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Solubility And Ph Of Five Endodontic Sealers – An In-Vitro Study

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Abstract
Aim: *The aim of this study was to measure the solubility and pH value of a comparably new tricalcium silicate–containing sealer (Endosequence BC ealer) and a MTA containing sealer (MTA Fillapex; Angelus, Londrina, Brazil) in comparison with a conventional epoxy resin–based root canal sealer (AH Plus;Dentsply DeTrey, Konstanz, Germany) , calcium -hidroksid based (Sealepex) zinkoksid -eugenol based (Tubliseal) during 24 ours in aqua ridestilata.*
Metodology: *In conformity with ISO6876:2012, the solubility test was performed. MTA –Fillapex, Edosequence BC sealer, Tubuliseal,AH-plus and Selapex were tested. Ring molds measuring 20 ± 0, 1 mm in internal diameter and 1, 5 ± 0, 1 mm in height were used to create similar specimens. Once the specimens were created, their masses were recorded digitally, both before and after they were immersed in double distilled water. Solubility was definited after 24 hours and statistically analyzed using the (Kruskal-Wallis ANOVA test). After 24 hours of manipulation, the pH value was determined using a digital pH meter.*
Results: *According to the Kruskal-Wallis ANOVA test, the difference between the average values of the five groups is significant for p<0.05 (Kruskal-Wallis test: H (4, N= 45) =39.55558 p =.0000). The significant difference according to the Kruskal-Wallis ANOVA test is mainly due to the statistically significant difference of the average solubility values between Ah+ versus MTA – Fillapex, Endosequenca and Selapex for p<0.05 (p=0.024223; 0.000000; 0.000539) and the significant difference of the mean values between Endosequenca versus Tubuliseal for p<0.05 (p=0.000313).*
Conclusion: *All of root canal sealers showed a weight loss of less than 3% and achieved the International Standard Organization 6876's standards for solubility except Endosequence and Selapex which demonstrated significantly higher solubility and higher pH. The finding of this study showed that resin-based sealers like AH-Plus are characterized by an acidic pH and low solubility.*
Keywords: *Solubility,root canal sealer, pH.*

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I. Introduction

There are multiple microscopic and macroscopic contacts between the root canal system, the periodontal ligament, and the surrounding bone in addition to the apical foramen, dentinal tubules, supplementary foramina, and lateral canals are examples of these structures.**(1)**

As a result, tissue fluid can easily access the root canal system, causing the sealer material to degrade, to avoid dissolution by bodily fluids in the root canal, root canal filler materials should be more or less insoluble **(2)**

Preventing the transfer of germs from coronal to apical or vice versa is the goal of a root canal filling, which creates a three-dimensional bacterial and fluid-tight closure across the root canal system**. (3, 4)**
To accomplish this goal, gutta-percha (GP) is combined with root sealers**. (5)**

The integrity of the sealer, not the core material, is what determines the root canal's long-term bacteria-proof seal**. (6, 7)**

Solubility is a fundamental characteristic of endodontic filling materials, because the dissolution of the endodontic sealers can greatly affect the success of the endodontic treatment**. (8)**

But how the solubility changes over an extended length of time is still unknown**.** Undoubtedly, long-term low solubility is crucial for the clinical outcome of root canal obturation**.(9)**

After endodontic therapy, the presence of microorganisms in the root canal has been associated to post treatment disease**. (10)**

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Due to their ability to create acids like lactic acid and lower the pH of their surroundings, bacteria are able to survive. Sealer's alkalinity may reduce this ideal environment, which will stop bacteria from developing. The pH of sealer directly affects its antibacterial action**. (11)**

The International Standard 6876 has established low solubility of a root canal sealer as a necessary condition**. (12).**This standard and ANSI/ADA Specifications No. 57. **(13)** specify that sealer's solubility should not pass 3% mass fraction during a 24-hour immersion in water.

The aim of this study was to measure the solubility and pH value of a comparably new tricalcium silicate–containing sealer (Endosequence BC ealer) and a MTA containing sealer (MTA Fillapex; Angelus, Londrina, Brazil) in comparison with a conventional epoxy resin–based root canal sealer (AH Plus;Dentsply DeTrey, Konstanz, Germany) , calcium -hidroksid based (Sealepex) zinkoksid -eugenol based (Tubliseal) during 24 ours in aqua ridestilata.

The null hypothesis tested was that there is no significant difference in solubility and pH percentages between the five different endodontic sealers.

II. Materials And Methods

Five different root canal sealers were examined.

Material used:

- Endosequence BC sealer (Brasseler , Savannah, GA,USA),
- MTA Fillapex (Angelus, Londrina, PR, Brazil)
- AH Plus sealer (Dentsply International INC, York, PA)
- Selapex (**1232799** | Kerr Corporation – 18432, Made in the U.S.A.)
- Tubuliseal (Kerr Corpoation 1717 West Collins Ave. Orange,CA 92867,USA)

MATERIAL	COMPOSITION
Endosequence BC Sealer	Zirconium Oxide, Calcium silicates, Calcium phosphate monobasic, Calcium hydroxide, Filler and thickening agents
MTA Fillapex	Salicylate resin, Diluting resin, Natural resin, Bismuth Trioxide, Nanoparticulated silica, MTA, Pigments
AH Plus sealer	Epoxide paste: Diepoxide, calcium tungstate, zirconium oxide, aerosil, pigment Amine paste: 1- adamantane amine, N,N'-dibenzyl-5-oxa nonandiamine-1,9, TCD-Diamine, calcium tungstate, zirconium oxide, aerosil, silicone oil
Selapex	**Base paste**: Calcium oxide,Bismuth trioxide,Zinc oxide,Sub-micron silica,Zinc stearate,Titanium dioxide, and Tricalcium phosphate **Catalyst paste**: Ethyl toluene sulfonamide,Poly (methylene methyl salicylate) resin, and Isobutyl salicylate.
Tubuliseal	Tubli-Seal™ is a paste/paste zinc oxide eugenol root canal sealant. It is light in colour, non-darkening and radiopaque.(1 x Tubli-Seal Base1 x Tubli-Seal Accelerator)

Solubility

According to International Standards Organization (ISO) 6876, solubility was verified.

After putting the samples in double-distilled water, the solubility has been determined based on the findings achieved. The weight variation of the specimens was noted. Stainless steel ring molds with an interior diameter of 20.0 mm (~0.1 mm) and a height of 1.6 mm (~0.1 mm) have been used for all sample preparation. For fifteen minutes, all molds were cleaned in an ultrasonic bath using acetone. Prior to usage, each mold was precisely weighed three times (to within 0.0001 g), and the average was determined.

Materials that did not require water for setting were mixed according to the manufacturer's instructions and placed in stainless steel molds and then in glass dishes and placed in to the cabinet for a period of time 50% longer than the fixing time declared by the manufacturer.

Materials that required moisture to setting, 2 g of material was mixed according to the manufacturer's instructions with 0.02 ml and the molds were filled and placed in glass plates, in our experiment Selapex and Endosequence were the materials that needed water for setting. All the sampels were then placed in the cabinet for 24 hours at 37˚C and >95% relative humidity.Then the mass of the sealer was determined to 0.001 g.After setting of the sealers, 50 samples were prepared for immersion in aqua ridestilata .The samples were put into a Petri dish with 50 ml of ridestilate water, and they were then kept in the same incubator for twenty-four hours at

37˚C and more than 95% relative humidity. After incubation time, the samples were rinsed with 3 mL of distilled water and the washings were allowed to drain back into the Petri dish. The samples were then discarded, and the Petri dishes were dried in an oven at 105 ˚C for 48 hours, reweighted after cooling in the same desiccator. To the closest 0.0001 g, the weight difference between the material's initial and final weights was recorded.

Assessment of pH

Based on the solubility test's experimental setup, a parallel pH measurement was conducted.

An electrode pH meter was used to calculate the pH value (ISO LAB Laborgerate GmbH).Prior to every pH measurement, calibration solutions were used to regulate the pH meter's accuracy.(BUFFER SOLUTION pH 7.00 +/- 0.02@25 C,and BUFFER SOLUTION Ph 4,00 +/-0.02@25C)

In order to prevent contaminating the next fluid, the electrode was also cleaned with AD after each individual measurement. To replicate the environment inside the oral cavity, the measurement was performed on all samples at a fluid temperature of 34°C.

III. Results

50 samples participate in the study, every tenth of which is a control.

Divided into five groups in relation to the Endodontic Sealers (MTA – Fillapex, Endosequenca, Tubuliseal, Ah+ and Selapex).

The initial (initial) weight is the same for all samples and is 2.8242, and for the control samples it is 0.8242. In control samples, the difference between initial and final weight is zero.

The average value of the final weight at 24 hours of solubility is the highest for Ah+ (Endodontic sealer) and is 2.82±0.003, in the range from 2.81 to 2.82 (table and graph 1a). The average value of the difference between the initial and final weight is low and amounts to 0.006±0.03 (table 1b).

The average value of the final weight for Tubuliseal (Endodontic sealer) is 2.77±0.021, in the range from 2.71 to 2.77 (table and graph 1a). The average value of the difference between the initial and final weight is 0.056±0.012 (table 1b).

The average value of the final weight for MTA - Fillapex (Endodontic sealer) is 2.74±0.012, ranging from 2.75 to 2.79 (table and graph 1a). The average value of the difference between the initial and final weight is 0.081±0.021 (table 1b).

The average value of the final weight of Selapex (Endodontic sealer) is 2.72±0.004, in the range from 2.71 to 2.73 (table and graph 1a). The average value of the difference between the initial and final weight is 0.1±0.004 (table 1b).

The average value of the final weight of Endosequenca (Endodontic sealer) is the lowest and is 2.63±0.036, in the range from 2.58 to 2.69 (table and graph 1a) The average value of the difference between the initial and final weight is the highest and is 0.197±0.036 (table 1b).

According to the Kruskal-Wallis ANOVA test, the difference between the average values of the five groups is significant for $p<0.05$ (Kruskal-Wallis test: H (4, N= 45) =39.55558 p =.0000).

The significant difference according to the Kruskal-Wallis ANOVA test is mainly due to the statistically significant difference of the average solubility values between Ah+ versus MTA – Fillapex, Endosequenca and Selapex for $p<0.05$ ($p=0.024223$; 0.000000; 0.000539) and the significant difference of the mean values between Endosequenca versus Tubuliseal for $p<0.05$ ($p=0.000313$) (table 1c).

Table (1a) Presentation of the average final weight after 24 hours of solubility in all samples in the five groups

Endodontic sealer/ Final weight	Average	N	Std. Dev	Minimum	Maximum
MTA - Fillapex	2.74	9	0.021153	2.707000	2.768200
Endosequenca	2.63	9	0.036394	2.585600	2.686200
Tubuliseal	2.77	9	0.012552	2.754600	2.792300
Ah+	2.82	9	0.003543	2.810100	2.822100
Selapex	2.72	9	0.004510	2.715500	2.729200

Graph 1a

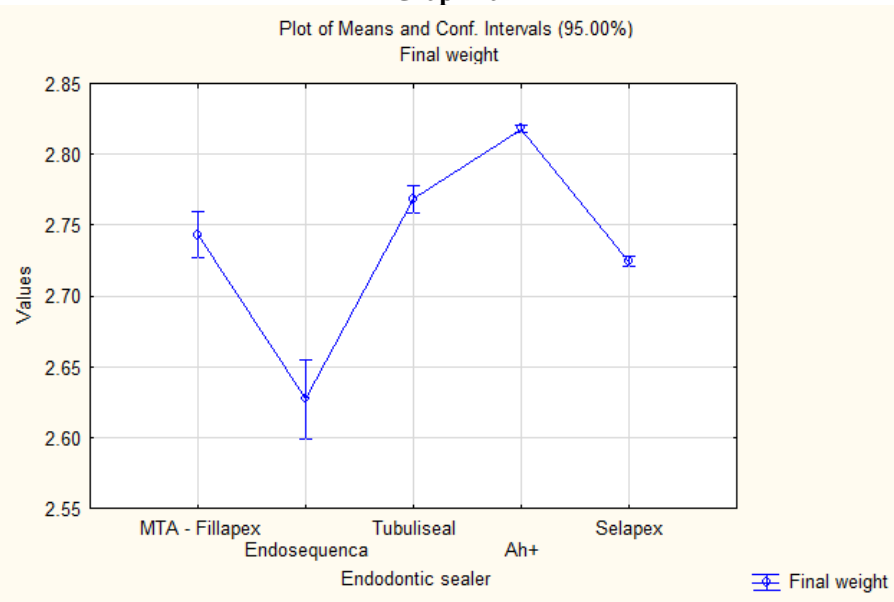


Table (1b) Presentation of the average difference (difference) between the initial and final weight in all samples in the five groups.

Endodontic sealer/ difference	Average	N	Std. Dev	Minimum	Maksimum
MTA - Fillapex	0.081	9	0.021153	0.056000	0.117200
Endosequenca	0.197	9	0.036394	0.138000	0.238600
Tubuliseal	0.056	9	0.012552	0.031900	0.069600
Ah+	0.006	9	0.003543	0.002100	0.014100
Selapex	0.1	9	0.004510	0.095000	0.108700

Table 1b

Multiple Comparisons p values (2-tailed); Final weight Independent (grouping) variable: Endodontic sealer Kruskal-Wallis test: H (4, N= 45) =39.55558 p =.0000					
	MTA - Fillapex R:22.222	**Endosequenca R:5.0000**	**Tubuliseal R:30.778**	**Ah+ R:41.000**	**Selapex R:16.000**
MTA - Fillapex		0.054085	1.000000	0.024223	1.000000
Endosequenca	0.054085		0.000313	0.000000	0.756242
Tubuliseal	1.000000	0.000313		0.987308	0.169945
Ah+	0.024223	0.000000	0.987308		0.000539
Selapex	1.000000	0.756242	0.169945	0.000539	

In the control samples in all five groups, the Ph value is 7, ie. is neutral

The average value of Ph 24 hours for Ah+ is 6.53±0.18, in the range from 6.35 to 6.84 ie. the environment is acidic (table and graph 2, picture 1).

The average value of Ph 24 hours for Tubuliseal is 6.62±0.22, in the range from 6.24 to 6.99 ie. the environment is acidic (table and graph 2, picture 1).

The average value of Ph 24 hours for MTA - Fillapex is 8.56±0.29, in the range from 8.19 to 8.98 ie. the environment is basic (alkaline) (table and graph 2, figure 1).

The average value of Ph 24 hours for Endosequenca is 9.45±0.34, in the range from 8.99 to 9.84 ie. the environment is basic (alkaline) (table and graph 2, picture 1).

The average value of Ph 24 hours for Selapex is 10.79 ±0.11, in the range from 10.68 to 10.98 ie. the environment is basic (alkaline) (table and graph 2, picture 1).

Table (2a) Display of the average Ph values 24 hours of the five groups

Endodontic sealer	Avergae	N	Std. Dev	Minimum	Maksimum
MTA - Fillapex	8.56	9	0.288579	8.19000	8.98000
Endosequenca	9.45	9	0.336208	8.99000	9.84000
Tubuliseal	6.62	9	0.223632	6.24000	6.99000
Ah+	6.53	9	0.181621	6.35000	6.84000
Selapex	10.79	9	0.111853	10.68000	10.98000

Graph 2

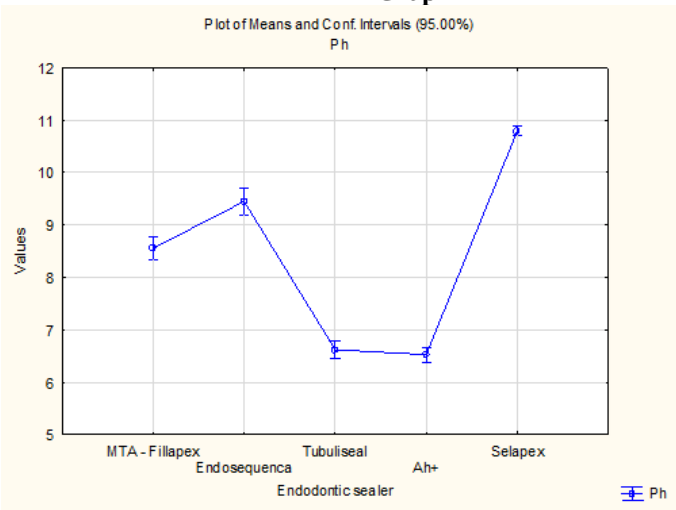
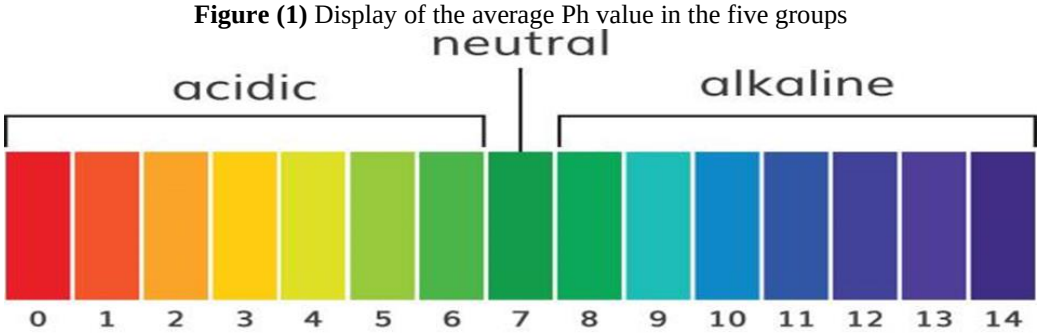


Figure (1) Display of the average Ph value in the five groups



IV. Discussion

To prevent pathogens and irritants from leaking into the periradicular tissues from the root canal system, root canal sealers must create an apical seal**. (14)**

Numerous factors, including as the media's pH, the setting time, the surrounding environment, the chemical composition of the material, and interaction with tissue fluid, may affect the solubility**. (15)**

When determining if a substance is suitable to be used as restorative material in dentistry, one of the most crucial factors to consider is its solubility. It has also been suggested that the lack of solubility is an important characteristic for root-end filling materials**. (16)**

According to the International Standards Organization 6876 standard or ANSI/ADA Specification No. 57, the solubility of the root canal sealers must not be greater than 3% mass fraction following a 24-hour immersion in water.

In this investigation, the null hypothesis was rejected. It has been demonstrated that the solubility percentages of the root canal sealers tested varies significantly.

Much researches proving the biocompatibility and bioactivity of calcium silicate-based root canal sealers has contributed to their great biological appeal, which has resulted in their increasing popularity in the field of endodontics as well .Not neglecting the biological benefits, some researchers have reported that calcium silicate-based root canal sealers exhibit significant solubility, which can greatly affect the overall quality of the obturation**.(8)**

The results of this study showed that Endosequence BC Sealer and Selapex had a significantly greater solubility, with a weight loss greater than 3%, in comparison with other tested endodontic sealers which were within the suggested range (Ah-plus,Tubuliseal,Mta-Fillapex).

The average value of the final weight of the Endosequence is the lowest because it was the sealer that dissolved the most during the 24-hour stand in redistilled water. This result of the high solubility of this sealer is

also in agreement with the study done by the author **Borges et al,** who suggested that the high solubility of this sealer was due to hydrophilic nanoparticles, which increase their surface area and let more liquid molecules to interact with the sealer**. (17).**

Also, the results achieved for Selapex were not in accordance with the recommendations of ANSI/ADA (2000), similar results were also achieved by the author **Borges et al (17)** in his stady which demonstrated that Sealapex had solubility values higher than the ANSI/ADA limit. Sealapex was also thought to have great solubility, according to **Eldeniz et al**. **(18),** even so the argument was made because of the substance's high pH and potent calcium ion release.

Furthermore, higher solubility levels might also be explained by the unstable setting of this sealers, according to earlier laboratory investigations such an endodntic sealer Edosequence BC sealer can take a long time to set, even do not set after long experimental times, even in the presence of humidity**.(19),(20).**

The final results of this study showed that the average of the final weight was higher for Ah-plus, because it was the sealer that was least dissolved during the stay in redistilled water for 24 hours.This result for Ah-plus is also in agreement with the results achieved in the studies carried out earlier by the authors Schäfer **& Zandbiglari 2003 (6), Versiani MA et al 2006 (21) Garrido AD et al 2010 (22).**

The endodontic seaers' alkaline pH may enhance their capacity for osteogenesis, biocompatibility, and antimicrobial activity**. (23, 24, 25).**

In this stady calcium-hidroksid based selaer Selapex pH (10.79) and bioceramic- based selaer Endosequence pH (9.45) showed higher alkalinity,following by Mta-based selaer Mta-Fillapex with pH(8.56) and with acidic pH zinkoksid-based sealer Tubuliseal pH(6.62) and epoxy-resine based AH-plus with pH(6.53).

It has been suggested that root canal sealers with an alkaline pH may neutralize the lactic acid produced by osteoclasts and stop the disintegration of the tooth's mineralized components. Therefore, by stimulating alkaline phosphatase, root canal sealers may help in the development of hard tissue**. (26)**

The bioceramic-based sealers (Edosequence) demonstrated high alkaline pH following the testing period. The high pH for Endosequence after all test periods was also confirmed by the author **Zhou et al.(2**7). While the lowest pH was for Ah-puls, according to recent studies, poor solubility and a relatively neutral pH are characteristics of resin-based sealers like AH- Plus concluded also author **Marciano et al** in his stady **(28).** After a long testing period Ah-Plus was not alkaline founded by the author **Kent Urban et al. (2).**

V. Conclusion

All of root canal sealers showed a weight loss of less than 3% and achieved the International Standard Organization 6876's standards for solubility except Endosequence and Selapex wich demonstrated significantly higher solubility and higher pH, the finding of this stady showed that resin-based sealers like AH -Plus are characterized by an acidic pH and low solubility.

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