Türkiye Klinikleri Diş Hekimliği Bilimleri Dergisi
Turkiye Klinikleri Journal of Dental Sciences Turkiye Klinikleri J Dental Sci. 2024;30(1):83-91

ORIGINAL RESEARCH ORİJİNAL ARAŞTIRMA

DOI: 10.5336/dentalsci.2023-98001

Physicochemical Properties of Hydraulic Calcium Silicate Sealers in Comparison with Resin and Calcium Hydroxide-Based Sealers: An *In Vitro* Study

Hidrolik Kalsiyum Silikat Bazlı Kanal Patlarının Fizikokimyasal Özelliklerinin Rezin ve Kalsiyum Hidroksit Bazlı Kanal Patları ile Karşılaştırılması: *İn Vitr*o Çalışma

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ABSTRACT Objective: This study aimed to compare the physicochemical properties of 3 bioceramic sealers (NeoSealer Flo, Well-Root ST, and CeraSeal) with mineral trioxide aggregate-like sealer (MTA Fillapex), epoxy resin-based sealer (AH Plus Jet), methacrylate resin-based sealer (EndoREZ), and calcium hydroxide/salicylate resin-based sealer (Apexit Plus) in terms of flow, setting time, film thickness, solubility, water absorption, radiopacity, immediate and delayed pH, compressive strength, and dimensional stability. **Material and Methods:** Flow, setting time, film thickness, and solubility of all sealers were evaluated by International Standards Organization (ISO) 6876:2012, while water absorption and radiopacity values were determined using ISO:4049:2019 and ISO:13116:2014, respectively. Five samples were prepared from sealers for each test (n=5). Data were analyzed with Kruskal-Wallis H test with 5% significance threshold. **Results:** Flow, setting time, film thickness, and radiopacity values of all canal sealers complied with ISO standards. The solubility of AH Plus Jet, EndoREZ sealers was acceptable, while those of NeoSealer Flo, Well-Root ST, CeraSeal, MTA Fillapex, and Apexit Plus showed greater solubility than recommended. The lowest pH value in freshly mixed samples was observed in EndoREZ sealer. **Conclusion:** All the sealers complied with ISO 6876:2012 and ISO 13116:2014 standards regarding flow, setting time, film thickness, and radiopacity. MTA Fillapex did not set during its reported working time. The solubility values of AH Plus Jet, EndoREZ pastes were found to comply with ISO 6876:2012 standards, while Apexit Plus and bioceramic sealers exceeded the threshold.

Keywords: Endodontics; epoxy resin-based root canal sealer; root canal filling materials

ÖZET Amaç: Bu çalışma, 3 farklı hidrolik kalsiyum silikat kanal patının (NeoSealer Flo, Well-Root ST ve CeraSeal) fizikokimyasal özelliklerini bir mineral trioksit agregat içeren kanal patı (MTA Fillapex), bir epoksi rezin bazlı kanal patı (AH Plus Jet), bir metakrilat rezin bazlı kanal patı (EndoREZ) ve bir kalsiyum hidroksit/salisilat reçine bazlı kanal patı (Apexit Plus) ile sertleşme zamanı, film kalınlığı, çözünürlük, su emilimi, radyoopasite, sertleşme esnasında ve sonrasındaki pH değerleri, baskı dayanımı ve boyutsal stabilite açısından karşılaştırmayı amaçlamıştır. **Gereç ve Yöntemler:** Akıcılık, sertleşme süresi, film kalınlığı, çözünürlük Uluslararası Standartlar Teşkilatı [International Standards Organization (ISO)] 6876:2012 ile değerlendirilirken su emme, radyoopasite, erken ve geç pH, basınç dayanımı ve boyutsal stabilite sırasıyla ISO:4049:2019 ve ISO:13116:2014 ile değerlendirildi. Her bir test için beşer örnek hazırlandı (n=5). Veriler %5 anlamlılık eşiği ile Kruskal-Wallis H testi ile istatistiksel olarak analiz edildi. **Bulgular:** Tüm kanal patlarının akıcılık, sertleşme süresi, film kalınlığı ve radyopaklık değerleri ISO standartlarına uygundu. AH Plus Jet, EndoREZ kanal patlarının çözünürlüğü kabul edilebilir düzeydeyken, NeoSealer Flo, Well-Root ST, CeraSeal, MTA Fillapex ve Apexit Plus arzu edilenden daha yüksek çözünürlük göstermiştir. Yeni karıştırılmış örneklerde en düşük pH değeri EndoREZ için bulundu. **Sonuç:** Bütün kanal patları akıcılık, sertleşme süresi, film kalınlığı ve radyoopasite açısından ISO 6876:2012 ve ISO 13116:2014 standartlarına uygun bulunmuştur. MTA Fillapex belirtilen sertleşme süresinde sertleşme göstermemiştir. AH Plus Jet ve EndoREZ patlarının çözünürlük değerleri ISO 6876:2012 standardına uygun bulunurken, Apexit Plus ve biyoseramik patlar standartta belirtilen eşiği aşmıştır.

Anahtar Kelimeler: Endodonti; epoksi-rezin bazlı kök kanal patları; kök kanal dolgu materyalleri

TO CITE THIS ARTICLE:
Teker B, Keskin C, Aydemir H. Physicochemical properties of hydraulic calcium silicate sealers in comparison with resin and calcium hydroxide-based sealers: An *in vitro* study. Turkiye Klinikleri J Dental Sci. 2024;30(1):83-91.

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Peer review under responsibility of Turkiye Klinikleri Journal of Dental Sciences.

Received: 16 May 2023 ***Received in revised form:*** 27 Oct 2023 ***Accepted:*** 21 Nov 2023 ***Available online***: 07 Dec 2023

One of the most critical steps in a successful root canal treatment is the three-dimensional sealing of the root canal system following thorough disinfection to treat or prevent apical periodontitis.[1] The ideal root canal filling aims to prevent the infiltration of microorganisms into the root canal due to coronal microleakage, the infiltration of tissue fluid into root canal space through the accessory canals and apical foramen, and the proliferation of the remaining microorganisms within the root canal system.[2] Root canal sealers are used to provide three-dimensional sealing covering the irregularities of canal anatomy, where the core material cannot reach.[3] The root canal sealers possess antibacterial properties and fill the voids between gutta percha and the dentin walls, thus ensuring that the canal space is filled and sealed.

Sealers have been classified differently according to their physical properties, setting times, materials they contain, and their ability to be resorbed. Each canal sealer has a different composition; therefore, they all exhibit different physical, chemical, and biological properties. Today, there is a wide variety of commercialized root canal sealers claiming to provide ideal properties for root canal filling. Epoxy resin-based canal sealers are considered the gold standard and the most known epoxy resin-based sealer AH Plus (Dentsply Sirona, DeTrey, Konstanz, Germany) is often used in studies for comparison.[4] They have favorable physicochemical properties including optimal working time, dimensional stability, adequate radiopacity, and low solubility.[5]

Development of sealers based on hydraulic tricalcium silicate, also known as bioceramics, aimed to achieve bioactivity exhibited by calcium hydroxide that forms as a result of a reaction between tricalcium silicate and phosphates from tissue fluids.[6] Hydraulic calcium silicate sealers offer a number of advantages compared to traditional canal sealers due to their biocompatibility, bioactivity, and bioinductivity.[7,8] Due to these biological properties, their clinical use has become widespread and led to the development of many commercially available products for hydraulic condensation technique.[9] MTA Fillapex (Angelus, Londrina, Brazil) has a mineral trioxide aggregate component but mainly consists salicylate resin matrix and silica.[10] CeraSeal (Meta Biomed, Cheongju, Korea) and Well-Root ST (Vericom, Chuncheon, Korea) are both premixed hydraulic calcium silicate sealers that have been reported to show favorable biological properties while studies also indicated that their physicochemical properties did not complied with International Standards Organization (ISO) standards.[9,11-13] NeoSealer Flo (Avalon Biomed, Houston, TX, USA) is a novel hydraulic calcium silicate sealer that has not been evaluated in terms of its physicochemical properties, while the manufacturer claims that it has high flow and radiopacity values.[14]

Physicochemical features of root canal sealers are investigated using standardized laboratory tests described by the ISO.[15] Although some requirements to modify test procedures with the development of different dental materials, ISO standards have been reported to be the best method for evaluating canal sealers.[4] The present study aimed to compare flow, setting time, film thickness, solubility, water absorption, radiopacity, immediate and delayed pH, compressive strength (CS), and dimensional stability of hydraulic calcium silicate sealers NeoSealer Flo, CeraSeal, Well-Root ST with AH Plus, MTA Fillapex, methacrylate resin-based EndoREZ (Ultradent, South Jordan, UT, USA), and calcium hydroxide/salicylate resin-based Apexit Plus (Ivoclar Vivadent, Schaan, Liechtenstein). The null hypothesis was that no significant difference would be detected among sealers in terms of their physicochemical properties.

MATERIAL AND METHODS

Automix dual syringe forms of AH Plus Jet, MTA Fillapex, EndoREZ and Apexit Plus were used, while NeoSealer Flo, CeraSeal, and Well-Root ST were premixed. Chemical composition of the sealers ISO is presented in Table 1. ISO 6876:2012 standard was used to evaluate flow, setting time, film thickness, and solubility; ISO:4049:2019 standard was used to evaluate water absorption; ISO:13116:2014 was used to evaluate radiopacity. Sample sizes were determined using G*Power (3.1 for Mac.; Heinrich Heine, Universitat Dusseldorf, Dusseldorf, Germany) using the appropriate effect size of the previous studies with an alpha-type error of 0.05 and a power beta of 0.80-0.95. Five samples were prepared for each test (n=5).[4,9]

TABLE 1: Chemical composition and manufacturer of the sealers used in this study.

Sealer	Sealer type	Manufacturer	Composition
AH Plus Jet	Epoxy resin based	Dentsply De Trey Gmbh, Konstanz, Germany	Paste A: Bisphenol-A epoxy resin, bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments Paste B: Dibenzyldiamine aminoadamantane, tricyclodecane-diamine, calcium tungstate, zirconium oxide, silica, silicone oil
EndoREZ	Methacrylate-based	Ultradent, UT, USA	Triethylene glycol dimethacrylate, bismuth chloride oxide, calcium lactate pentahydrate, diurethane dimethacrylate
NeoSealer Flo	Bioceramic	Avalon Biomed, Houston, TX, USA	Tricalcium silicate, tricalcium aluminate, dicalcium silicate, calcium aluminate, calcium sulphate, tantalum oxide, grossite
Well-Root ST	Bioceramic	Vericom Dental, Gangwon-Do, Korea	Calcium silicate, calcium sulphate dehydrate, calcium sodium phosposilicate, zirconium oxide, titanium oxide, thickening agents
CeraSeal	Bioceramic	Meta Biomed, Cheongju, Korea	Calcium silicate, zirconium oxide
MTA Fillapex	MTA-based	Angelus, Londrina, Brazil	Paste A: Salicylate resin, bismuth trioxide, fumed silicon dioxide Paste B: Fumed silicon dioxide, titanium dioxide, tricalcium silicate, dicalcium silicate, calcium oxide, tricalcium aluminate
Apexit Plus	Calcium hydroxide-based	Ivoclar Vivadent, Schaan, Liechtenstein	Base: Calcium hydroxide, hydrated collophonium Activator: Disalicylate, bismuth hydroxide

FLOW ANALYSIS

According to ISO 6876:2012 specification, 0.05 mL of each sealer was transferred in the center of a 40x40 mm glass 5 mm in thickness and weight of 20 g. An identical glass was placed over the first 180 s after the mixing, and a weight of 100 g was applied vertically to the glass. Maximum and minimum diameters of the compressed sealers were measured by a digital caliper (Civtec CTN Digital Micrometer, İstanbul, Türkiye), and the test was renewed if the difference between them was within 1 mm. ISO 6876:2012 required flow values should be at least 17 mm in diameter.[15] The mean value of 3 measurements was calculated.

SETTING TIME

Sealers were transferred in stainless steel molds (10 mm x 2 mm) were prepared and stabilized on cellophane coated glasses. MTA Fillapex, NeoSealer Flo, Well-Root ST, and CeraSeal samples were wrapped with moist gauze pieces with sterile distilled water (DW), as the specification required.[15] Since EndoREZ is a dual-cure sealer, the specimens were initially left in a dark room for 40 seconds and then light-cured. Vicat needle apparatus (Atom Teknik, Ostim, Ankara, Türkiye) was used to determine setting time. Each sample was placed on the plate of the Vicat apparatus and the needle was lowered vertically to touch the surface of the sample. If an indentation was created, the needle tip was removed, and cleaned the test was repeated every 10 min or hour according to the reported setting time. The time when the needle failed to cause indentation on the sample surface was recorded.

FILM THICKNESS

The film thickness was determined as described in ISO 6876/2012. The mixed sealers were placed between the glass plates. Following 180±5 seconds from the mixing, 150 N load was applied. After 10 minutes from the start of mixing, the thickness of the plates and the sealer was measured thrice.

SOLUBILITY AND WATER ABSORPTION

Solubility and water absorption were determined according to the guidelines recommended by ISO 6876/2012 and ISO 4049/2019, respectively. The solubility was determined according to the mass change after the samples were suspended in DW and phosphate-buffered saline (PBS). Sealers were prepared as disks (10 mm x 2 mm) and incubated for 24 hours at 37°C and >95% relative humidity. The set samples were weighed three times on a balance with an accuracy of 10^{-3} g, and the average value was recorded as $mass_1$ (m_1) in grams. The samples, which were kept in DW or PBS, were removed after 15 minutes and dried slightly with air, weighed again to determine the water absorption, and this value was recorded as mass$_2$ (m_2). The samples were dried in a desiccator for 24 hours and weighed thrice, and the average value was recorded as mass$_3$ (m_3). These steps were repeated separately in the 1st and 30th days using both DW and PBS. The solubility and water absorptions were calculated using the formula below:

Solubility=$(m_1-m_3)/m_1$X100

Water absorption=$(m_2-m_3)/m_1$X100

RADIOPACITY

Sealers were prepared as disks in metal molds 10 mm in diameter and 1 mm thickness and incubated (37˚C, >95% RH) according to the longest specified setting time. Digital radiographic images were obtained at 60 kVp and 7 mA with an intraoral radiography device (Sirona Variou DG, York, PA, USA) with a 10-step 99% pure aluminum step. The focal spot and object distance was 30 cm, and the exposure time was 0.2 s. A separate graph was created for each experimental sample using a computer program (Curve Expert 1.3, California, USA) for the grayness values of the steps in the aluminum assembly. Using the formula of this graph, the equivalents of the density values obtained from the canal sealer images to the aluminum thickness were calculated (Microsoft Excel 2000, Washington, USA). The mean value of 10 measurements were calculated.

pH ANALYSIS

Mixed samples were transferred to stainless molds (5 mm x 1 mm). Freshly mixed and set samples were immersed in DW at 37˚C. The pH of the solution was measured at certain periods (3, 15, and 60 min and 24 h for fresh samples and 24 h, 7, 14, 21, and 30 days for set samples), by using a digital pH meter (EDT Instruments Auto pH Meter, Dover, UK). The mean value of 10 measurements were calculated.

CS

Cylindrical samples (6 mm x 12 mm) were kept at 37˚C and 95% relative humidity for 30 days. The samples were mounted on a Universal test device (Instron, Norwood, MA, USA) towards a calibrated rod with a crosshead speed of 0.5 mm/min until failure, which was recorded as the loading failure (P). CS was calculated according to the formula (D is the diameter):

$$CS=4P/\pi D2.$$

DIMENSIONAL STABILITY

Samples were prepared as discs (6 mm x 12 mm) and incubated at 37°C and 95% relative humidity until set. Then, the distance between the flat ends of the samples was measured with a digital caliper. Three measurements were made for each sample, and the mean value was recorded as D_1. Afterward, the samples were kept in 10 mL DW at 37°C for 30 days, and the measurements were repeated and recorded as D_2. The mean change in D was calculated with the formula:

$$D=(D_2-D_1)/D_1\times100.$$

STATISTICAL ANALYSIS

SPSS (IBM, Chicago, IL, USA) software was used for all statistical analyses. Kolmogorov-Smirnov test was used to determine that the data conformed to the normal distribution. Comparison of the sealers in terms of the tested parameters was performed with the Kruskal-Wallis H test with a 5% significance threshold.

RESULTS

FLOW ANALYSIS

The flow, setting time, film thickness and radiopacity values of the sealers are shown in Table 2. All sealers showed flow values required by ISO 6876:2012 standards, and none of the samples were renewed (Table 2). Apexit Plus showed the highest, and MTA Fillapex showed the lowest flow values, although no significant difference was found among all sealers ($p>0.05$).

SETTING TIME

AH Plus exhibited the longest setting time, followed by NeoSealer Flo, with no significant difference between them ($p>0.05$). EndoREZ had the shortest setting time, which was significantly shorter than AH Plus, NeoSealer Flo, CeraSeal, and Apexit Plus ($p>0.05$). At the same time, no significant difference

TABLE 2: The flow (mm), setting time (min), film thickness (μm), and radiopacity (mmAl) values of the tested sealers (mean±standard deviation).

	AH Plus	EndoREZ	NeoSealer Flo	Well-Root ST	CeraSeal	MTA Fillapex	Apexit Plus
Flow	23.33^{a}±1.52	23.00^{a}±3.46	25.33^{a}±3.05	24.66^{a}±2.51	24.00^{a}±2.64	19.66^{a}±2.08	27.66^{a}±1.52
Setting time	682.0^{a}±17.33	33.25^{b}±3.09	453.25^{a}±14.68	138.25^{bc}±23.86	209.0^{ac}±7.74	-	221.0^{ac}±12.67
Film thickness	18.0^{ab}±2.0	18.33^{ab}±3.51	14.66^{ab}±2.08	16.0^{ab}±2.0	14.66^{ab}±0.57	19.66^{a}±1.52	11.33^{b}±1.52
Radiopacity	15.14^{a}±0.15	13.32^{a}±0.41	10.79^{a}±0.26	7.47^{ab}±0.21	5.41^{b}±0.32	3.48^{b}±0.33	11.31^{a}±0.31

Different superscript letters in each row indicate significant difference among sealer groups (p<0.05).

was found between Well-Root ST, CeraSeal, and Apexit Plus (p>0.05). MTA Fillapex sealer did not set completely during the working period. The remaining sealers were found to comply with ISO 6876:2012 standards.

FILM THICKNESS

The highest average film thickness value belongs to MTA Fillapex sealer, and the lowest average film thickness value belongs to Apexit Plus sealer, and there was a significant difference between them (p>0.05). The remaining sealers had similar film thickness values (p>0.05). All sealers showed film thickness values in accordance with ISO 6876:2012 standards.

SOLUBILITY AND WATER ABSORPTION

The solubility and water absorption values of the sealers are shown in Table 3. At the end of 1st day, all sealers show similar solubility in DW and PBS (p>0.05). In the samples immersed in DW, NeoSealer Flo, Well-Root ST, and CeraSeal showed a significant increase in solubility (p>0.05) between the 1st and 30th days, while the increase in solubility of the remaining sealers was not significant (p>0.05). However, in PBS, only Well-Root ST and CeraSeal showed a significant increase in solubility (p>0.05). All sealers dissolved less in PBS; however, the difference was not significant (p>0.05). AH Plus Jet and EndoREZ complied with ISO 6876:2012 standards (<3%) in all environments.

Between the 1st day to the 30th, CeraSeal showed significantly greater water absorption rates in both DW and PBS (p>0.05). Water absorption values of AH Plus and EndoREZ were significantly lower than other sealers (p>0.05) and did not change according to the medium (p>0.05).

RADIOPACITY

All canal sealers showed radiopacity values in accordance with ISO 6876:2012 standards, while MTA Fillapex exhibited the lowest opacity values,

TABLE 3: Solubility (%) and water absorption (%) values of the tested sealers immersed in distilled water or phosphate buffered liquid (mean±standard deviation).

	Solubility				Water absorption			
	DW		PBS		DW		PBS	
	1st d	30th d	1st d	30th d	1st d	30th d	1st d	30th d
AH Plus	0.47^{aA}±0.45	0.77^{aA}±0.29	0.40^{aA}±0.02	0.42^{a}A±0.23	1.35^{aA}±0.29	1.59^{aA}±0.48	1.00^{aA}±0.38	1.64^{aA}±0.20
EndoREZ	0.79^{aA}±0.12	0.87^{aA}±0.12	0.68^{aA}±0.17	0.56^{aA}±0.21	1.59^{aA}±0.24	1.82^{aA}±0.15	1.38^{aA}±0.03	1.77^{aA}±0.28
NeoSealer Flo	5.41^{aA}±1.94	9.42^{bA}±1.58	2.06^{aA}±0.14	5.68^{aA}±0.33	15.91^{bA}±1.17	20.51^{bA}±2.36	20.02^{bA}±1.43	22.55^{bA}±1.01
Well-Root ST	6.79^{aA}±0.49	9.77^{bA}±0.67	2.95^{aA}±0.1	7.00^{aB}±0.47	8.21^{abA}±1.54	16.97^{bB}±0.31	12.49^{bA}±2.07	20.07^{bA}±1.44
CeraSeal	4.24^{aA}±0.03	12.09^{bB}±0.43	2.71^{aA}±0.47	8.08^{aB}±0.95	11.93^{bA}±1.69	20.57^{bB}±1.58	13.01^{bA}±2.25	28.81^{bB}±1.37
MTA Fillapex	4.28^{aA}±0.41	6.09^{abA}±0.88	1.73^{aA}±0.67	3.44^{aA}±0.72	10.11^{bA}±1.23	14.73^{bA}±0.94	12.50^{bA}±1.49	19.03^{bA}±1.89
Apexit Plus	6.20^{aA}±0.09	8.63^{abA}±0.43	3.90^{aA}±0.06	5.08^{aA}±0.96	12.91^{bA}±2.53	16.54^{bA}±0.79	12.98^{bA}±1.03	19.75^{bA}±0.01

Different superscript lower-case letters in each column indicate significant difference between sealer groups at the tested period (p<0.05); Different superscript capital letters indicate significant difference between 1st d and 30th d of the same material (p<0.05); DW: Distilled water; PBS: Phosphate-buffered saline.

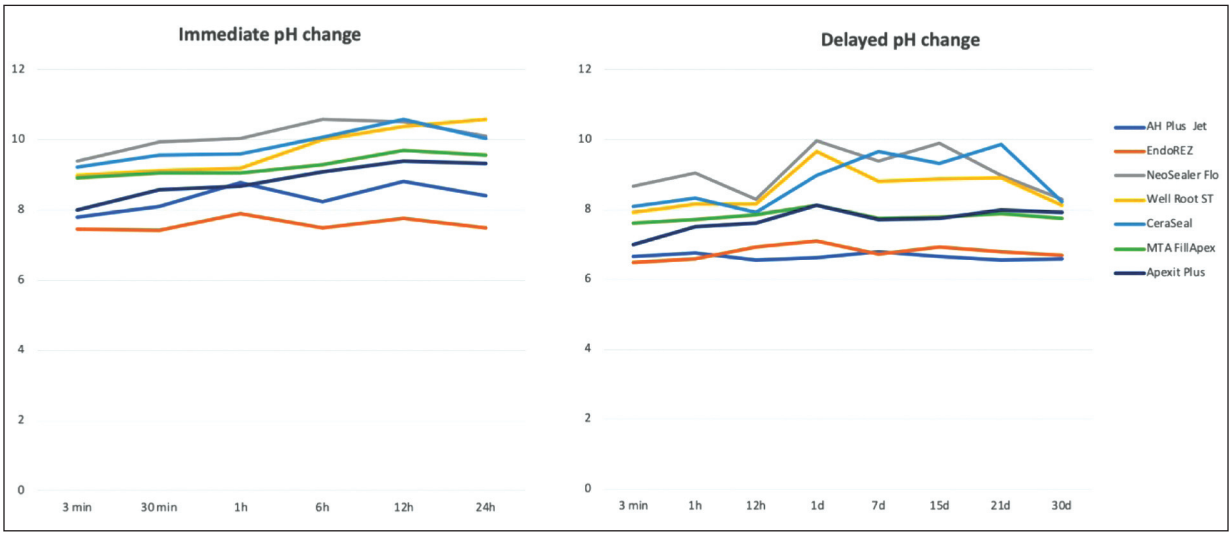

FIGURE 1: The pH values of the freshly mixed and set sealers at different intervals.

CeraSeal, and Well-Root ST (3.4, 5.4, and 7.4, respectively) ($p>0.05$).

pH ANALYSIS

The pH values of the "immediate" and "delayed" samples of the sealers are shown in Figure 1. All sealers show similar pH values throughout the measurement intervals ($p>0.05$), apart from 30 min after mixing. After 30 min of mixing, fresh EndoREZ showed significantly lower pH than other sealers ($p>0.05$). The pH of EndoREZ was neutral in both fresh and set samples, whereas AH Plus was neutral in freshly mixed samples. Despite no significant difference being detected between fresh and set samples, set samples of AH Plus, EndoREZ, MTA Fillapex , and Apexit Plus showed lower pH values ($p>0.05$).

CS

The CSs of MTA Fillapex and Apexit Plus were significantly lower than the remaining sealers ($p>0.05$) (Table 4).

DIMENSIONAL STABILITY

All sealers showed an increase in dimensions after 30 days. At the end of 1st day, the dimensional stability of EndoREZ, MTA Fillapex, and Apexit Plus were significantly higher than the remaining sealers ($p>0.05$). AH Plus, EndoREZ, and NeoSealer Flo showed significantly higher dimensional stability

TABLE 4: Dimensional stability (%) and compressive strength (MPa) of the tested sealers (mean±standard deviation).

	Dimensional stability		Compressive strength
	1 day	30 days	(MPa)
AH Plus	$0.17^{a}\pm0.02$	$0.42^{a}\pm0.02$	$16.66^{a}\pm0.45$
EndoREZ	$0.13^{b}\pm0.01$	$0.23^{a}\pm0.02$	$13.79^{a}\pm1.05$
NeoSealer Flo	$0.25^{a}\pm0.01$	$0.45^{ab}\pm0.04$	$10.82^{a}\pm0.46$
Well-Root ST	$0.33^{a}\pm0.01$	$0.61^{abc}\pm0.02$	$10.23^{a}\pm0.44$
CeraSeal	$0.16^{a}\pm0.02$	$0.77^{bc}\pm0.06$	$9.69^{a}\pm0.23$
MTA Fillapex	$0.13^{b}\pm0.02$	$0.60^{abc}\pm0.01$	$1.83^{b}\pm0.16$
Apexit Plus	$0.13^{b}\pm0.02$	$0.79^{c}\pm0.02$	$3.53^{b}\pm0.26$

Different superscript lower-case letters in the same column indicate significant differences among sealers in terms of dimensional stability at 1 and 30 days, and compressive strength.

compared to the remaining groups at 30th day ($p>0.05$) (Table 4).

DISCUSSION

The present study evaluated the physicochemical properties of 3 novel hydraulic calcium silicate sealers with commonly used epoxy resin-, methacrylate resin-, calcium hydroxide and salicylate resin-, and MTA-based sealers. Since the properties of the sealers showed significant differences, the null hypothesis was rejected. However, the data obtained from this study could also be used to compare the compliance of the sealers with the values determined in the ISO standards.

Ideally, sealers with good flow and low surface tension can be easily applied and provide a well-adapted root canal filling. In this study, Apexit Plus, which is hydroxide and salicylate resin-based sealer, showed the highest flow with 27.66 mm. This result was different from the manufacturer's report, however, it was within the acceptable determined by ISO 6876:2012 for root canal filling pastes and is compatible with the literature.[16] The results of AH Plus, MTA Fillapex , and CeraSeal were compatible with other studies while this is the first report regarding the flow of NeoSealer Flo.[9,17,18]

The setting time depends on the moisture in the dentinal tubules besides the formulation since it has been shown that setting time increases in dry environments.[19] In our study, the longest setting time was observed in AH Plus, and the shortest setting time was observed in EndoREZ; both were compatible with ISO standards. Hydraulic calcium silicate sealers generally have a shorter setting time than pastes based on traditional formulations such as AH Plus.[20] In our study, although NeoSealer Flo showed setting time similar to AH Plus, Well-Root ST, Apexit Plus, and CeraSeal.

The most interesting finding regarding the setting time was that MTA Fillapex did not completely set. The setting of MTA Fillapex consists of two chemical reactions the hydration reaction of orthosilicate ions and the reaction between MTA and salicylate resin.[21] Although moisture was added to support the hydration reaction, the complete setting of the material could not be observed in this study. These results were supported by the previous studies complete settings according to the ISO standards were also found.[22-24] Inconsistent results regarding the setting time of MTA Fillapex should be investigated by further studies since longer setting times may increase the possibility of toxic by-products of root canal sealers passing into the periapical tissue and may adversely affect its biocompatibility.[22-25]

According to the author's knowledge, no study evaluated the film thickness of EndoREZ, NeoSealer Flo, and CeraSeal. The average film thickness of 7 different canal sealers were below the upper limit of film thickness (50 µm) determined by ISO 6876:2012.[15] In our study, Apexit Plus showed the least film thickness with the highest flow values, which is supported by a previous study that associates increased flow rate with decreased film thickness.[26]

ISO 6876:2012 states that the solubility of a set sealer should not exceed 3% of its mass after 24 hours in DW.[15] In the present study, only the solubility of AH Plus and EndoREZ sealers complied with ISO standards and were not affected by the environment. Immersion in PBS decreased the solubility of hydraulic calcium silicates, MTA-based, and calcium hydroxide/resin-based sealers. Previous studies reported that hydraulic calcium silicate sealers form a hydroxyapatite structure and precipitate on the surface in contact with phosphate-containing liquids.[22,27] Hydraulic calcium silicate sealers showed greater solubility in DW at two different time points as reported previously.[28,29]

Similar to solubility rates, hydraulic calcium silicate and calcium hydroxide/resin-based sealers showed a greater amount of water absorption. Small hydrophilic particles in the composition of these sealers are associated with high solubility and absorption potential since the small particles enable increased surface area and contact with liquid.[30,31] Calcium hydroxide formation as a result of the hydration reactions might also interfere with these results. Dissociation of calcium hydroxide into calcium and hydroxyl ions are well-known to increase solubility and liquid absorbance of these sealers.[32]

MTA Fillapex contains bismuth oxide as a radiopacifer, and its radiopacity value was determined as 3.9 mmAl, which was in accordance with previous studies.[25,33] Similarly, Apexit Plus, which showed significantly higher radiopacity values than MTA Fillapex , contains bismuth oxide. The reason for showing different opacity values with MTA Fillapex may be due to the differences in filler ratio.

The alkaline pH of sealers plays a role in the healing process. It can increase their antibacterial effect, neutralize the lactic acid produced by osteoclasts, and increase the accumulation of mineralized components.[34] Moisture in the dentinal tubules accelerates the hydration reactions of calcium silicate-based sealers that release $Ca(OH)_2$ and elevates the pH.[35] The present study observed alkaline pH values in hydraulic calcium silicate NeoSealer Flo and CeraSeal, while resin-based sealers showed nearly neutral pH values.

An increase in dimension is attributed to water absorption and solubility of the sealers and could be considered a disadvantage in terms of increasing the potential to induce cracks or fractures that may occur in the dentin; however, it is also desired to some degree for three-dimensional sealing of canal system.[11,15] According to the ISO standards, the average expansion rate should not exceed 0.1%, which is exceeded by all sealers in this study. Although these properties are very important for the characterization and clinical use of the sealers, further *in vitro* and *in vivo* studies are warranted to evaluate their biological properties and effect on the treatment outcome to determine their performance more comprehensively.

CONCLUSION

All canal sealers tested in the study complied with ISO 6876:2012 standards in terms of flow, film thickness, and radiopacity. Setting times of the sealers were also in accordance with ISO standards except for MTA Fillapex , which did not set completely. In addition, calcium silicate-based sealers show higher solubility and pH values in relation to $Ca(OH)_2$ formation while setting, tend to expand due to water absorption with the effect of $Ca(OH)_2$ formed, and dimensional change criteria are ISO (6876:2012) found to be inconsistent with standards.

Source of Finance

During this study, no financial or spiritual support was received neither from any pharmaceutical company that has a direct connection with the research subject, nor from a company that provides or produces medical instruments and materials which may negatively affect the evaluation process of this study.

Conflict of Interest

No conflicts of interest between the authors and / or family members of the scientific and medical committee members or members of the potential conflicts of interest, counseling, expertise, working conditions, share holding and similar situations in any firm.

Authorship Contributions

***Idea/Concept:** Hikmet Aydemir, Bahar Teker, Cangül Keskin; **Design:** Hikmet Aydemir, Bahar Teker, Cangül Keskin; **Control/Supervision:** Hikmet Aydemir, Bahar Teker, Cangül Keskin; **Data Collection and/or Processing:** Bahar Teker, Cangül Keskin, Hikmet Aydemir; **Analysis and/or Interpretation:** Bahar Teker, Cangül Keskin, Hikmet Aydemir; **Literature Review:** Hikmet Aydemir, Bahar Teker; **Writing the Article:** Cangül Keskin, Bahar Teker, Hikmet Aydemir; **Critical Review:** Hikmet Aydemir, Cangül Keskin; **References and Fundings:** Bahar Teker, Cangül Keskin, Hikmet Aydemir.*

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