepared and obturated with Resilon cones. Then the specimens were assigned into three groups according to the light-curing unit (LCU) used; group 1: quartz-tungsten-halogen/40 seconds, group 2: light-emitting diode/20 seconds, and group 3: plasma arc/6 seconds. Failure modes were assessed quantitatively under a stereomicroscope and morphologically under SEM. Results revealed that both the type of LCU and the level of sectioning had significant effects on bond strength. The statistical ranking obtained for bond strength was as follows: quartz-tungsten-halogen > light-emitting diode > plasma arc. While coronal microleakage of specimens cured with the plasma arc was significantly greater than those of other groups [(80)].

Additionally the effect of 980 nm diode laser of different power and frequency on the bond strength of AH Plus and Epiphany sealers was investigated using the push out test. Results showed that the specimens irradiated with the diode laser and filled with AH Plus had significantly higher bond strength values than those irradiated and filled with Epiphany and the non-irradiated controls. It was concluded that 980 nm diode laser irradiation of root canal dentin increased the bond strength of AH Plus sealer, but did not affect the adhesion of Epiphany sealer [81].

While the effect of different root dentin surface treatment on the adhesion of Epiphany, Apexit Plus, and AH Plus sealers to root canal dentin was evaluated using the push-out test. Selected roots were instrumented and then were randomly assigned to four groups according to root dentin treatment: group 1: distilled water (control), group 2: 17% EDTAC, group 3: 1% NaOCl and group 4: Er:YAG laser. Results showed a significant difference among the dZntin surface treatments. The highest adhesion values were obtained with AH Plus when root dentin was treated with Er:YAG laser and 17% EDTAC. While Epiphany sealer presented the lowest adhesion values to root dentin treated with 17% EDTAC. It was concluded that resin based sealers had different adhesive behaviors, depending on the treatment of root canal walls [82].

In a recent study held in 2013, the effect of photoactivated disinfection (PAD) on the bond strength of root canal sealers to root canal dentin was compared using the push-out test. Root canals were prepared and the smear layer of the roots was removed using 17% EDTA followed by 5.25% NaOCl and distilled water. The roots were then randomly divided into 3 groups according to the final irrigation regimen. In group 1: PAD (FotoSan; CMS Dental, Copenhagen, Denmark) was applied to the root canals and light cured for 20 seconds. Group 2: was finally irrigated with a 2% solution of CHX, and group 3: served as the control group (NaOCl + EDTA). Results showed that there was no significant difference among the bond strength of PAD, CHX, and NaOCl. It was concluded that PAD does not adversely affect the bond strength of the AH Plus sealer to root canal dentin and that it can be used for the final disinfection of root canals [83].

In another recent study, the effect of different final irrigation activation techniques on the bond strength of an epoxy resin sealer (AH Plus) was evaluated .Roots were prepared and then randomly divided into 4 groups according to the final irrigation activation technique used as follows: no activation (control), manual dynamic activation (MDA), Canal Brush (Coltene Whaledent, Altststten, Switzerland) activation, and ultrasonic (US) activation. Results showed that bond strength values mostly decreased in the coronoapical direction. It was concluded that the bond strength of AH Plus sealer to root canal dentin may improve with ultrasonic activation in the coronal and middle thirds and MDA in the apical third [84].

Aim of the study

This study was directed to evaluate the bond strength of adhesive obturating materials using the push out test over a period of time.

Materials and Methods

Section outline:

4.1. Selection and preparation of the teeth

4.2. Grouping of the teeth

4.3. Cleaning and shaping of the root canals

4.4. Obturation of the root canals

4.4.1. Second generation methacrylate based sealer (EndoREZ)

4.4.2. Fourth generation methacrylate based sealer (MetaSEAL)

4.5. Incubation and storage of the root portions

4.6. Evaluation and statistical analysis of the root specimens

4.1. Selection and preparation of the teeth

Fifty six freshly extracted single rooted human mandibular premolars with fully formed root apices were selected to be used in this study. The selected teeth were examined radiographically from the buccal and mesial views to exclude any tooth having abnormalities such as root fractures, pulp stones or internal root resorption. The roots of the selected teeth were planned using an ultrasonic scaler[1] to remove any hard deposits on the root surfaces. This is followed by immersion in 5.25 % NaOCl[2] for 1 hour to remove any soft tissue debris that was present on the external root surface of the roots. Following immersion, the selected teeth were stored in normal saline at room temperature until the time of use.

4.2. Grouping of the teeth (Table 1&2):

The roots were divided into 2 main groups (28 each) according to the obturating material used (variable "A");

- Methacrylate-based sealer, EndoREZ (group A_1: 28 root portions).
- A 4-META based sealer, MetaSEAL (group A_2: 28 root portions).

1 WOODPEAKER, Made in China.
2CLOROX, Made in Egypt.

Each main group was further subdivided into 4 subgroups (7 each) according to the different storage period (variable "B");

- After 1 week (B_1),
- After 1 month (B_2),
- After 3 month (B_3),
- After 6 month (B_4)

Table (1): The different variables which were investigated.

Variables	Design	Description
Obturating material (A)	A_1 A_2	EndoREZ MetaSEAL
Storage period (B)	B_1 B_2 B_3 B_4	1 week 1 month 3 months 6 months

Table (2): Interactions between different variables (Factorial design)

B \ A	A_1	A_2	Total
B_1	A_1B_1	A_2B_1	14
B_2	A_1B_2	A_2B_2	14
B_3	A_1B_3	A_2B_3	14
B_4	A_1B_4	A_2B_4	14
Total	**28**	**28**	**56**

4.3. Cleaning and shaping of the root canals

Prior to cleaning and shaping, resection of crowns of the selected teeth was transversely done using a carborandum disc mounted on straight hand piece[3] under water coolant to produce standardized 14 mm root portions. After resection, patency and working length were established using a size #15 k-file[4] by introducing the K-file into the root canal until it reached the apical foramen and withdrawing 1 mm from this length. The root canals were instrumented in a crown down manner using

[3] D-mate,PM-LOW01 S, Delma Made.
[4] Mani, Inc,Tochigi, Japan

REVO-S Ni-Ti rotary system[5] in a file sequence SC1, SC2, SU, AS30, AS35 and AS40 with a speed and torque control setting as recommended by the manufacturer.

Between each file, irrigation was done using 1.8 ml of NaOCl (5.25 %) in a 30 gauge endodontic irrigating needle[6] to within 1-2 mm of the working length. All root canals were finally irrigated with 17% ethelynediaminetetraaceticacid EDTA[7]. Then EDTA was finally flushed out by rinsing the root canals with normal saline to stop the action of EDTA.

4.4. Obturation of the root canals

4.4.1. Second generation methacrylate based sealer (EndoREZ)

EndoREZ is a urethane dimethacrylate (UDMA) resin based sealer that has a filler content of approximately 50% by weight of bismuth oxychloride, calcium lactate pentahydrate and silicon dioxide. Resin coating contains gutta percha, zinc oxide, barium sulphate and coloring agents and is entirely coated with a thin layer of polymerized urethane dimethacrylate resin (UDMA).

5 Micro-Mega ,france.
6 (Max-I-Probe)Dentsply/Maillefer,Made in USA.
7 Dentsply,USA.

The EndoREZ[8] kit (Figure 1) is composed of TwoSpense (double barrel) EndoREZ Syringe, Ultra-Mixer tips, Skini syringes, Assorted NaviTip tips and EndoREZ Points.

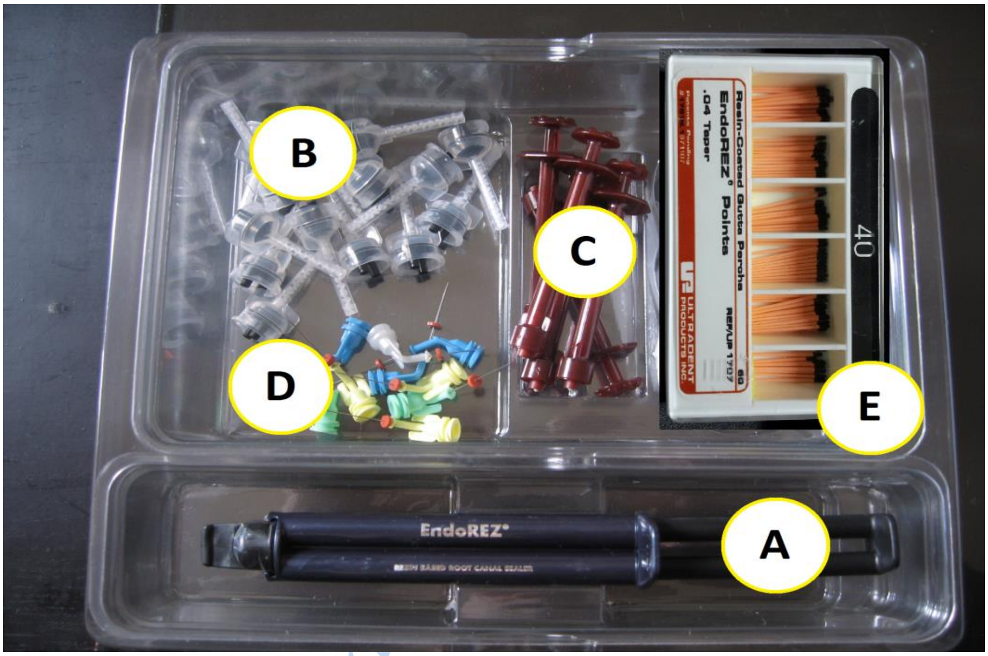

Figure (1): A photograph showing EndoREZ kit including 6ml TwoSpense EndoREZ Syringe (A), Ultra-Mixer tips (B), Skini syringe (C), NaviTips of different gauges and lengths (D) and EndoREZ points (E)

After cleaning and shaping of the root canals, a size # 40 resin coated gutta percha point was checked and its fit was verified radigraphically. Once the fit was verified, the master cone was removed from the canal. The mixing tip was attached

8 Ultradent Products Inc, South Jordan,UT.

to the EndoREZ syringe and the flow of two components (base and catalyst) of EndoREZ syringe was checked by injecting a small amount of material on a glass slab. The plunger of the Skini syringe was removed and the syringe was back-filled through the mixing tip of EndoREZ syringe. Then the plunger was slowly inserted to avoid trapping any air bubbles in the Skini syringe. The NaviTip was attached to the Skini syringe and the flow of EndoREZ was checked before delivery into the canal. The NaviTip was placed into the canal at a level 2-3 mm of the working length and EndoREZ was slowly injected into the canal. Once the canal was filled with EndoREZ, the NaviTip was removed and the prefitted size # 40 resin coated gutta percha point was placed into the canal to the full working length. Additional resin coated gutta percha cones were laterally compacted into the canal till it was filled using a finger spreader size # 30. Excess resin coated gutta percha was severed using a heated plugger 1mm below the canal opening.

4.4.2. Fourth generation methacrylate based sealer (MetaSEAL)

The MetaSEAL[9] kit (Figure: 2) is composed of powder and liquid, plastic measuring spoon, plastic spatulas and mixing pads. MetaSEAL Liquid: consists of monomer components

9. Parkell Inc, Edgewood, NY, USA.

include 4-methacryloxyethyl trimellitate anhydride (4-META) and 2-hydroxyethyl methacrylate (HEMA) while MetaSEAL Powder: consists of zirconium oxide, silica amorphous, water-soluble polymerization initiator.

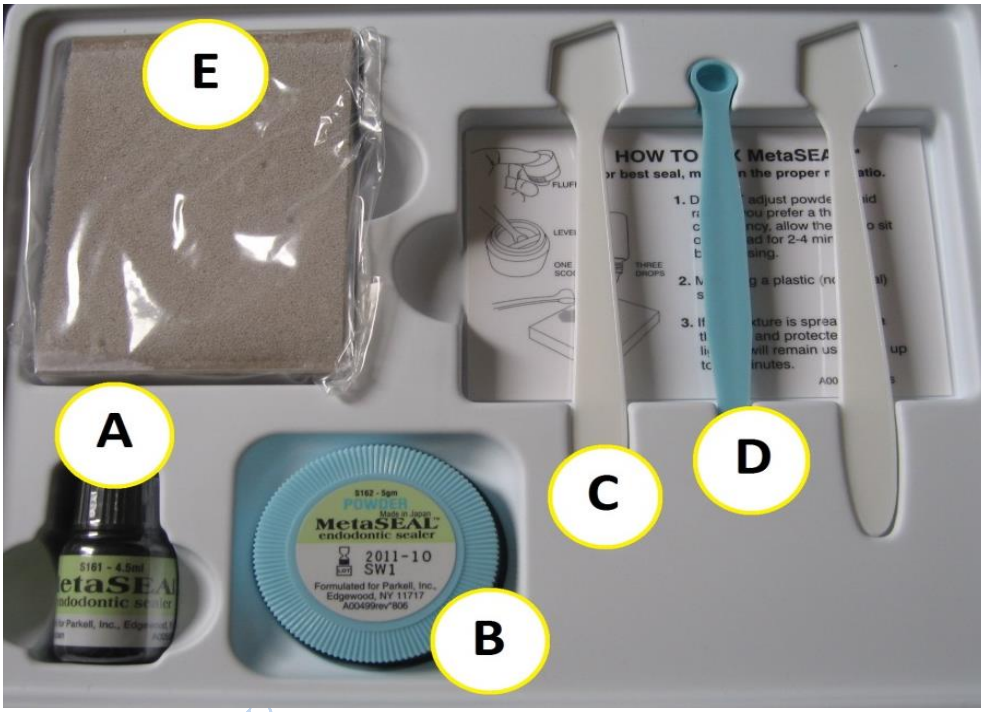

Figure (2): A photograph showing MetaSEAL kit including; 4.5ml MetaSEAL liquid (A), 5g MetaSEAL powder (B), Plastic spatula (C), Plastic measuring spoon (D) and Mixing pad (E)

MetaSEAL was hand mixed under aseptic condition with a plastic spatula according to manufacturer instructions. After mixing, the sealer was applied to the prepared canal using the gutta percha[10] master cone size # 40 followed by inserting the prefit gutta percha cone to the full working length. Additional gutta percha cones were laterally compacted into the canal till it was filled using a finger spreader size # 30. Excess gutta percha was severed using a heated plugger 1mm below the canal opening.

After obturation of the root canals in both groups, light curing was done for 40 seconds to initiate the polymerization process of sealers and create an instant coronal seal. This was followed by coronal sealing of the root specimens with Cavit[11] .

5. Incubation and storage of the root portions

After sealing the coronal portion of the root specimens with Cavit, all the specimens were placed in dry, cotton plugged glass tubes at 37° C in the incubator[12] for 48 hours to allow complete setting of all sealers. Following setting of the obturation materials, the specimens were stored for either, 1 week, 1 month, 3 months or 6 months (variable "B") at 37° C in a completely sealed glass tubes containing 0.9 % normal saline.

10. Dentsply-Maillefer, Ballaigues, Switzerland.

11 Cavit (3M ESPE, st Paul, MN).

12 PS.3A Advanced Technology, Egypt.

6. Evaluation and statistical analysis of the root specimens:

(Figures: 3-6)

After the end of each storage period, the root portions were prepared to evaluate their bond strength using the push out test. A specific cylindrical copper mold (14 mm in height and 10 mm in diameter) was fabricated for specimen housing (Figure:3). Copper mold was filled with self-cure acrylic resin and each root specimen was individually imbedded in the acrylic resin. After complete curing of the acrylic resin, the specimens were removed from the mold and serially sectioned using a low speed motorized disk under water cooling. Sections were prepared along the apical, middle and cervical segments of each root to produce three root slices of 2 mm thickness each. This resulted in 21 horizontal sections per subgroup with a total number of 168 horizontal sections for the two experimental groups. After sectioning, each root slice was marked from both sides to determine the apical and coronal sides with a marker.

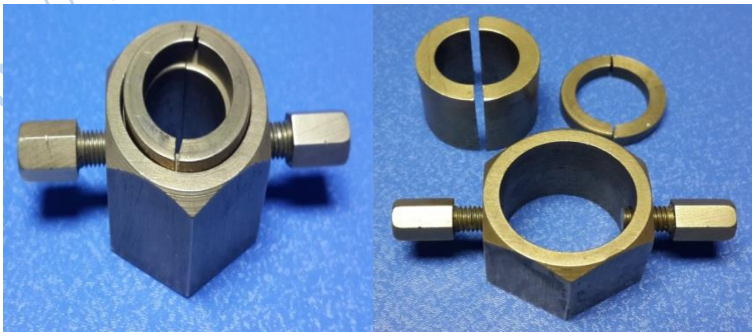

Figure (3): A photograph showing cylindrical copper mold for specimen housing before (left) and after (right) assessment.

Then each root slice was photographed from both sides using a digital microscope[13] to measure the apical and coronal radii using image analysis software.

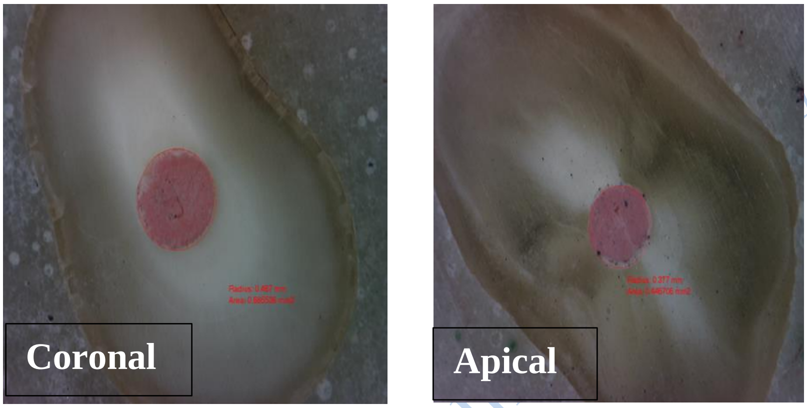

Figure (4): A photograph showing measurement of coronal (left) and apical (right) radii.

A computer controlled materials testing machine[14] was used to evaluate the push out bond strength of each specimen. The machine is composed of metallic block for specimen housing, stainless steel plungers of different sizes for load application and connected to computer software[15].

The specimens were placed in the metallic block with a circular cavity at the middle and subjected to a compressive loading at a crosshead speed of 1 mm/min.

13 Scope capture.Digital microscope, Guandong, China.
14 Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK.
15 Nexygen-MT; Lloyd Instruments.

Load was applied by a stainless steel cylindrical plunger of 0.9 mm diameter for coronal sections, 0.7 mm for middle sections and 0.5 mm for apical sections. The plunger tip was sized and positioned to touch only the filling, without stressing the surrounding dentin, in apical coronal direction to push the filling material toward the larger diameter, thus avoiding any limitation to the filling movement possibly owing to the canal taper (figure 5).

After load application, failure was manifested by extrusion of the filling material and confirmed by sudden drop along load-deflection curve recorded by Nexygen computer software[16]. The amount of load required to fracture or dislodge the sealer was recorded in Newton. To express the bond strength in Megapascals, the load at failure recorded in Newton was divided by the area of the bonded interface according to the formula used by ***Lopes et al. 2010*** [(8586)].

Bond strength (MPa) = F/A

Where **F** is the load recorded in Newton

And **A** is the bonding surface area

16 LIyod Instruments Ltd.

The bonding surface area was calculated from the following formula:

$$[A = (\pi\ h\ (r_1+r_2)],$$

Where,

π is the constant 3.14,

r_1 apical radius, r_2 coronal one,

and **h** is the thickness (height) of the sample in millimeters

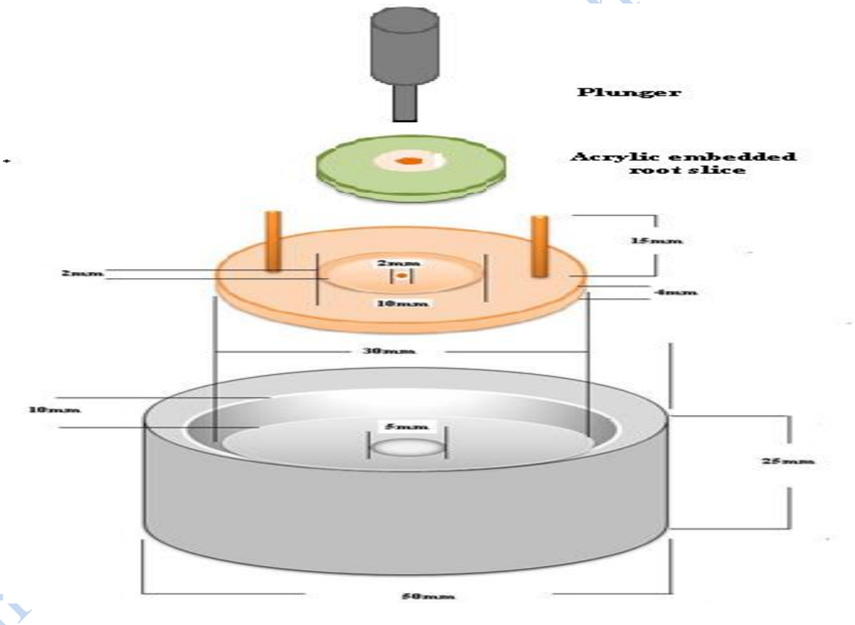

Figure (5): A Schematic drawing of the specimen positioned on the apparatus for alignment and load application in the push-out bond strength

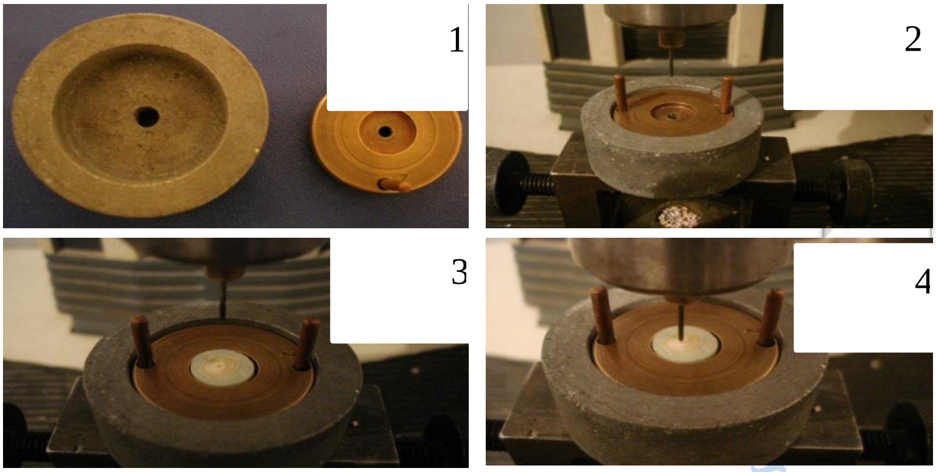

Figure (6): A photograph showing Materials Testing Machine with root slice inside resin block for measuring Push-out bond strength.

The obtained data was collected, tabulated and statistically analyzed. Push-out bond strength data were presented as mean and standard deviation (SD) values. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. Data showed non-parametric distribution, so Mann-Whitney U test was used to compare between the two sealers. Kruskal-Wallis test was used for comparisons between the storage periods. Mann-Whitney U test was used for pair-wise comparisons between techniques when Kruskal-Wallis test is significant. Friedman's test was used to compare between root segments. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

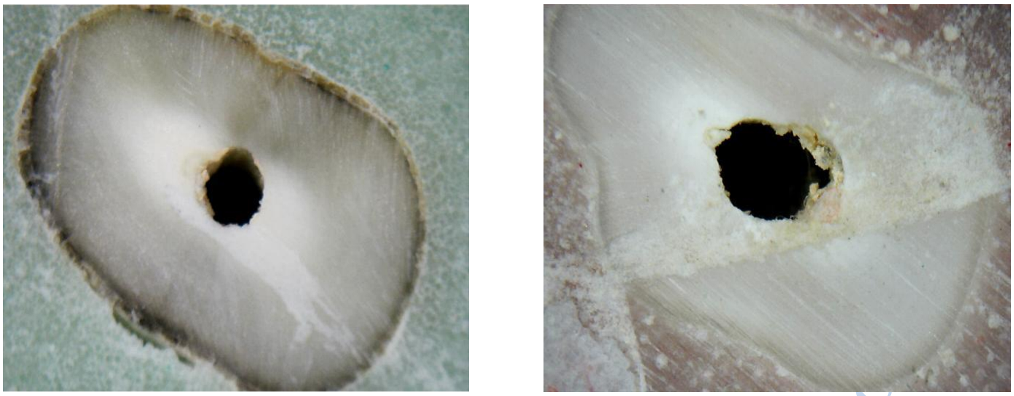

Figure (7): A photograph showing 2 samples of Endo REZ and Meta SEAL after load application and failure of specimens

® IBM Corporation, NY, USA., ® SPSS, Inc., an IBM Company

Experimental design

Fifty six freshly extracted single rooted human mandibular premolars

The root canals were instrumented using REVO-S rotary system to size 40

Specimens were divided into 2 main groups (28)

GroupA1 **EndoREZ** | Group A2 **MetaSEAL**

All samples were stored in an incubator for either:

1 W | 1Mon | 3 Mon | 6 Mon

Sections were prepared along the apical, middle and cervical segments of each root to produce three root slices of 2 mm thickness each.(168 root slices)

Diameter of filling material measured for each specimen to calculate bonded area A

Push out test using universal testing machine gave debonding force F

Bond strength calculated using formula F/A

Statistical analysis of data

Results

Section outline:

5.1. Comparison between the obturating materials used.

5.2. Comparison between different storage periods.

5.3. Comparison between root segments.

5.1. Comparison between the obturating materials used

The data in this section was statistically analyzed using Mann-Whitney U test.

a. <u>After 1 week</u>

At the cervical segment, the mean push out bond strength was significantly higher when using MetaSEAL (3.60 ± 1.95) than when using EndoREZ (0.63 ± 0.38). *P*-value = 0.001.

At the middle segment, the mean push out bond strength was significantly higher when using MetaSEAL (3.75 ± 2.42) than when using EndoREZ (1.09 ± 0.89). *P*-value = 0.017.

At the apical segment, the mean push out bond strength was significantly higher when using MetaSEAL (2.23 ± 1.74) than when using EndoREZ (0.28 ± 0.06). *P*-value = 0.042.

b. <u>After 1 month</u>

At the cervical segment, the mean push out bond strength was significantly higher when using MetaSEAL (6.49 ± 1.82) than when using EndoREZ (1.33 ± 1.69). *P*-value = 0.002.

At the middle segment, the mean push out bond strength was significantly higher when using MetaSEAL (4.89

± 1.90) than when using EndoREZ (1.23 ± 1.19). *P*-value = 0.004.

At the apical segment, there was no significant difference between the mean push out bond strength recorded for MetaSEAL (3.81 ± 2.34) and EndoREZ (3.95 ± 4.97) .*P*-value = 0.456.

c. <u>After 3 months</u>

At the cervical segment, the mean push out bond strength was significantly higher when using MetaSEAL (5.13 ± 2.13) than when using EndoREZ (1.12 ± 1.05). *P*-value = 0.002.

At the middle segment, the mean push out bond strength was significantly higher when using MetaSEAL (5.52 ± 3.26) than when using EndoREZ (0.86 ± 0.98). *P*-value = 0.002.

At the apical segment, there was no significant difference between the mean push out bond strength recorded for MetaSEAL (4.29 ± 4.33) and EndoREZ (1.48 ± 1.11) .*P*-value = 0.165.

d. <u>After 6 months</u>

At the cervical segment, the mean push out bond strength was significantly higher when using MetaSEAL (4.39 ± 1.52) than when using EndoREZ (0.64 ± 0.32). *P*-value = 0.001.

At the middle segment, the mean push out bond strength was significantly higher when using MetaSEAL (5.14

± 1.91) than when using EndoREZ (1.17 ± 0.83). *P*-value = 0.002.

At the apical segment, the mean push out bond strength was significantly higher when using MetaSEAL (8.35 ± 5.02) than when using EndoREZ (1.37 ± 1.25). *P*-value = 0.004.

Table (3): Mean push out bond strength values, Standard Deviations (SD) and P- values when comparing MetaSEAL versus EndoREZ.

Storage period	Sealer / Root segment	EndoREZ		MetaSEAL		*P*-value
		Mean	SD	Mean	SD	
1 week	Cervical	0.63	0.38	3.60	1.95	0.001*
	Middle	1.09	0.89	3.75	2.42	0.017*
	Apical	0.28	0.06	2.23	1.74	0.042*
1 month	Cervical	1.33	1.69	6.49	1.82	0.002*
	Middle	1.23	1.19	4.89	1.90	0.004*
	Apical	3.95	4.97	3.81	2.34	0.456
3 months	Cervical	1.12	1.05	5.13	2.13	0.002*
	Middle	0.86	0.98	5.52	3.26	0.002*
	Apical	1.48	1.11	4.29	4.33	0.165
6 months	Cervical	0.64	0.32	4.39	1.52	0.001*
	Middle	1.17	0.83	5.14	1.91	0.002*
	Apical	1.37	1.25	8.35	5.02	0.004*

**: Significant at $P \leq 0.05$*

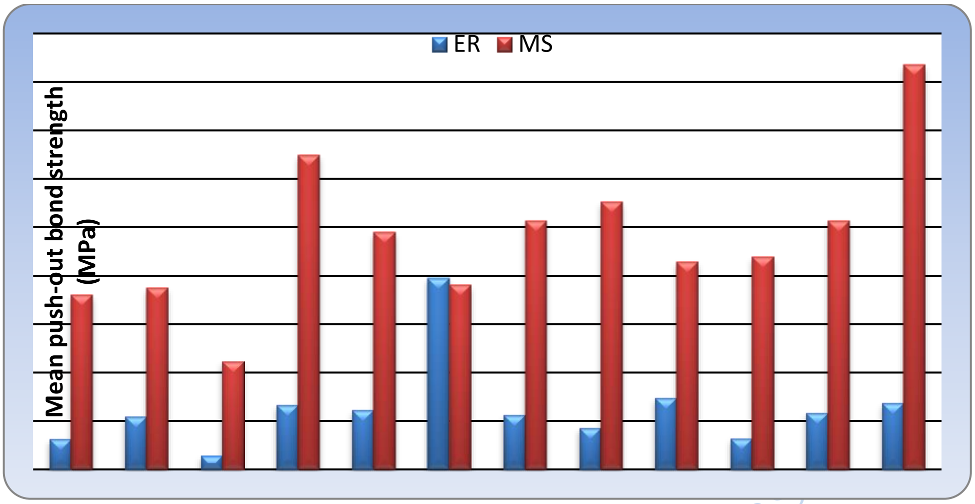

Figure (8): Bar chart representing the mean push-out bond strength values when comparing MetaSEAL vs. EndoREZ.

5.2. Comparison between different storage periods

The data in this section was statistically analyzed using Kruskal-Wallis test.

a. <u>Using EndoREZ</u>

At the cervical segment, there was no significant difference between the mean push out bond strength after 1 month (1.33 ± 1.69), 3 month (1.12 ± 1.05), 6 month (0.64 ± 0.32) and 1 week (0.63 ± 0.38). *P*-value = 0.952.

At the middle segment, there was no significant difference between the mean push out bond strength after 1 month (1.23 ± 1.19), 6 month (1.17 ± 0.83), 1 week (1.09 ± 0.89) and 3month (0.86 ± 0.98). *P*-value = 0.884.

At the apical segment, there was no significant difference between the mean push out bond strength after 1 month (3.95 ± 4.97), 3 month (1.48 ± 1.11), 6 month (1.37 ± 1.25) and 1week (0.28 ± 0.06). *P*-value = 0.086.

b. <u>Using MetaSEAL</u>

At the cervical segment, the highest mean push out bond strength value was recorded after 1 month (6.49 ± 1.82) followed 3 month (5.13 ± 2.13) and 6 month (4.39 ± 1.52) with no significant difference between them. While the lowest mean push out bond strength value was recorded after 1week (3.60 ± 1.95). *P*-value = 0.043.

At the middle segment, there was no significant difference between the mean push out bond strength after 3 month (5.52 ± 3.26), 6 month (5.14 ± 1.91), 1 month (4.89 ± 1.90) and 1 week (3.75 ± 2.42). *P*-value = 0.665.

At the apical segment, there was no significant difference between the mean push out bond strength after 6 month (8.35 ± 5.02), 3 month (4.29 ± 4.33), 1 month (3.81 ± 2.35) and 1week (2.23 ± 1.74). *P*-value = 0.064.

Table (4): Mean push out bond strength values, Standard Deviations (SD) and P- values when comparing both of MetaSEAL and EndoREZ at different storage periods.

Sealer	Storage period / Root segment	1 week		1 month		3 months		6 months		*P*-value
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	
EndoREZ	Cervical	0.63	0.38	1.33	1.69	1.12	1.05	0.64	0.32	0.952
	Middle	1.09	0.89	1.23	1.19	0.86	0.98	1.17	0.83	0.884
	Apical	0.28	0.06	3.95	4.97	1.48	1.11	1.37	1.25	0.086
MetaSEAL	Cervical	3.60^{c}	1.95	6.49^{a}	1.82	5.13^{b}	2.13	4.39^{b}	1.52	0.043*
	Middle	3.75	2.42	4.89	1.90	5.52	3.26	5.14	1.91	0.665
	Apical	2.23	1.74	3.81	2.34	4.29	4.33	8.35	5.02	0.064

**: Significant at $P \leq 0.05$, Different superscripts in the same row are significantly different.*

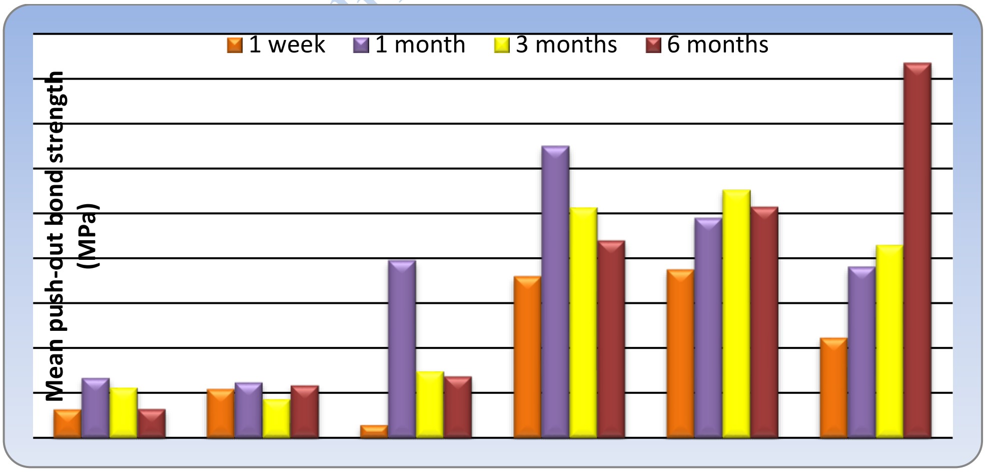

Figure (9): Bar chart representing the mean push-out bond strengths when comparing both of MetaSEAL and EndoREZ at different storage periods.

5.3. Comparison between root segments

a. Using EndoREZ

After 1 week, there was no significant difference between the mean push out bond strength at the middle (1.09 ± 0.89), cervical (0.63 ± 0.38) and apical (0.28 ± 0.06) root segments. *P*-value = 0.472.

After 1 month, there was no significant difference between the mean push out bond strength at the apical (3.95 ± 4.97), cervical (1.33 ± 1.69) and middle (1.23 ± 1.19) root segments. *P*-value = 0.368.

After 3 month, there was no significant difference between the mean push out bond strength at the apical (1.48 ± 1.11), cervical (1.12 ± 1.05) and middle (0.86 ± 0.98) root segments. *P*-value = 0.368.

After 6 month, there was no significant difference between the mean push out bond strength at the apical (1.37 ± 1.25), middle (1.17 ± 0.83) and cervical (0.64 ± 0.32) root segments. *P*-value = 0.565.

b. Using MetaSEAL

After 1 week, there was no significant difference between the mean push out bond strength at the middle (3.75 ± 2.42), cervical (3.60 ± 1.95) and apical (2.23 ± 1.74) root segments. *P*-value = 0.066.

After 1 month, there was no significant difference between the mean push out bond strength at the cervical (6.49 ± 1.82), middle (4.89 ± 1.90) and apical (3.81 ± 2.34) root segments. *P*-value = 0.565.

After 3 month, there was no significant difference between the mean push out bond strength at the middle (5.52 ± 3.26), cervical (5.13 ± 2.13) and apical (4.29 ± 4.33) root segments. *P*-value = 0.565.

After 6 month, there was no significant difference between the mean push out bond strength at the apical (8.35 ± 5.02), middle (5.14 ± 1.91) and cervical (4.39 ± 1.52) root segments. *P*-value = 0.368.

Table (5): Mean push out bond strength values, Standard Deviations (SD) and P- values when comparing EndoREZ at different root segments.

Storage period / Root segment	1 week		1 month		3 months		6 months	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Cervical	0.63	0.38	1.33	1.69	1.12	1.05	0.64	0.32
Middle	1.09	0.89	1.23	1.19	0.86	0.98	1.17	0.83
Apical	0.28	0.06	3.95	4.97	1.48	1.11	1.37	1.25
P-value	0.472		0.368		0.368		0.565	

**: Significant at P ≤ 0.05*

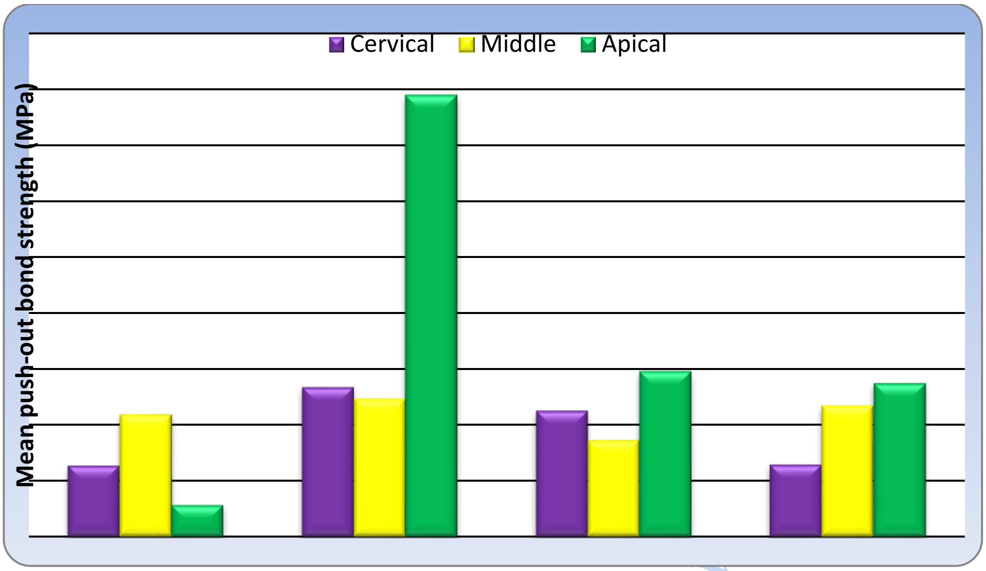

Figure (10): Bar chart representing the mean push-out bond strengths when comparing EndoREZ at different root segments.

Table (6): Mean push out bond strength values, Standard Deviations (SD) and P- values when comparing MetaSEAL at different root segments.

Storage period / Root segment	1 week		1 month		3 months		6 months	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Cervical	3.60	1.95	6.49	1.82	5.13	2.13	4.39	1.52
Middle	3.75	2.42	4.89	1.90	5.52	3.26	5.14	1.91
Apical	2.23	1.74	3.81	2.34	4.29	4.33	8.35	5.02
P-value	0.066		0.565		0.565		0.368	

*: *Significant at* $P \leq 0.05s$

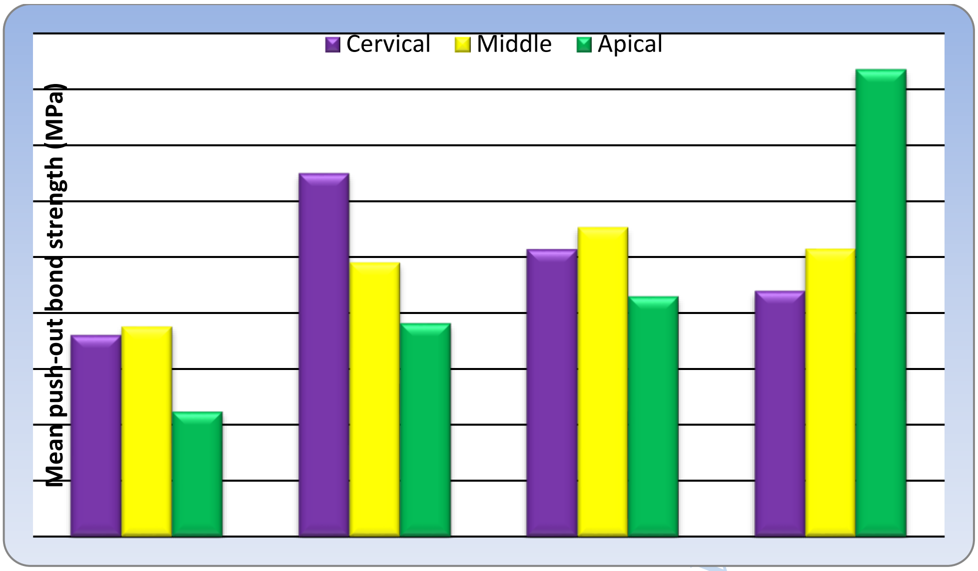

Figure (11): Bar chart representing the mean push-out bond strengths when comparing MetaSEAL at different root segments.

Discussion

Adhesion of an endodontic sealer is defined as its capacity to adhere to the root canal walls and promote the union of gutta percha cones to each other as well as to root dentin (64). Adhesion is of paramount importance because it could help to avoid both fluid percolation between the spaces of obturating materials and the displacement of materials during procedures (87,88).

There are many methods for measuring the adhesion of endodontic root canal sealers, but none has yet been widely accepted. One of these methods is the tensile strength test which is sensitive test. Despite the load is applied perpendicular to the bond line inducing pure tensile loads, an alignment errors can occur inducing a bending stresses that results in premature failure and consequently yielding incorrect low results. Additionally, shear bond strength test has been used with difficulty to align the shear-loading device with the bond interface. The load is offset at some distance from the bonded interface, resulting in unpredictable torque loading on the specimen (53).

Alternatively, the push out test which is an efficient and reliable test as it allows assessment of regional differences in bond strength among root levels with acceptable variability of the data distribution [14,89].Another advantage of this method is that it allows root canal sealers to be evaluated even when bond strengths are low [15, 49].Therefore, the push out test was used in the present study to evaluate the bond strengths of the selected materials at different storage periods.

Lately, attempts have been made to introduce obturating systems with adhesive properties to obtain a *"monoblock"* in which the core material, sealing agent, and root canal dentin form a single cohesive unit. Unfortunately, achievement of a *"monoblock"* has been hampered by the lack of chemical union between the polyisoprene component of gutta percha and root canal sealer. To overcome this problem, a coated gutta percha cone with a polybutadienediisocyanate methacrylate adhesive was introduced. This proprietary adhesive resin includes a hydrophobic portion that is chemically compatible with the hydrophobic polyisoprene substrate in gutta percha and a hydrophilic portion that is chemically compatible with a hydrophilic methacrylate resin sealer. With the use of this adhesive resin coating, a strong chemical union is achieved between the gutta percha and the methacrylate resin based sealer. This thermoplastic resin-coated gutta percha cone is recommended for use with the EndoREZ obturating system [90,91].

Recently, a self etch, dual cured resin based sealer (MetaSEAL) was introduced into the market. This material was developed for root canals and has hydrophilic characteristics especially in terms of its catalyst system and self-conditioning monomer. A 4META, an acidic monomer engineered in Japan, a component of numerous dentin adhesives, is used. Acidic monomers allow conditioning of the dentin surface and penetrate into the patent collagen network, which creates a hybrid layer simultaneously. This rather assumes a softening of the gutta percha surface by the solvent of the self conditioning sealer and hence forming an equivalent intermixture [92].

The primary goal of this study was to evaluate the bond strength of a self adhesive EndoREZ and self etching MetaSEAL at different storage periods using push out bond strength test at different root levels. The push out test was done using Universal Testing Machine to standardize the test and make it reproducible with reliable results [49].

Within the parameters of this study, MetaSEAL showed higher push out bond strength than EndoREZ when comparing between both materials. This may be attributed to the nature of MetaSEAL that include formation of hybrid-like layer between the obturating core and MetaSEAL sealer interface in addition to presence of strong resin tags to micropores in root canal dentin [93,94].Also, the slower polymerization process allows MetaSEAL to flow in the pregel stage, which provides some stress relief from polymerization contraction at the resin-dentin interface [95]. Alternatively, the lower push out bond strength of EndoREZ may be attributed to its lower cohesive strength than adhesive strength in addition to pulling of resin sealer tags out of the tubules during polymerization shrinkage of the sealer that might create gaps along the sealer-dentin interface [33,37,96,97]. Additionally, stresses occurred when using EndoREZ (*Tertiary Monoblock*) is more than the stress when using MetaSEAL (*Secondary monoblock*) as a result of increasing in the number of the adhesive interfaces[62].Hiraishi and cooperators attributed the weak bond of EndoREZ to lack of free radicals from the prepolymerized coating of EndoREZ gutta percha cones due to the removal of the oxygen inhibition layer for packing purposes[39].While, Jensen & Fischer attributed the lower bond strength of EndoREZ to the inconsistency of the external proprietary resin coating in the form of uneven circumferential thickness or partial detachment [90].Alternatively, Tay &Pashley stressed on using EndoREZ obturating system with either a single cone technique or a technique that involves the passive placement of accessory cones without lateral compaction, to avoid disruption of these external coatings. They advocated that, lateral compaction may cause disruption of those external resin coatings [11].

However at the apical root segment, there was no significant difference when comparing between both materials after 1 and 3 month storage period which may be attributed to increasing the push out bond strength of EndoREZ as it undergoes the highest peak of water sorption after about 1month then decline again up to 6 month [98]. There was another explanation related to pretest failure of 3 samples belonging to EndoREZ group that resulted in significant difference recorded apically between both materials after 1 week. Another assumption of the statistical significance at the apical root level may be due to the extremely over increase of the bond strength values of MetaSEAL (8.35 ± 5.02) after 6 month storage periods.

While when comparing among different storage periods (1 week, 1, 3 and 6 month), there was no significant difference between push out bond strengths when using both of EndoREZ and MetaSEAL. The only exception was recorded when using MetaSEAL at the cervical segment of the root portions .The results showed that, the highest push out bond strength was recorded after 1 month followed by 3& 6 month while the lowest push out bond strength was recorded after 1 week . Such results may be attributed to reaching the high peak of water sorption after about 1 month then the push out bond strength decline over time due to polymer degradation with subsequent increasing the interfacial leakage that resembles in vivo aging [99,100]. While at 1 week, complete polymerization of the sealer was not yet occurred [97].This was in agreement with Archegas et al. that suggests a seven day period of storage may be insufficient to evaluate the real values of water sorption in resin based materials as water sorption varied from 24 hours to 28 days [101].

Recently, Moon[102]and coordinators reported that release and activation of endogenous matrix metalloproteinases (MMPs) from dentin during dentin bonding were thought to be responsible for the in vitro manifestation of thinning and disappearance of collagen fibrils from incompletely infiltrated hybrid layers in aged, bonded dentin over time. Studies reported that the use of MMPs inhibitors such as chlorohexidene may prevent decrease in the bond strength over time [103].

With regarding to the significant regional push out bond strength at the cervical segment of the root portions when using MetaSEAL after 1 month, it was found that greater resin tag density with better formation of resin/dentin inter- diffusion zones (RDIZ) in the cervical third of the root (higher density of dentinal tubules) than in the middle and apical thirds (lower density of dentinal tubules) in human teeth. Therefore, this utilization of different root regions may be partly responsible for the difference in the push-out bond strength values in addition to the previous explanation of high peak of water sorption after 1 month [18,19,104].Also, incomplete removal of the smear layer at the middle and apical portions of the root canals may prevent adhesive penetration into the dentinal tubules [105].Moreover, Structural deficiencies originated from the air entrapped in the sealer mass during mixing or transferring the sealer into the canal may delay the setting reaction and weaken the resin sealer and result in debonding between the resin cement and the root dentin[106] .This was in disagreement with Tay et al. [107] which reported that immediate light-curing from the coronal side of the roots may also create a large polymerization stress during setting by preventing flow of resin-based sealers and may lead to de-bonding of the resin from the root canal walls, which results in gap formation. The controversial between both studies may be attributed to the differences in methodology.

Summary

Success in endodontic treatment is mainly determined by a hermitically three dimensional obturation of root canal system after chemo-mechanical preparation. Unfortunately, Hermetic seal of the root canal system using the traditional obturating systems have not been proved yet.

Recently, a resin based root canal sealers have been introduced to overcome this problem based on the *"monoblock"* concept. Lately, A dual curable methacrylate-based resin sealer (EndoREZ) has been introduced that can be used in combination with gutta percha or with resin-coated gutta percha to form a "monoblock". More recently, (4-META) methacrylate-based endodontic sealer (MetaSEAL) has also been introduced to bond to radicular dentin.

This study was directed to evaluate the bond strength of EndoRez and MetaSEAL with the push out test over a period of time using a computer controlled materials testing machine.

Fifty six freshly extracted single rooted human mandibular premolars with fully formed root apices were selected to be used in this study.

The root portions were divided into 2 main groups (28 each) according to the obturating material used (variable "A");

- Second generation Methacrylate-based sealer, EndoREZ (group $A_{1:}$ 28 root portions).
- Fourth generation 4-META based sealer, MetaSEAL (group A_2: 28 root portions).

Each main group was further subdivided into 4 subgroups (7 each) according to the different storage time (variable "B");

- After 1 week (B_1),
- After 1 month (B_2),
- After 3 month (B_3),
- After 6 month (B_4)

The root canals were instrumented in a crown down manner using REVO-S rotary system with a speed and torque control setting as recommended by the manufacturer. The root canals were obturated using either EndoRez or MetaSEAL then light curing was done for 40 seconds to initiate the polymerization process of sealers.

Following setting of the obturation materials, the specimens were stored for either, 1 week, 1 month, 3 months or 6 months (variable “B”) at 37° C in a completely sealed glass tubes containing 0.9 % normal saline.

Sections were prepared along the apical, middle and cervical segments of each root to produce three root slices of 2 mm thickness each.To express the bond strength in megapascals, the load at failure recorded in Newton was divided by the area of the bonded interface according to the formula.

Bond strength (MPa) = F/A

Where F is the load recorded in Newton

And A is the bonding surface area

The obtained data was collected, tabulated and statistically analyzed.

Within the parameters of this study the following conclusions were drawn;

1. MetaSEAL resin sealer showed overall higher push out bond strength than EndoREZ resin sealer.
2. MetaSEAL resin sealer showed improvement in its bond strength over time.
3. The highest push out bond strength value recorded for MetaSEAL resin sealer was found after 1month storage period.
4. Root segment level did not have an overall significant influence on bond strength.

5. According to this study, MetaSEAL demonstrated promising results in terms of push-out bond strength while EndoREZ did not perform well.

Conclusions

Within the parameters of this study the following conclusions were drawn;

1. MetaSEAL resin sealer showed overall higher push out bond strength than EndoREZ resin sealer.
2. MetaSEAL resin sealer showed improvement in its bond strength over time.
3. The highest push out bond strength value recorded for MetaSEAL resin sealer was found after 1month storage period.
4. Root segment level did not have an overall significant influence on bond strength.
5. According to this study, MetaSEAL demonstrated promising results in terms of push-out bond strength while EndoREZ did not perform well.

Recommendations

1. Further studies should be done to correlate the effect of storage period and the efficacy of bond between both of EndoREZ and MetaSEAL sealers and the core obturating material.
2. Further studies should be microscopically done to evaluate the nature of failure mode either cohesive, adhesive or mixed when using both tested materials.
3. Further studies should be done to evaluate the hybrid layer formation between the different obturating materials and root canal dentin.
4. Further modifications should be conducted to improve the bond strength of EndoREZ system.

الملخص العربي

يعتمد نجاح المعالجة اللبية في الاساس علي السد المحكم ثلاثي الأبعاد لنظام قناة الجذر بعد الاعداد الكيماوي ميكانيكي. لسوء الحظ ، لم يثبت حتى الآن السد المحكم لنظام قناة الجذر باستخدام نظم السد التقليدية.

حديثا، أدخلت المواد الراتنجيه لنظم قناة الجذر للتغلب على هذه المشكلة استنادا إلى مفهوم تكوين "قطعة واحدة".وقد تم في الآونة الأخيرة إدخال مزدوج الراتنج سيلر (اندوريز) الذي يمكن استخدامه في تركيبة مع جاتا بيرشا أومع جاتا بيرشا المغلفة مع الراتنج لتشكيل "سدة أحادية".هذا وقد تم في الآونة الأخيرة تصنيع مادة (4-ميتا) (ميتاسيل) لزيادة الترابط بعاج الجذر.ولذلك فقد وجهت هذه الدراسة لتقييم قوة الترابط بين اندوريز وميتاسيل على مدى فترة من الزمن باستخدام آلة اختبار المواد بالكمبيوتر .
هذا وقد تم اختيار ست وخمسون سنا من أشباه الضروس مكتملة الجذور ومستخرجه حديثا من ضواحك الفك السفلي للإنسان لاستخدامها في هذه الدراسة.

وتم تقسيم الأجزاء الجذرية الي مجموعتين رئيسيتين (عدد كل منهما28) وفقا لمادة السد المستخدمة (متغير "أ")؛

- سداده على أساس الميثاكريليت، اندوريز الجيل الثاني (المجموعة أ1: 28 أجزاء الجذر).
- سداده على أساس4-ميتا ميتاسيل الراتنجيه الجيل الرابع (المجموعة أ2: 28 أجزاء الجذر).

تم تقسيم كل مجموعة رئيسية الى مزيد من المجموعات الفرعيةالي اربع مجموعات (عدد كل منهم 7) وفقا لاوقات التخزين المختلفة (متغير "ب")؛

- بعد الأسبوع 1 (ب1)،
- بعد شهر 1 (ب2)،
- بعد 3 أشهر (ب3)،
- بعد 6 أشهر (ب4).

تم تحضير قنوات الجذور في أسفل التاج باستخدام مبارد ريفو- اس الدوارة مع تحديد سرعة وعزم الدوران على النحو الموصى به من قبل الشركة المصنعة.

وتم إحكام غلق القنوات الجذرية باستخدام اندوريز أو ميتاسيل ثم عمل تجفيف ضوئي لمدة 40 ثانية لبدء عملية بلمرة السدادات.

بعد وضع مواد السد، تم تخزين العينات لمدد مختلفة لمدة أسبوع، وشهر، ثلاثة أشهر أو ستة أشهر (متغير "ب") عند 37 درجة مئوية في أنابيب زجاجية محكمة الإغلاق تماما تحتوي على محلول ملحي عادي.

أعدت المقاطع على هيئة قطاعات قمية ومتوسطة وعنقيه من كل جذر للحصول علي ثلاث شرائح جذريه سمك الواحدة منها 2 مم.

وللتعبير عن قوة الترابط بالميجاباسكال، تم تقسيم قوة التحمل المسجلة بالنيوتن ومساحة سطح الترابط وفقا للصيغة الاتية:

قوة الترابط = ق / م

حيث **ق** هي الحمولة المسجلة في نيوتن

م هي مساحة الترابط

وقد تم جمع البيانات التي تم الحصول عليها، وجدولتها وتحليلها إحصائيا.

هذا وقد تم استخلاص الاستنتاجات التالية ضمن حدود هذه الدراسة :

1. أظهرت سداده ميتاسيل الراتنجيه أعلى قوة ترابط عامة من سداده اندوريز الراتنجيه.
2. أظهرت سداده ميتاسيل الراتنجيه تحسن في قوة الترابط بمرور الوقت.
3. تم العثور على أعلى دفعة من قيمة قوة الترابط المسجلة لسداده ميتاسيل الراتنجيه بعد فترة التخزين من 1شهر.
4. مستوى قطاع الجذر لم يكن له تأثير كبير وفعال على قوة الترابط.
5. وفقا لهذه الدراسه ، أظهرت ميتاسيل نتائج واعدة من حيث قوة الترابط في حين ان ذلك لم يحدث مع اندوريز.

References

1. Kardon B., Kuttler S., Hardigan P., Dorn S.: An in vitro evaluation of the sealing ability of a new root-canal-obturation system. J Endod 2003; 29; 10:658-61.
2. Magure M., Kafrawy A., Brown C., Newton C.: Human saliva coronal microleakage in obturated root canals: an in vitro study. J Endod 1991; 17:324 –31.
3. Shipper G., Orstavik D., Teixeira F., Trope M.: An evaluation of microbial leakage in roots filled with a thermoplastic synthetic polymer-based root canal filling material (Resilon). J Endod 2004; 30:342–7.
4. Shipper G., Teixeira F., Arnold R., Trope M.: Periapical inflammation after coronal microbial inoculation of dog roots filled with gutta-percha or resilon. J Endod 2005; 31:91– 6.
5. Grande N., Plotino G., Lavorgna L., Ioppolo P., Bedini R., Pameijer C. et al.: Influence of different root canal-filling materials on the mechanical properties of root canal dentin. J Endod 2007; 33: 859–63.
6. Bouillaguet S., Bertossa B., Krejci I., Wataha J., Tay F., Pashley D.: Alternative adhesive strategies to optimize bonding to radicular dentin. J Endod 2007; 33:1227–30.

7. Orstavik D., Eriksen H., Beyer-Olsen E.: Adhesive properties and leakage of root canal sealers in vitro. Int Endod J 1983; 16:59–63.
8. Grossman L.: Physical properties of root canal cements. J Endod 1976; 2:166 –75.
9. Gesi A., Goracci C., Pashley D., Tay F., Ferrari M.: Interfacial strength of resilon and gutta-percha to intraradicular dentin. J Endod, 2005; 31: 11:809-13.
10. Kim Y., Grandini S., Ames J., Gu L., Kim S., Pashley D.: Critical review on methacrylate resin-based root canal sealers. J Endod 2010; 36:383-399.
11. Tay F., Pashley D.: Monoblocks in root canals -a hypothetical or a tangible goal, J Endod 2007; 33: 391-398.
12. Zmener O., Pameijer C.: Resin coated gutta-percha cones coupled with a resin-based sealer: a new alternative for filling root canals. Endod Pract 2007; 10:21-5.
13. Belli S., Ozcan E., Derinbay O., Eldeniz A.: A comparative evaluation of sealing ability of a new, self-etching, dual-curable sealer: Hybrid Root SEAL (MetaSEAL). Oral Surg Oral Med Oral Pathol Oral Radiol Endod, 2008; 106:45–52.

14. Ungor M., Onay E., Orucoglu H.: Push-out bond strengths: the Epiphany- Resilon endodontic obturation system compared with different pairings of Epiphany, Resilon, AH Plus and gutta-percha. INT ENDOD J , 2006; 39:643-7.
15. Jainaen A., Palamara J., Messer H.: Push-out bond strengths of the dentin sealer interface with and without a main cone. INT ENDOD J , 2007; 40:882–90.
16. Salehrabi R., Rotstein I.: Endodontic treatment outcomes in a large patient population in the USA: an epidemiological study. J Endod 2004; 30:846–50.
17. Schwartz R.: Adhesive dentistry and endodontics. Part 2: bonding in the root canal system-the promise and the problems: a review. J Endod 2006; 32:1125-1134.
18. Okuda M., Pereira P., Nakajima M., Tagami J., Pashley D.: Long-term durability of resin dentin interface: nanoleakage vs. microtensile bond strength. Oper Dent 2002; 27:289-096.
19. Ferrari M., Mannocci F., Vichi A., Cagidiaco M., Mjor I.: Bonding to root canal: structural characteristics of the substrate. Am J Dent 2000; 13:255-60.

20.Gaston G., Lesely A., Frederick R., Carlos F., Pashely D.: Evaluation of regional bond strength of resin cement to endodontic surfaces. J Endod 2001; 27(5)321:324.

21.Salz U., Zimmermann J., Salzer T.: Self-curing, self-etching adhesive cement systems. J Adhes Dent 2005; 7:7–17.

22.Al-Assaf K., Chakmakchi M., Palaghias G., Karanika-Kouma A., Eliades G.: Interfacial characteristics of adhesive luting resins and composites with dentin. Dent Mater 2007; 23:829–39.

23.Benkel B., Rising D., Goldman L., Rosen H., Goldman M., Kronman J.: Use of a hydrophilic plastic as a root canal filling material. J Endod 1976; 2:196-202.

24.Yesilsoy C. Radiographic evidence of absorption of Hydron from an obturated root canal. J Endod 1984; 10:321–3.

25.Hosoya N, Nomura M, Yoshikubo A, Arai T, Nakamura J, Cox CF. Effect of canal drying methods on the apical seal. J Endod 2000; 26:292– 4.

26.Petschelt A. Drying of root canals. Dtsch Zahnarztl Z 1990; 45:222– 6.

27. R, J, Reid, D. F, Wilson, K, K. Chau, G, S, Heithersay & P, S. Heijkoop Tissue responses to Hydron, assessed by

intraosseous Implantation. INT ENDOD J (1992) 25,192-198

28. Rhome BH, Solomon EA, Rabinowitz JL. Isotopic evaluation of the sealing properties of lateral condensation, vertical condensation, and Hydron. J Endod 1981; 7:458–61.

29. Tanzilli J., Nevins A., Borden B.: A histologic study comparing Hydron and gutta-percha as root canal filling materials in monkeys. J Endod 1981; 7:396-401.

30. De Munck J., Vargas M., Van Landuyt K., Hikita K., Lambrechts P., Van Meerbeek B.: Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater 2004; 20:963-71.

31. Hammad M., Qualtrough A., Silikas N.: Extended setting shrinkage behavior of endodontic sealers. J Endod 2008; 34:90-3.

32. Bergmans L., Moisiadis P., De Munck J., Van Meerbeek B., Lambrechts P.: Effect of polymerization shrinkage on the sealing capacity of resin fillers for endodontic use. J Adhes Dent 2005; 7:321-9.

33. Tay F., Loushine J., Monticelli F., Norman R., Breschi L., Ferrari M., David H.: Effectiveness of resin-coated gutta-percha cones and a dual-cured, hydrophilic

methacrylate resin-based sealer in obturating root canals. J Endod 2005; 31, 9: 659-664

34. Fisher A., Berzins W., Bahcall K.: An in vitro comparison of bond strength of various obturation materials to root canal dentin using a push-out test design. J Endod 2007; 33:856-858.

35. Rahimi M., Jainaen A., Parashos P., Messer H.: Bonding of resin-based sealers to root dentin. J Endod 2009; 35:121–124.

36. Nagas E., Altundasar E., Serper A.: The effect of master point taper on bond strength and apical sealing ability of different root canal sealers. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2009; 107:61-64.

37. Eldeniz U., Erdemir A., Belli S.: Shear bond strength of three resin based sealers to dentin with and without the smear layer. J Endod 2005; 31: 293–296.

38. Gillespie W., Loushine R., Weller R.: Improving the performance of EndoREZ root canal sealer with a dual-cured two-step self-etch adhesive II. Apical and coronal seal. J Endod 2006; 32:771–5.

39. Hiraishi N., Loushine R., Vano M.: Is an oxygen inhibited layer required for bonding of resin-coated gutta-

percha to a methacrylate-based root canal sealer? J Endod 2006; 32:429-33.

40. Watanabe I., Nakabayashi N., Pashley D.: Bonding to ground dentin by a phenyl-P self-etching primer. J Dent Res 1994; 73:1212–20.

41. Kokkas A., Boutsioukis A., Vassiliadis L., Stavrianos C.: The influence of the smear layer on dentinal tubule penetration depth by three different root canal sealers: an in vitro study. J Endod 2004; 30:100–2.

42. Tay F., Pashley D.: Aggressiveness of contemporary self-etching systems: I- depth of penetration beyond dentin smear layers. Dent Mater 2001; 17:296–308.

43. Tay F., Loushine R., Weller R.: Ultrastructural evaluation of the apical seal in roots filled with a polycaprolactone-based root canal filling material. J Endod 2005; 31:514 –9.

44. Souza SFC, Bombana AC, Francci C, Gonc¸alves F, Castellan C, Braga RR. Polymerization stress, flow and dentin bond strength of two resin-based root canal sealers. International Endodontic Journal, 42, 867–873, 2009.

45. Perdigão J., Lopes M., Gomes G.: Interfacial adaptation of adhesive materials to root canal dentin. J Endod 2007; 33:259 –263.

46. Nunes V., Silva R., Alfredo E., S-Neto Yara T., Sousa S.: Adhesion of Epiphany and AH Plus sealers to human root dentin treated with different solutions. Braz Dent J 2008; 19: 224-227.

47. Kaya B., Keçeci A., Orhan H., Belli S.: Micropush-out bond strengths of gutta-percha versus thermoplastic synthetic polymer-based systems – an ex vivo study. INT ENDOD J 2008; 41:211–218.

48. Kazandag M., Sunay H., Tanalp J., Bayirli G.: Fracture resistance of roots using different canal filling systems. INT ENDOD J 2009; 42:705-710.

49. Tay F., Hiraishi N., Pashley D., Loushine R., Weller R., Gillespie W.: Bondability of Resilon to a methacrylate-based root canal sealer. J Endod 2006; 32:133-137.

50. Radovic I., Monticelli F., Goracci C., Vulicevic Z., Ferrari M.: Self-adhesive resin cements: a literature review. J Adhes Dent 2008; 10:251–8.

51. Lawson M., Loushine B., Mai S.: Resistance of a 4-META-containing, methacrylate-based sealer to dislocation in root canals. J Endod 2008; 34:833–7.
52. Mai S., Kim Y., Hiraishi N., Ling J., Pashley D., Tay F.: Evaluation of the true self-etching potential of a fourth generation self-adhesive methacrylate resin–based sealer. J Endo 2009; 35:870-874.
53. Onay E., Ungor M., Ari H., Belli S., Ogus E.: Push-out bond strength and SEM evaluation of new polymeric root canal fillings. Oral Surg Oral Med Oral Path Oral Radio Endod 2009; 107:879–885.
54. Ori T., Otsuki H., Wakamatsu S., Kawashima T., Matsushima K., Ikemi T.: SEM evaluation of roots obturated with adhesive root canal sealers, Sun Medical Co., Ltd, Moriyama, Japan, Department of Dental Caries Control and Aesthetic Dentistry, Nihon University School of Dentistry at Matsudo, Chiba, Japan, 3Department of Endodontics, Nihon University School of Dentistry at Matsudo, Chiba, Japan, 4Nihon University, Chiba, Japan.
55. Akman M., Akman S., Derinbay O., Bellic S.: Evaluation of gaps or voids occurring in roots filled with three different sealers. Eur J Dent. 2010; 4: 101–109.

56. Babb R., Loushine J., Bryan E., Ames M., Causey S., Kim J. et al: Bonding of self-adhesive (self-etching) root canal sealers to radicular dentin. J Endod 2009; 35:578-582.
57. Ori T., Otsuki H., Wakamatsu S., Kawashima T., Matsushima K., Ikemi T.: Push-out testing and SEM evaluation of adhesive root canal sealers, Sun Medical Co., Ltd, Moriyama, Japan, Department of Dental Caries Control and Aesthetic Dentistry, Nihon University School of Dentistry at Matsudo, Chiba, Japan, Department of Endodontics, Nihon University School of Dentistry at Matsudo, Chiba, Japan.
58. Costa J., Rached-Júnior F., Souza-Gabriel A., Silva-Sousa Y., Sousa-Neto M.: Push-out strength of methacrylate resin-based sealers to root canal walls. Int Endod J 2010; 43: 698–706.
59. Arata M., Ori T., Wakamatsu S., Yamamoto N., Kawashima T., Matsushima K., Ikemi T.: Effect of aging on bondability of a 4-META-containing methacrylate-based sealer. Sun Medical Co., Ltd, Moriyama, Japan, Department of Dental Caries Control and Aesthetic Dentistry, Nihon University School of Dentistry at Matsudo, Chiba, Japan, Department of Endodontics,

Nihon University School of Dentistry at Matsudo, Chiba, Japan.

60. Stiegemeier D., Baumgartner J., Ferracane J.: Comparison of push-out bond strengths of Resilon with three different sealers. J Endod 2010; 36:318-321.
61. Saeki K., Zartoshtimanesh S., Staninec M., Watanabe L., Marshall S.: Mechanical properties of novel self-etch resin based endodontic sealer. University of California - San Francisco, San Francisco, CA, UCSF School of Dentistry, San Francisco, CA.
62. Belli S., Eraslan O., Eskitascioglu G., Karbhari V.: "monoblocks" in root canals: a finite elemental stress analysis study. Int Endod J 2011; 44:817-826.
63. Ersev H., Yılmaz B., Pehlivanoğlu E., Özcan-Çalışkan E., Erişen F., Resistance to vertical root fracture of endodontically treated teeth with MetaSEAL. J Endod 2012; 38: 653-656.
64. Wachlarowicz J., Joyce P., Roberts S., Pashley D.: Effect of endodontic irrigants on the shear bond strength of epiphany sealer to dentin. J Endod 2007; 33: 152–155.
65. Shokouhinejad N., Sharifian R., Jafari M., Sabeti A.: Push-out bond strength of Resilon/Epiphany self-etch and gutta-percha/AH26 after different irrigation

protocols. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2010; 110: 88–92.

66. Pinna L., Loushine R., Bishop F., Cotti E., Weller R., Pashley D., Tay F.: Hybrid Root SEAL (MetaSEAL) creates hybrid layers in radicular dentin only when EDTA is used as the final rinse. American Journal of Dentistry 2009, 22:299-303.

67. Plum J.: The influence of different irrigation protocols on bond strength of the sealer Hybrid Root SEAL and ActiV GP to root canal dentin 11.10.2012 Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU).

68. Shokouhinejad N., Meraji N. , Shamshiri A., Khoshkhounejad M., Raoof M.: Effect of different final irrigants on bond strength of Resilon/Epiphany and Resilon/Epiphany self-etch. Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran 2013; Vol. 10, No.4.

69. Hashem A., Ghoneim G., Lutfy A., Fouda Y.: The effect of different irrigating solutions on bond strength of two root canal–filling systems. J Endod 2009; 35:537–540.

70. Sousa-Neto M., Silva-Sousa Y., Vilanova W., Carvalho-Junior J., Alfredo E.: Effect of intracanal irrigants on the

bond strength of epoxy resin-based and methacrylate resin based sealers to root canal walls. INT ENDOD J 2012; 45:42–48.

71. De-Deus G., Namen F., Galan J., Zehnder F.: Soft chelating irrigation protocol optimizes bonding quality of resilon/epiphany root fillings. J Endod 2008; 34(6):703–705.

72. Elsakaa S., Elnaghyb A.: Effect of addition of chitosan to self-etching primer: antibacterial activity and push-out bond strength to radicular dentin. Journal of Biomedical Research 2012; 26:288–294.

73. Elsakaa S., Elnaghyb A.: Antibacterial activity of calcium hydroxide combined with chitosan solutions and the outcomes on the bond strength of RealSeal sealer to radicular dentin. Journal of Biomedical Research 2012; 26: 193–199.

74. Erdemir A., Ari H., Güngüneş H., Belli S.: Effect of medications for root canal treatment on bonding to root canal dentin. J Endod 2004; 30: 113–116.

75. Barbizam B., Erica C., Teixeira B.: Effect of calcium hydroxide intracanal dressing /on the bond strength of a resin-based endodontic sealer. Braz Dent J 2008; 19: 224-227.

76. Weston H., Ito S., Wadgaonkar B., Pashley D.: Effects of time and concentration of sodium ascorbate on reversal of NaOCl-induced reduction in bond strengths. J Endod 2007; 33: 879–881.
77. Ayad M., Farag A., Garcia-Godoy F.: Effect of lactic acid irrigant on shear bond strength of Epiphany adhesive sealer to human dentin surface. Oral Surg Oral Med Oral Path Oral Radio Endod 2010; 109: 100-106.
78. Erdemi A., A.Eldeniz A., Belli S., Pashley D.: Effect of Solvents on Bonding to Root Canal Dentin J Endod 2004; 30: 589-592.
79. Nagas E., Cehreli Z., Durmaz V.: Effect of light-emitting diode photo polymerization modes on the push-out bond strength of a methacrylate-based sealer. J Endod 2011; 37:832-5.
80. Nagas E., Cehreli C., Durmaz V., Vallittu K., Lassila V.: Regional push-out bond strength and coronal microleakage of Resilon after different light-curing methods. J Endod 2007; 33: 1464–1468.
81. Alfredo E., Silva S., Ozório J., Sousa-Neto M., Brugnera-Júnior A., Silva-Sousa Y.: Bond strength of AH Plus and Epiphany sealers on root dentine irradiated with 980 nm diode laser. INT ENDOD J 2008; 41: 733–740.

82. Haragushiku G., Sousa-Neto M., Silva-Sousa Y., Alfredo E., Silva S., Silva R. Adhesion of endodontic sealers to human root dentine submitted to different surface treatments. Photo medicine and Laser Surgery 2010, 28: 405-410.
83. Ok E., Ertas H., Saygili G., Gok T.: Effect of photoactivated disinfection on bond strength of root canal filling. J Endod 2013; 39: 1428–1430.
84. Topçuoğlu H., Tuncay O., Demirbuga S., Dinçer A., Arslan H. The Effect of Different Final Irrigant Activation Techniques on the Bond Strength of an Epoxy Resin–based Endodontic Sealer: A Preliminary Stud. J Endod 2013.
85. Lopes G., Ballarin A., Baratieri L.: Bond strength and fracture analysis between resin cements and root canal dentin. Aus Endod J; 2010:1-7 (Article in press).

86. Saleh I., Ruyter I., Haapasalo M., Orstavik D.: The effects of dentin pretreatment on the adhesion of root canal sealers. Int Endod J 2002; 35:859–66.
87. Tagger M., Tagger E., Tjan A., Bakland L.: Measurement of adhesion of endodontic sealers to dentin. J Endod 2002; 28:351-4.

88. Goracci C., Tavares A., Fabianelli A.: The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements. Eur J Oral Sci 2004; 112:353– 61.
89. Haschke E.: Adhesive endodontic cones and related methods. United States Patent Application 20040202986. US Patent & Trademark Office, October 14, 2004.
90. Jensen S., Fischer D.: Method for filling and sealing a root canal. United States Patent & Trademark Office. Patent Number 6,811,400, November 2, 2004.
91. Chang J., Hurst T., Hart D., Estey A.: 4-META use in dentistry: a literature review. J Prosthet Dent 2002; 87:216–24.
92. Ori T., Otsuki H., Wakamatsu S.: Chemical surface analyses of a 4-META-containg methacrylate resin-based sealer. J Dent Res 2009; 88: 33-35.
93. Sun medical co ltd. 2009. Available at: http://www.sunmedical.co.jp/english/productinfo/hybrid-root seal/general/index.html.
94. Braga R., Ferracane J., Condon J.: Polymerization contraction stress in dual-cure cements and its effect on interfacial integrity of bonded inlays. J Dent 2002; 30:333– 40.

95. Pommel L., About I., Pashley D., Camps J.: Apical leakage of four endodontic sealers. J Endod 2003; 29:208 –10.

96. Pashley D., Ciucchi B., Sano H., Carvalho R., Russell C.: Bond strength versus dentin structure: a modelling approach. Arch Oral Biol 1995; 40:1109 –18.

97. Donnelly A., Sword J., Nishitani Y. : Water sorption and solubility of methacrylate resin– based root canal sealers. J Endod 2007; 33:990–994.

98. Unemori M., Matsuya Y., Matsuya S., Akashi A., Akamine A.: Water sorption of poly (methyl methacrylate) containing 4-methacryloxyethyl trimellitic anhydride. Biomaterials 2003; 24:1381–7.

99. Gopferich A.: Mechanisms of polymer degradation and erosion. Biomaterials1996; 17: 103–14.

100. Archegas L., Caldas D., Rached R., Vieira S., Souza E.: Sorption and Solubility of Composites Cured with Quartz-tungsten Halogen and Light Emitting Diode Light-curing Units. J Contemp Dent Pract 2008; 2:073-080.

101. Moon P., Weaver J., Brooks C.: Review of matrix metalloproteinase's effect on the hybrid dentin bond

layer stability and chlorhexidine clinical use to prevent bond failure. Open Dent J. 2010; 4:147-52.

102. Tay F., Pashley D., Loushine R., Weller R., Monticelli F., Osorio R.: Self-Etching Adhesives Increase Collagenolytic Activity in Radicular Dentin. J Endod 2006; 32:862–868.

103. Ferrari M., Vichi A., Grandini S., Goracci C.: Efficacy of a self-curing adhesive-resin cement system on luting glass-fiber posts into root canals: An SEM Investigation. Int J Prosthodont 2001; 14:543-9.

104. Mjör I., Smith M., Ferrari M., Mannocci F.: The structure of dentine in the apical region of human teeth. Int Endod J 2001; 34:346-53.

105. Mutal L., Gani O.: Presence of pores and vacuoles in set endodontic sealers. Int Endod J 2005; 38:690-6.

106. Tay F., Loushine R., Lambrechts P., Weller R., Pashley D.: Geometric factors affecting dentine bonding in root canals: a theoretical modeling approach. J Endod 2005; 31:584-9.

Introduction and review of literature

Success in endodontic treatment is mainly determined by complete obturation of root canal system. The objective of root canal obturation is to eliminate coronal and apical microleakage, creating a fluid tight seal along the dentinal wall.[1] Gutta percha in combination with sealer does not completely prevent bacterial leakage due to lack of adhesion to radicular dentin. There has been a continuous quest throughout the history of endodontics for a sealing material that bond to canal walls as well as to the core material to form a “monoblock”. [2]

New polymer-based obturation materials in combination with resin sealer have been developed in order to overcome the disadvantages of gutta percha and conventional sealer i.e Resilon. [3] Although these materials have proved to provide a better seal than gutta percha, unfortunately they have undesirable properties including low push-out bond strength, low cohesive strength and incomplete achievement of an apical seal.[4]

New dual curable methacrylate-based resin sealer has been developed to circumvent the disadvantages of Resilon, can be used with gutta percha or with resin-coated gutta percha to form a “monoblock”. [5]

A 4-methacryloloxyethyl trimellitate anhydride (4-META) containing methacrylate-based endodontic sealer (MetaSEAL) has been recently introduced .It bonds to radicular dentin via the formation of hybrid layer.[6] It also bonds to gutta percha through 4-META which is a good penetrating agent, permits the MetaSEAL to penetrate into the microstructure of gutta percha creating a “hybrid layer” .This allows the two materials to bond.[7]

Several studies have investigated the adhesion of root canal filling materials to the root canal walls. Among of the methods is the push out test which gives an accurate quantitative value for each specimen.(11) Very little studies have been done to evaluate the push out bond strength of these new obturation materials.

Aim of study

The present study will be directed to evaluate the bond strength of various adhesive obturation materials.

Supervisors

Prof. Dr. Taher Medhat Islam

Associate professor, Department of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University,

Cairo (Boys)

Dr. Ashraf Samir Mahmoud Refai

Lecturer, Department of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University,

Cairo (Boys)

Dr. Motaz-Bellah A.Al-khawas

Lecturer, Department of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University,

Cairo (Boys)

المشرفون

ا. د / طاهر مدحت إسلام

أستاذ مساعد علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

دكتور/ أشرف سمير محمود رفاعي

مدرس علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

دكتور/ معتزبالله أحمد الخواص

مدرس علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

Materials and methods

Freshly extracted single rooted mandibular premolars will be utilized in this study. Teeth will be stored in distilled water until tested. Crowns of teeth will be removed to facilitate root canal preparation. After that, preparation of the root canal will be done according to standardized technique. Samples will be divided equally into two groups, then, obturated according to obturation material;

Group 1: obturated with methacrylate based obturation material.

Group 2: obturated with 4-META based obturation material.

Samples will then be transversally sectioned at various levels for their evaluation using the push-out test.

The data will be collected, tabulated and statistically analyzed.

An evaluation of the bond strength of various adhesive root canal filling materials

Protocol Submitted in partial fulfillment of the requirements for Master Degree in Endodontics

By

Amr Abdelwahab Abdelhamed Bayoumi

B.D.S (Al-Azhar University 2004G),

Resident of Endodontics,

Faculty of Dental Medicine,

Al-Azhar University (Cairo-Boys).

Endodontic Department,

Faculty of Dental Medicine,

Al-Azhar University (Cairo-Boys)

2009G - 1430H

تقييم قوة الترابط لحشوات قنوات الجذوراللاصقة المختلفة

خطة بحث

مقدمة كجزء من مقومات الحصول على درجة الماجستير فى علاج الجذور

مقدمة من

الطبيب / عمرو عبد الوهاب عبد الحميد بيومى

بكالوريوس طب الفم و الأسنان جامعة الأزهر(القاهرة-بنين2004 م)

طبيب مقيم بقسم علاج الجذور

كلية طب الأسنان جامعة الأزهر(القاهرة- بنين)

قسم علاج الجذور

كلية طب الأسنان

جامعة الأزهر(القاهرة- بنين)

2009 م - 1430 هـ

References

1. Kardon BP, Kuttler S, Hardigan P, Dorn SO. An in vitro evaluation of the sealing ability of a new root-canal-obturation system. *J Endod*, 2003;29;10:658-61.

2. Gesi AR, Goracci C, Pashley DH, Tay FR, Ferrari M. Interfacial strength of resilon and gutta-percha to intraradicular dentin. *J Endod*, 2005;31;11:809-13.

3. Tay FR, Loushine RJ, Monticelli F, Weller RN, Breschi L ,Ferrari M, et al. Effectiveness of resin-coated gutta percha cones and a dual-cured, hydrophilic methacrylate resin based sealer in obturating root canals. *J Endod*, 2005;31:659-64.

4. Shipper G, Orstavik D, Teixeira FB, Trope M. An evaluation of microbial leakage in roots filled with a thermoplastic synthetic polymer-based root canal filling material (Resilon). *J Endod*, 2004;30:342–7.

5. Zmener O, Pameijer CH. Resin coated gutta-percha cones coupled with a resin-based sealer: a new alternative for filling root canals. *Endod Pract*, 2007;10:21-5.

6. Belli S, Ozcan E, Derinbay O, Unverdi Eldeniz A. A comparative evaluation of sealing ability of a new, self-etching, dual-curable sealer: Hybrid Root SEAL (MetaSEAL). *Oral Surg Oral Med Oral Pathol Oral Radiol Endod*, 2008;106:45–52.

7. Ungor M, Onay EO, Orucoglu H. Push-out bond strengths: the Epiphany- Resilon endodontic obturation system compared with different pairings of Epiphany, Resilon, AH Plus and gutta-percha. *Int Endod J*, 2006;39:643-7.

8. Sevimay S, Kalayci A. Evaluation of apical sealing ability and adaptation to dentine of two resin-based sealers. *J Oral Rehabil*, 2005;32:105-10.

9. Zmener O, Spielberg C, Lamberghini F, Rucci M. Sealing properties of a new epoxy resin-based root-canal sealer. *Int Endod J*, 1997;30:332-4.

10. Azar NG, Heidari M, Bahrami ZS, Shokri F. In vitro cytotoxicity of a new epoxy resin root canal sealer. *J Endod*, 2000;26:462–5.

11. Jainaen A, Palamara JE, Messer HH. Push-out bond strengths of the dentin sealer interface with and without a main cone. *Int Endod J*, 2007;40:882–90.

لجنة الحكم والمناقشة

أ.د / طاهر مدحت إسلام

أستاذ مساعد و رئيس قسم علاج الجذور

كلية طب الأسنان- جامعة الأزهر(القاهرة – بنين) مشرفاً ومناقشاً

أ.د / أحمد عبد الرحمن هاشم

أستاذ مساعد علاج الجذور

كلية طب الأسنان- جامعة عين شمس- مناقشا

أ.د / وائل حسين كامل

أستاذ مساعد علاج الجذور

كلية طب الأسنان- جامعة الأزهر(أسيوط)- مناقشاً

السادة المشرفون

ا . د / طاهر مدحت إسلام

أستاذ مساعد ورئيس قسم علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

دكتور/ أشرف سمير محمود رفاعي

مدرس علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

دكتور/ معتزبالله أحمد الخواص

مدرس علاج الجذور

كلية طب الأسنان

جامعة الأزهر

القاهرة-بنين

تقييم قوة الترابط لحشوات قنوات الجذوراللاصقة المختلفة

رســـالة

مقدمة كجزء من مقومات الحصول على درجة الماجستير

فى علاج الجذور

مقدمة من

الطبيب / عمرو عبد الوهاب عبد الحميد بيومى

بكالوريوس طب الفم و الأسنان- جامعة الأزهر (القاهرة- بنين 2004 م)

معيد بقسم علاج الجذور

كلية طب الأسنان جامعة الأزهر (القاهرة- بنين)

قسم علاج الجذور

كلية طب الأسنان

جامعة الأزهر(القاهرة- بنين)

1435 هـ - 2014 م