Release of hydroxyl ions from calcium hydroxide preparations used in endodontic treatment

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Abstract

Purpose: The purpose of our study was to compare the in vitro release of hydroxyl ions from several calcium hydroxide preparations used in endodontic treatment.

Material and methods: Equal quantities of the materials – nonsetting (pure calcium hydroxide, Biopulp, Calcicure), setting – canal sealers (Sealapex, Apexit) and points were placed in dialisis tubes which were then immersed in deionized water. The release of hydroxyl ions from the preparations was measured by the median pH of the deionized water used for dialisis, by means of a pH-meter. The results of our study were analized by means of Tukey's resoneable correlation. Significance difference (one-way variance analysis ANOVA) and Pearson's linear correlation coefficient (r^2).

Results: Nonsetting preparations of calcium hydroxide have a significantly higher capability of hydroxyl ions release in comparison with sealers and points, irrespective of time ($p<0.05$). Sealapex and "plus" points released hydroxyl ions to a much greater extent than both Apexit and "regular" points at most periods of the experiment ($p<0.05$). Apexit released significantly more of hydroxyl ions than "regular" points, and Sealapex more than "plus" points in the later periods of the experiment ($p<0.05$). The pH values of dialysis samples of all materials correlated positively with time and the pH. Almost all materials reached a maximum on the 8-th day of the experiment.

Conclusions: To achieve maximum concentration of hydroxyl ions in tissues: • for temporary root fillings nonsetting preparations of calcium hydroxide should be chosen rather than points and they should be placed for at least one week, • for permanent root fillings it is more recommended to use Sealapex than Apexit as a sealer.

Key words: calcium hydroxide, release of hydroxyl ions.

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Introduction

Calcium hydroxide is widely used in dentistry and especially in endodontics as a dressing in periapical tissue inflammation, in cases of tooth root fracture, mechanical or inflammatory perforations as well as in the process of apexification [1-4]. The therapeutic effectiveness of calcium hydroxide dressing materials is based on the release of hydroxyl ions causing an increase in pH [5]. This alkaline pH promotes the action of alkaline phosphatase, a vital enzyme in the calcification, which requires an optimal pH of 10.2 [5,6].

The calcium hydroxide preparations used as dressing materials for temporary or final root canal filling are available as either setting, or nonsetting compounds and points and may release hydroxyl ions in varying degrees thus exerting different therapeutic effects. From the clinical point of view, it is therefore reasonable to measure the release of hydroxyl ions from different types of calcium hydroxide preparations available. It is also important to know whether the pH of these preparations changes with time.

The aim of our study was firstly to compare the in vitro release of hydroxyl ions from the various calcium hydroxide preparations used in endodontic treatment and secondly to determine whether their pH altered with time.

Material and methods

The calcium hydroxide preparations used in our study are summarized in *Tab. 1.* The methodology provided by Economides et al. was used in the study [7].

Table 1. Calcium hydroxide preparations used in present study.

Group	Product	Code	Manufacturer	Batch number	Composition
1 - nonsetting preparations	"pure" calcium hydroxide	PCH	Dental Therapeutics AB/Gillevagen, Sweden	510200	The ready – made paste consisting of pure calcium hydroxide and destilled water q.s.
	Biopulp *	BP	Chema Elektromet/ /Rzeszów, Poland	010201	Calcium hydroxide, bibasic calcium phosphate, magnesium oxide, sodium chloride, potassium chloride, dehydrated sodium carbonate
	Calcicure #	CR	VOCO/Cuxhaven, Germany	10946/M/97	Radiopaque water – based calcium hydroxide paste
2 - setting preparations	Apexit	AT	Vivadent Ets./Schaan, Liechtenstein	533281AN	Calcium hydroxide, hydrogenized calaphony, silicon dioxide, paraffin oil, calcium oxide, zinc oxide, calcium phosphate, polidimethylosiloxane, alkyl ester of phosphorid acid, trimethylohexanediosalicylate
	Sealapex	SX	Kerr Italia S.p.A/ /Salerno, Italy	68432	Calcium oxide, barium sulphate, zinc oxide, sub-micron silica, titanium dioxide, zinc stearate
3 - calcium hydroxide points	"Regular"	RCHP	Roeko GmbH/ /Langenau, Germany	367097	Calcium hydroxide, guttapercha, colouring agent
	"Plus"	PCHP	Roeko GmbH/ /Langenau, Germany	366896	Calcium hydroxide, guttapercha, sodium chloride, surfactant, colouring agent

* Biopulp was mixed with destilled water (pH 7.5) (Polpharma SA/Starogard Gdański, Poland, 031199) in proportion 0.07 g/0.03 g, respectively

\# The manufacturer does not give details about the composition

The materials of groups 1 and 2 were prepared according to the manufacturers' instructions. Equal quantities (0,1g) of the substance were placed in dialysis tubes (Sigma Aldrich Chemie GmbH, Steinheim, Germany, batch number D9277) which were then placed in deionized water of 6.6 pH. Four samples of each of the test materials were prepared.

The release of hydroxyl ions from each preparation was determined by measuring the pH value of the deionized water used for dialysis by means of a pH-meter model 5170 (ELWRO, Częstochowa, Poland). The pH value was measured directly after placing the samples in the water and after 30, 60, 90, 120 minutes and on the 2-nd, 8-th and 15-th day. The materials were kept in an incubator model Cultura (Almedica AG, Galmiz, Swiss) at a temperature of 37°C throughout the period of observation.

The results of our studies were analyzed by means of Tukey's reasonable significant difference (one-way variance analysis ANOVA) and Pearson's linear correlation coefficient (r^2). Statistical analysis was performed on the basis of computer programm Statistica 5.0 PL.

Results

The results obtained in our study are presented in *Fig. 1* and *Tab. 2,* and *3*. The figure shows that the greatest increase in pH occurred in the first 30 minutes of experiment. The pH of the samples increased gradually reaching maximum values on day 8 of the experiment and then decreased over the next seven days. A further slight increase in pH was only observed in the calcium hydroxide "plus" points and in "pure" calcium hydroxide.

As it can be seen in *Tab. 2*, a positive correlation between pH and time was found for all materials in this experiment – the highest for "pure" calcium hydroxide and the "plus" points (r^2=0.72) and the lowest for "regular" points (r^2=0.18). A positive correlation was observed between the pH values of particular samples – the highest for Biopulp, a nonsetting paste, and "pure" calcium hydroxide (r^2=0.99) and the lowest for "plus" and "regular" points (r^2=0.74).

The correlation between the pH values of almost all the test materials and time (with the exception of "regular" points) and the correlation between pH values in particular samples was statistically significant ($p<0.05$).

Tab. 3 shows that nonsetting calcium hydroxide preparations (group 1) presented the highest initial alkaline pH, ranging from 9.93±0.06 to 12.24±0.01 for "pure" calcium hydroxide, from 10.10±0.08 to 12.08±0.05 for Biopulp and from 10.39±0.20 to 12.09±0.04 for Calcicure. These differences were statistically significant with regard to Calcicure and "pure" calcium hydroxide, Calcicure and Biopulp after 30, 60, 120 minutes and also on the second day of the experiment ($p<0.05$).

Canal sealers (group 2) and calcium hydroxide points (group 3) presented lower values of pH (*Tab. 3*).

In group 2, Sealapex pH – from 7.89±0.03 to 11.77±0.05

Figure 1. Correlation between pH and time with regard to particular materials.

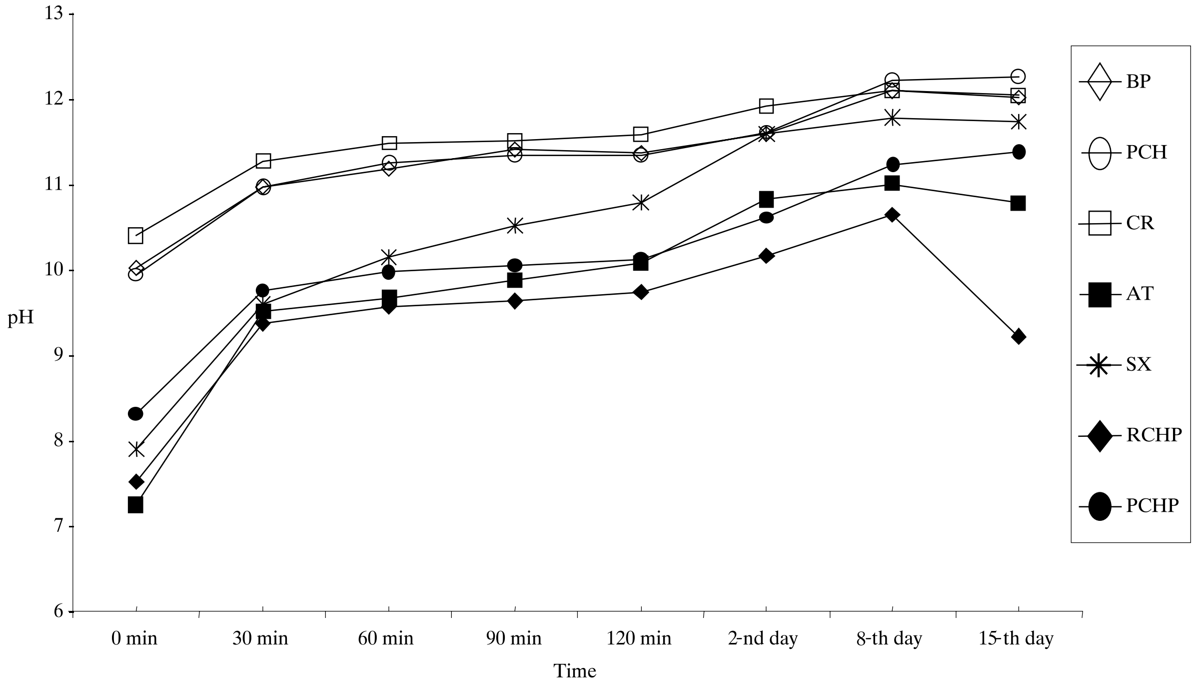

Table 2. Correlation between the pH values of particular materials and the time, and correlation between preparations in the whole period of the experiment.

	Time	BP	PCH	CR	AT	SX	RCHP	PCHP
Time	–	◊	■	◊	◊	◊	○	■
BP	◊	–	◄	◄	◄	◄	■	◄
PCH	■	◄	–	◄	◄	◄	■	◄
CR	◊	◄	◄	–	◄	◄	■	◄
AT	◊	◄	◄	◄	–	◄	■	◄
SX	◊	◄	◄	◄	◄	–	■	◄
RCHP	○	■	■	■	■	■	–	■
PCHP	■	◄	◄	◄	◄	◄	■	–

◄ – almost complete correlation $0.9 < r^2 < 1$; ■ – very high correlation $0.7 < r^2 < 0.9$; ◊ – high correlation $0.5 < r^2 < 0.7$;
○ – low correlation $0.1 < r^2 < 0.3$

– was higher than that for Apexit – from 7.24±0.12 to 10.99±0.21 – and these differences were statistically significant at almost all periods of the experiment (except at 30 minutes) ($p<0.05$).

In group 3, alkaline pH ranged from 7.51±0.43 to 10.66±0.07 for "regular" points and from 8.30±0.43 to 11.22±0.22 for "plus" points and these differences were statistically significant in all periods of the experiment ($p<0.05$).

Moreover, significantly higher values of pH were observed in group 1 in comparison with group 2 and 3 at almost all periods of the experiment (except the 2-nd and the 15-th day) ($p<0.05$).

Among the materials of group 2 and 3, the pH of Sealpex was higher than the pH of both types of points after 60, 90, 120 minutes, and also in the 2-nd and 8-th day of the experiment. The pH of Apexit was higher than the pH of "regular" points only in the last five periods of the experiment. The differences were statistically significant ($p<0.05$).

Discussion

The therapeutic effect of calcium hydroxide is due to its breakdown into calcium and hydroxyl ions. Hydroxyl ions show an affinity to various biologically active substances [8]. Their antimicrobal activity is important since microbes cause the majority of endodontic diseases [9].

The antibacterial activity of hydroxyl ions is related to the formation of a potent alkaline medium leading to the destruction of lipids, the main component of bacterial cell membrane, and their causing structural damage to bacterial proteins and nucleic acids [8]. Numerous publications support the theory regarding bactericidal activity of calcium hydroxide [10-14].

The results of our in vitro studies, indicating that nonsetting calcium hydroxide preparations (group 1) release hydroxyl ions most easily, are consistent with other authors' findings [1,3,7,9,15,16]. The pH values obtained in our study proved

Table 3. Mean pH values of calcium hydroxide preparations analyzed during the experiment.

Preparations	Time							
	0 min	30 min	60 min	90 min	120 min	2-nd day	8-th day	15-th day
BP (a)	10.01 ±0.08	10.96 ± 0.01	11.17 ± 0.04	11.40 ± 0.05	11.36 ±0.02	11.58 ± 0.00	12.08 ±0.05	12.00 ±0.09
PCH (b)	9.93 ±0.06	10.96 ±0.03	11.24 ± 0.05	11.33 ± 0.03	11.33 ± 0.09	11.60 ±0.01	12.21 ±0.04	12.24 ± 0.01
CR (c)	10.39 ±0.20	11.25 ±0.04	11.47 ± 0.02	11.50 ± 0.03	11.56 ± 0.02	11.90 ± 0.02	12.09 ± 0.04	12.03 ± 0.01
AT (d)	7.24 ±0.12	9.50 ±0.03	9.66 ±0.03	9.87 ± 0.03	10.06 ± 0.06	10.81 ± 0.07	10.99 ± 0.21	10.77 ± 0.31
SX (e)	7.89 ±0.03	9.58 ±0.06	10.14 ± 0.08	10.50 ± 0.04	10.77 ± 0.02	11.58 ± 0.09	11.77 ± 0.05	11.72 ±0.04
RCHP (f)	7.51 ± 0.43	9.36 ± 0.13	9.56 ±0.10	9.62 ± 0.12	9.73 ±0.10	10.15 ± 0.07	10.63 ± 0.07	9.21 ± 0.96
PCHP (g)	8.30 ± 0.43	9.74 ± 0.13	9.96 ±0.9	10.04 ± 0.10	10.11 ± 0.11	10.60 ± 0.11	11.22 ± 0.02	11.37 ± 0.13
The differences statistically significant for p<0.05 between particular preparations	a,b,c – d,e,f,g d – e,g f – g	a,b – c,d,e,f,g c – d,e,f,g d,f – g e – f	a,b – d,e,f,g c – d,e,f,g d – e,g e – f,g f – g	a – d,e,f,g b – c,d,e,f,g d – e,f,g e – f,g f – g	a,b – c,d,e,f,g c – d,e,f,g d – e,f e – f,g f – g	a,b – c,d,f,g c – d,e,f,g d – e,f,g e – f,g f – g	a,b,c – d,e,f,g d – e,f,g e – f,g f – g	a,b,c – d,f d – e,f e – f f – g

to be significantly higher for Calcicure than for the rest of the materials in group 1 after 30, 60 and 120 minutes as well as on the second day of the experiment ($p<0.05$). Beltes et al. [1] also mention a high alkaline potential of Calcicure. Canal sealers (group 2) and points (group 3) were characterized by much weaker capability of releasing hydroxyl ions. As for sealers, a higher alkaline pH was found for Sealapex in comparison with Apexit and these differences were statistically significant at almost all periods of the experiment, except for 30 minutes ($p<0.05$). This was despite a higher percentage of calcium hydroxide in Apexit (31.9%) than in Sealapex (24%). Staehle et al. [16] presented similar findings when examining root dentine alkalization produced by applying various calcium hydroxide materials, particularly Sealapex, in root canals. However, they observed that application of Apexit did not cause an increase in the pH of hard tissue. Gordon and Alexander [17] also indicated a greater ability of Sealapex to release hydroxyl ions, in comparison with other calcium hydroxide sealers.

Among calcium hydroxide points, the pH of "regular" points rose from an initial 7.51±0.43 at the beginning of the experiment to 10.63±0.07 after 8 days. In the second week, the pH of the samples decreased rapidly to the level of 9.21±0.96. Our results differ from the results obtained by Economides et al. [7], who observed that the maximum pH of "regular" points was 9,5. The above divergence could be caused by the different times of the pH measurements.

"Plus" points were characterized by a significantly higher release of hydroxyl ions in comparison with "regular " points. Their pH increased from the first to the last day of the experiment and ranged from 8.30±0.43 to 11.37±0.13. We could not find any data on calcium hydroxide "plus" points found in the literature, which could be compared to the results of our studies. These materials are characterized by a lower percentage quantity of calcium hydroxide (52% of $Ca(OH)_2$) in comparison with "regular " points (58% of $Ca(OH)_2$), but additionally, they contain sodium chloride and surfactant a surface active agent which improves the solubility of points and facilitates the release of ions.

This proves that ion accessibility is connected with the addition of substances that inhibit or initiate the release of ions, not with the calcium hydroxide content in preparations. This conclusion is also shared by other authors [9,16-18].

Time seems to be a vital factor in relation to the effective therapeutic action of calcium hydroxide preparations. In the present studies, the pH of all the dialysis samples increased gradually with time reaching the highest values on the 8-th day of the experiment. An exception was the "plus" points in which the pH reached the highest value after 2 weeks. A positive correlation between the pH values of the dialysis samples and time was found for all materials. This corresponds with other authors findings [5,17].

The low solubility and poor ability to diffuse make it difficult for compounds based on calcium hydroxide [8] to reach maximum pH levels in a short period of time. The results of our in vitro studies show that they should be applied to canals for a minimum of 7 days to achieve maximum therapeutic effectiveness.

In conclusion, present in vitro experiment suggest that, to achive maximum concentration of hydroxyl ions in tissues two issues should be contemplated: 1) for temporary root fillings nonsetting preparations of calcium hydroxide should be chosen rather than points and they should be placed in situ for at last one week, 2) for permanent root fillings it is more recommended to use Sealapex than Apexit as a sealer.

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