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Solubility of three different pulp capping materials: a comparative study

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Aim: The aim of the study was to evaluate the solubility of new pulpotomy materials; Pulpine mineral (PMIN) and Pulpine NE (PNE) in comparison to Mineral Trioxide Aggregate (MTA).
Materials and Methods: 9 split Teflon ring molds of a height of 1.5 mm and an internal diameter of 7.75 mm were performed, specimens were randomly distributed into three equal groups (PMIN, PNE and MTA), the specimens with molds were immersed in 100 ml separate sealed cups, filled with 60 ml deionized water and stored in the incubator for 24 h and 1 week. Solubility was recorded by the percentage of material mass loss over time.
Results: There was a statistically significant difference between each group, the highest value of solubility test was recorded to Pulpine mineral group, followed by pulpine NE while the least solubility value was recorded to MTA.
Conclusion:MTA revealed the best solubility value among different groups

Keywords: Mineral trioxide aggregate, Pulpine mineral, Pulpine NE, solubility

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Introduction

Solubility is a critical item when assessing the adaptability of restorative dental materials. Decreased solubility is a necessary property for tooth repair materials (1), a long-term sealing ability is a crucial characteristic of dental materials in the oral or periapical tissue. Thus, according to the (ISO) standards 6876:2001, solubility value shouldn't exceed 3% in distilled water. calcium silicate particles can combine with phosphate-containing body fluid, creating hydroxyapatite crystals according to previously published researches (2), (3).

Recently, Hoffman's Pulpine Mineral and Pulpine NE products are introduced as vital pulp therapy medicaments, having the same liquid (propolis and ethanol) but differ in their powder, PMIN is consisting of (Hydroxyapatite, Calcium compounds) but PNE is composed of (Calcium compounds, Zinc compounds). Manufacturers claimed that they can be an alternative to MTA in vital pulp therapy by means of their biocompatibility, antibacterial effect, easy application, quick setting time, and very good adhesion. Our study can be considered the preliminary study to investigate the solubility test related to those materials.

Flavonoids are the most essential active composition in Propolis, which have anti-inflammatory, antibacterial, antifungal, antioxidant and antiviral properties, Studies on Propolis have increased because of its biological and therapeutic characteristics (4) Hydroxyapatite crystals presented in the powder of pulpine mineral material have biocompatibility, bio-inductivity, antibacterial property, osteogenicity, and the capability to stimulate hard tissue formation (5).

Materials and Methods

The solubility test was estimated in accordance with previous investigations (6)(7), following the American National Standard Institute/American Dental Association (ANSI/ADA) Specification No. 57.

Specimens of MTA (n=3), Pulpine Mineral (PMIN) (n=3), Pulpine NE (PNE) (n=3) were established utilizing split Teflon ring molds (1.5 mm height ± 0.1 mm and 7.75 mm internal diameter ± 0.1 mm). The molds were filled with the prepared material which was adopted on a glass slide. Another glass slide was placed over the specimens exerting light pressure to get rid of excess material, then the materials were left to set for three times greater than the initial setting time determined by the manufacturers, setting time of MTA (10-15) min, PMIN (1-3:30) min, and PNE (1-3:45) min. A high precision scale with 0.0001-g accuracy (Precision electrical weighing balance, Sartorius MAX 220 g, Germany) was used to record the net weight of each specimen with mold (W0). Then, nylon thread was stuck to the internal side of the cup cover and to the metal part of the mold to hang the specimens and not to touch the wall, thereafter, the specimens with molds were immersed in 100 ml separate sealed cups, filled with 60 ml deionized water, and stored in the incubator for 24 hour and 1 week (37 C, 95% relative humidity), after that, specimens were slightly dried with absorbent papers, and placed in a desiccator containing silica for 24hrs.

Then, specimens with molds were reweighed to the nearest 0.001 (W1), specimens were unmolded and the weight of the molds only was measured to get the accurate weight of the specimens without molds (W2), The percentage of material mass loss over time was used to determine the solubility. The

percentage of solubility for each sample was calculated as follows, with a little change to a previously published article (10)

% Wight loss = (W0-W2)-(W1-W2)/(W0-W2) × 100.

Statistical test:

Following the normality check, the ANOVA test was used to compare the different groups, followed by the Post Hoc test to establish the significance. For intragroup comparison, Repeated ANOVA was employed.

Results

Solubility was measured as a percentage weight loss, then tabulated and graphically displayed in table (1).

I- Regarding different materials:

At 24 hours, the PMIN group had the largest value (-2.5), followed by PNE (-2.4) while MTA had the least value (-1.2) with no statistically significant difference between PMIN and PNE but a statistically significant difference (P=0.027) between MTA and the other groups.

At one week, the largest value was recorded to the PMIN group (-12.8), followed by PNE (3.4), while the least mean percentage value was showed in the MTA group (-1.6) with a statistically significant difference between the three groups (P<0.000) utilizing ANOVA test.

II-Regarding different time interval:

Within the MTA group, there was a slight increase in the mean weight percentage loss from 24hrs to one week (-1.2 to -1.6) respectively, with no statistically significant difference P=0.093, while in the PMIN group there was a high increase in the mean weight percentage loss from 24hrs to one week (-2.5 to -12.8) respectively with a significant difference P<0.001, and in PNE group there was an increase from 24hrs to one week (-2.4 to-3.4) with no statistically significant difference P=0.301 using dependent samples t-test.

Table (1) showing mean (SD) values of solubility test among different groups at different time interval.

Time	MTA mean (SD)	PMIN mean (SD)	PNE mean (SD)	P.value
After 24hrs	-1.2 ^{Aa} (0.251)	-2.5 ^{Ab} (0.305)	-2.4 ^{Ab} (0.556)	0.027*
After one week	-1.6 ^{Aa} (0.200)	-12.8 ^{Bb} (0.200)	-3.4 ^{Ac} (0.556)	0.000*
P.value	0.093	0.001*	0.301	

Means with different capital superscript letters in the same column are statistically significant different, the different small letters between each row are statistically significant difference (P<0.05).

Discussion

Solubility is the percent loss of weight after setting, immersion in water, and should not exceed 3%, according to ANSI/ADA Standardization 57 (2000). A slight modification was achieved by previous studies (6)·(7) which revealed comparable results with decreased material volume, they used a smaller volume of tested materials, but got satisfactory results. Thus, we utilized a split teflon mold with lesser dimensions than ANSI/ADA Standards 57, to support the materials during the experiment, particularly with PMIN, as it became crumbly when it undergoes stress during mold removal, resulting in cracks, followed by disintegration of the sample, as this material might have low cohesive strength.

Our present study demonstrated that at 24 hrs the least value of solubility among the three studied materials was MTA followed by PNE and PMIN with no distinct difference, however at one week, PMIN showed a high increase in solubility (12.8%) that greatly exceed the ANSI/ADA recommendation, followed by pulpine NE (3.4%), and MTA demonstrated the best value which was (1.6%) at 7 days.

MTA solubility (1.6%) is similar to various studies (7), (8), (9) and can be explained by leaching of calcium and hydroxide ions at the initial setting (10).

which is a key factor for successful vital pulp therapy because of the role of calcium on pulp cell differentiation, proliferation and calcific tissue formation (11), resulting in an increase in pH in the medium.

However, on the other hand, **Gandolfi et al** (12)**,** discovered a higher level of MTA solubility, which they attributed to the use of newly mixed MTA, which is susceptible to washout and causes early cement disintegration when in contact with tissue fluid or blood (13),(14).

PMIN showed a significant increase in solubility in comparison to MTA and PNE at one week (12.8%), The integration of propolis into the hydroxyapatite powder (HA) induces a decrease in the agglomeration degree of the particles due to its spherical form, according to **Scatolini et al**. (15). Another study indicated that adding 10% or 20% HAP to IRM resulted in disintegration of these materials in bovine serum and in buffered phosphate after 8 weeks (1). In addition, the fast evaporation of the ethanol liquid during manipulation, resulted in the inadequacy of the mix.

Furthermore, our high solubility results of PMIN can also be related to the chemical composition of propolis. Propolis phenolic compounds are acidic groups that are vulnerable to deprotonation in a slightly alkaline medium, creating phenoxide ions and raising solubility in an aqueous solution. (16). Hesperidin which is the most essential flavonoids located in propolis, demonstrated a large increase in solubility of the pure powder in distilled water after 28 days, as the existence of hydroxyl rings combine with those of water (17).

PNE showed decreased solubility in comparison to PMIN (3.4% at one week) which was slightly exceed the maximum recommendation, and that might be contributed to the addition of zinc compound in its powder, **Tanomaru et al** (18) , revealed the hydration reaction of MTA with ZnO particles, resulted in a material with low solubility, other researches proved that the addition of ZnO affect the solubility (19),(20).

Conclusion

MTA has the best solubility value among the different groups, further researches of physical and biological properties are required to emphasize the whole characteristics of these novel materials.

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