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Physicochemical Properties of Four Bioactive Root Canal Sealers

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Abstract

The purpose of present study was to assess and to compare the physicochemical properties of four bioactive root canal sealers. **Materials and Methods**: four bioactive root canal sealers that are available on the market were tested (Well-Root; Apexit Plus; Dia-Root and MTA Fillapex). The initial and final setting time was determined by Gilmore needle. Flow Test and solubility test conducted according to ISO standard 6876. The film thickness was determined according to the ISO 3107 standard. pH measurement for different sealers that immersed in deionized water at 5minutes, 1h, 12h, 24h and 48h time intervals. Fourier transform infrared spectroscopy (FTIR) analysis was done for the chemical structure of set samples for tested sealers. Data were analyzed by ANOVA and Tukey's tests ($p<0.05$) using SPSS. **Results:** The setting time of Well-Root significantly less than other sealers ($p=0.00$) for both initial and final setting time. Higher flow mean value presented with Well-Root and MTA Fillpex compared with than Apexit plus and Dia-Root respectively. Well-Root sealer shows significantly more solubility than other tested sealer. Film thickness of Well-Root sealer significantly less than other sealers. Well-Root sealer showed the highest alkaline value at all evaluation time followed by Dia-Root, Apexit Plus and MTA Fillapex , respectively. FTIR analysis revealed the chemical composition similarity and difference in set samples observed with change in intensities of the peaks. **Conclusion:** All the tested bioactive root canal sealers showed acceptable physicochemical properties.

Introduction:

Endodontic sealers have traditionally served to: (1) achieve sealing along the root canal systems including different canal complexity (isthmus, numerous foramina, lateral and accessory canals e.g), (2) bonding or bridging the capes between canal filling materials itself and with root canal walls, (3) providing mean of lubrication that promote positioning of canal filling, (4) reducing and/or killing of residual canal flora [1]. Sealers' chemical and physical properties have received substantial study because to their respective biological and technical value since their invention in the early twentieth century [(2)]. In the past thirty years, there is increased in the demand of bioceramic innovations in dentistry[(3)]. The interaction of bioceramic sealers with the surrounding periapical tissue determines whether they are bioactive or bioinert substance[(4)]. Bioactive substance is defined as "the capacity of a materials to elicit a specific biological responses at the interface of the material which results in the formation of a bond between the tissues and the material" [(5)]. The various bioactive materials are calcium hydroxide, mineral trioxide aggregate, calcium-enriched mixture, Biodentine, Inert material (isobutyl cyanoacrylate and tricalcium phosphate ceramic)[(6)] , MTYA1-Ca filler, tetracalcium phosphate [(7)], sol-gel-derived bioactive glass [(8)], ceramic containing silver ions, calcium phosphate [(9)]. Although various branded bioactive root canal sealers are available on the market, several are still relatively new, necessitating further laboratory and clinical trial to establish their performance.The study designed to evaluated and compare the physicochemical properties (setting time, flow, film thickness, pH, solubility and FTIR of setting sealers) for bioactive root canal sealers.

Material and Methods:

Four bioactive root canal sealers that are available on the market were tested: Well-Root (Vericom co. LTD, Korea); Apexit Plus (Ivoclar Vivadent, Schaan, Liechtenstien); Dia-Root (DiaDent, Europe B.V., Korea) and MTA Fillapex (Angelus, Londrina, Brazil).

Setting Time

The setting time for the four bioactive sealers were measured using a method described by Boyadzhieva et *al*., (2017) [(10)]. The sealer were filled a Paris mold congaing 12 openings with 10mm diameter and 2mm depth. The paris molds were first stored at 37ºC in a water bath for 24hrs, and then the sealer filled the mold. The premixed sealers (Well-Root and Dia-Root) were directly dispensed to mold, whereas, the other sealer (MTA Fillapex and Apexit Plus) manipulated according to the manufacturer instructions (n=5 for each). A Gilmore needle (mass 100±0.6g and diameter of 2.0±0.1mm) was used to determine the initial setting time. The sealer surface tested every 30min by lowering the tip of needle vertically on the sample surface until no longer indentation was visible. The final setting time started immediately after initial setting time by using a needle of 466.5g with 1mm diameter, again lowering the needle until no longer indentation was visible. During all the study, the samples kept in incubator at 37ºC and 95% humidity.

Flow Test

According to ISO standard 6876 [(11)] the test flow was carried out. A 0.5ml of each sealer was placed on the central surface of glass plate and leave it for 180seconds (n=10). Another glass plate (20g) was applied carefully above sealer and glass slab. A load of 100gm was placed on the top plate for 10min the major and minor diameters of compressed sealer had been measured using digital caliper with resolution 0.01mm (Mitutoyo MTI Corporation Tokyo, Japan). Only a difference of less than 1mm was reported in the study.

Film Thickness

The film thickness was determined according to the ISO 3107 standard [(12)]. The film thickness was determine as the difference in thickness of two square glass plate (area 200mm^2 and 5mm thickness) with and without the sealer interposed by a digital mirometer cliper (Mitutoyo Corp., Tokyo, Japan). The first measurement done when two glass plates togeather, whereas the second measurement done after application of 0.015g sealer between two plates. The glass plates with interposed sealer were pressed with 150N for 10min. five measurements for each sealer were done.

Solubility

The solubility was determined respecting the (ISO) 6876 [(11)]. A Teflon ring molds with internal diameter of 20mm and thickness of 1.5mm used to produce specimens for each sealers. The mold placed on thin polyethylene sheets supported by glass plates and was filled with the evaluated sealers, to slight excess. A nylon thread was inserted through the specimen of sealer before setting for hanging the specimen. After three times of setting time, the sealers were removed from the molds and every remaining particles were removed by microbrush. The specimens (n=10) were weighed in an analytical balance to precision 0.0015 (Sartorius Cubis, gottingen, Germany) and considered as specimen net weight (M0). Every specimen suspended by nylon thread in tightly seal glass bottle containing 50ml of distilled water, whereas the specimen positioned in such way not touched bottle walls at constant temperature of 37ºC for 28 days. After this period, the specimen removed from the bottle, gently washed with distilled water, dried firstly with filter paper and finally dehumidify at 37ºC for 1h. each specimens re-weight to obtain their final weight (M1). The solubility of the substance was calculated as the percentage of the weight lost as opposed to the initial weight as follow[13]:

Percentage of mass loss=(M0-M1/M0)*100

M0=initial specimen net weight

M1=final specimen net weight

Measurement of pH

To determine the pH value, a polyethylene tubes measuring 5mm in length and 1mm in diameter were filled with each sealers (n=10). The tubes were immersed in tightly sealed plastic tube containing 10ml of deionized water and kept in incubator at 37 ºC during the study. pH measurements were done at 5minutes, 1h, 12h, 24h and 48h. the measurement perfumed using a previously calibrated digital pH meter (Hanna instruments, HI 2211, Romania). The mean of 3 measurements was taken for each sample and calculated statistically.

FTIR Analysis

FTIR analysis was performed using ALPHA FTIR (Bruker, Germany) in the 500-3500 cm^{-1} wavenumber range. A sample for each sealer was prepared by a teflon ring molds with internal diameter of 9mm and thickness of 5mm. As this procedure did not require prior preparation, samples were directly placed on the diamond crystal for examination after complete setting of the sealer.

ANOVA and Tukey's tests were used to analyze test results at ($p<0.05$) using SPSS program (Version 25 for Windows, Inc., USA).

Results:

The setting time of Well-Root significantly less than Dia-Root, Apexit Plus and MTA Fillapex, respectively (p=0.00) for both intial and final setting time Table (1).

There is a significant difference between flow of sealer. Higher mean value presented with Well-Root and MTA Fillpex compared with than Apexit plus and Dia-Root respectively Table (1). Well-Root sealer shows significantly more solubility than other tested sealer Table (1). The result of film thickness indicated that film thickness of Apexit Plus, MTA Fillapex and Dia-Root was not statistically significant differ from each other but, significantly differ from Well-Root sealer Table (1). Well-Root sealer shows significantly greater solubility Apexit Plus, MTA Fillapex and Dia-Root, respectively. All the tested sealers showed significant differences of pH among themselves at all examined time intervals (p=0.00). Well-Root sealer showed the highest alkaline value at all evaluation time followed by Dia-Root, Apexit Plus and MTA Fillapex, respectively Table (2).

FTIR spectra of Apexit Plus sealer showed band of free hydroxyl group of calcium hygdroxid at 3642 cm^{-1} [15]. All the sealers presented band at 1000-1100 cm^{-1} which is related to Calcium silicate hydrate [16]. A band of the sulfate group (calcium sulfate hydrate) were detected at 1153 cm^{-1} for all sealers[17]. Around 750 cm^{-1} MTA Fillapex, Dia-root and Apexit Plus sealers showed a peak of tricalcium aluminate[17].

All the sealers showed bands at 1410, 869 and 660 cm^{-1} related to calcium carbonate in the C-O group [15. phosphate phase bands at 1097 cm^{-1} for Dia-Root, Apexit

Plus , 960 cm^{-1} for MTA Fillapex , 607 cm^{-1} and 570 cm^{-1} for MTA Fillapex [16,17], are clearly observed. The band at 1070–1098 cm^{-1} is ascribed to the overlapping of the PO4 groups of hydroxyapatite and the band at 1020–1040 cm^{-1} also belongs to the PO4 groups of hydroxyapatite for all tested sealers[18] Fig. (1).

Discussion:

Nowadays, with many commercially available and successively developed bioactive root canal sealers, it is more crucial for the endodontist to comprehend the physicochemical properties of various sealers that allow them to work perfectly in clinical situations.

Until now, there is no definitive standard setting time of endodontic sealers but, according to the ANSI/ADA[19] the setting time of a sealers should vary only 10% in comparisons to established by manufacturer. According to the present results, no any sealers tested are fulfill the requirements of ANSI/ADA with regard to setting time. Ørstavik *et al.,* 2001[20], stated that the setting time of a sealer depend on sealer components, particle size, temperature, and humidity. The long setting time for Apexit Plus and MTA Fillapex can be related to the presence of epoxy resin in former and salicylate resin in later[21]. The result in accordance with Camilleri (2015) [21]. Also the delay in setting time of Apexit Plus has been associated with calcium oxide, which is converted to calcium hydroxide in contact with water, which delays the setting time[22]. In comparisons to the other sealers in this study, the short setting time of Well-root may be due to absence of calcium hydroxide on its composition[23]. In general the a number of radiopacifying agents add to bioactive sealers and these agents can be responsible for the long setting time, since these agents have low solubility in water[20].

According to ISO 6876[11] specification required that sealer shall have a diameter of no less than 20mm. Although all the sealers met the dependent specification. Theoretically, good flow contributes to good penetration into complexity of root canal system (isthmus, dentinal tubules, accessory and lateral canals), it is important to realized that excessive flow might cause leaks into the periapical tissues, that may complicate the healing process depending on degree of sealer cytotoxicity[24]. Moreover, some authors believed that it is not crucial that the flow of bioactive sealers are in agreement with ISO and previous studies [25,26]. This thought is based on the bioactivity of the sealers, once mineral infiltration zone is occur, and the bioactive sealer becomes part of the canal dentine[26]. A thin film thickness sealer is believed to moisten the surface more effectively than a thick one, giving in a superior seal[27]. The findings of the film thickness data for all tested sealers were in accordance with the ISO 6876[11] specification, which states that no more than 50μm required to provide a superior seal [11].

Solubility is an important criterion for assessing validity of dental materials. It is defined as "the ability of a substance to dissolve in another and it's expressed as the concentration of the saturated solution of the former in the latter"[28]. According to the ISO 6876 [11] or ANSI/ADA[19] Specification No. 57, the solubility of sealer should not exceed 3% . The lesser solubility for Apexit Plus and MTA Fillapex can be related to the presence of epoxy resin in former and salicylate resin in later which could be lead to their strong cross-linking polymers [21]. Because root canal sealers can come into close contact with periapical tissues fluids in the apical area, this type of investigation is crucial [29].

The pH value of sealer determine physicochemical, antibacterial and biological properties. The capacity of root canal sealers to be osteogenic, biocompatible, and antibacterial may be enhanced by an alkaline sealer [30,31]. Present study, the result indicated the sealers (Well-Root and Dia-Root) showed significantly higher pH values than the resin containing bioactive sealers (MTA Fillapex and Apexite Plus) in all experimental time intervals (p=0.00). Well-Root sealer showed the highest alkaline value followed by Dia-Root, MTA Fillapex and Apexit Plus respectively. The diffrences between non resin containing bioactive sealers (Well-Root and Dia-Root) and resin containing bioactive sealers (MTA Fillapex and Apexite Plus) probably reflect the fact the former is pure bioactive sealers, whereas the latter are combined of bioactive and resin based sealers. from the other hand the alkalizing effect can be explained by the presence of calcium hydroxide in matrix of former sealers and similar result confirmed by many previous studies[25,30-32]. The result of FTIR indicated that all sealers used in this study contain calcium silicate (Ca3SiO5 and Ca2SiO4), which is the base compounds of bioactive and bioceramic sealer, together with radiopacifying ingredient such as zirconia, bismuth, tantalum or tungsten oxides[33,34]. Calcium silicate-based sealers, particularly those containing bismuth oxide radiopacifier, have some drawbacks such as tooth discolouration, a lengthy setting period, and challenge in handling. [35]. The investigated root canal sealers showed the presence of free –OH (band at 3642cm^{-1}) in the Apexit Plus sealer. It is important to know that upon hydration, it is not necessary that all calcium silicate based materials produce calcium hydroxide[36]. Even with the presence of calcium hydroxide as a reaction product, some additive component could react with it and reduce its amount agreement with Camilleri *et al.*, (2015)[21] and Abu Zeid *et al.*, (2017) [37]. The presence of phosphate phase in all tested sealer mean the bioactivity capacity to mineralization induction[16,36]. The presence of sulfur in the FTIR analysis of all sealers indicating the presence of gypsum (calcium sulfate) phase of Portland cement which is plays important role to harden the material[18, 38].

Conclusions:

Based on the present study, the tested bioactive endodontic sealers showed in general acceptable physicochemical properties but Well-Root seems to be more soluble. Although the differences in the composition of the tested sealers, the FTIR analysis showed close similarity in the composition of the final set sealer.

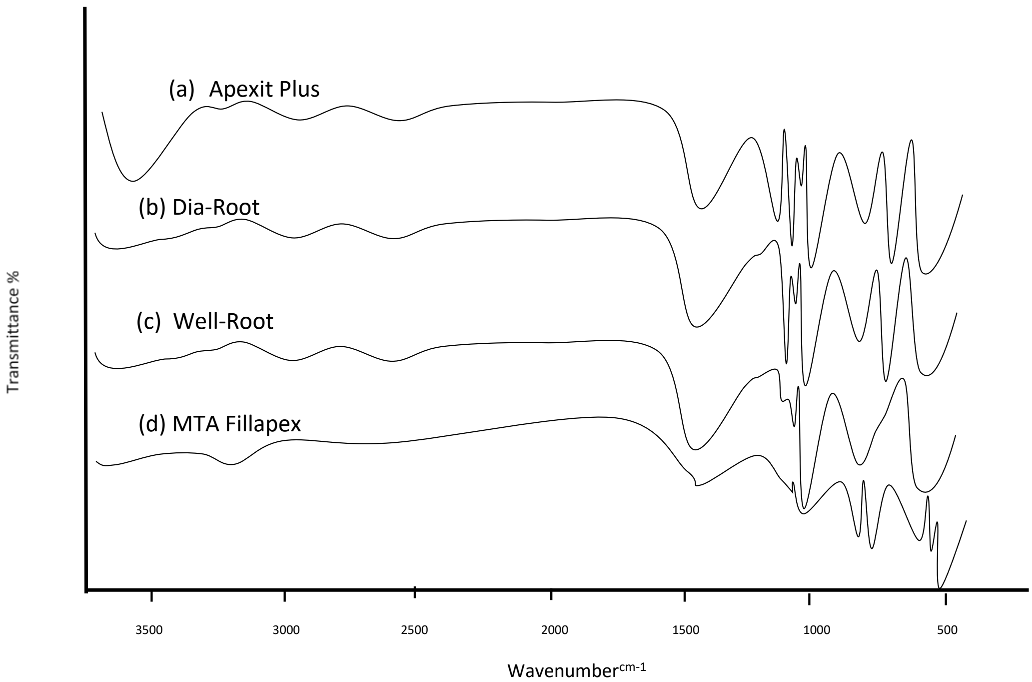

Figure (1): FTIR of set bioactive sealers (a) Apexit Plus, (b) Dia-Root, (c) Well-Root and (d) MTA Fillapex.

Table 1: shows the mean values for the physicochemical properties of the tested sealers.

Sealer	Setting time(min)		Flow (mm)	Solubility (%)	Film thickness (μm)
	Initial	final			
Apexit Plus	434[c]	1038[c]	20.7[b]	1.2[b]	20[b]μm
Dia-Root	350[b]	767[b]	20.8[b]	1[b]	20[b] μm
MTA Fillapex	801[d]	1092[d]	28.6[a]	1.1[b]	23[b]μm
Well-Root	138[a]	586[a]	30.2[a]	6[a]	30[a] μm

[a,b,c,d] Significant differences between sealers are indicated by different letters in each column.

Table 2: pH measurements of different sealers at different time intervals.

Sealers	Times intervals				
	5 min	1h	12h	24h	72h
Apexit Plus	9.80±.07 d	10.25±.05d	10.46±.03d	10.56±.05d	10.53±.04d
Dia-Root	11.38±.51 b	11.50±.04b	11.51±.03b	11.48±.04a	11.74±.41b
MTA Fillapex	9.34±.03c	9.60±.11c	10.18±.12c	10.16±.15c	10.16±.15c
Well-Root	11.51±.03 a	11.58±.04 a	11.67±.05a	11.79±.12b	11.79±.12a

[a,b,c,d] Significant differences between sealers are indicated by different letters in each column at examined time intervals.

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