ORIGINAL ARTICLE

Mixture of Indonesian White Portland Cements, Bi_2O_3, and Light Cured Methacrylate-based resin as a potential candidate for pulp capping material

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ABSTRACT

Introduction: The bioactivity property of pulp capping materials is determined by the hydroxyl ion release ability, which may alter the environmental pH to become alkaline. Calcium silicate-based materials are antibacterial, able to induce reparative dentin and have a good bacteria-tight seal. The poor handling of calcium silicate-based materials can be overcome by adding resin, so the materials will be easier to handle. Therefore, the purpose of this study was to synthesize a mixture of Indonesian White Portland Cements, Bi_2O_3, Light Cured Methacrylate-based Resin and analyze its hydroxyl ion release ability. **Methods:** The study was quasi experimental. Sample in this research is the Indonesian White Portland Cements. The Indonesian White Portland Cements, Bi_2O_3 mixture was prepared using the simple solution method and mixed with Light Cured Methacrylate-based Resin. Population was WPC, Bi_2O_3 and light cured methacrylate-based resin. The sample's ability to release hydroxyl ions was measured using a calibrated pH meter and titration test 60, 120, 180 and 320 minutes. Analyzed **Results:** This study showed that the mixture of Indonesian White Portland Cements, Bi_2O_3, Light Cured Methacrylate-based Resin had an initial pH of 11.04 and increased to its peak on 168 hours mark, or on day seven, to pH 11,77 with hydroxyl ion release value of $10^{-2.23}$

Conclusions: The mixture of Indonesian White Portland Cements, Bi_2O_3, Light Cured Methacrylate-based Resin has an alkaline pH, is able to release hydroxyl ion and has a potential candidate for pulp capping material.

KEYWORDS

Indonesia white portland cements, Bi_2O_3, light cured methacrylate-based resin, hydroxyl ion release ability

INTRODUCTION

Conservative dentistry aims to preserve the pulp vitality and pulp capping is one of several treatments that can preserve pulp vitality.[1,2] Bioactive materials are ones that are able to elicit a specific biological response at the interface of the material. According to this, bioactive materials in vital dental pulp therapy are remineralizing agents that can induce reparative dentinogenesis when applied close to the pulp. This result might be achieved due to the ability of bioactive materials in releasing hydroxyl ions which will make the pH become alkaline.[1] The pH value plays an essential role in determining bioactive materials properties. Since these materials have an alkaline pH of over 7.5, they have antimicrobial characteristics as well as the ability to initiate mineralization on dentin. Dentin mineralization is triggered by the alkaline pH which stimulates the alkaline phosphatase and Bone Morphogenic Protein-2 (BMP-2). The increase in pH value is perpendicular to hydroxyl ion concentrations that are produced.[2-5]

Nowadays, most bioactive materials that are being developed contain calcium silicate and Portland cement, and one of these materials is Mineral Trioxide Aggregate (MTA). Both calcium hydroxide and MTA produce hydroxyl ions, however studies have shown that the use of MTA was better when compared to calcium hydroxide. MTA is proven to have an alkaline pH, with the pH right after manipulation is 10,2 and will rise up to 12,5 after three hours. Although MTA has a lot of advantages as a pulp capping material, it is not the first material of choice for pulp capping since it is not cost-effective, difficult to manipulate, require a longer setting time and less compatible with other restorative materials.[6,7]

Type III Portland Cement or white Portland is the type of Portland cement used in MTA mixture. Indonesia is one of white Portland cement manufacturers, known as Indonesian White Portland Cement (IWPC), which makes this material readily available and more affordable. The radiopacity of this material can be improved by mixing white Portland cement with Bismuth Oxide (Bi_2O_3), so that radiography evaluation post pulp capping procedure is possible.[8]

To achieve easier manipulation and faster polymerization, Light Cured Methacrylate-based Resin (Urethane Dimethacrylate/UDMA), which is one of the polymer resin-based materials (PRM), can be added into the pulp capping mixture. The pulp capping material added by UDMA not only has bioactive properties but also has a good biological seal and is compatible with the restorative material.[9,10] A study by Erfanparast et al[11], showed that resin-modified Portland cement-based material (TheraCal-LC) had a success rate of 91,8%.[11,12] Despite the result of the study, one of the main disadvantages of the material used was the fairly high price, especially for developing countries. Based on this issue, the objective of this study was to synthesize a mixture of IWPC, Bi_2O_3, Light Cured Methacrylate-based Resin and analyze its hydroxyl ion release ability as well as its potential to be used as a pulp capping material.

METHODS

The study was quasi experimental. Materials and sample pre materials used in this study were Indonesian White Portland Cement (Tiga Roda Brand, PT. Indocement Tunggal Prakarsa), Bi_2O_3 (Shanghai Xinglu Chemical Technology Co. Ltd., Shanghai, China), isopropanol 99,9% (Merck® Emsure, Germany), camphorquinone (Sigma-Aldrich Inc., USA), Triethylene Glycol Dimethacrylate (TEGDMA) (Sigma-Aldrich Inc., USA), UDMA (Sigma-Aldrich Inc., Germany) and trimethoxysilyl propyl methacrylate (Sigma-Aldrich Inc.). Approximately 80 grams of IWPC was mixed with 20 grams of Bi_2O_3 and dissolved in 150ml 99,9% isopropanol in a chemical glass (beaker glass). This mixture then stirred for 30 minutes using a magnetic stirrer at 200 rpm until it was homogeneous. The homogeneous mixture was subsequently put into the centrifuge tube and centrifugation was done for 10 minutes at 5000 rpm.

After centrifugation, the pellet obtained was spread on petri dishes and dried with a vacuum drying oven at 60° C for 60 minutes. When the drying process was done, the IWPC- Bi_2O_3 powder was collected. This powder was then characterized with an X-Ray Fluorescence machine (XRF) NexCG brand, Rigaku type, before and after 24 hours of hydration.

The addition of resin material was done by mixing 1,16 grams dry the IWPC- Bi_2O_3 powder with 0,014 grams of camphorquinone, 0,66 grams of UDMA, 0,02 grams of TEGDMA and 0,14 grams of trimethoxysilyl propyl methacrylate. These materials were stirred with a magnetic stirrer until homogeneous and the final product was a mixture of the IWPC, Bi_2O_3, and light cured methacrylate-based resin. Afterwards, the mixture was re-characterized using the XRF machine, pH value was measured and a titration was conducted.

Hydroxyl ion release measurement. The release of Hydroxyl ions was measured by two methods, one was pH measurement and the other a titration test: (1). pH measurement, The mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin were polymerized using a light-curing unit with a light intensity of 1000 mW/cm^2 for 20 seconds. Next, the polymerized materials were prepped and immersed in 10 ml of deionized water. The liquid from these samples was stored in an incubator at 37° C and 100% humidity. The pH measurements were performed with a calibrated pH meter (pH - 207, Lutron Co., Taipei, Taiwan) right after the samples were polymerized, at 3 hours, 24 hours, 168 hours, 336 hours, 576 hours and 720 hours mark. Measurements of hydroxyl ion release from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin were calculated using the equation of Mc Mahon et al[13] ; (2). Titration test 60, 120, 180 and 320 minutes, A 50ml sample liquids from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin was prepared and poured into an erlenmeyer flask. The solution was stirred for 60, 120, 180 and 320 minutes.

The sodium hydroxide (1N, 2ml) was added in each sample until the pH reached a range of 12-13. Adding about 30 -50 mg murexide indicator and titration done with Na_2-EDTA standard solution to achieve color changes. The volume of Na_2-EDTA standard solution used is then recorded. Calcium levels are then calculated, and when the value is obtained in mg Ca/L units calculation of total calcium concentration percentage is done. If the total calcium concentration is known, hydroxyl ion release of the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin can be done by using this equation % OH= $Mr\ 2\ OH/Mr\ Ca$ x% Ca.[14]

RESULTS

Table 1. XRF Characterization Result of The Mixture of Indonesian White Portland Cements and Bi_2O_3

Elemental Composition	Concentration before hydration (%)	Concentration after hydration (%)
Magnesium (Mg)	7.91	5.17
Aluminum (Al)	6.01	4.62
Sulfur (S)	1.14	0.34
Potassium (K)	0.341	0.467
Chrome (Cr)	0.004	0.004
Ferrum (Fe)	0.277	0.271
Zirconium (Zr)	0.831	0.968
Bismuth (Bi)	12.1	13.5
Calcium (Ca)	55.9	63.4
Silica (Si)	15.4	11.3
Titanium (Ti)	0.069	0.062

Based on the result of XRF characterization (table 1), it showed that the highest concentration of mineral elements contained in the IWPC - Bi_2O_3 powder before and after hydration was Calcium (Ca) at 55,9% and 63,4%, respectively. Following Calcium (Ca), other mineral elements with highest concentrations were Silica (Si), Bismuth (Bi) and Magnesium (Mg). In the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin, Calcium also had the highest concentration with a percentage of 54%. There was a decrease in Calcium concentration in the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin compared to the IWPC - Bi_2O_3 powder. In the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin, two new elements were discovered, copper (Cu) and Zinc (Zn) with a percentage of 0,056% and at 0,254%, respectively.

The pH and hydroxyl ion release measurement from the sample of the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin are shown in Table 2.

Table 2. pH measurement and hydroxyl ion release value

Time	pH	hydroxyl ion release (Molar)
0	11,04	$10^{-2.96}$
3 hours	11,09	$10^{-2.91}$
24 hours	11,31	$10^{-2.69}$
168 hours	11,77	$10^{-2.23}$
336 hours	10,22	$10^{-3.78}$
576 hours	9,35	$10^{-4.65}$
720 hours	8,88	$10^{-5.12}$

According to the data in Table 2, the mixture of the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin had an initial pH of 11.04 but then increased to its peak on 168 hours mark, or on day seven, to a pH of 11,77 with hydroxyl ion release value of $10^{-2.23}$. After reaching its peak on the seventh day, the pH decreased to 8,88 on hour 720, or day 30, with a hydroxyl ion release value of $10^{-5.12}$.

Titration test was conducted to analyze calcium (Ca) ion release from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin. The titration test was a total of calcium ion release from the mixture sample on percentage (%) unit which can be seen in the blue bar chart in figure 1.

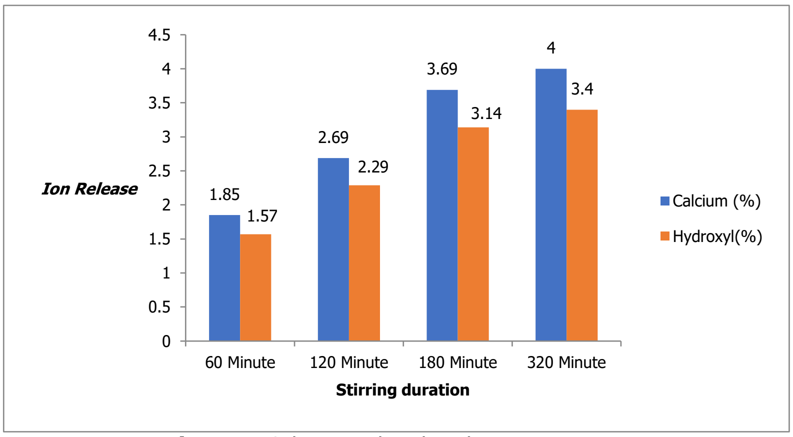

Figure 1. Calcium and Hydroxyl Ion Diagram

Based on figure 1 it can be seen that the longer the stirring, the higher total of calcium ion release from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin samples. The lowest calcium ion release (1.85%) was obtained when the stirring was done in 60 minutes, while the highest value (4%) was achieved when stirring was done in 320 minutes. Chemical analysis of hydroxyl ion (OH^-) broken down from $Ca(OH)_2$ allows the percentage of hydroxyl ion release (OH^-) calculated from the sample's total calcium value (Ca^{2+}). The measurement result can be seen in the red bar chart in Figure 1.

The percentage shown in the red bar chart is a result of hydroxyl ion calculation based on the titration test result towards the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin. According to the result shown in Figure 1, it can be seen that hydroxyl ion release is perpendicular to total calcium contained in the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin. The lowest hydroxyl ion is on 60 minutes stirring and keeps increasing up to 3,4% on 320 minutes stirring.

DISCUSSION

Calcium hydroxide is one of the widely used pulp-capping materials. This material has the ability to form reparative dentin along with its antimicrobial properties. However, calcium hydroxide also has a lot of disadvantages, such as tunnel defect formation and easily dissolved; therefore, nowadays numerous pulp capping materials are being developed. Based on a few studies, calcium silicate-based materials can stimulate formation of reparative dentine, have antimicrobial properties and good bacteria-tight seal. The difficulty in calcium silicate-based materials manipulation requires the addition of resin into these materials so it will be easier to handle.[15]

In several studies, incorporation of resin monomer matrix into calcium silicate-based pulp-capping material will decrease the Ca^{2+} and OH^- ions release as well as reducing the formation of apatite precipitation on the material's surface. This might be due to the decrease of calcium silicate solubility since the resin monomer had already polymerized into the material.[16]

Resin-based calcium silicate pulp-capping material is expected to undergo some shrinkage following polymerization due to the resin matrix component. Nevertheless, it has proven that hydration reactions from the particles of calcium silicate compounds will still take place because of the hydrophilic matrix component (e.g.TEGDMA). The fluid of dentin tubules will be absorbed into the resin-based calcium silicate pulp-capping material and compensate for the shrinkage, where the apatite compound will act as a seal between the material and tooth structure. Previous studies reported that resin-based calcium silicate pulp-capping material had a better seal and bond strength compare to MTA.[17]

Pulp tissue inflammation seems to be longer in duration and greater in intensity when resin-based calcium silicate material is applied. Several studies also showed that chronic pulp inflammation took place for a long time before finally subsiding around the 28th day. Like any other calcium silicate-based material, resin-based pulp capping material is capable of forming an apatite layer on the surface when in contact with physiologic fluid that contains phosphate. Overtime, the formation of this apatite layer will reduce the elution of resin component, which is considered to be cytotoxic, and will result in healing of the pulp tissue.[18]

Measurement of pH on the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin showed that this mixture had an initial pH of 11,04 a moment after polymerization, as shown in table 2. The pH value of this study's sample was much higher compared to Angelus MTA used in a study conducted by Natale et al[19], in which the initial pH was only 9,9. Gandolfi et al[22], reported that the pH of Proroot MTA on the first three hours was 11,52 and the pH of TheraCal was 10,96.[19,20,21] The pH value of the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin can be categorized as alkaline. Alkaline pH resulting from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin had allowed this material to possess antimicrobial properties as well as the ability to initiate mineralization in dentine. According to study by Gandolfi et al[22], although pulp capping materials contained resin, which is a hydrophobic monomer, these materials were able to maintain their ability to release hydroxyl ions in moist areas, such as dental pulp and dentin. These materials are also capable of interacting with the hydrophilic properties of dentine. In a study of water resorption by pulp capping materials that contain resin, the materials' formula had allowed water resorption, which has a role in initiating the stage of hydrating particle reaction on the Portland cement, so portlandite or calcium hydroxide is formed.[6,19,21]

In this present study, the pH raise was apparent from the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin. The pH value of this mixture increased from the initial pH of 11.04 to its peak on the seventh day to pH 11,77 then decreased on the 14th day to pH 10,22 (table 2). This result was in accordance with a study conducted by Lucjaz-Cepowicz et al (2017), which reported that in the first one hour gray ProRoot MTA had a pH value of 11,50 and continued to rise up to pH 12,17 at 504 hour mark. If this is compared to the result of Gandolfi et al[22] study that investigated TheraCal pulp capping material, the pH of TheraCal increased in the first three hours to pH 9,53 and then decreased to pH 8.12 on day 28.[2,22]

The hydroxyl ion release of the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin decreased after the seventh day towards the physiological pH which is a suitable environment for pulp cells and its metabolic activity to form a new reparative dentin. In this study, the hydroxyl ion was continuously released over time. Clinically, the effect of hydroxyl ion on vital tissues can induce deposition from hard tissues and has an antibacterial effect. When the chemical elements of pulp capping materials dissolve, they may penetrate into dentin and its surroundings.[20,22]

The mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin contains calcium silicate (table 1), like other formula of MTA-like calcium silicate materials, which make the content of calcium and hydroxyl ion higher compared to conventional calcium hydroxide. Hydration reaction of calcium silicate particles can trigger surface to dissolve by formation of calcium silicate hydrate gel and $Ca(OH)_2$, that coincide with calcium and hydroxyl ion release. Based on this, hydroxyl ion value can be obtained if calcium ion value is already known. In a study by Natale et al (2015), calcium ion value of pulp capping materials can be obtained through titration test. In this present study, ki calcium ion release obtained on biodentine and Angelus MTA is each 0,21 mmol/L and 0,22 mmol/L, and hydroxyl ion release at 0,42 mmol/L.[15,19] On this study, calcium and hydroxyl ion release from the IWPC - Bi_2O_3 - UDMA mixture is obtained after stirring at 60 minutes which is 1,85% and 1,57% and rises to 4% and 3,40% on 320 minutes of stirring (figure 1).

Resin contained in the mixture of IWPC, Bi_2O_3, and light cured methacrylate-based resin can decrease the material's ability of releasing ions, but based on a study by V.E.S Gajewski et al (2012), UDMA has a degree of conversion at 72,4% at 24 hours with water sorption ability at 42,3 μg/mm^3 and solubility at 20,4 μg/mm^3. This allows particles that are not polymerized to exist and can experience hydration together with dentinal liquids so the material can release ions. The slow bioactivity release process on pulp capping materials can be advantageous because it allows balance between material resorption for it to regenerate. Materials that can release calcium ion also have the ability to accelerate osteoblast differentiation.[8,11,19] Unpolymerized monomers will cause a longer inflammation in the pulp. However, over time the process of forming a layer of apatite will reduce inflammation and will result in healing of the pulp tissue.[18] Further study is needed to determine the degree of polymerization.

CONCLUSION

The mixture of Indonesian White Portland Cements, Bi_2O_3, Light Cured Methacrylate-based Resin has an alkaline pH, is able to release hydroxyl ion and has a potential candidate for pulp capping material.

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