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Original article

Evaluation of the physical properties of glass ionomer cement modified by ethanolic extract of propolis

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ABSTRACT

Background: *Glass ionomer cement (GIC) is a dental material often used in clinical practice. However, its use is limited due to its drawbacks. Natural resources such as propolis have been used to mainly enhance GIC's antibacterial properties, but other properties attributed to this enhancement also require evaluation.* ***Purpose:*** *The study aims to evaluate the surface hardness, the surface roughness, and the water absorption of GIC containing ethanolic extract of propolis (EEP) from Trigona spp.* ***Methods:*** *Samples in this study were divided into four groups: GIC (control group); GIC + 25% EEP; GIC + 30% EEP; and GIC + 35% EEP. Surface roughness was measured using a surface roughness tester. Surface hardness was measured using a micro-Vickers hardness tester. Water absorption was measured by weighing the samples before and after immersion in distilled water. Surface characterization was carried out using a scanning electron microscope (SEM). Data was statistically analyzed using the Shapiro–Wilk normality test and one-way variance analysis along with the post-hoc Tukey's test to determine significant differences between all four samples.* ***Results:*** *The statistical test showed significant differences in surface hardness and surface roughness between the control group and the GIC + EEP group. However, the test demonstrated no significant differences in water absorption between the control group and the GIC + EEP group.* ***Conclusion:*** *The addition of EEP to conventional GIC can affect its physical properties.*

Keywords: *glass ionomer cement; hardness test; propolis; surface roughness; water absorption*
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INTRODUCTION

Glass ionomer cement (GIC) has been used for more than 40 years in dentistry glass ionomer cement.[1] It has wide application in dentistry as a luting, restoration, and lining material.[2] GIC is one of the most frequently used materials for cavity lining and cementation for fixed partial dentures.[3,4] Compared to other dental restorative materials, GIC offers advantages such as fluoride release, excellent biocompatibility with pulp, and chemical bonding with the tooth structure. Fluoride release in GIC can protect teeth from secondary caries.[4] However, despite these advantages, the material has some drawbacks, including sensitivity to moisture, low compressive strength and wear resistance, and high solubility.[5,6]

Recently, many modifications have been made to GIC's composition.[1] These modifications aim to enhance GIC's properties, including physical and mechanical,[7] by adding various materials such as hydroxyapatite, metal, and propolis.[8]

Propolis is a natural substance produced by honeybees and has many benefits that include antibacterial, anti-inflammatory, and antioxidant effects.[9] Propolis is obtained by extracting honeycombs[10] and is used in dentistry as a toothpaste and mouth rinse for its antibacterial activity. *Trigona* spp., a type of honeybee, produces higher amounts of propolis.[11] The most commonly used propolis is ethanolic extract of propolis (EEP).[9] Studies have proven that adding propolis to GIC can enhance GIC's antibacterial effect on *Streptococcus mutans.*[12] A study conducted by

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El Ghazouly et al.[12] found that GIC with a higher EEP concentration (50%) exhibited lower microhardness than GIC with a lower EEP concentration (25%).GIC itself showed minimum sensitivity in caries depth, indicating that adding propolis to GIC as lining cement provides optimal results in minimizing sensitivity and improving its antibacterial properties.[13] Other studies have investigated the effect of EEP on GIC's properties.[12] Although research on GIC containing propolis has been documented, further research is necessary to evaluate propolis' effect on GIC. In addition to antibacterial properties, GIC as a luting and lining cement (GC Fuji I) requires properties such as suitable surface roughness to promote biological response, appropriate surface hardness to ensure it remains intact after being applied inside the cavity, and less water absorption to improve its mechanical property such as hardness.[12] This study aims to evaluate the surface roughness, the surface hardness, and the water absorption of GIC (GC luting and lining cement) that contains EEP from *Trigona* spp.

MATERIALS AND METHODS

EEP was obtained by dissolving propolis from the *Trigona* spp. honeybee in ethanol.[13] GC Gold Label luting and lining cement was obtained from the GC Corporation in Tokyo, Japan. The study was conducted from November to December 2022 at the biochemistry laboratory, Bogor Agricultural University, Bogor, for extract formulation and at the DMTCore Laboratory, Universitas Trisakti, Jakarta, for sample testing.

Initially, GIC powder and liquid were mixed with EEP on a paper pad for 45 seconds using a plastic spatula. The mixture was then poured into a cylindrical mold with a diameter of 10 mm and a thickness of 2 mm. Samples were divided into three groups based on EEP concentrations of 25%, 30%, and 35% (Table 1).

Surface hardness was measured using a micro-Vickers hardness tester (HMV-G31DT, Shimadzu, Tokyo, Japan) with a 200-gram indenter load applied for 5 seconds. Each sample was measured in three different places, and the average value was noted. Surface roughness in terms of roughness average (Ra) was measured using a surface roughness tester (Suntronik S-100 Series, Taylor Hobson, Leicester, UK). A stylus was placed on the sample surface until it was leveled, the tester was started, and the Ra value was noted. The resulting measurement in each sample was the average value taken from three different places. Water absorption was measured based on the equation below:[14,15]

$$W = m2 - m1$$

W: Water absorption of the samples (g)
m1: Initial mass before immersion (g)
m2: Sample mass after immersion (g)

Table 1. GIC and EEP composition of each group

Group	EEP (g)	GIC Liquid (g)	GIC Powder (g)
GIC (control)	-	0.189	0.386
GIC + 25% EEP	0.047	0.142	0.386
GIC + 30% EEP	0.057	0.132	0.386
GIC + 35% EEP	0.066	0.123	0.386

Table 2. Effect of EEP on GIC's surface hardness, surface roughness, and water absorption

Group	Surface Hardness (VHN)	Surface Roughness (μm)	Water Absorption
	Mean ± SD	Mean ± SD	Mean ± SD
GIC (control)	60.34 ± 2.94	0.44 ± 0.10	0.0049 ± 0.0055
GIC + 35% EEP	44.67 ± 0.59	0.50 ± 0.08	0.0130 ± 0.0010
GIC + 30% EEP	38.32 ± 0.22	0.81 ± 0.07	0.0120 ± 0.0162
GIC + 35% EEP	37.14 ± 1.53	0.91 ± 0.16	0.0046 ± 0.0010

Table 3. Comparison of GIC's surface roughness, surface hardness, and water absorption between all groups

Group		Significance (p-value)		
		Surface Roughness	Surface Hardness	Water Absorption
GIC (control)	GIC + 25% EEP	0.879	0.000*	0.557
	GIC + 30% EEP	0.001*	0.000*	0.652
	GIC + 35% EEP	0.000*	0.000*	1.000
GIC + 25% EEP	GIC (control)	0.879	0.000*	0.557
	GIC + 30% EEP	0.007*	0.000*	0.998
	GIC + 35% EEP	0.001*	0.000*	0.520
GIC + 30% EEP	GIC (control)	0.001*	0.000*	0.652
	GIC + 25% EEP	0.007*	0.000*	0.998
	GIC + 35% EEP	0.613	0.759	0.614
GIC + 55% EEP	GIC (control)	0.000*	0.000*	1.000
	GIC + 25% EEP	0.001*	0.000*	0.520
	GIC + 30% EEP	0.613	0.759	0.614

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Sample weights were initially measured (m1), placed in a container, immersed in distilled water, and then stored at a temperature of 37°C. After 24 hours, the samples were removed from the container and dried with filter paper, and their weight was measured (m2). The surface morphology was observed with an SEM (JSM 6510-LA, JEOL, Tokyo, Japan) with magnifications of 2000x, 3500x, and 5000x. Before SEM analysis, the samples were coated with gold-palladium alloy. The obtained data was statistically analyzed using the Shapiro–Wilk normality test and one-way variance analysis along with a post-hoc Tukey's test to determine significant differences between all of the samples. The significance level was $p<0.05$.

RESULTS

The results of this study, including surface hardness, surface roughness, and water absorption, are shown in Table 2. Differences between all groups are shown in Table 3. Significant differences in surface hardness and

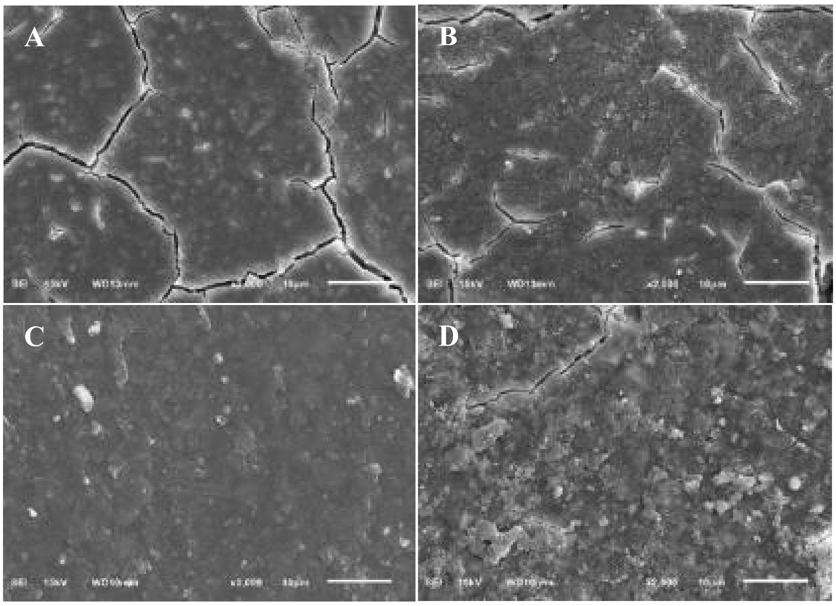

Figure 1. Surface characterization under SEM with 2000x magnification. (A) GIC without EEP; (B) GIC + 25% EEP; (C) GIC + 30% EEP; (D) GIC + 35% EEP.

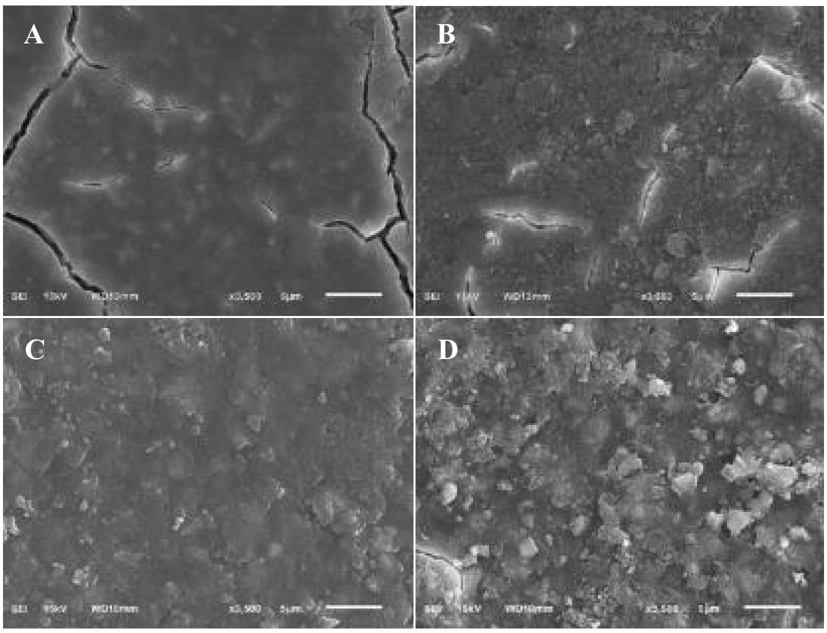

Figure 2. Surface characterization under SEM with 3500x magnification. (A) GIC without EEP; (B) GIC + 25% EEP; (C) GIC + 30% EEP; (D) GIC + 35% EEP.

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surface roughness were found between some groups, but there were no significant differences in water absorption among all groups.

Results also showed that there were statistically significant differences between the surface roughness scores of the control group and the experimental groups. The control group (GIC) had a mean surface roughness of 0.44 μm compared to 0.50 μm for the GIC + EEP 25% group, 0.81 μm for the GIC + EEP 30% group, and 0.91μm for the GIC + 35% EEP group (Table 2). The surface roughness scores aligned with the SEM results in this study. SEM examination was carried out with 2000x magnification (Figure 1), 3500x magnification (Figure 2), and 5000x magnification (Figure 3). The surface morphological structure of samples with a higher EEP concentration appeared to be transformed, exhibiting a rougher surface. This result aligned with the surface roughness value shown in Table 3.

Water absorption scores in this study showed no significant differences between the control group and the experimental groups (Table 2). However, surface hardness results demonstrated a significant difference in mean surface hardness between the control group and the experimental group (Table 2).

DISCUSSION

GIC is a commonly used dental restoration material in dentistry due to its advantages such as fluoride release and ability to bond to the tooth's surface in a moist environment.[9] It also exhibits adhesion, dimensional stability, and a coefficient of thermal expansion similar to dental hard tissues.[16] However, GIC cannot be used as restoration material in teeth that receive high occlusal forces because of its brittleness.[9,17] Therefore, some of GIC's properties need to be improved.[9]

Propolis is a thin film that covers honey and bee bread. Bees use propolis to protect themselves from predators.[11] Many studies have reported that propolis has antibacterial, antiviral, anti-inflammatory, antifungal, and antioxidant effects.[18] Propolis has been processed and incorporated into several everyday consumer products such as toothpaste, mouthwash, soap, and cosmetics. It contains substances called flavonoids that help reduce inflammatory reactions and improve the human immune system.[17] Several forms of propolis are available on the market, but the most common form is EEP.[9] Propolis extraction with 70% ethanol produces the highest propolis antioxidant activity.[10] Research has shown that EEP has antibacterial effects on *Streptococcus mutans.*[9] A study by Topcuoglu et al. stated that GIC containing 25% and 50% EEP showed antibacterial activity against *Streptococcus mutans.*[19] Another study concluded that adding 25% and 50% EEP to GIC did not alter the microleakage. Although there has been significant research about the antibacterial effects of GIC containing propolis, further research on its other properties in preventing material deterioration is required.[19]

Surface hardness is a property that demonstrates a dental material's wear resistance and durability.[9] Altunsoy et al. evaluated the microhardness of GIC with EEP concentrations of 10%, 25%, and 50% and showed that the microhardness value of GIC containing EEP increased as the ratio of EEP increased.[9] However, the current study showed that adding EEP to GIC at concentrations of 25%, 30%, and 35% reduced surface hardness. Another research revealed that the compressive strength of GIC containing propolis decreased. GIC's reduced properties after adding propolis could be due to changes in GIC's setting reaction.[17] Propolis contains flavonoids, a composition that has antibacterial

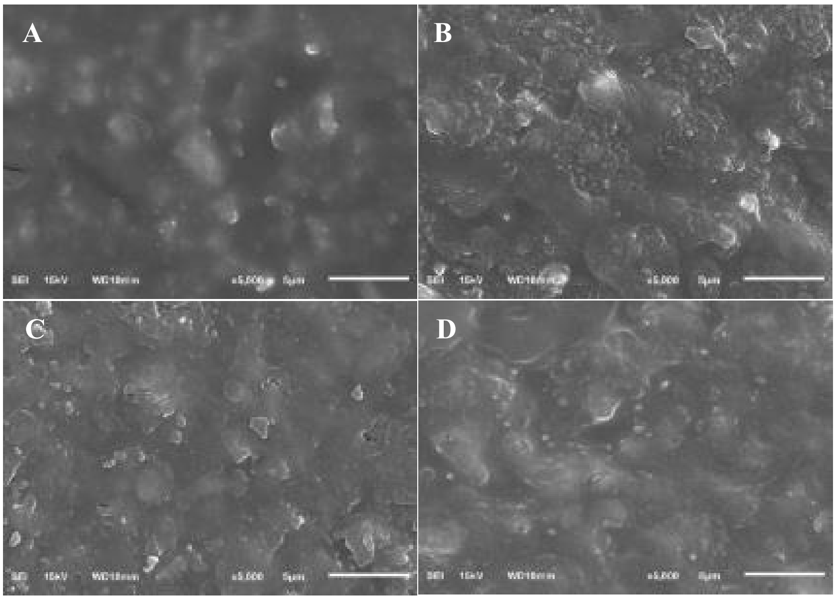

Figure 3. Surface characterization under SEM with 5000x magnification. (A) GIC without EEP; (B) GIC + 25% EEP; (C) GIC + 30% EEP; (D) GIC + 35% EEP.

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effects. However, it does not have functional groups that can bond with functional groups in the GIC.[20] Propolis might cause unreacted polyacrylic acid particles and glass particles to increase,[21] resulting in a delayed setting time of GIC containing propolis. Adding EEP to GIC liquid changes the liquid's consistency, making it less viscous and taking longer to work.[17] This study showed that the highest concentration of EEP (35%) had the lowest value of microhardness. This finding aligns with El Ghazouly et al., which concluded that GIC with 50% EEP had a lower microhardness value than GIC with 25% EEP.[12]

The current study's surface microhardness results also aligned with the surface roughness results and the surface characterization using an SEM. Rough surfaces may degrade the material, encourage plaque accumulation, and cause the material to lose its shine.[22] The control group (GIC) had the lowest surface roughness value, while the GIC + 35% EEP group had the highest surface roughness value. A higher percentage of EEP added to conventional GIC correlated with a higher surface roughness value, meaning more EEP added to conventional GIC results in a rougher surface. This result was also demonstrated by the SEM examination. Conventional GIC had a homogeneous surface texture, while GIC + EEP had a rough texture, with GIC + 35% EEP exhibiting the roughest texture.

Wulandari et al. conducted a study on the porosity of GIC with 2.5%, 5%, and 10% of added gourami fish scale powder (GFSP).[23] Higher percentages of GFSP showed a higher porosity level, which was likely due to the absence of a bond between the particles. Higher concentrations of GFSP may render the reaction ineffective, reducing the matrix that is formed.[23] Another study added 5% and 10% of nano chitosan to GIC and found that SEM images showed that GIC containing a higher percentage of nano chitosan exhibited a rougher surface, which may have been caused by many undissolved particles.[24] Based on these studies, researchers concluded that higher percentages of ingredients added to GIC may interfere with the bonds between particles, which can also affect GIC's properties. Still, further research is necessary. GIC with added EEP had reduced hardness compared to the GIC control group, indicating a lower mechanical property. However, as lining cement, this low mechanical property would not significantly affect GIC's performance since its application would be covered by restoration (e.g., a resin composite).

In addition to its physical properties, a restorative material must be resistant to the environment in the oral cavity, such as water absorption. Water absorption can alter the physical and mechanical properties of the material through hygroscopic expansion and plasticization effects.[25] Water absorption might change a restorative material's matrix structure and volume, affecting its durability. The current study obtained the water absorption value by measuring the samples' weight gain from the elution of monomers and the diffusion of water molecules and other small molecules. However, the study showed that adding EEP did not affect GIC's water absorption. In conclusion, adding EEP increased GIC's surface roughness and decreased its surface hardness but did not affect its water absorption of the GIC. *In vitro* and *in vivo* evaluations need to be conducted to study the effects of adding propolis on GIC's antibacterial properties.

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