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Evaluation of Microhardness and Roughness of Conventional and Experimental Glass-Ionomer Cements Modified with Sesamum Indicum Extract

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Abstract

Purpose: The purpose of this research was to evaluate the influence of Sesamum indicum extract on the microhardness and roughness of two formulations of the cement of glass ionomer (GIC). **Patients and methods:** A total of 60 samples of GIC were prepared according to the test type. The samples were divided into two equal groups ($n = 30$) according to the type of GIC; group (1): Conventional GIC, group (2): Experimental GIC. Each group was further subdivided into three subgroups ($n = 10$) according to the ratio of Sesame oil, subgroup (A): GIC without Sesame oil, subgroup (B): GIC with Sesame oil ratio 1 (v/v%), subgroup (C): GIC with Sesame oil ratio 4 (v/v%). All the samples were submitted to evaluate the following properties microhardness and roughness. **Results:** GICs modified with 1 (v/v%) Sesame oil significantly reduced surface roughness but a non-significant decrease in microhardness. GICs modified with 4 (v/v%) Sesame oil exhibited Sesame oil showed a considerable increase in surface roughness, but no significant decrease in microhardness. Experimental GIC exhibited nonsignificantly higher microhardness but was nonsignificantly lower in surface roughness. **Conclusion:** Sesame oil enhanced the surface roughness of GIC at low concentrations, while it showed a detrimental effect on surface roughness at high concentrations and with a nonsignificant effect on microhardness at both concentrations for both GIC formulations.

Keywords: Glass-ionomer cement, Microhardness, Roughness, Sesame oil, Sesamum indicum extract

1. Introduction

The use of resin-based and water-based glass ionomer cement (GIC) for aesthetic restorative procedures is being considered as an alternative to old metallic restorations due to their detrimental effects on patient health. GIC has unique properties such as chemical adhesion, biocompatibility, remineralization of dental tissues, coefficient of thermal expansion similar to the tooth, and anti-cariogenic effect through fluoride release [1]. For these reasons, it is commonly used in the dental field for numerous uses, including luting agents, fissure sealants, orthodontic bracket adhesives, and liners and bases [2].

Despite their advantages, conventional GICs can only be used at specific bearing areas with minimal stress, due to their limitations, which include susceptibility to dehydration, and poor physical and mechanical properties [3]. Numerous experimental trials involving adding filler components such as silica, zirconia, glass fiber, hydroxyapatite, silver amalgam particles, and bioactive glass particles have been attempted since the introduction of GIC. The physico-mechanical characteristics of cement have been considerably altered by the addition of these filler particles to GIC [4].

To enhance the qualities of glass ionomer, natural oils—particularly sesame oil, which is rich in minerals including manganese, copper, zinc, iron,

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magnesium, and phosphorus have been employed [5]. Sesame oil has been demonstrated to possess antibacterial effects that decrease bacterial adherence to tooth structure while enhancing oral hygiene because of its viscosity and emulsification process. It also contains vitamin E and other unsaturated fatty acids which minimize the free-radical damage of the oral tissues [6].

Although previous studies investigated the influence of sesame oil on remineralizing effect, antibacterial and antioxidant activities [5–7], however, a lack of knowledge about its impact on the properties of GIC that was modified with sesame oil. Thus, the current study aimed to examine whether sesame oil could strengthen GIC. The null hypothesis was that the mechanical properties of GIC could not be improved by sesame oil.

2. Patients and methods

Two materials were used in this study; conventional GIC powder and liquid (Prevest Den Pro Limited, Jammu, India), experimental GIC prepared by sol-gel method, and Sesame oil (Harraz herbal pharmaceutical company, Cairo, Egypt). The study was approved by the Research Ethics Committee, Faculty of Dental Medicine for girls, Al-Azhar University, with the code (REC-MA-24-01).

2.1. Sample size calculation and sample grouping

Based on power analysis and sample size calculation in a previous study [7], 60 samples of GIC were used in the current study. The samples were divided into two equal groups ($n = 30$) according to the type of GIC. According to the sesame oil ratio, we divided each group into three subgroups ($n = 10$). All the samples were submitted to evaluate the following properties microhardness and roughness.

Group (1): Conventional-GIC ($n = 30$).

Subgroup (A): Conventional GIC without sesame oil (control) ($n = 10$).
Subgroup (B): Conventional-GIC with sesame-oil ratio 1 (v/v%) ($n = 10$).
Subgroup (C): Conventional GIC with sesame oil ratio 4 (v/v%) ($n = 10$).

Group (2): Experimental GIC ($n = 30$).

Subgroup (A): Experimental GIC without sesame oil (control) ($n = 10$).
Subgroup (B): Experimental GIC with sesame oil ratio 1 (v/v%) ($n = 10$).
Subgroup (C): Experimental GIC with sesame oil ratio 4 (v/v%) ($n = 10$).

2.2. Preparation of glass powder by sol-gel method

Glass powder was prepared by the sol-gel method. Various reagents and solutions with specific concentrations were selected as a source for SiO_2, Al_2O_3, CaO, and P_2O_5, Na_2O) (all reagents were manufactured by Sigma-Aldrich, St. Louis, MO, USA), for the preparation of 100 gm of sol-gel formulated GIC powder (50% SiO_2, 15% Al_2O_3, 20% CaO, and 5% P_2O_5, 10% Na_2O [8,9].

GIC was modified with different concentrations of Sesame oil; 1 and 4 (v/v%) using the magnetic stirrer (Labnet AccuPlate Digital Hotplate Stirrer, Labnet International) for 24 h to obtain a homogenous_mix.

2.3. Samples preparation for microhardness test and surface roughness test

The samples were prepared using a specially designed Teflon mold (6 mm diameter and 4 mm height) the powder for the conventional and experimental GIC was mixed with different liquid formulations according to the manufacturer's instructions. Then, each mixed cement was placed in the Teflon mold on a glass plate covered with celluloid strips pressed with another glass plate, and left for setting. All samples were incubated at 37 °C in deionized water for 24 h until testing [7].

2.4. Testing procedures

2.4.1. Microhardness assessment

The microhardness tests were conducted by a digital Vickers microhardness tester (Model-HVS-50, Laizhou-Huayin-Testing-Instrument Co., Ltd. China). A diamond indenter was loaded with 50 g for 10 s. A microhardness equation will be used to calculate the Microhardness based on three measurements [10] (Fig. 1).

$$HV = 1.854 \cdot P / d^2$$

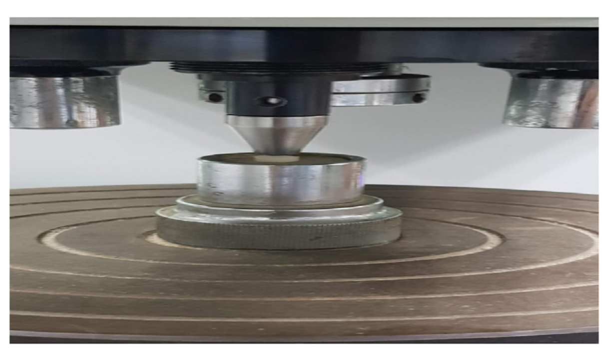

Fig. 1. Microhardness test.

where HV is Vickers hardness in Kgf/mm^2, P is the load in Kgf and d is the length of the diagonals in mm.

2.4.2. Surface roughness assessment

Surface roughness was estimated using optical profilometry on disc-shaped samples (6 mm × 4 mm) using a USB-digital microscope with a built-in camera (U500 × DigitalMicroscope, Guangdong, China) connected to a compatible personal computer.

A three-dimensional (3D) image of the surface profile of cropped images for the specimens was created and analyzed using WSxM software. Within the WSxM software, all limits, sizes, frames, and measured parameters are expressed in pixels. Therefore, system calibration was done to convert the pixels into absolute real-world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software.

Subsequently, 3D images were collected for each specimen, in the central area and the sides at the area of 10 μm × 10 μm. This area was chosen based on the dimension of the typical bacteria expected to adhere to the restoration surface in vivo. WSxM software was used to calculate an average of heights (Ra) expressed in μm, which can be assumed as reliable indices of surface roughness [11].

2.5. Statistical analysis

The Windows program Graph Pad Instat (Graph Pad, Inc.) was used to analyze the data. *P* values less than 0.05 were regarded as statistically significant. The standard deviation and mean were used to express continuous variables. Following confirmation of the homogeneity of variance and the normal distribution of errors, a one-way analysis of variance (ANOVA) was carried out, and if significant results were found, Tukey's post-hoc test was run. For comparison pairings, a Student *t*-test was conducted. The effects of each element (material and concentration) were compared using two-way ANOVA. Large effect sizes for main effects and pairwise comparisons could be detected with the sample size ($n = 10$/group), with a sufficient level of power set at 80% and a 95% confidence level. Correlation between microhardness and surface roughness was detected by Pearson linear correlation.

3. Results

3.1. Microhardness test results (Kg/um^2)

The Microhardness-test results (kg/mm^2) for both GIC groups with different sesame oil extract concentrations showing the mean values and SD are summarized in Table 1.

3.1.1. Conventional GIC (group 1)

The group 1 A recorded the highest mean value of Vickers hardness (49.18 kg/mm^2) followed by group 1 B which recorded the mean value of 49.11 kg/mm^2 while group 1C recorded the lowest mean value 48.46 kg/mm^2. Statistically, no significant variances were detected between the subgroups in one-way ANOVA ($P = 0.8341 > 0.05$).

3.1.2. Experimental GIC (group 2)

The group 2 A was found to have the highest Vickers hardness value 49.51 kg/mm^2, followed by group 2B which recorded the mean value 49.39 kg/mm^2, and group 2C was found to have the lowest mean value 49.38 kg/mm^2. According to Table 1, there was no statistically significant difference between subgroups ($P = 0.9921 > 0.05$).

3.1.3. Results of comparison between conventional and experimental GIC groups

a) Subgroup (A): 0% Sesame oil modification

A higher, Vickers hardness mean, value 49.51 kg/mm^2 was noted in the experimental GIC (group 2 A)

Table 1. Comparison of microhardness (Mean values ± SDs) between both GIC groups with different Sesame oil extract concentration.

Variables	Conventional GIC (group 1) Mean ± SDs	Experimental GIC (group 2) Mean ± SDs	*t*-test *P* value	Total Mean ± SDs
Sesame oil extract concentration				
Subgroup A	$49.18^A \pm 2.183$	$49.51^A \pm 1.523$	0.7892ns	$49.35^A \pm 1.853$
Subgroup B	$49.11^A \pm 1.714$	$49.39^A \pm 1.984$	0.8146ns	$49.25^A \pm 1.849$
Subgroup C	$48.46^A \pm 2.246$	$49.38^A \pm 1.95$	0.5113ns	$48.92^A \pm 2.098$
Total	48.92 ± 2.047	49.43 ± 1.819	0.4775ns	
ANOVA				
P value	0.8341ns	0.9921ns		0.8757ns

Different letters in the same column indicating statistically significant difference ($P < 0.05$).
*Significant ($P < 0.05$).
ns; nonsignificant ($P > 0.05$).

than the conventional one (group 1 A) (49.18 kg/mm^2), as showed in Table 1 by *t*-test ($P = 0.7892 > 0.05$) the difference was statistically nonsignificant.

b) Subgroup (B): 1% Sesame oil modification

A higher Vickers hardness mean value 49.39 kg/mm^2 was noted in group 2 B than the group 1 B 49.11 kg/mm^2, according to Table 1 by *t*-test $P = 0.8146 > 0.05$ the difference was statistically nonsignificant.

c) Subgroup (C): 4% Sesame oil modification

A higher Vickers hardness mean value was noted in the group 2C (49.38 kg/mm^2) than that in group 1C 48.46 kg/mm^2, as indicated by a *t*-test ($P = 0.5113 > 0.05$). This difference was statistically nonsignificant.

As determined by two-way ANOVA ($P = 0.4775 > 0.05$), group 2 verified statistically nosignificant higher microhardness number (49.43 kg/mm^2) than group 1 (48.92 kg/mm^2). Despite the GIC type, the highest Vickers hardness mean value 49.35 kg/mm^2 was detected by subgroup A followed by the subgroup B mean value 49.25 kg/mm^2 while the lowest mean result 48.92 kg/mm^2 recorded by the subgroup C the difference was statistically nonsignificant ($P = 0.8757 > 0.05$) based on two-way ANOVA as shown in Table 1.

3.2. Surface roughness test results (μm)

The mean values and standard deviation of surface roughness test results (μm) for both groups with different Sesame oil extract concentrations are summarized in Table 2.

3.2.1. Conventional GIC (group 1)

According to this study, group 1C recorded the highest average surface roughness value of 0.2505 μm, followed by group 1 A mean value of 0.2494 μm, and the lowest mean value was determined by group 1 B which recorded the mean value of 0.2469 μm. Table 2 and Fig. 2 indicate that there was a statistically nonsignificant difference between subgroups ($P = 0.0530 > 0.05$).

Table 2. Comparison of surface roughness (Mean values ± SDs) between both GIC groups with different Sesame oil extract concentration.

Variables	Conventional GIC (group 1) Mean ± SDs	Experimental GIC (group 2) Mean ± SDs	*t*-test *P* value	Total Mean ± SDs
Sesame oil extract concentration				
Subgroup A	$0.2494^{A} \pm 0.0016$	$0.248^{A} \pm 0.0006$	0.0873ns	$0.2487^{AB} \pm 0.001$
Subgroup B	$0.2469^{A} \pm 0.0031$	$0.2486^{A} \pm 0.0004$	0.2569ns	$0.2477^{B} \pm 0.002$
Subgroup C	$0.2505^{A} \pm 0.0014$	$0.2495^{A} \pm 0.0013$	0.2393ns	$0.25^{A} \pm 0.001$
Total	0.2489 ± 0.002	0.2487 ± 0.001	0.6046ns	
ANOVA				
P value	0.0530ns	0.0537ns		0.0052*

Different letters in the same column indicate a statistically significant difference ($P < 0.05$).
*Significant ($P < 0.05$).
ns; nonsignificant ($P > 0.05$).

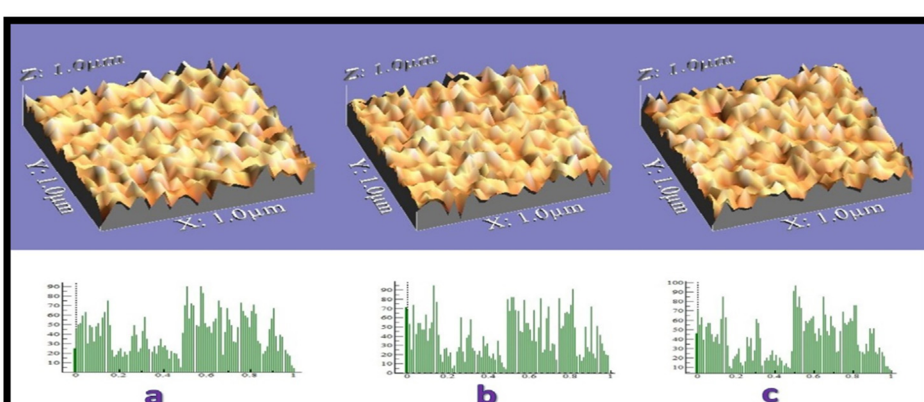

Fig. 2. Three-dimensional images and Histogram showing surface topographic features of Conventional GIC modified with: a) 0% Sesame oil, indicating the presence of elevations (hills) and depressions (valleys), b) 1% Sesame oil, indicating a slight decrease in the number of elevations and depressions, and c) 4% Sesame oil, showed that elevations and depressions have increased slightly.

3.2.2. Experimental GIC (group 2)

In group 2C the mean surface roughness (0.2495 μm) was the highest, followed by that of group 2 B which recorded a mean value of (0.2486 μm), and the lowest mean surface roughness (0.248 μm) was recorded in the group 2 A. In Table 2 and Fig. 3, it is evident that the difference between subgroups was statistically non-significant ($P = 0.0537 > 0.05$).

3.2.3. Results of comparison between conventional and experimental groups

(a) Subgroup (A): 0% Sesame oil modification

The Conventional GIC (group 1) measured a higher mean value of surface roughness 0.2494 μm than the experimental GIC (group 2) 0.248 μm, and this was statistically non-significant as indicated by *t*-test ($P = 0.0873 > 0.05$) as shown in Table 2.

(b) Subgroup (B): 1% Sesame oil modification

Statistically, the experimental GIC (group 2) had higher mean surface-roughness values 0.2486 μm than the conventional GIC (group1) 0.2469 μm, indicating that the results were not statistically significant ($P = 0.2569 > 0.05$) as shown in Table 2.

(c) Subgroup (C): 4% Sesame oil modification

The conventional GIC (group 1) recorded higher surface roughness mean value 0.2505 μm than experimental GIC (group 2) 0.2495 μm and this was statistically nonsignificant as indicated by the *t*-test ($P = 0.2393 > 0.05$) in Table 2.

As determined by two-way ANOVA, The conventional GIC (group 1) recorded statistically non-significantly higher surface roughness (0.2489 μm) than experimental GIC (group 2) (0.2487 μm) regardless of sesame oil extract concentration ($P = 0.6046 > 0.05$). Despite the GIC type, subgroup C recorded the highest surface roughness mean value 0.25 μm followed by the subgroup A mean value 0.2487 μm but subgroup B recorded the lowest mean value 0.2477 μm the difference was statistically significant as indicated by two-way ANOVA ($P = 0.005 < 0.05$) as shown in Table 2.

Topographical 3D image of investigated groups showing surface topographic features using a digital microscope.

3.3. The correlation between microhardness (kg/mm^2) and surface roughness (μm)

It was found that there was a significant correlation between microhardness and surface roughness as indicated by Pearson linear correlation (Correlation coefficient (r) = 0.4842, r^2 = 0.2345, and $P = 0.0471 < 0.05$) shown in Fig. 4.

4. Discussion

Investigators are constantly looking for improvements in dental materials, as there is no dental material like GIC available with all the necessary properties for every dental application. Additional compounds could be incorporated with dental materials to achieve this objective.

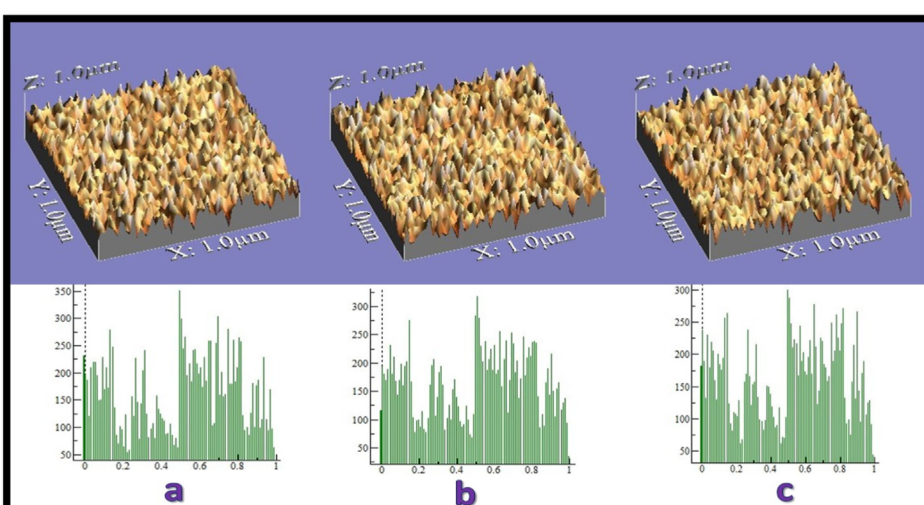

Fig. 3. Three-dimensional images and Histogram showing surface topographic features of Experimental glass ionomer cement modified with: a) 0% Sesame oil, indicating the presence of elevations (hills) and depressions (valleys), b) 1% Sesame oil, indicating a slight decrease in the number of elevations and depressions, and c) 4% Sesame oil, showed that elevations and depressions have increased slightly.

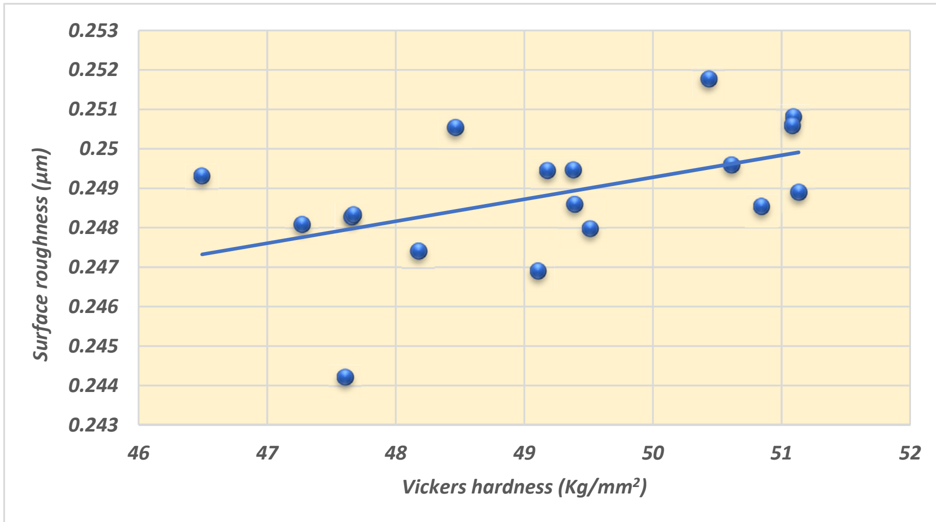

Fig. 4. Linear chart showing the correlation between Vickers hardness and roughness.

Medicinal plants are thought to be a major source of a wide range of chemical compounds with various purposes, in addition to being used to treat a variety of other diseases [12]. Thus, was oil extracted from Sesame seeds chosen for the current study because of its well-established medicinal benefits in oral health such as its -efficacy in treating dental plaque, -dental caries, and halitosis in addition to its easy accessibility [6,13].

Sesame oil has the potential to make cement stronger, tougher, and more scratch-resistant by increasing the degree of interlocking and cross-linking inside the cement matrix. Sesame oil was incorporated based on a previous study with two concentrations 1 and 4 (v/v%) to determine the proper concentration that enhances the mechanical properties [7].

The sol-gel method is a suitable and easier alternative to the traditional melt-and-quench method for producing GIC. Due to its relatively low synthesis temperature and ability to use thermal energy less than the temperature of oxide element crystallization through the use of liquid chemical precursors to create highly homogeneous, pure glass-like ceramic. However, a comparison of sol-gel and melt-derived glasses with similar compositions also seems to have similar structure and atomic correlations [9].

GIC's wear resistance and deformation degree are predicted by surface hardness [14]. This parameter can be used to compare the tooth structure with materials such as GIC. In this study, the Vicker hardness method was used due to its ability to determine the hardness of small-sized samples and brittle materials like GIC. For this study, a disc-shaped specimen was selected because it was suitable for testing [15].

The null hypothesis is partially rejected in some properties and accepted in others. Experimental GIC recorded comparable Vickers hardness values with conventional one. The addition of Sesame oil decreased the microhardness of GIC, which might be due to the delayed- reaction of acid-base through which Sesame oil may diminish the COOH group available for the final setting of GIC by its reaction with AL^{+3} [16]. Another explanation for this result might be due to the softening or plasticizing effect of Sesame oil on the poly acid salt network of the set GIC [4].

Surface roughness is an important surface parameter because this property affects the optical properties such as light reflection, color, and aesthetics, in addition to favoring biofilm accumulation which might increase the risk of carious lesions and periodontal disease. In the current study optical profilometry technique through digital imaging and a surface analyzer program was used due to easier access, affordability, and reduced time [17].

The current study displayed that conventional GIC recorded non-significant higher surface roughness than experimental one. It is suggested that The pores within the cement are closed with sesame oil or its components. Which increases packing density by reducing the roughness of the surface of the cement modified with 1% Sesame oil [7].

It was proposed that the porosity entrapped during manipulation could be mostly attributed to the viscous liquid of the GIC. Additionally, it was informed that the hand-mixed cement contains more voids than encapsulated versions [18]. The 4%

modified GIC's increased surface roughness may be attributed to greater porosity during hand mixing due to higher oil concentration, which makes the handling more difficult [18].

In line with Bastawy *et al.*, who found that natural bioactive did not enhance the mechanical properties of traditional GIC, the results of the study are partly consistent [11], while another study indicated the mechanical properties of GIC weakened when bioactive glass was added [19]. While partial disagreement with Aref NS [7] demonstrated that sesame oil improves microhardness and decreases the surface roughness of conventional GIC. This may be explained by dissimilar concentrations, testing protocols, and differences in GIC formulations.

4.1. Conclusion

Within the limitation, it was concluded that the experimental GIC showed comparable results with the conventional one regarding microhardness and surface roughness. Both Sesame oil concentrations have a negative impact on the microhardness of both GIC formulas. Sesame oil exhibited a better surface texture at low concentrations, while it showed a detrimental effect on surface roughness at high concentrations.

4.2. Recommendations

Further studies are needed to evaluate sesame oil's other properties, such as its solubility, water sorption, and ion leaching, despite its potential for strengthening GIC.

Ethics information

The study was approved by the Research Ethics Committee, Faculty of Dental Medicine for girls, Al-Azhar University, with the code (REC-MA-24-01).

Biographical information

Study was done at Faculty of Dental medicine for Girls Al Azhar University.

Funding

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Conflicts of interest

There are no conflicts of interest.

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