

## Evaluation of an experimental zinc phosphate cement powder

Safwat EM<sup>1</sup>, Saniour SH<sup>2</sup>, Zaki DY<sup>1</sup>, El-Batran MM<sup>3</sup>, Mousa IM<sup>2</sup>

<sup>1</sup>Restorative and Dental Material Research Department, National Research Centre. Cairo, Egypt

<sup>2</sup>Biomaterials Department. Faculty of Oral and Dental Medicine. Cairo University.

<sup>3</sup>Basic Dental Science Department. National Research Centre. Cairo, Egypt.

**Corresponding author:** Engie\_safwat@hotmail.com

**Abstract:** The aim of this study was to evaluate the properties of an experimentally prepared zinc phosphate cement powder. The working time, setting time, film thickness, compressive strength and solubility were tested for the experimental cement powder and compared with one of the commercially available zinc phosphate cement. Testing was done according to the ANSI/ADA specification No. (8) for zinc phosphate cement and No. (96) for dental water-based cements. Results revealed that the experimental cement produced working time, setting time, film thickness and solubility comparable with that specified by the ADA specification No. (8) and (96), and with that of the commercial cement, however the compressive strength (42.09 MPa) was significantly lower than that specified by the ADA No.(96) (70 MPa) but was not significantly different than that of the commercial cement (49.6 MPa).

[Safwat EM, Saniour SH, Zaki DY, El-Batran MM, Mousa IM. **Evaluation of an experimental zinc phosphate cement powder.** Journal of American Science 2011;7(1):264-268]. (ISSN: 1545-1003). <http://www.jofamericanscience.org>.

**Key words:** zinc phosphate cement, ANSI/ADA specification No.(8) and No.(96), working time, setting time, film thickness, compressive strength, solubility, disintegration.

### 1. Introduction

A number of materials are available for cementation, luting and bonding purposes in dentistry. Zinc phosphate cement has been used for the longest period compared with other cement types.

Dental zinc phosphate cement is supplied in the form of powder and liquid. The typical formulation of zinc phosphate cement powder is zinc oxide (90.2%wt), magnesium oxide (8.2%wt), silicon dioxide (1.4%wt), bismuth trioxide (0.1%wt) and other minimum ingredients e.g. BaO<sub>2</sub>, Ba<sub>2</sub>SO<sub>4</sub> and Ca O (0.1%wt). However, the liquid is free phosphoric acid (38.2%wt), phosphoric acid combined with aluminium and zinc (16.2%wt), aluminium (2.5%wt), zinc (7.1%wt) and water (36.0%wt). Craig et al.,(2004);(2006). The set zinc phosphate cement is essentially a hydrated amorphous network of zinc phosphate that surrounds incompletely dissolved particles of zinc oxide. Although no crystalline phosphate is involved in the setting process of the cement, there can be subsequent growth of crystalline hopeite, (Zn<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. 4H<sub>2</sub>O) in the presence of excess moisture during setting, Craig et al.,(2006).

The initial hardening of zinc phosphate cements normally occur within 4-7 minutes, although the strength continues to increase for some time after that. The ADA specification No.(96) American Standards Institute (1977;1994) carries no requirement for the working time, so the results of the working time test of the commercial cement were taken as reference to the experimental cement. However, the ADA

specification No.(96) stated that the net setting time is 2.5 minute minimum and 8 minutes maximum. Therefore, the total setting time (working time + net setting time) would be 7.5 minutes minimum and 13 minutes maximum, Walls and McCabe(1998).

The desired consistency of the zinc phosphate cement mixture depends on the particular purpose of the material and the working convenience needed, as expressed by the setting time, where the inlay seating require maximum film thickness of 25µm i.e, light consistency, American Standards Institute (1994). However, the cement base should have a heavier consistency to be used as a thermal and chemical insulating barrier over thin dentin Craig et al.,(2004).

The ADA specification No.(96) stated that the compressive strength of zinc phosphate cement should be 70Mpa minimum and that the disintegration percentage should be 0.1 % maximum, American Standards Institute(1994)This study was designed to compare the properties of an experimentally prepared zinc phosphate cement powder with a commercially available one, and to evaluate their properties according to the ANSI/ADA specification No. (8) for zinc phosphate cement and No. (96) for dental water-based cements.

### 2. Materials and methods

#### 2.1. Materials:

##### 2.1.1. Cement powder:

One of the commercially available zinc phosphate cement powder (Alpha-dent, Dental technologies manufacturer, USA) was selected to

compare its properties with that of the experimental cement powder.

An experimental zinc phosphate cement powder was prepared according to Safwat et al (2007) The chemical composition of the experimental cement powder is listed in table (1).

**Table (1): The chemical composition of the experimental cement powder.**

Zinc oxide	Magnesium oxide	Aluminum oxide	Silica	Bilebovine	Borax
91.66 %	2.2%	3.31%	0.126%	1.43 %	0.9%

## **2.2.Methods:**

### **2.2.1.Sample preparation:**

Preparation of the commercial cement samples was done according to the manufacturer instructions, using liquid to powder ratio (L/P) of 3:1. On the other hand the experimental cement samples were prepared according to Safwat et al (2007) using L/P ratio of 3:1.

### **2.2.2. Evaluation of the physical and mechanical properties:**

#### **2.2.2.1. Evaluation of working time:**

A teflon ring mold approximately 4.8mm high and 9.5mm internal diameter was used to prepare the cement specimens. An indenter of 28gm weight, having a needle with flat end of 2mm diameter was used. The tip of the indenter was cylindrical for approximately 5mm, plane and perpendicular to the long axis of the needle. A holding ring was also used to insure vertical and perpendicular loading of the indenter. The working time was measured as the time elapsed from beginning of mixing till the needle no longer penetrates the surface. The test was repeated five times; the mean and standard deviation (S.D) of both the experimental and the commercial zinc phosphate cement were calculated.

#### **2.2.2.2.Evaluation of setting time:**

The same teflon ring mold used for the working time evaluation was used to prepare specimens for the setting time testing. An indenter of 400gm weight, having a needle with flat end of 1mm diameter was used. The needle tip was also cylindrical for approximately 5mm, plane and perpendicular to the long axis of the needle and a holding ring was used to insure vertical and perpendicular loading of the indenter. Three and half minutes after starting of mixing, the indenter was

carefully lowered vertically through the holding ring onto the surface of the cement in the ring mold, left for five seconds under its own weight then a trial run was carried out. This procedure was repeated at 30 seconds intervals until the needle failed to make a complete circular indentation on the surface of the specimen. The setting time was recorded as the time elapsed from the start of mixing till the needle failed to make a complete circular indentation on the surface of the specimen. The setting time was recorded to the nearest minute and the test was repeated five times. The mean and standard deviation for both the experimental and the commercial zinc phosphate cement were calculated.

#### **2.2.2.3.Evaluation of film thickness**

The thicknesses of two flat square glass plates of 5mm uniform thickness and 45mm length, having contact surface area of 200mm<sup>2</sup> were measured while stacked in contact using a digital micrometer accurate to the nearest 1.25mm. The cement mixtures were then mixed and placed between the two plates. Ten seconds before the previously determined working time, a loading device with a force of 15Kg was applied vertically and centrally on the upper glass slab for ten minutes. The thicknesses of the two plates were measured with the mixed cement in-between. The film thickness was calculated as the difference between the two measurements. The test was repeated five times; the mean and standard deviation for both the experimental and the commercial zinc phosphate cement were calculated.

#### **2.2.2.4.Evaluation of solubility and disintegration:**

Two empty graduated glass beakers were thoroughly dried and weighed. Four cylindrical specimen, 20mm diameter and 1.5mm thick, were prepared using a metal mold, three minutes after mixing. specimens were stored for 1 hour in a relative humidity of 100% at 37°C. The specimens were then submerged in the two beakers containing 50ml-distilled water each for 23 hours at 37°C then removed from the beakers, leaving behind their remnants in the distilled water. Distilled water was then completely evaporated at temperature just below its boiling point i.e., approximately 90°C, leaving only the remnants of the cement in the beakers. The two beakers were then reweighed and the amount of disintegration was calculated as the difference between the two weight measurements. The weight gained by the beakers divided the original weight of the specimens' multiplied by 100 gives the percentage of disintegration reported to the nearest 0.1%.

**2.2.2.5. Evaluation of compressive strength:**

A split teflon circular mold 6mm in height and 4mm in diameter with a holding ring to hold the mold plates together was used. The largest convenience portion of the cement is applied to one side of the mould 60 minutes after the end of mixing and filled in excess to consolidate the cement and avoid trapping air; another glass slab was applied over the mold to obtain a smooth surface. Five specimens were prepared then removed from the mould after one hour and immediately immersed in distilled water at 37°C for 23 hours. Twenty-four hours after the end of mixing, the prepared samples were individually and vertically mounted on a computer controlled universal testing machine (model LRX-plus; Lloyd instruments Ltd., Fareham, UK) with a load cell of 5KN and a crosshead speed of 0.75mm/min. The load at failure was recorded and the compressive strength values in MPa were calculated for five replicas; the mean and standard deviation for both the experimental and the commercial zinc phosphate cement were calculated.

**2.2.3. Statistical analysis:**

The mean and standard deviation were recorded and statistically analyzed using ANOVA test to compare the ranks of the levels of the experimental and commercial groups. SPSS for windows software, release 15.0 (SPSS, Chicago, IL) was used.

**3. Results****3.1. Results of working time, setting time and film thickness:**

Results of working time, setting time and film thickness are shown in table (2). Regarding the results of working time, no statistical significant difference could be detected between the experimental cement and the commercial cement (5 and 4 minutes respectively) ( $P=0.4$ ). Results also indicated that both the experimental and the commercial cement have the same setting time (7.5 min), which meets that specified by the ADA specification (96). American Standards Institute (1994). As regards the film thickness, both the experimental and the commercial cements have the same film thickness (25 $\mu$ m) which also meets that specified by the ADA specification (96), American Standards Institute (1994).

**3.2. Results of solubility and disintegration:**

Results of solubility are shown in table (3). The experimental cement gave a higher disintegration percentage than the commercial cement however, when reported to the nearest 0.1% as stated in the ADA specification No (8), the two results were equal (0.1%).

Table (2): The mean and standard deviation (S.D) of the working time, setting time and film thickness of the commercial and experimental cement.

<b>Properties</b> <b>Mixture type</b>	<b>L/P ratio</b> <b>(ml/gm)</b>	<b>Mean working</b> <b>time(in</b> <b>minutes)</b>	<b>Mean setting</b> <b>time (in</b> <b>minutes)</b>	<b>Mean film</b> <b>thickness (in</b> <b><math>\mu</math>m)</b>
Commercial cement	3:1	5 $\pm$ 1	7.5 $\pm$ 0.5	25 $\pm$ 0.5
Experimental cement	3:1	4 $\pm$ 0.5	7.5 $\pm$ 0.2	25 $\pm$ 0.1

Table (3): The mean disintegration % of the commercial and experimental cement

<b>Table (3): The mean</b> <b>disintegration % of the</b> <b>commercial and</b> <b>experimental cement</b>  <b>Mix type</b>	<b>L/P ratio</b> <b>(in ml/gm)</b>	<b>Disintegration %</b>
Commercial cement	3:1	0.099% 0.1%
Experimental cement	3:1	0.126% 0.1%

### 3.3. Results of compressive strength

Results of compressive strength are shown in table (4). Results revealed that the experimental cement gave a lower compressive strength (42.09 MPa) compared with that specified by the ADA

specification No. (96) (70 MPa), however there was no statistical significant difference between the experimental and the commercial zinc phosphate cement (49.6 MPa).

**Table (4): The mean and standard deviation of the compressive strength of the commercial and experimental zinc phosphate cements.**

Mix type	L/P ratio (in ml/gm)	Compressive strength (in MPa)
Commercial cement	3:1	49.6 ±1.2
Experimental cement	3:1	42.09 ±2.2

### 4. Discussion

Dental zinc phosphate cements use a special form of zinc oxide that has been deactivated by mixing with magnesium oxide. Silica and alumina are added to reinforce the set cement, followed by being sintered together at temperatures between 1000°C and 1400°C then grounded to a fine powder John (2002). According to Safwat et al,(2007) several additives were tried to justify the fluidity, the working and setting time. The additives used were carefully selected in the light of the published literature on similar materials, their availability at local markets and their cheapness. Although, these additives should improve some properties, yet they should not alter or destroy other properties. The bile bovine additive is a yellowish powder obtained after grinding of ox-gall stones. It is used as a wetting agent in marbling paper industry. The bile bovine alters the surface tension of the cement allowing it to spread out over the surfaces. The bile bovine additive promotes the penetration of the particle clumps with water because part of the bile salts present in its composition, is soluble in water Wolfe (1990). The addition of 1.43% bile bovine and 0.9% borax improved working time and setting time of zinc phosphate cement. The added borax influenced the rate of cement hydration resulting in reduction in the setting time and increasing the early strength development. Moreover it disperses cement particles; thus increasing the flow of the experimental cement resembling that of the commercial cement. This may be also due to its adsorption on the particles surfaces leading to a mutual repulsion of individual particles and reduction in interparticles friction, Zaki et al.,(2006). Consequently the combination of borax and bile bovine increase the setting time allowing more particles in the mixture to react.

The mean compressive strength of the commercial cement and experimental cement (49.6

MPa), (42.09 MPa) respectively, did not reach the ADA specification limits (70 MPa). This may be due to the porosity in the set mass resulted from human errors during samples preparation. This result is in agreement with Goto et al (1999) where 25% of the mixed cement had strength values below 40 MPa. They emphasized that the ideal mixing conditions of the cement components are seldom achieved.

From the foregoing discussion, it can be concluded that, the prepared experimental cement properties simulated that of the commercial cement and met the specification requirements except for the compressive strength. Further investigations are required to improve the properties of the experimental cements to reach that of the commercial cement as regards compressive strength.

### 5. References

1. ANSI-ADA specification No (96). Water based dental cement. American National Standard Institute, Revised 1994.
2. ANSI-ADA specification No (8). Zinc phosphate cement. American National Standard Institute, Revised 1977.
3. Craig RG, Powers JM, Wataha JC. Dental materials: properties and manipulation. 8th ed. St. Louis: Mosby; 2004; 298-301.
4. Craig RG, Powers JM, Sakaguchi RL. Craig's restorative dental materials. 12th ed. St. Louis: Mosby Elsevier; 2006:480-511.
5. Goto T, Adachi M, Otani Y and Marquis AC: The influence of clinically induced variability on the distribution of

- compressive strength of hand-mixed zinc phosphate dental cement: Dent Mater, 1999; 15: 87-97. Walls AWG and McCabe JF: Applied Dental Materials. 8th ed. Black well Science Ltd, UK, 1998; 226-229
6. John WN: The chemistry of medical and dental materials. MPG Books Ltd, Bodmin, Cornwall, 2002; 157-159.
  7. Safwat EM, Saniour SH, Zaki D, El-Batran MM and Mousa IM: Preparation and evaluation of zinc phosphate cement powder from Egyptian raw materials. Thesis submitted to the faculty of oral and dental medicine, Cairo University, in partial fulfillment of the requirement of master degree in dental materials. 2009.
  8. Walls AWG and McCabe JF: Applied Dental Materials. 8th ed. Black well Science Ltd, UK, 1998; 226-229
  9. Wolfe J: Marbled paper; its history, techniques, and patterns. 2nd Edition, University of Pennsylvania Press, 1990; 50-66,.
  10. Zaki D, Khodary A, Mousa T, and Meligy A: Preparation and evaluation of phosphate bonded investment from Egyptian raw materials. Thesis submitted to the faculty of oral and dental medicine, Cairo University, in partial fulfillment of the requirement of PhD degree in dental materials. 2006

12/14/2010