Dental Cement Physical and Mechanical Properties by In Vivo Approach

Prihartini Widiyanti[1,2], Siswanto[2] and Idha Sa'Ada[1]
[1]Biomedical Engineering Study Program, Department of Physics, Faculty of Science and Technology, Universitas Airlangga, Indonesia
[2]Physics Study Program, Department of Physics, Faculty of Science and Technology, Universitas Airlangga, Indonesia

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Abstract: The current production technology of dental cement material is growing rapidly compared to 50 years ago. Cement as dental restoration material must be elastic and have a low conductivity property. There are four kinds of dental cement commonly used in dentistry, namely zinc phosphate cement, polycarboxylate cement, glass ionomer cement, and zinc oxide and eugenol cement. There is no study about physical and mechanical properties based on in-vivo condition. This study aims to know the physical and mechanical properties of dental cement, by the in-vivo approach and using rabbit as an experimental object. Dental cements used in this research were zinc phosphate cement (ZPC), polycarboxylate cement (PC), glass ionomer cement (GIC), and zinc oxide and eugenol cement (ZOEC). The method of this study was the preparation of tools, materials and experimental animals. We used six male - 5 months in age - rabbits. Before being treated (fill-teeth material insertion), the rabbits were anesthetized by an anaesthetist from the Animal Hospital, Faculty of Veterinary Medicine, Universitas Airlangga. Then the rabbit teeth were drilled and formed to become box cavity. Information which can be obtained from this in-vivo experiment is its physical and mechanical properties i.e. compressive strength, tensile strength, and microstructure of dental cement. Based on the physical and mechanical characterization value, the best compressive strength was 101.888 MPa and refers to zinc phosphate cement and the best tensile strength value was glass ionomer cement with 6.555 MPa. The morphological features, mainly surface structure of dental cement, showed less well sealed for zinc phosphate cement, strongly bonded with teeth for polycarboxylate cement, there were lumps of unreacted powder particles for glass ionomer cement and there was a very hard lump formed for zinc oxide and eugenol cement.

1 INTRODUCTION

The technology of dental cement nowadays is emerging compared to 50 years ago. This condition facilitates the dentists to have more choice to restore the broken teeth or even the loose teeth. One of the dental cement alternatives is by using polymer. Some scientists have developed the polymer-based material to be close to the characteristics and appearance of the natural teeth (Wagh, 2016; Manappallil JJ, 2016)

The polymer is a long chain molecule consisting of several repetitions of units (Combe, 2013). Most of the polymer was used in the industry or medical area. One example of polymer used in the medical area is teeth filling material (dental cement). Cement as a teeth filler should be elastic (low strength materials). This cement could be synthesized by mixing the powder material with some liquid. The cement composition varies in chemical composition, characteristics, or the usage. This material also has low conductivity compared to the metal filling material.

Four types of dental cement are normally used in dentistry, zinc-phosphate cement, polycarboxylate cement, glass ionomer cement, and zinc oxide and eugenol cement (Noort, 2002).

There was a study that synthesized the zinc phosphate dental cement zinc oxide and phosphate acid (Wagh AS, 2016). This study results showed that the mechanical properties on the zinc phosphate dental cement increased with the increase of powder-liquid ratio until their mass was the same. But, it would decrease if the liquid ratio increased in the composition ratio. The best composition ratio in

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the zinc phosphate dental cement was at 60:40 respective to powder and liquid.

The study of the addition of polystyrene as an additive substance in the dental cement based on zinc oxide eugenol has been performed. Polystyrene is a linear polymer that has thermoplastic property. This material would be melted at a temperature around 95°C and become viscous solution at 120-180°C and become liquid above 250°C, then degraded above 320-350°C. This study concluded that the addition of polystyrene to the eugenol, as much as 10%, could give the best mechanical properties. Fourier Transform Infrared (FT-IR) spectrophotometer is a device to make a chemical-physical identification, especially in the qualitative analysis on the functional group of organic or an-organic materials based on the absorbance towards infrared. FTIR test showed that the mixing of polystyrene and eugenol is a simple mixture.

From that study, there was a lack in the physical and mechanical properties testing which was no in-vivo test using living organism yet. This study would use the rabbit as a living organism for an in-vivo test. The teeth of the rabbit would be recessed as in the class III caries. This type of caries usually occurs on the anterior teeth and could occur on the medial or distal surface of the incisor or canine. From the in-vivo test, the information about the physical and mechanical properties could be obtained comprising compressive strength, tensile strength, and microstructure from several types of dental cements.

2 MATERIALS AND METHODS

2.1 Materials

The first step of this study was the preparation of the tools to drill and patch the teeth. They were round bur, fissure bur and tapered bur for class III cavity preparation, cement spatula for mixing and taking the dental cement materials, straight probe for detecting the cavity, dental tweezers for helping to take the bur eye, debris and cotton, plastic filling instrument for inserting the dental cement materials in the cavity, the glass plate and mixing paper for mixing the materials, and cotton roll for blocking the saliva.

The materials for dental patching were zinc phosphate cement, polycarboxylate cement, glass ionomer cement, zinc oxide and eugenol cement. Those four types of dental cements were a package of powder and liquid that could be obtained in the market.

The preparation of the animal trial started with six male rabbits with age of five months. Before the treatment, the rabbit was anesthetized first to know their condition. To ease the preparation of the cavity on the rabbit's teeth, the rabbits were stunned. The anesthesia was performed by the vet of Universitas Airlangga as shown in Table 1.

Table 1: Anesthesia of the animal

	F1	F2	F3	F4	F5	F6
Weight (kg)	1.8	2	1.5	1.9	1.6	1.7
Xylazine (ml)	0.5	0.5	0.4	0.5	0.4	0.5
Antrophine (ml)	0.4	0.4	0.3	0.4	0.3	0.4
Ketamine (ml)	0.9	1	0.8	0.9	0.8	0.9

2.2 Testing Sample Preparation

After the preparation of the tools and materials, the animal trial sample was prepared. Two incisors of the rabbit were drilled as shown in Figure 1. The preparation of class III cavity used round bur, fissure bur and tapered bur. The preparation was only at the dentin. After the preparation, the cavity was cleaned with alcohol-dipped cotton. The caries was detected by using tweezers.

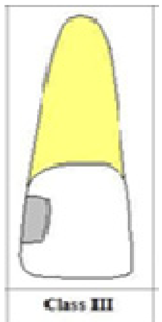


Figure 1: Class III Cavity based on the position of Caries

After the cavity was dry and clean, the patching process was performed. The patching area was isolated by cotton roll to prevent the saliva from inserting the cavity. The patching materials were prepared in the glass plate based on the usual procedure and stirred with cement spatula. Specific to glass ionomer cement, the patching material was stirred on the mixing paper with plastic cement spatula. The patching materials were then inserted in the cavity by using the plastic filling instrument. After one or two minutes, the patching was pressed with amalgam stopper and formed.

On the Rabbit I, the cavity was patched by using zinc phosphate cement. On the Rabbit II, the cavity was patched by using polycarboxylate cement. On the Rabbit III, the cavity was patched by using glass ionomer cement and on the Rabbit IV, the cavity was patched by using zinc oxide and eugenol cement. On the Rabbit V, the right cavity was patched by using zinc phosphate cement and the left cavity was patched by using polycarboxylate cement. On the Rabbit VI, the right cavity was patched by using glass ionomer cement and the left cavity was patched by using zinc oxide and eugenol cement. The total of the overall samples were 12 pieces as shown in Table 2.

Table 2: In-Vivo Test Sample

No.	Sample Type	Amount (tail)	Dental Cement
1.	A	3	zinc phosphate cement
2.	B	3	polycarboxylate cement
3.	C	3	glass ionomer cement
4.	D	3	zinc oxide and eugenol cement

The categorization of A, B, C, D was based on the type of cements applied in the tooth cavity

After the patching process, the sample caring was performed for 21 days to observe the strength of the patch after patched to the teeth through in-vivo test. After that, the teeth were alienated using incisor teeth alienating pliers and characterized.

2.3 The Sample Characterization

The aim of this characterization was to know the compressive strength, tensile strength and microstructure of the dental cement.

2.3.1 Compressive Strength

Compressive strength testing was using Autograph. The load used in this test was 100 kN. From this test, the compressive strength given to the sample until it breaks of fractures was obtained. By using equation (1), the compressive strength could be determined.

$$\tau = F/A \qquad (1)$$

τ is the compressive strength (Pa), F the load on the sample (N), and A is area (m^2).

2.3.2 Tensile Strength

For knowing the stickiness of the dental cement, tensile strength testing was used by giving a tensile load directly to the sample. To ensure the sample held firmly, the tip of the sample was made bigger than the middle part of the sample. The tensile strength measurement was using Autograph. By using equation (2), the tensile strength was determined.

$$TS = \frac{F}{A} \qquad (2)$$

TS is the tensile strength (Pa), F is the load (N), and A is the area (m^2).

2.3.3 Microstructure

To know the microstructure, a Scanning Electron Microscope (SEM) was used. The sample was prepared first by coating with a specific material (gold) in the stub from metal with a diameter of 9 mm. The sample was then inserted into the specimen chamber and illuminated with the electron beam (20 kV). The reflected electron was detected by the scintillator detector amplified by an electrical circuit that could produce a figure from a Cathode Ray Tube (CRT). The capturing process was performed after choosing a specific part of the sample with the correct magnification so that a good and clear image was obtained. To print the film of the capturing result, a vacuum evaporator JEOL JEE-4X was used.

3 RESULT

This study performed an in-vivo test by using a living organism. The in-vivo test gained the information of the physical and mechanical properties, such as compressive strength, tensile strength, and microstructure from zinc phosphate cement, polycarboxylate cement, glass ionomer cement), and zinc oxide and eugenol cement.

3.1 Sample Testing Result

This study was performed experimentally by measuring the mechanical properties, which were compressive strength and tensile strength. The sample characterization result is shown in Table 3.

Table 3: The Result of Sample Characterization

No.	Sample Type	Compressive Strength (MPa)	Tensile Strength (MPa)
1.	A	101.888	5.777
2.	B	56.555	6.111
3.	C	70.777	6.555
4.	D	46.111	3.111

Description :

A = zinc phosphate cement

B = polycarboxylate cement

C = glass ionomer cement

D = zinc oxide and eugenol cement

From the result of sample characterization, there was a relation between the type of dental cement and their mechanical properties. The graphs of compressive strength and tensile strength are shown in Figures 2 and 3.

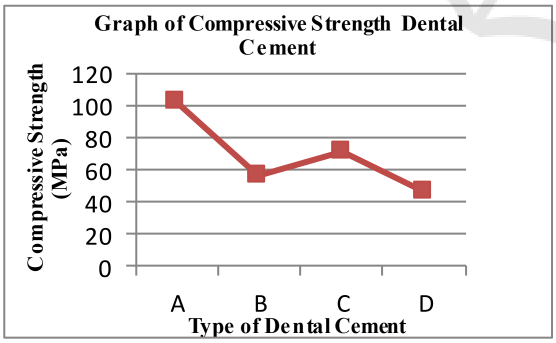


Figure 2: The Compressive Strength of Several Dental Cements

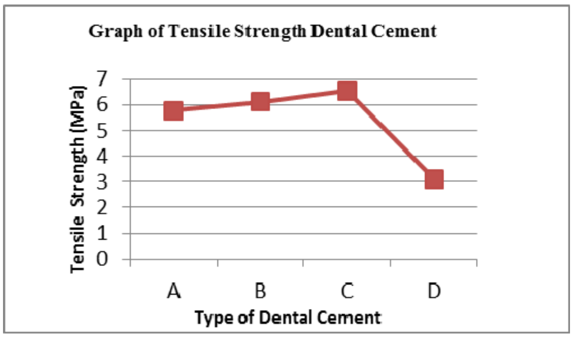
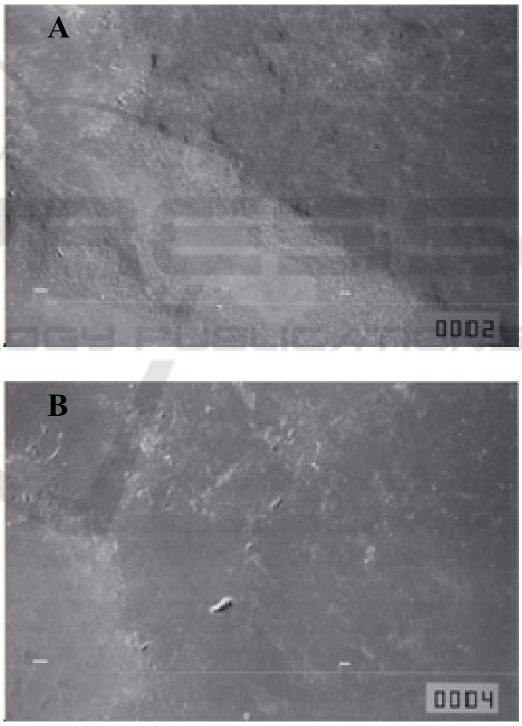


Figure 3: The Tensile Strength of Several Dental Cements

The microstructure of the dental cement could be observed from the result of SEM. The microstructure of the dental cement is shown in Figure 4.



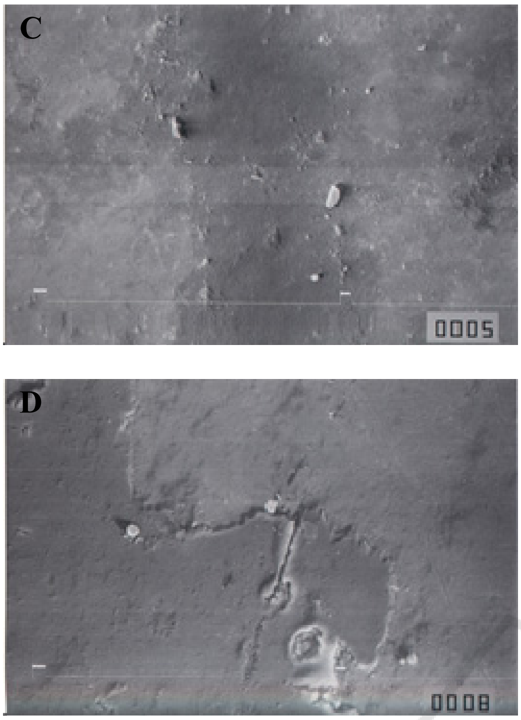


Figure 4: (a) The microstructure of zinc phosphate cement. (b) The microstructure of polycarboxylate cement. (c) The microstructure of glass ionomer cement. (d) The microstructure of zinc oxide and eugenol cement

4 DISCUSSION

The results on the mechanical properties of the dental cement show that the best compressive strength was the zinc phosphate cement at 101.888 MPa and the best tensile strength was glass ionomer at 6.555 MPa. If manipulated correctly, the zinc phosphate cement could have the compressive strength of 104 MPa and tensile strength of 5.5 MPa (Anusavice, 2003).

The compressive strength and tensile strength were various based on the ratio of the powder and liquid. The increase in the strength was obtained by adding more powder than was recommended and it was obvious compared to the decrease of the strength caused by the decrease of the powder in the mixture. The decrease of the ratio of powder and liquid would produce a weak cement. The lack or increase of water content from the liquid would decrease the tensile strength and compressive strength of the cement. Like zinc phosphate cement, glass ionomer cement would be easy to break when hardening. After that, the recess of the cement could be thrown away by gouging or breaking the cement away from the restoration side. This cement is sensitive to water contamination in the hardening process. Thus, the restoration side should be coated to protect the cement from early contact with a liquid.

The compressive strength from polycarboxylate cement was lower than the zinc phosphate cement but had a slightly higher tensile strength. This cement was not as brittle as zinc phosphate cement, so that it was more difficult to eliminate the recess of the cement after the cement hardened.

The mechanical properties of the zinc oxide and eugenol cement were lower than the other cements. This cement was hard to manipulate in the mouth cavity. The thickness of its layer tended to be higher and the recess of the hardened cement was hard to remove.

The difference in compressive strength and tensile strength was caused by the speed of mixing between the powder and the liquid, the mixture plate and the temperature of the mixing tools. The speed of the mixing between powder and liquid could affect the hardness of the dental cement because the powder that was mixed with the liquid gradually with a small amount would increase the working time and hardness so that it could decrease the heat produced and allow more powder to be added to the mixture.

The mixture plate and the temperature of the mixing tools could also affect the mechanical properties of the dental cement. The high temperature on the mixing tools could increase the hardening reaction from the dental cement. On the other hand, if the temperature of the mixing tools was low, then the hardening reaction of the dental cement could be longer so that the matrix formation could be slowed down. Besides that, what needs to be taken into account is the technique of mixing the powder and liquid. The inappropriate mixing could cause a crack in the dental cement so that it would complicate the mechanical properties measurement.

The microstructure of the zinc phosphate cement in Figure 4(a) showed that the dental cement and the teeth were not stuck together. When the powder was mixed with the liquid, the phosphoric acid had contact with the particle surface and released zinc ions to the liquid. The aluminium, which has been bonded to the phosphoric acid, reacts to the zinc and produces zinc gel as aluminophosphate on the surface of the residual particles. This hardened cement is the main structure and consisted of unreacted zinc oxide particles, coated with a solid matrix that has not been formed from the zinc aluminophosphate. Because water affects the acidbase reaction, the composition of the liquid should be maintained to ensure a consistent reaction. The change in composition and reaction speed could occur due to the evaporation of the water. So, the change in composition could affect the reaction.

The microstructure of the polycarboxylate cement in Figure 4(b) showed that the cement was bonded tightly to the tooth structure. The hardening reaction of this cement involved the dissolution of the particle surface by the acid that released the zinc, magnesium, and tin ions and merged to the polymer chain though carboxyl group. These ions react to the carboxyl group and polyacid chain near them and form a salt with crosslink while the cement was hardening. The hardenend cement consisted of non-uniform matrix gel with a spread of unreacted particles inside. The microstructure image was similar to the zinc phosphate cement.

The microstructure of the glass ionomer cement in Figure 4(c) showed that there was a lump of powder particles that did not react. When the powder and the liquid were mixed to form a paste, the glass particle surface would be dissolved in the acid. The calcium, aluminium, sodium, and fluorine ions were released to the watery media. The polyacrylic acid chain would crosslink with the calcium ions and form a solid mass. For the next 24 hours, a new phase was formed in which aluminium ions bond in the cement mixture and form a brittle cement. Sodium and fluorine ions did not have a part in the crosslinking of the cement. Some of the sodium ions could replace hydrogen ions from the carboxylic group, and the rest would join the fluorine to form natrium fluoride that spread evenly in the hardened cement. Along the hardening process, the crosslinking phase was also hydrated by the same water as the medium. The parts that did not react with the glass particles would be coated by the silica gels that have been formed during the cation release from the particles surface. Thus, the hardened cement consisted of lumps of powder particles that have not reacted and been surrounded by the silica gels in the amorphous matrix of calcium hydrate and a mixture of aluminium salt.

The microstructure of the zinc oxide and eugenol cement in Figure 4(d) showed that there was a hard lump. In the right condition, the reaction between zinc oxide and eugenol resulted in a hard relative mass. The hardening mechanism of the zinc oxide eugenol materials consisted of hydrolysis of zinc oxide and eugenol to form lumps. Zinc acetic dihydrate accelerated it, that was more soluble than zinc hydroxide and could give zinc ions faster. The high temperature could increase the hardening speed.

The main property of the dental cement is that it should last in the solubility and disintegration in the mouth cavity. The cement had continuous contact with several types of acid produced by the microorganism and food processing. Some of them were carried to the mouth as food and beverages. pH and temperature in the mouth cavity were always changing. So, no cement could fulfill all desired ideal characteristics. A cement system is maybe suitable for one use compared to the other system. Every condition must be evaluated based on the environment and biological and mechanical factors.

5 CONCLUSIONS

Based on the in-vivo test, the physical and mechanical properties were obtained from four types of dental cements. The mechanical properties were determined through compressive strength and tensile strength. The best compressive strength was shown by zinc phosphate dental cement at 101.888 MPa and tensile strength from glass ionomer cement at 6.555 MPa. The dental cement from zinc oxide and eugenol had the lowest physical properties compared to the other dental cements.

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