

The Flexural Strength of Tricalcium silicate-White Portland Cement-Resin Versus Resin-Modified Calcium Silicate

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Abstract

The Mineral trioxide aggregate (MTA) has been recommended for its effectiveness as a pulp capping material. However, this material has some weaknesses. TheraCal LC is a resin-modified calcium silicate-based developed to cover the MTA weaknesses. One of the important qualities of pulp capping materials is their flexural strength. Some studies show that white portland cement (WPC) and MTA share similar characteristics, yet the quality difference in WPC raw materials can affect its potential as a pulp capping material.

The current research aims to find flexural strength value of the Tricalcium Silicate-White Portland Cement (TS-WPC)-Bi₂O₃-UDMA mixture and TheraCal LC and analyze their value differences. The pure experimental method was used in this research at a laboratory. The 32 samples were divided into 4, i.e., groups A (TS-WPC- Bi₂O₃-UDMA mixture), B (TS-WPC - Bi₂O₃-UDMA mixture submerged in ringer lactate), C (TheraCal LC), D (TheraCal LC submerged in ringer lactate). The flexural strength values were tested based on ISO 4049. The data were analyzed using ANOVA, followed by a post hoc independent t-test. The mean values of flexural strength for groups A, B, C, and D were 77.260, 80.200, 48.525, and 17.579 respectively. Based on ANOVA, the data between groups were significant at a p-value <0.05. From the post hoc independent t-test, it was found that differences between groups B and D, groups D and C, groups D and A, groups C and A, and groups C and B were significant.

The flexural strength value of the TS-WPC- Bi₂O₃-UDMA mixture was higher than that of TheraCal LC. This was because of the filler composition, the monomer used, and the addition of coupling agent silane.

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Introduction

Caries lesions that reach two-thirds of the dentin depth, with the pulp being at risk of getting exposed, are known as deep caries lesions or caries profunda.^{1,2} The attempt to prevent the pulp from being exposed and, at the same time, to maintain its vitality is by applying indirect pulp capping.³ This indirect pulp capping is a treatment to maintain the affected dentin and

leaves a thin layer of the infected dentin to cover all of the affected zones using a cavity liner. Meanwhile, mineral trioxide aggregate (MTA) has been studied and recommended for its effectiveness as a pulp capping material. It is also better in the process of dentin bridge formation stimulation than its predecessor, i.e., calcium hydroxide.⁴⁻⁶ As a pulp capping material, the mineral trioxide aggregate has some weaknesses such as lengthy setting time, difficult handling, incompatibility with some restoration materials when used using the layering technique, as well as relatively lower compression and flexural strength than dentin.^{7,8} Flexural strength value is an important property in predicting the ability of a material to withstand stress or high pressure from mastication forces.^{9,10}

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In the last few years, several researchers have focused their attention on the common characteristics of white portland cement and MTA.^{11,12} These studies show that they have similar composition, physical property, mechanical property, and biocompatibility, except that Bismuth oxide (Bi_2O_3) is inexistent in the white portland cement.¹¹⁻¹³ This might pose a concern, considering that Bi_2O_3 is critical for its function as a radiopacifier, one of the requisites for a pulp capping material. Some countries have investigated the commonality between white portland cement and MTA. However, the quality difference in the raw materials of white portland cement might affect its potential to be a pulp capping material. Indonesia is a country with its wealth in sources of white portland cement. Indonesian white portland cement has a high chemical composition similarity with MTA, thus it has the potential of having the same reaction when used as a pulp capping material.¹⁴

Recently, a new pulp capping material has been developed to fix the physical-mechanical properties of MTA. TheraCal LC (BISCO Inc.) is a new resin-modified, light-cured calcium silicate-based material consisting of type-III Portland cement (20–60%), poly (ethylene glycol) dimethacrylate (10– 50%), bis-GMA (5–20%), and barium zirconate (1–10%).¹⁵ It is fairly good to be used as a pulp capping material. Yet, in its use in dentistry daily practice, its low economic price becomes its main weakness. Based on this background, the writer conducted the research to make a Tricalcium Silicate-White Portland Cement - Bi_2O_3 - Urethane Dimethacrylate (UDMA) mixture and see its flexural strength value as compared to the resin-modified calcium silicate (TheraCal LC).

Materials and methods

The materials used in this study were Tricalcium silicate-white Portland cement (TS-WPC) (PT. Indocement, Cirebon, Indonesia), Bi_2O_3 (Shanghai Xinglu Chemical Technology Co. Ltd., Shanghai, China), distilled water, 99.9 % isopropanol, and commercial TheraCal LC (BISCO Inc) was used as the control. This research used a pure experimental method and 32 samples. This research had 4 treatment groups, with each group being tested 8 times, thus making a total of 32 samples being tested in

this study. Group A was a TS-WPC - Bi_2O_3 - UDMA mixture which was tested after polymerization. Group B was the TS-WPC - Bi_2O_3 -UDMA mixture which was tested after being submerged in ringer lactate for 24 hours at 37°C upon polymerization. Group C was resin-modified calcium silicate (TheraCal LC) that was tested after polymerization, and Group D was resin-modified calcium silicate (TheraCal LC) that was tested after being submerged in ringer lactate for 24 hours at 37°C upon polymerization.

Making TS-WPC - Bi_2O_3 mixture

The TS-WPC- Bi_2O_3 mixture was made at the Laboratory of the Center for Nano Technology and Graphene Institution Research and Development (Print-G) of Universitas Padjadjaran. As much as 80 gr TS-WPC and 20 gr Bi_2O_3 were mixed and put into the beaker glass. Furthermore, 100 ml of isopropanol solution 99% was added to the mixture and stirred using a magnetic stirrer for 30 minutes until it was homogeneous. The homogeneous mixture was then put into 6 centrifuge tubes evenly for a centrifugation process at 5000 rpm for 10 minutes. The resulting pellets were saved in a petri dish and inserted into a drying oven, at 60°C for more or less 2 hours until TS-WPC- Bi_2O_3 dried powder was obtained. This dried powder was saved in an airtight container for further procedure.

Making TS-WPC - Bi_2O_3 - UDMA mixture

The TS-WPC - Bi_2O_3 - UDMA mixture was made at the Integrated Research Laboratory Faculty of Dentistry at Universitas Padjadjaran. As much as 11.66 gr TS-WPC- Bi_2O_3 dried powder and 1.4 gr Trimethoxy(propyl)silane were mixed in a beaker glass and stirred using a magnetic stirrer. Furthermore, 6.6 gr Diurethane dimethacrylate (UDMA) and 0.2 gr Tri-ethylene glycol dimethacrylate (TEGDMA) were added to the mixture. The beaker glass containing the mixture was covered by aluminium foil to make it light-tight. Then, 0.14 gr camphor quinone was added to the mixture. All of these materials were stirred using a magnetic stirrer for 20 minutes until they were homogeneous. The mixed materials were then put into a mold using a 3 cc sput. For the first layer, the mold was filled up to 1 mm. The first layer was polymerized using a light curing unit at 4 points, each of which was polymerized for 20 seconds. This was because

the specimen's length was greater than the 6-mm diameter of the tip light curing unit. The second layer was filled a little over 1 mm, and further layered by mylar stripe. Polymerization was done using a light curing unit at 4 points for the second layer, with each point being polymerized for 20 seconds. Every time the polymerization process was about to begin, the light intensity was first measured using a light meter. The light intensity was set at 975 mW/cm². The procedure for making the TS-WPC - Bi₂O₃-UDMA mixture was repeated until 16 samples were obtained. Upon the polymerization, the 16 samples were divided into two groups, groups A and B. The first group (A) received a flexural strength test immediately, and the second group (B) was submerged in ringer lactate for 24 hours and saved in an incubator at 37°C, then tested for flexural strength afterward.

TheraCal LC control sample

The TheraCal LC was put into a (25±2) mm x (2±0.1) mm x (2±0.1) mm mold. The first layer of the mold was filled up to 1 mm. This first layer was polymerized using a light curing unit at 4 points, with each point being polymerized for 20 seconds. The second layer was filled a little over 1 mm, and then was layered by a mylar stripe. The polymerization was done using a light curing unit at 4 points for the second layer, with each point being polymerized for 20 seconds. Upon polymerization, the 16 TheraCal LC samples were divided into two groups, i.e., groups C and D. The first group (C) received a flexural strength test immediately, and the second group (D) was submerged in ringer lactate for 24 hours and saved in an incubator at 37°C, then tested for flexural strength afterward.

Flexural strength test

The sample and control groups were tested for their flexural strength based on ISO 4049 using the three-point bending method by employing the universal testing machine (Lloyd Instrument LRX Plus). The obtained flexural strength values were then analyzed statistically.

Results

The result of the flexural strength measurement of TS-WPC-Bi₂O₃-UDMA mixture and TheraCal LC specimen can be seen in Table 1. Table 1 shows that the mean value of group A was 77.260 Mpa, with their standard deviation

value being 7.6564. Group B's mean value was 80.200 MPa and the standard deviation was 8.5769. Group C's mean value was 48.525 MPa and the standard deviation was 8.3067. Group D's mean value was 17.579 MPa and the standard deviation was 1.2307. Using ANOVA, the statistical analysis obtained p value < 0.05, which indicated a significant test property between one group and other groups as shown in Table 2.

Sample	A	B	C	D
1	72.75	82.03	52.5	19.07
2	81.57	77.24	58.49	17.90
3	79.63	87.79	61.75	17.84
4	87.08	90.84	46.46	18.29
5	81.20	77.94	42.46	18.87
6	80.32	80.89	46.56	16.66
7	62.10	62.29	38.90	15.68
8	73.43	82.58	41.08	16.32
Mean value	77.260	80.200	48.525	17.579
Standard deviation value	7.6564	8.5769	8.3067	1.2307

Table 1. Result of mean and standard deviation measurements of flexural strength values in Mpa.

Source	SS	Df	MS	F	p-value
Treatment	20,557.1376	3	6,852.37921	135.22	9.19 x 10 ⁻¹⁷
Error	1,418.9001	28	50.67500		
Total	21,976.0378	31			

Table 2. ANOVA.

	D	C	A	B
Mean	17,579	48,525	77,260	80,200
D	17,579			
C	48,525	1,92 x 10 ⁻⁹		
A	77,260	3,90 x 10 ⁻¹⁶	8,63 x 10 ⁻⁹	
B	80,200	1,14 x 10 ⁻¹⁶	1,18 x 10 ⁻⁹	0,416

Table 3. Post hoc analysis p-values for pairwise t-tests.

A post hoc test with t-independent statistic was carried out to discover which groups of the four groups were different, as shown in Table 3. The values in the table show the p-value of the test results between one treatment and other treatments (in pairs). When group D (at a mean value = 17.759) was compared to group B (whose mean value = 80.200), the p-value was found at 1.14 x 10⁻¹⁶. This indicates a significant difference. The same applied to the comparison results between groups D and C, groups D and A, groups C and A, and groups C and B.

Discussion

The three-point flexural strength test was designed to be a test for primary strength for restorative dental material containing resin based on ISO 4049 and a suitable test method for comparing the flexural strength property of materials such as in this research.¹⁶ The statistical test results presented in Table 3 show that the flexural strength value of mixture groups A and B was significantly higher ($p < 0.01$) than groups C and D. Two important variables affected the mechanical strength in resin calcium silicate cement mixture; they were filler and matrix. The flexural strength values in all groups were mainly affected by the filler composition which reached 55% of the total mixture. The inorganic filler in all groups was calcium silicate cement deriving from portland cement whose major constituents consisted of tricalcium silicate and dicalcium silicate. Mirică et al., (2020) suggest that the percentage of inorganic filler content affects the mechanic property of flowable resin composite. The high inorganic filler content indicated a high flexural strength value. However, filler content was not the only determinant of flexural strength value. The organic matrix also contributed to the high flexural strength value.¹⁷ The calcium silicate cement volume in groups A and B was higher at 1.64% than in groups C and D. The correlation between mechanical property and filler volume was directly proportional. Finally, an increase in the filler volume led to an increase in the mechanic property, including the flexural strength.¹⁸

The second variable was the organic matrix. The organic matrix of the calcium silicate cement mixture consisted of dimethacrylate resin. Groups A and B used urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) monomers, and groups C and D used A diglycidildimethacrylate (Bis-GMA) and PEGDMA monomers. The molecular structure and viscosity of the comonomer mixture significantly affected the mechanical and physical properties of the cured resin. A study at a laboratory conducted by Szczesio-Włodarczyk et al., (2021) reveals that UDMA addition results in the formation of a denser polymer network. UDMA resin had a higher reactivity than Bis-GMA resin thanks to its more flexible molecular structure, possible hydrogen abstraction, and chain transfer

reaction mechanism. The flexural strength of resin with no filler based on UDMA/TEGDMA ranged between 44 and 78 MPa, while for Bis-GMA/TEGDMA it ranged between 51 and 66 MPa.¹⁹

Urethane dimethacrylate had the highest flexural strength value among other monomers. However, UDMA had an issue in terms of manipulation because of its high viscosity. UDMA resin was one of those monomers that had high viscosity as a result of the interaction of intermolecular hydrogen bonds between its amino groups (-NH-) and carbonyl (-C=O). UDMA had lower viscosity than Bis-GMA since the hydrogen bonds in the amino group (-NH-) were lower than the hydroxyl group (-OH) in Bis-GMA. Yet, because the viscosity of both monomers was so high, UDMA and Bis-GMA could not be formulated independently without any addition from the diluent monomer with low viscosity such as TEGDMA.¹⁹ A study by Szczesio-Włodarczyk A et al., (2022) on the effect of low molecular weight monomer (TEGDMA, HDDMA, HEMA) on the Bis-GMA and UDMA-based matrix and composite properties, suggests that UDMA/TEGDMA-based composite resin shows higher flexural strength and flexural modulus than Bis-GMA/TEGDMA-based composite resin. This is a result of the increased density of cross-linking in the polymer network.²⁰⁻

²¹ Based on this, the writer preferred UDMA to Bis-GMA to be mixed with TS-WPC- Bi_2O_3 , and thus TS-WPC- Bi_2O_3 -UDMA mixture was formed. In this mixing process, the writer also combined UDMA with monomer that had lower viscosity such as TEGDMA to obtain an optimal flexural strength value.

Another factor other than the two main variables that influenced the significance of the flexural strength value of groups A and B than groups C and D was the addition of coupling agent silane. Calcium silicate hydrate (C-S-H) constituted a major inorganic phase in Portland cement paste that played a role in determining the cohesion and mechanical properties. The C-S-H phase filled in most of the cement hydration product and had a random structure layer. The silane's molecular structure contained two different types of groups. The first group was the polar group which could make a strong chemical bond with inorganic particles. The second group was the non-polar one that could cause a reaction

with organic components. This could lead the inorganic and organic components to bond well.²² Feng H et al., (2016) suggest the use of silane as a coupling agent to bridge the organic and inorganic phases of C-S-H matrix and to connect some layers of C-S-H.²³ A study at a laboratory carried out by Aydinoğlu A et al., (2017) reveals that the silanization of silica filler using methacryloxy silane as a coupling agent could increase the mechanical properties (compression strength, angular flexural strength, flexural strength, and elasticity modulus) significantly, as a result of the chemical bond between the silanized filler and organic matrix.²⁴

The treatment using simulated body fluid (SBF) ringer lactate was intended to mimic the body fluid ion concentration state when the pulp capping material was applied to the teeth. When the dental materials were applied to the mouth cavity, the materials would absorb water and release the unreacted monomer. The absorbed water would be dispersed molecularly into the polymer matrix and play the role of a plasticizer, thus causing a decrease in the mechanical properties. The more water was absorbed, the more likely it was for hydrolytic degradation to occur.²⁵ The biocompatibility of dental material was affected directly by the water solubility property, which reflected the number of unreacted monomers released from the materials. Using SBF as a medium had a negative effect on mechanical strength and hardness because of the aggressive attack from free cations and anions in SBF. However, its mechanical strength and hardness values were still pretty high for the use of a biomaterial in a dental application.²⁶ This was not especially consistent with the result of this study. The highest and lowest flexural strength values in this research were found in those groups submerged in ringer lactate. The highest value was found in group B and this was possibly because of the effect of silane addition. Such an assumption was supported by a previous study that found that adding silane could hinder the transfer of unsaturated water. The methyl functional group in silane was hydrophobic with a relatively high molecular mass, allowing it to sterically hinder the transportation of water molecules in C-S-H gel pores.²⁷ The hydrophilic and hydrophobic properties of a material had something to do with water absorption. The higher water content

percentage was a result of the high hydrophilic property and lower water absorption percentage, directly proportional to the high hydrophobic property.²⁸ Based on this analysis result, it could be concluded that the silanization in groups A and B made their flexural strength mean values not significantly different.

The pulp capping material would receive the bending stress from the mastication force. It is important for clinicians to understand the flexibility property of the pulp capping material to be used. Flexural strength is a material's ability to bend which is obtained when its ultimate flexibility is achieved before the proportional limit. If the mastication load is still below the proportional limit, then no permanent deformation will occur and the material will return to its initial dimension. The test of this flexural strength produces a measurement between the compressive strength on the upper surface of a specimen and the tensile stress on the lower surface. The flexural strength test is preferred for brittle materials such as cement or composite, since the stress distribution is closer to simulating what happens during a clinical function.⁹

The use of ISO 4049 allowed the test results from various studies to be compared. This test protocol had been standardized, yet the research result data indicated great variations as proven by the high standard deviation. The variation could occur since some variables could affect the results such as voids and light intensity. The voids were formed because some air bubbles were trapped after the curing process. These voids could reduce the stress resulting from polymerization shrinkage and, at the same time, could be a pressure point when pressure was exerted and served as the initial point of fracture in the matrix and made it easy for a material to break. An increase in voids had the opposite effect on mechanical strength.²⁹ Based on this, to reduce the air trapped in the cement-resin mixture, it was suggested to use a vibrator during condensation and press the air to the mold wall. The intensity of light received could also affect the difference in results. In the current study, the light intensity was measured using a light meter every time the polymerization was to be performed. However, the distance between the tip and surfaces to be polymerized as well as the different angles of the light curing unit in each layer could affect the result of flexural strength

values. Jayanthi N et al., (2013) suggest that a 0 mm contact distance or direct contact with the sample surface will always have a high microhardness value in all samples tested. The light intensity declined as the tip of the light source moved further from the restoration surface. This could be addressed by adding the curing time.³⁰

This research had some limitations. First, its results could not be used as the only reference to rate the clinical performance of a material, since the ISO 4049 for flexural strength test only recommended submerging the sample for 24 hours. Another limitation was that the flexural strength test in this research was only shown for one mechanical cycle before a fracture eventually occurred. Such a specification did not reflect a material's long-term ability.^{31,32} A further study that uses TS-WPC- Bi₂O₃-UDMA mixture is needed to maintain the standardized protocol of a re-test. In conducting a further study, it is expected that researchers make a specimen with precision to avoid the formation of voids and curing time addition since they significantly affect the flexural strength value.

Conclusions

The flexural strength mean values of TS-WPC- Bi₂O₃-UDMA mixture were 77.260 MPa and 80.200 Mpa for a treatment using submersion in ringer lactate. The flexural strength mean values of resin-modified calcium silicate TheraCal LC were 48.525 Mpa and 17.579 MPa for a treatment using submersion in ringer lactate. There was a difference in the flexural strength values between TS-WPC- Bi₂O₃-UDMA mixture and resin-modified calcium silicate TheraCal LC. The flexural strength value of TS-WPC- Bi₂O₃-UDMA mixture was higher than that of resin-modified calcium silicate TheraCal LC. This was because of the filler composition, the monomer used, and the addition of coupling agent silane.

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Declaration of Interest

The authors report no conflict of interest.

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