



## Comparison the PH of roots after intra canal placement of four types of calcium hydroxide (An in vitro study)

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### Abstract

The successful outcome of endodontic treatment depends on the reduction or elimination of bacteria present in an endodontic infection. It has been reported that chemo-mechanical instrumentation alone is not enough for this purpose.

The purpose of this in vitro study was to compare the pH changes that occurred over a period of 14 days using four types of calcium hydroxide. Four types of calcium hydroxide materials were filled in fifty extracted single rooted teeth which were kept in individual vials containing distilled water at a pH of (7.4). Digital pH-meter was used to measure the pH of different groups at two hours, 24 hours, 7 days & 14 days after immersion of the specimens. The mean pH was found for all groups and statistical analysis was carried out using ANOVA and POST HOC TEST (LSD).

Calcium hydroxide was found to significantly raise the root PH. Therefore calcium hydroxide is an effective choice in clinical practice to be used as intra canal medicament between appointments.

**Keywords: Calcium hydroxide, Intracanal medicaments, pH.**

### Introduction

Dentists have been using calcium-based chemicals in clinical practice for over a century. Calcium hydroxide was introduced to endodontics by Herman in 1920. It is a strong alkaline substance, which has a pH of approximately (12.5). In an aqueous solution, calcium hydroxide dissociates into calcium and hydroxyl ion <sup>(1)</sup>. Various biological properties have been attributed

to this substance, such as antimicrobial activity,<sup>(1)</sup> inhibition of tooth resorption<sup>(2)</sup> and induction of repair by hard tissue formation, because of such effects, calcium hydroxide has been recommended for use in several clinical situations.<sup>(3)</sup> Most

of the endodontic pathogens are unable to survive in the highly alkaline environment provided by calcium hydroxide.<sup>(4)</sup> Antimicrobial activity of calcium hydroxide is related to the release of hydroxyl ions in an aqueous environment. Hydroxyl ions are highly oxidant free radicals that show extreme reactivity with several biomolecules.<sup>(5)</sup>

The aim of this present in vitro study was to compare the pH changes following intracanal placement of four types of calcium hydroxide materials, Conventional Calcium Hydroxide Paste, 95% pure calcium hydroxide powder (KGA, Darmstadt, Germany), two Commercial Ca(OH)<sub>2</sub> Pastes (Calxyde and Endocal) and calcium

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hydroxide plus iodoform paste over different time intervals ,2 hours,24 hours,7 days and 14 days. Thereby, to evaluate which of these products has a better alkalinizing potential over time to be used as an inter appointment intra canal dressing.

## Materials and Methods

Fifty extracted single rooted teeth were collected for the study. The soft tissue remnants on the tooth were removed with a soft brush. The teeth were examined for fractures, cracks, or any other defects. All the teeth were stored in freezer in distilled water until sufficient number of samples was collected. Access cavities were created using diamond burs in air turbine hand piece using round bur and round end tapering diamond burs. The canals were explored using No10 K file, (Dentsply / Maillefer). Teeth with two canals or calcified canals were excluded from the study.

Following this, working length was established at 1 mm from apical foramen. Root Canal Preparation was performed by a step back technique using K-flex files; apically to a master apical file size # 35 and cervically flared sufficiently with step back technique up to size # 60 K-flex file. Throughout the entire cleaning and shaping procedure, a total of 10 ml of 1.5% sodium

Hypochlorite solution was used for irrigation of root canal. At termination of instrumentation, canals were flushed with 3ml of 16% EDTA solution for 3 minutes followed by 1.5% of NaOCl solution to remove the smear layer and finally rinsed with 5 ml of distilled water.

Between each stage of study, the teeth were stored in air tight containers in incubator at 100% humidity and 37°C for 3 days<sup>6</sup>.

## Placement of intra canal dressing

Teeth were randomly divided into 5 groups of 10 teeth each for placement of different calcium

Hydroxide formulations as intra canal dressing.

**Group I:** Conventional Calcium Hydroxide Paste, 95% pure calcium hydroxide powder (KGA, Darmstadt, Germany) was taken, measured with analytical balance and fractional weight box to 50 milligrams. This powder was mixed on a clean sterile glass slab with distilled water to a P/L weight ratio of 50% using a cement spatula. The teeth were dried with absorbent paper points and dressing was placed in the canals using lentulo spirals (Dentsply/Maillefer, Ballaigues, Switzerland) and then condensed with finger pluggers (Dentsply/ Maillefer). For each specimen new sterile instruments were used. A cotton pellet was placed in the pulp chamber, and sealed by intermediate restoration.

**Group II:** First type Commercial Calcium Hydroxide Paste Calxyde (Spofadental-Spain) was used. After drying the canals, small amount of paste was placed into the canal with the syringe delivery system provided by manufacturer according to manufacturer instructions and complete filling of canal was done using lentulo spirals and then condensed with finger pluggers. A moist cotton pellet was placed over it and sealed by intermediate restoration.

**Group III:** Second type Commercial Calcium Hydroxide Paste Endocal (Septodent Ltd –France) was used. After drying the canals, small amount of paste was placed into the canal with the syringe delivery system provided by manufacturer according to manufacturer instructions and complete filling of

canal was done using lentulo spirals and then condensed with finger pluggers. A cotton pellet was placed over it and access cavity was sealed with intermediate restoration.

**Group IV:** Calcium Hydroxide past plus iodoform (Dia Dent corporation-Korea) was used. After drying the canals, small amount of paste was placed into the canal with the syringe delivery system provided by manufacturer according to manufacturer's instructions and complete filling of canal was done using lentulo spirals and then condensed with finger pluggers. A cotton pellet was placed over it and access cavity was sealed with intermediate restoration.

**Group V:** Control group

Canals were dried, a cotton pellet was placed in the chamber and access cavities were sealed with intermediate restoration.

Following intracanal placement of different calcium hydroxide products all the samples were radiographed in BL & MD direction by using intra oral digital X-ray machine (Owandy-France) exposure time 0.05 s, 63 KV, 8 mA, , the digital sensor was fixed by rubber base on the dental chair so that not moved during taking radiograph ,the samples radiographed in BL direction by fixing the tooth in BL direction within a transparent nylon tube that placed over the surface of sensor and fixed by rubber base.

Then the samples radiographed in MD direction in the same way as described BL direction.

Distance between end of the cone and the sample was 2cm fig(1). Two specialists (blinded to the groups) evaluated the adaptation of calcium hydroxide to canals walls.

To exclude the intra-calibration errors two readings was made for each specialist with interval of three days. Any sample that showed voids or incomplete adaptation to the walls was excluded from the study and replaced by another one.

### **Storage and recording the reading**

After preparation of all specimens, they were stored individually in glass vials containing 3 ml of deionized water as a storage medium fig (2). The diffusion of ions and recording of PH was done for this medium. PH readings were then recorded using calibrated digital pH meter (Caberscan 510-Eutech -Singapore) fig (3). Measurements were done at different time intervals namely 2 hours, 24 hours, 7days and 14 days. Each vial was shaken properly and electrode rinsed with double distilled water was inserted to record the pH value. The reading was allowed to stabilize and recorded. The procedure was repeated for each sample and for each time interval. In between each stage the vials were stored in incubator.

### **Statistical Analysis**

Data were collected and analyzed using mean value and standard deviation. Differences between groups were examined by ANOVA; post hoc test (LSD) was used to compare between means of two each specific group.

P-values of 5% and more were regarded as statistically insignificant, whereas values less than 5% ( $p < 0.05$ ) were considered as significant and those values less than 1% ( $p < 0.01$ ) were considered as highly significant.

### **Results**

Tables (1-4) list the mean and standard deviations of the groups. The pH values indicated that the materials tested in Groups 1, 2, 3 and 4 produced a high alkalinity score. The results of the established that the pH changes were determined by the composition of the materials tested and the time interval at which the test was conducted. After 2 hrs, the pH of calcium hydroxide/distilled water, calxyde, Endocal and Ca(OH)<sub>2</sub> plus iodoform had experienced a rapid increase with no significant difference, the alkalinity continued to increase; also after 24 hr, 7 days and 14 days there was no statistically significant difference between the four experimental groups. Table (5-8) lists POST HOC TEST (LSD) which was used to compare between means of two each specific group, no statistically significant differences were established between four groups at different time intervals.

The followings are the mean of PH changes of five groups at different time intervals:

**Group I: calcium hydroxide + Distilled water**

It showed a mean pH of 10.31 after 2 hours, 11.38 after 24 hours, 11.51 after 7 days and 11.62 at 14th day.

**Group II: First type Commercial Calcium hydroxide paste (Calxyde)**

This group showed a mean pH of 9.67 at 2 hours, 10.74 at 24 hours, 11.77 at 7th day and 11.91 at 14th day interval.

**Group III: Second type Commercial calcium hydroxide Paste (Endocal)**

The mean pH observed at different time intervals was: 9.85at 2hours, 10.76 after 24 hours, 11.78 at 7th day and 12.27 after 14 days.

**Group IV: Calcium hydroxide past plus iodoform**

The mean pH observed at different time intervals was: 9.52 at 2hours,

10.66 after 24 hours, 11.74 at 7th day and 12.05 after 14 days .

**Group V: Control group**

It showed minimum changes in pH over time and readings recorded were: 7.47at 2 hours, 7.42 after 24 hours, 7.39 at 7th day and 7.26 after 14 days.

**Discussion**

The importance of the alkalinizing effects of calcium hydroxide products used as intracanal dressings and their capacity to produce hydroxyl ions in the periapical environment has been extensively reported <sup>(6,7)</sup>. It has been demonstrated that the action of inflammatory and clastic cells is enhanced by an acidic pH, leading to disintegration and subsequent resorption of hard tissues <sup>(8)</sup>. The action of calcium hydroxide that is the hydroxyl ions are diffusing through the dentinal tubules and the apical foramen, with the latter being the most effective <sup>(9)</sup>. The alkalinity of the periapical tissues that can be achieved by the use of calcium hydroxide compounds and the method of diffusion of hydroxyl ions through the apical foramen is therefore worthy of investigation.<sup>9</sup> The dressed teeth were immersed in 3ml of deionized water, the quantity of the distilled water in the vials was just the amount sufficient to record pH changes. The design of this study provided a simple method for reproducible measurements of pH changes, thus allowing a comparison between different calcium hydroxide materials.

In the present in vitro experiment, four observations were made over a 14-days period to determine the release of hydroxyl ions from four calcium hydroxide preparations through apices. The results of Table (1) demonstrated that the paste of calcium hydroxide/distilled water as well

as two Commercial Ca(OH)<sub>2</sub> Pastes (Calxyde and Endocal) and Ca(OH)<sub>2</sub> plus iodoform had good alkalinizing properties .

This suggests that hydroxyl ions were able to diffuse into the distilled water. The fast alkalinizing effects of these materials after 2 and 24 hrs was because of the immediate contact of the calcium hydroxide with the water, resulting in an instantaneous release of hydroxyl ions. The calcium hydroxide/distilled water released hydroxyl ions more rapidly over a 2 hr time period, whereas after 24 hrs no difference with Commercial Ca (OH)<sub>2</sub> Pastes (Calxyde and Endocal) and Ca(OH)<sub>2</sub> plus iodoform was observed. The faster releases of hydroxyl ions within the 2-hrs period may be inherent properties of calcium hydroxide powder/distilled water formulations <sup>10</sup> or it may be because of the much higher percentage of calcium hydroxide (powder/water weight percent ratio of 50%). For instance, Commercial Ca (OH)<sub>2</sub> Paste Calxyde is composed of calcium hydroxide (41.07%), barium sulfate (8.33%) and other ingredients in a sterile isotonic saline solution. The formulation of Commercial Ca(OH)<sub>2</sub> Paste Endocal is calcium hydroxide (52.20%) and a contrast- enhancing material in a saturated aqueous solution. while calcium hydroxide paste plus iodoform is composed of calcium hydroxide 38.0% ,silicone oil and others.<sup>11</sup> One may speculate that the slower PH increase from two Commercial Ca(OH)<sub>2</sub> Pastes (Calxyde and Endocal) and Ca(OH)<sub>2</sub> plus iodoform was caused by a reduced availability of calcium hydroxide because of the presence of additional components in these formulations, until over time a saturation level had been reached .<sup>11</sup> After 7 days the alkalinizing effects of Commercial Ca(OH)<sub>2</sub> Pastes and Ca(OH)<sub>2</sub> plus iodoform increased gradually, owing to

a slow but steady release of hydroxyl ions, whereas the calcium hydroxide/distilled water mixture appeared to have reached its maximum output .<sup>12</sup> The differences in PH recorded for Ca(OH)<sub>2</sub> (Calxyde and Endocal) Pastes and Ca(OH)<sub>2</sub> plus iodoform may be due to different concentrations of calcium hydroxide in three pastes. At the end of the experiment after 14 days, Ca(OH)<sub>2</sub> Paste Endocal showed the highest alkalinity; however, no statistical significance could be demonstrated between Ca(OH)<sub>2</sub> (Calxyde and Endocal) Pastes and Ca(OH)<sub>2</sub> plus iodoform whereas all of these materials had reached a statistically significant higher pH than the calcium hydroxide/distilled water mixture. The alkalinizing effects recorded in this experiment need to be viewed in the context of other published research. The vials containing the test materials were kept tightly sealed with rubber caps and only during the pH measurements were the materials exposed to aerobic conditions.

The results of Simon et al., differ from results in current study , the difference may be due to the experimental design The PH values recorded in this experiment were lower than what has been reported by Simon et al. <sup>(13)</sup>. They placed different calcium hydroxide materials in glass tube with an inner diameter of the glass tubes of 1.0 mm; the experimental design closely resembled a clinical situation of immature teeth or cases with apical root resorption. Therefore, it was no surprise that the diffusion of hydroxyl ions through this fairly large opening was much higher than for mature teeth <sup>(13,14)</sup> in which the diameter of the prepared apical area was much smaller .At the end of the experiment reported., According to Fuss et al. <sup>(15)</sup>, the efficacy of calcium hydroxide compounds may be affected by



chemical alteration when exposed to ambient air that contains CO<sub>2</sub>, which will produce an insoluble compound calcium carbonate.

A reduction in PH levels was reported after carbonation of calcium hydroxide-based

Materials<sup>(15)</sup>. In a clinical situation, CO<sub>2</sub> may also originate from metabolism of microorganisms within the canal or dentinal tubules and from the surrounding tissues<sup>(16,17,18)</sup>, a phenomenon that cannot occur under the experimental conditions of this study.

The PH level for the calcium hydroxide/distilled water mixture, Calxyde and Endocal and calcium hydroxide plus iodoform was at level that most bacteria cannot grow. The sustained alkalinizing effects of the tested materials are of clinical significance. Nerwich et al<sup>(19)</sup> measured PH changes in root dentin over a 2-week period and considered this a reasonable time interval to expect effective therapeutic benefits from calcium hydroxide-based materials as intra canal medicament.

## Conclusion

Calcium hydroxide was found to significantly raise the root PH. The calcium hydroxide/ distilled water released hydroxyl ions more rapidly over a 1 hr time period, whereas after 24 hrs no difference with Commercial Ca(OH)<sub>2</sub> Pastes (Calxyde and Endocal) and Ca(OH)<sub>2</sub> plus iodoform was observed. Therefore calcium hydroxide is an effective choice in clinical practice to be used as intra canal medicament between appointments.

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Table (1): The PH means of different groups after 2 hours.

Groups	N	Mean	SD
1	10	10.296	.201
2	10	9.674	.151
3	10	9.854	.295
4	10	9.524	.194
5	10	7.479	.157

Table (2): The PH means of different groups after 24 hours.

Groups	N	Mean	SD
1	10	11.384	.231
2	10	10.744	.173
3	10	10.760	.170
4	10	10.663	.207
5	10	7.429	.137

Table (3): The PH means of different groups after 7 days.

Groups	N	Mean	SD
1	10	11.516	.177
2	10	11.776	.119
3	10	11.780	.102
4	10	11.741	.171
5	10	7.392	.109

Table (4): The PH means of different groups after 14 days.

Groups	N	Mean	SD
1	10	11.621	.178
2	10	11.911	.382
3	10	12.173	.334
4	10	12.058	.136
5	10	7.265	.079

Table(5) : Comparison between groups after 2 hours.

Time intervals	Comparison between groups	P-value	Sig.	
2 HRS	1	2	0.440	N
		3	.0.658	N
		4	0.499	N
		5	0.0379	S
	2	3	0.595	N
		4	0.170	N
		5	0.828	N
	3	4	0.052	N
		5	0.0632	N
	4	5	0.0159	S

Table(6) : Comparison between groups after 24 hours.

Time intervals	Comparison between groups	P-value	Sig.	
24 HRS	1	2	0.950	N
		3	0.222	N
		4	0.342	N
		5	0.522	N
	2	3	0.860	N
		4	0.380	N
		5	0.460	N
	3	4	0.159	N
		5	0.165	N
	4	5	0.380	N

Table(7) : Comparison between groups after 7 days.

Time intervals	Comparison between groups	P-value	Sig.	
7 days	1	2	0.832	N
		3	0.845	N
		4	0.826	N
		5	0.287	N
	2	3	0.825	N
		4	0.769	N
		5	0.257	N
	3	4	0.825	N
		5	0.249	N
	4	5	0.267	N

Table(8) : Comparison between groups after 14 days.

Time intervals	Comparison between groups	P-value	Sig.	
14 days	1	2	0.154	N
		3	0.143	N
		4	0.328	N
		5	0.002	HS
	2	3	0.392	N
		4	0.284	N
		5	0.001	HS
	3	4	0.213	N
		5	0.002	HS
	4	5	0.002	HS



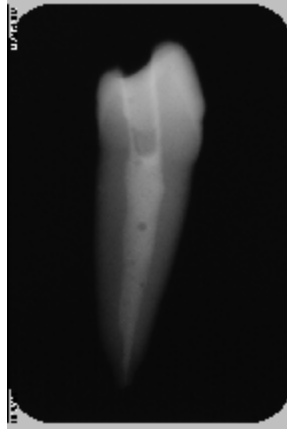


Fig.(1) : Radiograph show calcium hydroxide paste filled in the canal.



Fig (2): Sample stored in deionized water.



Fig (3):Calibrated digital PH meter used in this study.