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(RESEARCH ARTICLE)

# Colour changes and topography of resin modified glass ionomer cements with the addition of zinc oxide nanoparticles

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#### Abstract

**Background**: Resin-modified glass ionomer cement (RMGIC) is one of the restorative materials for cavity restoration in the anterior and cervical areas where aesthetics are crucial, especially for class III (anterior) cavity restorations. Adding zinc oxide nanoparticles (ZnONP) may enhance its mechanical and physical properties.

**Objective**: This study aims to evaluate the color change of RMGIC after the addition of ZnONP as much as 5%, 10% and 15% of the total mixture of RMGIC and ZnONP powder also to examines the topography of ZnONP distribution within RMGIC's interstitial space using scanning electron microscope (SEM).

**Methods**: Four groups of RMGIC were studied, a control group without ZnONP powder, 3 treatment groups with ZnONP powder as much as 5%, 10%, and 15%. Each group consists of 4 cylindrical samples (diameter of 10 mm, height of 2 mm). Color change was measured using VITA Easyshade V spectrophotometer and SEM was used for topography analysis. Data were analyzed with one-way ANOVA test and Tukey HSD post hoc test.

**Results**: One-way ANOVA test showed significant color differences (p<0.05) among all groups. Tukey HSD test showed significant color differences (p<0.05) in color change between control group and treatment groups also between group 2 and group 4. SEM observations confirmed ZnONP distribution in RMGIC's interstitial space in treatment groups.

**Conclusion**: Addition of 5%, 10%, and 15% ZnONP powder of the total mixture of RMGIC and ZnONP powders changed RMGIC's color to be brighter (lightness) and more saturated (chroma) also ZnONP distributed within RMGIC's interstitial space.

Keywords: Resin-modified glass ionomer cement; Zinc oxide nanoparticles; Color change; Topography

## 1. Introduction

Caries is one of the oral cavity diseases with the highest incidence in Indonesia. According to RISKESDAS 2018, the incidence of caries in Indonesia reached 88.8%. Tooth structure lost due to caries can be replaced with restorative materials [1]. One of the restorative materials that is often used to restore cavities is resin-modified glass ionomer cement (RMGIC).

Resin-modified glass ionomer cement was introduced as a restorative material modifying glass ionomer cement (GIC) with resin to extend working time and accelerate setting time [2]. The advantages of RMGIC have better aesthetics than glass ionomer cement (GIC) so it is used to restore cavities in the anterior (class III) and cervical (class V) regions [2]. These cements are also tooth-colored and available in various shades and have better translucency than GIC [3].

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Mechanical properties of RMGIC such as diametral tensile strength, flexural strength, compressive strength, compressive modulus have greater values than GIC [2]. The disadvantages of RMGIC, namely having a surface hardness that is still relatively low (64-85 KHN) when compared to type II GIC (87-177 KHN) [4]. The aesthetics of RMGIC are also still lower when compared to resin composites [3].

One attempt to improve the mechanical and physical properties of a restorative material is the addition of zinc oxide nano particles (ZnONP). Zinc oxide nano particles are made from zinc oxide produced through the sintering process of white zinc hydroxide [5]. Agarwal et al. (2018, p. 16071) showed that the addition of 1%, 5%, 10%, and 15% ZnONP powder to GIC powder increased the flexural strength and shear strength of GIC [6]. Research by AlMatar et al. (2022, p. 1515) showed that the addition of 5% ZnONP and 5% silver nanoparticles from the total mixture of RMGIC powder with ZnONP and silver nanoparticles resulted in stable color stability. The study also conducted a topography test using SEM to see the distribution of ZnONP particles in the interstitial space of RMGIC [7]. Topography tests using a scanning electron microscope (SEM) were also carried out to confirm the distribution of particles in the sample. The test shows the porosity and surface morphology of the sample.

Based on the background of the above problems, it is necessary to conduct further research on color changes in RMGIC with the addition of ZnONP as much as 5%, 10%, and 15%. This research will also confirm the topography of ZnONP distribution in the interstitial space of RMGIC using a scanning electron microscope (SEM).

## 2. Material and methods

Color change test and topography of RMGIC with the addition of ZnONP as much as 5%, 10%, and 15% of the total mixture of RMGIC and ZnONP powder using Fuji II LC Gold Label RMGIC, Japan and ITNANO ZnONP powder, Indonesia. This study examined 4 groups, namely the control group without the addition of ZnONP powder and 3 treatment groups with the addition of ZnONP powder as much as 5%, 10%, and 15%, respectively. Each group consisted of 4 replications of cylindrical samples (diameter 10 mm, height 2 mm) [8]. The tools and materials used in the color change test are analytical scales with accuracy of 0.001 g, centrifugal tubes, vortex mixer, glass slab, sample mold, agate spatula, plastic filling instrument, cement stopper, 1 kg weights, Light Emitting Diodes (LED) curing light, LED light meter, hand-piece micromotor, superfine diamond bur, glass pot container, incubator, VITA Easyshade V spectrophotometer, SEM, RMGIC (Fuji II LC Gold Label, Japan), ZnONP powder (ITNANO, Indonesia), celluloid strip, paper pad, distilled water, and tissue paper.

## 2.1. Sample Making

The ratio of RMGIC powder and liquid to make cylindrical samples with a diameter of 10 mm and a height of 2 mm follows the manufacturer's rules, namely 0.64 g RMGIC powder and 0.2 g RMGIC liquid. Details of the material ratio of each sample group can be seen in Table 1.

Group	RMGIC powder	ZnONP powder	<b>RMGIC liquid</b>
Group 1 (Control)	0.6400 g	-	0.2 g
Group 2	0.6080 g	0.0320 g	0.2 g
Group 3	0.5760 g	0.0640 g	0.2 g
Group 4	0.5440 g	0.0960 g	0.2 g

Table 1 Research material ratio of each sample group

The research material ratio of each sample in Table 1 was used for 1 repetition per group. Resin-modified glass ionomer cement and ZnONP powders in group 2 were weighed according to the ratio in Table 1. The mixture of RMGIC and ZnONP powder was poured into a centrifugal tube, then mixed using a vortex mixer for 5 minutes. The mixed RMGIC-ZnONP powder was poured into a glass pot container. Mixing was done in the same way in groups 3 and 4.

RMGIC powder for the control group or RMGIC-ZnONP powder for the treatment group was mixed with RMGIC liquid until homogeneous. The cement mixture was put into the sample mold that had been placed on the glass slab and celluloid strip using a plastic filling instrument and cement stopper. The top of the cement mixture was given a celluloid strip, thin glass slab, and 1 kg weights for 30 seconds. The cement mixture was cured for 20 seconds. The sample was released from the sample mold and the parts were smoothed using a fine finishing diamond bur on the excessive side.

#### 2.2. Sample Storage

All samples in one group were placed in a glass pot container and the container was closed tightly, then left for 24 hours. Distilled water was poured into the glass pot container containing the samples until all samples were submerged and placed in an incubator at 37°C for 24 hours. All glass pot containers were removed from the incubator after 24 hours [9]. All samples were removed from the glass pot containers containing distilled water and dried using tissue paper to perform color change tests and topography tests.

## 2.3. Sample Testing

The samples were tested for color change using the VITA Easyshade V Spectrophotometer. The color measurement results will show the E (color) value which is the measurement result of the L (Lightness/value), C (Chroma), and H (Hue) values. The E value between groups is calculated using the formula  $\Delta E^* = \sqrt{(\Delta L)^2 + (\Delta C)^2 + (\Delta H)^2}$  to determine the total color change that occurs.

The sample was topographically tested using a flexSEM1000 with a magnification of 10000× to confirm the distribution of particles in the sample.

#### 3. Results and discussion

Research on the color change and topography of RMGIC with the addition of ZnONP powder has been carried out. The average value of the results of the study which is getting bigger indicates a change in color to be lighter in the sample. The results of the color change test for each sample group can be seen in Table 2.

**Table 2** Mean and standard deviation of color change of each sample group.

Research Group	n	Average	Standard Deviation
Group 1 (Control)	4	2.2575	1.32303
Group 2	4	9.8900	0.71726
Group 3	4	11.5393	0.57097
Group 4	4	12.9300	1.41112

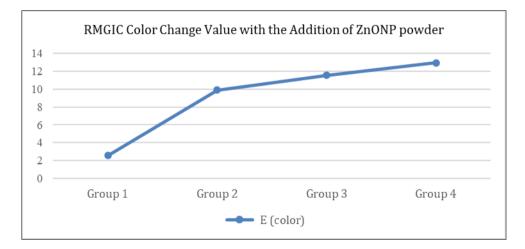


Figure 1 Graph of the average color change value of RMGIC with the ZnONP powder

This study was conducted to evaluate the color change of RMGIC without and with the addition of ZnONP powder as much as 5%, 10% and 15% of the total mixture of RMGIC and ZnONP powder. Color change is an important factor that can affect the quality of the restoration. Such changes can be caused by the penetration of colored substances into the material [2].

The color changes that occurred in RMGIC after adding 5%, 10% and 15% ZnONP powder to the total mixture of RMGIC and ZnONP powder were interpreted visually with the help of the Munsell color system. The lightness/value indicates the brightness of the color, so an increasing value indicates a change in brightness to be lighter and a decreasing value indicates a change in brightness to be darker. The chroma value indicates the saturation of the color, so increasing values indicate a change in color saturation to higher (intense) and decreasing values indicate a change in color saturation to lower (faded). The hue value indicates the direction of the dominant color change. An increasing hue value indicates a change in color towards yellowish, while a decreasing value indicates a change in color towards reddish.

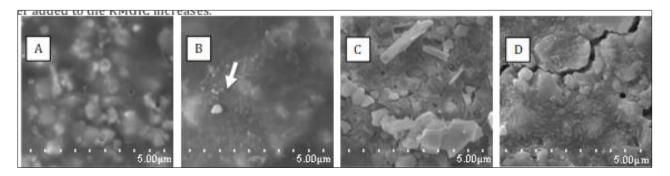
The results of statistical analysis of one-way Anova test of research data showed significant differences in color changes of RMGIC with the addition of ZnONP powder as much as 5%, 10%, and 15% of the total mixture of RMGIC and ZnONP powder in all sample groups. This shows that the results of adding ZnONP as much as 5%, 10%, and 15% to RMGIC cause the sample to change color significantly.

1	2	3	4
-	0.000*	0.000*	0.000*
-	-	0.184	0.008*
-	-	-	0.304
-	-	-	-
	1 - - - -	1 2	- 0.000* 0.000*

Table 3 Tukey HSD post hoc test data analysis results

\*: Significant difference

The results of the statistical analysis of the Tukey HSD post hoc test (Table 3) showed a significant difference in the value of color change (E) between group 1 and group 2, group 3 and group 4, respectively. The results of the statistical analysis showed that there was a significant change in the color of RMGIC to become lighter and more intense with the increase in the addition of ZnONP powder by 5%, 10%, and 15% of the total mixture of RMGIC and ZnONP powder. This is likely due to the white ZnONP particles filling the interstitial space of RMGIC changing the surface color to be lighter and more intense as the percentage of ZnONP powder added to RMGIC increases. The bond that occurs between ZnONP and RMGIC is caused by the reaction between ZnONP particles and polyacrylic acid in the RMGIC liquid. Zinc oxide nano particles have a surface area that is significantly larger than the original size of the particle so that the nano particles have a high surface free energy. This causes the ZnONP particles to bond strongly with each other or with RMGIC particles [10]. The bond between ZnONP particles and RMGIC particles occurs chemically, namely covalent and ionic bonds [11]. The SEM observation of the control group in Figure 2A shows the empty RMGIC interstitial space in black (yellow arrow). Figure 2B-D shows white ZnONP particles scattered throughout the RMGIC interstitial space (white arrows). The number of ZnONP particles filling the RMGIC interstitial space also increases as the percentage of ZnONP powder added to the RMGIC increases.



**Figure 2** SEM observation results with 10000× magnification A. Group 1, B. Group 2, C. Group 3, D. Group 4. (Yellow arrows indicate RMGIC interstitial spaces, white arrows indicate ZnONP particles filling RMGIC interstitial spaces)

The results of statistical analysis of Tukey HSD post hoc test of RMGIC color change research data with the addition of ZnONP powder showed significant color changes between group 2 and group 4. The addition of ZnONP powder at 10% intervals between group 2 and group 4 caused significant color changes. This is confirmed by the SEM observation results in Figure 2B and Figure 2D. Figure 2D shows that more ZnONP particles bind and fill the interstitial space of RMGIC than Figure 2B. Insignificant color changes were shown between group 2 and group 3 and between group 3 and

group 4. This is likely due to the addition of ZnONP powder at 5% intervals between group 2 and group 3 and group 3 and group 4 did not show significant color changes. The addition of ZnONP at 5% intervals filled the interstitial space, but could not show a difference in color change, while the addition of ZnONP at 10% intervals showed a significant difference in color change.

**Table 4** Total Color Chage Results ( $\Delta E^*$ )

Group	1	2	3	4
1	-	8.028	9.713	11.078
2	-	-	1.91	3.167
3	-	-	-	1.46
4	-	-	-	-

Color change can be visually detected by the observer if the total color change value ( $\Delta E^*$ ) = 1.2 (ISO) and 0.4 – 4.0 [4]. The results of this study show that the total value of color change ( $\Delta E^*$ ) between group 1 and group 2 is 8.028 (Table 4). This can be interpreted that the color change is visually detectable by the observer. Color change is aesthetically acceptable when  $\Delta E^*$  = 2.7 (ISO) dan 2.0 – 6.8 [4]. The value of  $\Delta E^*$  between group 1 and group 2 of 8.028 (Table 4) means that the color change is not aesthetically acceptable. The increasing addition of ZnONP powder to RMGIC in group 2, group 3, and group 4 caused the  $\Delta E^*$  value to increase. This indicates that the addition of ZnONP powder by 5%, 10%, and 15% to RMGIC from the total mixture of RMGIC and ZnONP powder causes a color change that can be detected by the observer visually and is not aesthetically acceptable.

## 4. Conclusion

Based on the results of the study, it can be concluded that the addition of ZnONP powder as much as 5%, 10%, and 15% of the total mixture of RMGIC and ZnONP powder changes the color to be lighter (lightness) and saturated (chroma). The color change of RMGIC with the addition of ZnONP can be detected by observers and is not aesthetically acceptable so it is not recommended for anterior tooth restoration. SEM observation showed that ZnONP particles were scattered in the interstitial space of RMGIC.

#### **Compliance with ethical standards**

#### Disclosure of conflict of interest

No conflict of interest to be disclosed.

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