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DIFFERENCE IN SETTING TIME AND WORKING TIME INTERVAL OF CONVENTIONAL ZINC PHOSPHATE CEMENT AND NANOPARTICLE ZINC PHOSPHATE CEMENT
(Research report)

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ABSTRACT
Introduction: *Zinc phosphate cement is one of cavity base material frequently used for pulp protection in the field of dentistry. This material demonstrates superiority in mechanical resilience and manipulation simplicity yet inducing pulp irritation as the reflection of high level acidity. Utilizing nano-particle ZPC may vanquish the shortcoming in conventional mechanical properties. However, brief setting time in nano-particle ZPC will generate higher exothermic reaction accompanied by the shortening of working period.* ***Purpose:*** *To assess the difference in setting time and working time interval of conventional ZPC and nano-particle ZPC.* ***Methods:*** *It is a true experimental laboratory study with post-test control group design. Four treatment groups were presented in five respective repetitions, comprising one scoop (0.318 gr) of conventional ZPC with three drops liquid in Group I, one scoop (0.386 gr) of nano-particle ZPC with four drops liquid in Group II, one scoop (0.386 gr) of nano-particle ZPC with three drops liquid in Group III, and one scoop (0.386 gr) of nano-particle ZPC with two drops liquid in group IV. ZPC was then manipulated in circular motion from powder to liquid. The setting time and working time was determined using stopwatch in second unit which then statistically analyzed using one-way ANOVA and post hoc Tukey HSD test.* ***Result****: Nano-particle ZPC with two drops liquid (Group IV) exhibited the shortest setting time value (mean 96.60s) which unveiled concurrently with the briefest working time interval (mean 34s). The longest setting time was presented in conventional ZPC (Group I) in the average of 627.80 seconds which also depicted the lengthiest working period with 84.20 seconds average. Statistical analysis revealed significant disparity in setting time value of conventional ZPC and nano-particle ZPC in each treatment group ($p<0.05$) while no significant difference was identified in working time data analysis ($p>0.05$).* ***Conclusion:*** *There is a significant difference in setting time and working time interval of conventional ZPC and nano-particle ZPC.*

Keywords: *Setting time, working time, zinc phosphate cement.*
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INTRODUCTION

Zinc phosphate cement is one of the most frequent pulp protective materials used in dentistry. It is easily manipulated with strong base properties which is beneficial in its application. The disadvantages of using ZPC comprise of its pulp irritating ability, weak antibacterial activity, poor adhesive potential, and high solubility in oral cavity fluids.[1]

Nano material is a very small particle sized from 1 to 100 nm. Nanomaterials have at least 1 dimension on a nanometer scale, which is equal to 1×10^{-9} m.[3] Nano-shaped dental material does not only refer to nano-sized material, but is a

development of new nanoscale structure which is controlled with condescending effect.[4] The size, diameter, distribution and nanoparticle bonding ability will definitely affect nano-particle mechanical properties.[5] The smaller the size of ZPC particle, the faster the setting time will be.[6] Greater surface area will generate higher and faster material surface reaction in smaller-sized particle resulting in almost no toxicity level when undergoing 100% setting process.[7]

The increase in ZPC powder and liquid ratio will accelerate phosphate matrix formation to escalate setting time reaction.[7] Brief setting time will induce faster exothermal or heat reaction in the time of manipulation thus not only reducing the level of toxicity but also shortening working time period. Zinc phosphate cement is reported with the highest exothermal reaction than other cements thus causing irritation of the pulp.[8]

The change in ZPC particle structure is expected to reduce pulp irritation potential and improve the properties of conventional ZPC. This study aims to demonstrate the disparity in setting time and working time interval of conventional ZPC and nano-particle ZPC.

METHODS

This study represented a true experimental laboratory framework with post-test only control group design. Conventional ZPC and nano-particle ZPC were investigated to confirm the disparity in setting time and working time value.

Four treatment groups (K-I, K-II, K-III, and K-IV) were presented in five respective repetitions, comprising one scoop (0.318 gr) of conventional ZPC with three drops liquid in Group I, one scoop (0.386 gr) of nano-particle ZPC with four drops liquid in Group II, one scoop (0.386 gr) of nano-particle ZPC with three drops liquid in Group III, and one scoop (0.386 gr) of nano-particle ZPC with two drops liquid in group IV. Zinc phosphate cement from GC Elite Cement 100 35g and nano-particle ZPC powder was employed with four drops, three drops, and two drops ZPC liquid. The weight of one scoop was 0.31 gram for conventional ZPC powder and 0.38 gram for nanoparticle ZPC powder.

ZPC manipulation was conducted on a glass plate where three separate portion of powder were initially acquired using fabricated measuring spoon number 3. The liquid was then located near the powder by dropping it in perpendicular bottle position. The manipulation was initiated by drawing ZPC powder into liquid in circular motion using cement spatula. Working time calculation was performed from initial manipulation to homogenous mixture indicated by uninterrupted string when pulled using cement spatula. Setting time calculation established from the beginning of ZPC manipulation until the hardened of cement presented by unscratched surface using dental explorer.

Saphiro Wilk test was performed to assess data normality in samples less than 50 which demonstrated normal data distribution in each treatment groups ($p>0.05$). Homogeny variants were presented in overall treatment samples after analyzed using Levene's variant test ($p>0.05$). Setting time and working time disparity was evaluated using Oneway ANOVA test which resulted in significant difference in setting time and working time value of treatment groups ($p<0.05$).

Post Hoc Tukey HSD test was conducted to inspect significant difference between K-I, K-II, K-III, and K-IV group. Significant disparity was observed in K-I to K-II and KII to K-IV setting time value ($p<0.05$), yet no significant difference presented in K-III to K-II group ($p>0.05$). Post Hoc analysis on working time value revealed significant difference in KI to K-II and K-III to K-IV treatment group ($p<0.05$) without any significant difference presented in K-II, K-III, and K-IV group ($p>0.05$).

RESULT

Setting time and working time tabulation of conventional ZPC and nanoparticle ZPC are presented as follow:

Table 1. Mean value of ZPC Setting Time

Group	*Setting Time* (second)	P
K-1 Conventional	627.80±49.017	.000
K-II Nano 4 drops *liquid*	278.40±42.776	.000
K-III Nano 3 drops *liquid*	137.60±18.284	.000
K-IV Nano 2 drops *liquid*	96.60±16.196	.000

Table 1 presents the shortest ZPC setting time in group IV (*mean* 96.60s) while the longest interval was presented in Group I with 627.80 seconds average.

Table 2. Means of ZPC Working Time

Kelompok	*Working Time* (second)	p
K-1 Conventional	84.20±14.687	.000
K-II Nano 4 drops *liquid*	38.60±5.771	.000
K-III Nano 3 drops *liquid*	39.00±13.210	.000
K-IV Nano 2 drops *liquid*	34.00±11.937	.000

Based on table 2, the shortest working time is depicted in treatment group IV (*mean* 34 seconds) while the longest working period can be observed in treatment group I (*mean* 84.20 seconds).

DISCUSSION

The result of this study reveals that average setting time in group IV is faster than group I.

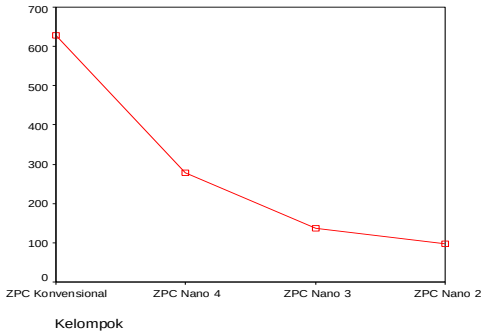

Figure 1. Graphic of ZPC Setting Time Mean Value

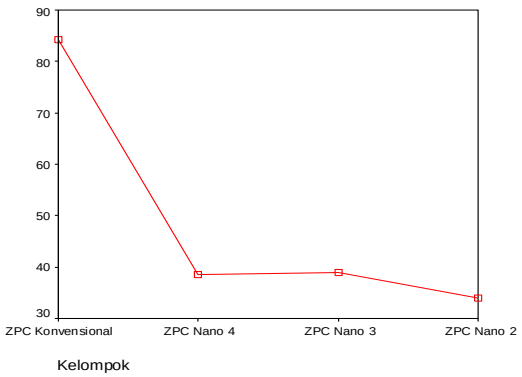

Figure 2. Graphic of ZPC Working Time Mean Value

Shorter setting time and working time in nano-particle ZPC occurs as the result of smaller ZPC particle which induce acceleration in setting time reaction.[6] Reaction begins when ZPC powder and liquid are manipulated. Phosphoric acid in liquid will react with particle surface resulting in the release of zinc ion into liquid. Aluminium and phosphoric acid in liquid react with zinc ion to form zinc aluminophosphate gel. Aluminophosphate gel is zinc oxide covered by aluminophosphate and zinc phosphate gel. Zinc phosphate cement which experienced final setting contains zinc oxide particle core structure scattered in zinc aluminophosphate matrix.[7]

$$3ZnO + 2H_3PO_4 + H_2O \rightarrow Zn_3(PO_4)_2 \,.\, 4H_2O$$

Figure 3. ZPC Setting Time Reaction

Smaller size in ZPC powder particle will increase the wide of surface area.[9] In nano-sized particle, faster reaction exists due to higher reaction presented in greater surface area.[6] This powder and liquid reaction represented exothermic reaction producing heat in the time of ZPC powder and liquid manipulation. Exothermal reaction in ZPC displays the highest reaction than other cement thus may induce irritation of the pulp.[8]

Other factor influencing ZPC setting time and working time is powder and liquid ratio. Working time and setting time will increase in the reduction of ZPC powder and liquid ratio.[7] An increase in powder and liquid ratio will accelerate setting time interval by advancing phosphate matrix formation.[8] Recommended ZPC powder and liquid ratio is 2.5 to 3.5 g/ml, which is equal to 1.4 g powder/ 0.5 ml liquid.[7]

This theory has been proven by the result of this study where an increase in powder and liquid ratio of ZPC nanoparticle, or reduction in liquid drop, were reported to accelerate working time and

setting time value. This demonstrates the shortened in ZPC nanoparticle setting time and working time by increasing powder and liquid ratio or reducing liquid drops.

The speed of ZPC powder and liquid manipulation will also influence working time and setting time value where fast manipulation of powder and liquid will accelerate existing reaction, so does slow manipulation conversely.[11] Gradual manipulation of ZPC powder to liquid will increase setting time and working time interval, and reduce exothermic or heat reaction at the time of manipulation.

Zinc phosphate cement setting time and working time may also be influenced by manipulation interval. Prolonged manipulating time will generate matrix destruction resulting in longer interval for new matrix production. Composed of zinc aluminophosphate, this matrix is formulated through exothermic reaction in the time of ZPC powder and liquid manipulation. Extended time for new matrix formation will prolong ZPC working time and setting time.[6]

Nano-sized particle cement expresses different characteristics than macro-sized particle cement. The difference in nano-particle and macro-particle cement is resulted by the reduced in scale. The reduced scale causes greater surface area per unit mass. An increase in surface area will result in the increasing number of low-coordinated atoms presented on material surface. Superficial atom presents less stability resulting in more reactive nanoparticle material.[10] Wider surface results in greater generated-reaction area thus accelerating the time of setting reaction.[6]

In this study, the most optimal ratio for ZPC nano-particle is one scoop (0.38 gr) powder with four drops liquid. Four liquid drops show better homogeneity when compared with other treatment group. The addition of liquid drops can cause a decrease in pH as the result of phosphoric acid contained in ZPC liquid. This liquid composition beholds a very high level of acidity which is highly potential in generating pulp irritation. The more liquid used, the more irritating ZPC will be.[8] It can be concluded that additional liquid drops in conventional and nanoparticle ZPC powder will increase setting time and working time interval. In conclusion, there is a significant difference in setting time and working time interval of conventional ZPC and nano-particle ZPC.

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