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Research Article

Physicomechanical Characterization of a Novel Resin-Modified Glass Ionomer Luting Cement Functionalized with a Phosphate Functional Monomer

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Background. Resin-modified glass ionomer cements (RMGICs) are characterized by their ability to chemically bond with the tooth structure and their fluoride release, making them commonly used to retain indirect restorations. However, inferior mechanical properties and solubility (SO) are their main drawbacks compared to the most recent resin-based cement. *Aim of the Study*. Formulate a novel brand of experimental RMGIC (eRMGIC), based on RMGIC by incorporating 2-(methacryloxy) ethyl phosphate (2-MEP), an organophosphorus monomer with the potential to enhance mechanical properties along with low SO. *Materials and Methods*. eRMGICs were prepared by the inclusion of 2-MEP monomer with different weight percentages (0–40 wt%) into the RMGIC's liquid (Fuji PLUS, GC. Corp.), then their compressive strength (CS), flexural strength (FS), film thickness (FT), setting time (ST), SO, and water sorption were examined and compared to the conventional RMGIC. Furthermore, a scanning electron microscope analyzed their surface homogeneity and integrity. Shapiro–Wilk test of normality was used to analyze data, one-way analysis of variance, Dunnett T3, and Tukey's honest significant difference post-hoc tests. *Results*. After 28 days and 180 days of storage, the values of CS of the eRMGICs were significantly higher. However, after 24 hr of storage, the values were comparable to the control group. The FS results showed a double-fold increase in different concentrations of eRMGICs through all the time intervals ($p < 0.001$) compared to conventional RMGIC. Furthermore, the inclusion of 2-MEP increased water uptake and decreased SO. The FT of experimental eRMGICs cement showed a statistically significant increase with increasing 2-MEP concentration. However, it was within the specification given by ISO 9917-1:2007. There was a decrease in the ST of eRMGICs compared to control cement; however, it was within the specification given by ISO 9917-2:2017. *Conclusions*. 2-MEP monomer showed encouraging results and could be used in producing new (eRMGICs) with enhanced physicomechanical properties, which can increase the longevity of cement and improve its ability to resist occlusal stresses without fracture.

1. Introduction

The efficacy of fixed prosthodontic therapy in achieving long-term clinical results depends upon using a luting agent to establish a hermetic seal between the restorative materials and tooth substrate [1]. The Latin word lutium, meaning "mud," is the source of the English term "luting." Dental luting cements establish a link between the prepared tooth and restoration, facilitating their bonding by various forms of surface attachment. These attachment mechanisms include mechanical, micromechanical, chemical, or combination [2].

An ongoing effort is to search for an optimal luting cement that can effectively preserve and protect tooth tissues, exhibit superior tensile and compressive strength (CS), establish a long-lasting link between tooth tissues and fixed restorations, and inhibit dental caries at the cement contact.

In general, luting cements are classified into either water-based; like zinc polycarboxylate, zinc phosphate, zinc silico-phosphate, glass-ionomer (glass-polyalkenoate cements); and resin-modified glass ionomer cement (RMGIC), or oil bases like zinc oxide eugenol or anhydrous luting cements like; resin cements based on methyl methacrylate, poly-acid modified composites (compomer), or based on aromatic di methacrylates and self-adhesive resin cements [3].

Most fixed prostheses are cemented using resin cements or RMGIC, two of the many types of dental cements on the market. Compared to resin cement, glass ionomer cement has several benefits, including better biocompatibility with the pulp and protection against secondary caries due to cement's capability to make a chemical bonding to the tooth substrate and fluoride recharging and releasing property. However, one of the most notable drawbacks of RMGIC cements is their weaker strength than resin cements and their high solubility (SO) due to 2-hydoxymethyl methacrylate (HEMA) [4].

Two notable issues are associated with the liquid of RMGICs: (1) carboxylic acid groups (COOH) attached directly or closely to the acrylic backbone, which may impact the complete transformation of carboxylic acid to carboxylate complexes. This, in turn, can influence the salt bridges formation and the migration of ions, potentially compromising the fracture toughness and the strength of the cement [5]; (2) increasing the molecular weight and concentration of the polyacids can be beneficial in the improvement of the CS and flexural strength (FS) of the cement. However, it can also introduce complications in terms of mixing, reduce the shelf life, and minimize the presence of nonstructural water content. The setting response of the RMGIC is highly dependent on the nonstructural water content [6, 7].

Previous research has primarily focused on enhancing the properties of glass ionomer restorative cement (GICs) through the functionalization of cement by introducing organic space-maintaining compounds or functional monomers. This approach aims to improve the compatibility between the inorganic and organic components of the cement, leading to enhanced salt-bridge formation and higher adhesive and mechanical properties of the glass ionomer cements [8–10].

Other research focused on using phosphate functional monomers in self-adhesive systems and resin-based cements as adhesion promoters [11, 12]. These phosphate functional monomers interact with minerals on the tooth substrate, forming a strong bond due to their reactivity and acidity [13, 14]. As shown by their chemical interaction and adhesion with the oxide layer in nonprecious and noble metals, zirconia, composite materials, and silica-based ceramics, these functional monomers also offer strong bond strength with dental indirect restorative materials [15].

2-(Methacryloxy) ethyl phosphate (2-MEP) is one of polymerizable phosphate-based monomers that is both photoreactive and proton conductive, with a pendant phosphate group that is most often used as an adhesive promoter; the interaction of the phosphate groups with their polarity results in a dense matrix that improves mechanical properties and increases the resistance to water intrusion, which in turn decreases water diffusion and SO [16, 17].

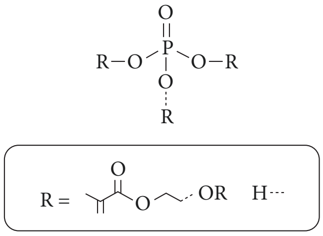


Figure 1: Chemical structure of 2-(methacryloxy) ethyl phosphate.

Although RMGIC is often used for luting indirect restorations, it still lacks the mechanical characteristics of resin cements, which might adversely influence the restoration's performance and longevity. A thorough review of the available literature is worth noting that this particular investigation represents the first use of 2-MEP in conjunction with RMGIC luting cement, as far as the author is informed to assess its impact on the mechanical properties (CS and FS), setting kinetics, film thickness (FT), and fluid uptake of the eRMGICs and to examine the efficacy of this novel cement to be used for luting of permanent restorations. The null hypothesis was that there would be no discernible change in the physicomechanical characteristics of a commercial RMGIC when varied weight amounts of 2-MEP (0–40 wt%) were added.

2. Materials and Methods

2.1. Main Materials of the Study. Polysciences Europe GmbH, Germany, supplied 2-(methacrylate) ethyl phosphate (batch number 52638-03-2; MW 210.12 g/mol; density 1.37 g/mL). Figure 1 depicts the molecular structure. The standard was the commercial RMGIC Fuji PLUS shade A3 (batch numbers 210713A, GC Corp., Europe). The liquid consists of a 7,7,9 (or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1; 2-HEMA 25%–50%; 2-hydroxy-1,3 dimethacryloxypropane 1%–5%; 16-dial bis methacrylate 1%–5%; water, chemical initiators, and the powder consisting of fluoro-aluminosilicate glass, initiator, and pigment (Figure 1).

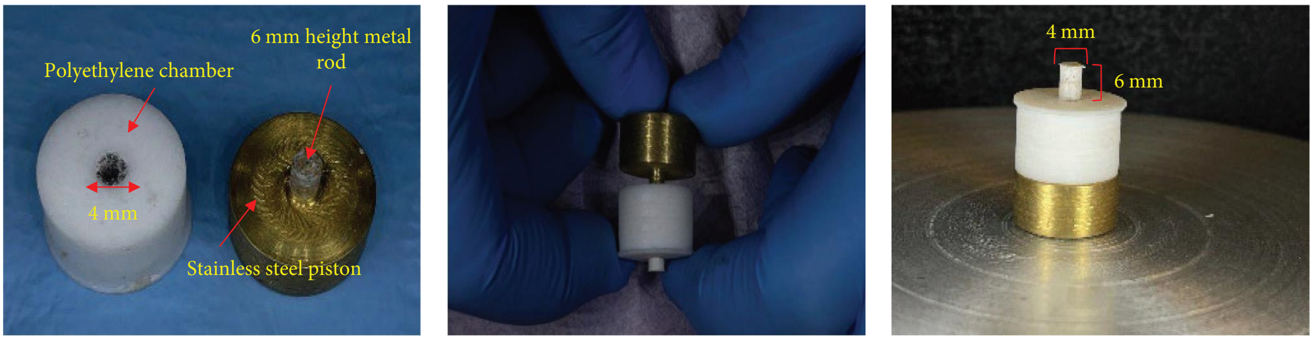
2.2. Formulation of the Experimental RMGIC (eRMGIC) and Grouping. The novel cement was developed by adding 2-MEP monomer to the liquid phase of commercial Fuji PLUS at varying percentages (0, 10, 20, 30, and 40 wt%). All different kinds of cement utilized the RMGIC powder without any adjustments.

The experimental cements with (10, 20, 30, and 40 wt%) of 2-MEP were prepared depending on the below equation [18, 19]. According to Equation (1), 0.2, 0.40, 0.60, and 0.80 g of 2-MEP, which represent (10, 20, 30, and 40 wt%), respectively, were weighted using a digital electron balance of three digits blended into 2 mL of original liquid after subtraction of equivalent weight of original liquid equal to the corresponding amount of added monomer, then the modified liquid blended by using a magnetic stirrer (LMMS300/Chaina) for 5 min to ensure homogeneously dispersion of the 2-MEP in cement liquid according to previous literature [20].

$$\mathrm{Con} = \frac{\mathrm{Wt}}{v}\%, \tag{1}$$

Table 1: Composition of the control RMGIC and experimental eRMGIC cements.

Codes/liquid	Powder	2-MEP (wt%)	P/L ratios
2-MEP 0 (Fuji PLUS liquid)	GC Fuji PLUS	0 (control)	1.0/3.0
2-MEP 10	GC Fuji PLUS	10	1.0/3.0
2-MEP 20	GC Fuji PLUS	20	1.0/3.0
2-MEP 30	GC Fuji PLUS	30	1.0/3.0
2-MEP 40	GC Fuji PLUS	40	1.0/3.0



(a) (b) (c)

Figure 2: The compressive strength mold: (a) polyethylene chamber with a metal piston; (b) pushing off the piston on the opposing side of the mold; (c) the prepared sample.

where Con: the concentration %, Wt: weight of solute (g), and v: volume of 100 mL of solution.

The experimental groups (2-MEP 10%, 2-MEP 20%, 2-MEP 30%, and 2-MEP 40%) were made by mixing a commercial Fuji PLUS powder with the modified liquid manually using a powder/liquid ratio of 1/3 (one large scoop of powder to three drops of liquid) according to the manufacturer's instruction at (23.2°C) ambient temperature and 35.5% relative humidity. The control (2-MEP 0%) group was unaltered Fuji PLUS cement. The samples for each particular study were divided into five groups: 2-MEP 0%, 2-MEP 10%, 2-MEP 20%, 2-MEP 30%, and 2-MEP 40% groups (each abbreviation's matching full material name may be found in Table 1).

2.3. Spectral Analysis by Fourier Transform-Infrared Spectroscopy (FTIR). The structural and chemical properties of 2-MEP 30% were analyzed and compared to 2-MEP 0% using FTIR spectroscopy. This analysis revealed the existence of a new peak, which may be attributed to the inclusion of the phosphate moiety in 2-MEP 30%. At the Ministry of Science and Technology in Baghdad, Iraq, samples were examined using a Burker FTIR Spectrometer (ALPHA P, USA) in the absorbance mode between 400 and 4,000 cm^{-1}. The sample was pressed against the ATR-FTIR lens to attain the maximum signal intensity of infrared (IR) radiation. The diagram displays the relationship between the absorption of IR radiation and the various functional groups in the sample, with wavelength shown in wavenumber units (cm^{-1}).

2.4. Mechanical Properties

2.4.1. Compressive Strength (CS) Test. One hundred twenty cylindrical specimens (24 specimens for each group) were prepared for the CS test according to the ISO standard 9917-1:2007 [21]. The specimens were prepared by using a custom-made polyethylene mold composed of a polyethylene chamber (4 mm diameter, 6 mm height), which is used to occupy the mixed cement and stainless steel piston with a metal rod used as a depth detector when inserted in the polyethylene mold to give 6 mm vertical space inside the mold precisely and also to simplify the removal of the samples when removed from the mold first and pushed through the opposing side of the mold when the cement has completely set (Figure 2). The specimens were prepared by filling the mold with an experimental cement that had been mixed following the manufacturer's specifications (the metal piston in this step is in its location engaged by the polyethylene mold to act as a depth detector). The mold was left overfilled without any attempt to level, which was done within the working time of the material. Immediately apply polyester film(s) and glass plate, then a (500 g) load is placed over the mold to extrude any excess material and to produce a flat end specimen [22]. At 37°C and relative humidity of 40%, all samples were left to set for 60 min from the end of mixing, then the plate was removed, and the ends of the samples were ground flat at right angles to their long axis. The specimens' diameter was assessed at three distinct points with a digital micrometer (Mitutoyo digimatic caliper 500–136, Kawasaki, Japan) to an accuracy of 0.01 mm. Subsequently, the mean diameter was calculated before the tests were conducted. The compressive test was conducted at three different time intervals: 1, 28, and 180 days of storage in distilled water at a temperature of 37°C. The distilled water was replaced weekly for the 28 and 128-day storage periods. For each group of five groups, the 24 samples were randomly subgrouped according to the storage duration: subgroup 1 for 1 day, subgroup 2 for 28 days, and subgroup 3 for 180 days ($n = 8$ per each period); the compressive test was conducted using an (Instron model 5569, USA), loaded with a 500 N load cell and a crosshead speed of 0.5 mm/min. To simulate the parameters of the oral environment, the specimens were examined while in a state of wetness. The procedure above included inserting a moist filter paper (specifically, Whatman No. 1, manufactured by Whatman International Ltd. in Maidstone, England) between the specimen's two ends and the testing apparatus's platens.

The specimens were subjected to compressive load, which was applied to their long axis, and the highest load at which failure occurred was documented. The calculation of the CS, denoted as C (MPa), for each cylindrical specimen included dividing the fracture force (p) by the area of the specimen. The diameter of the specimen, represented as D, was used in Equation (2) for this purpose [23].

$$C = \frac{4p}{\pi D^2}. \tag{2}$$

2.4.2. Flexural Strength (FS) Test. One hundred twenty test specimens (24 specimens for each group) were prepared for this test following ISO (9917-2:2017) standard for the RMGICs, Geneva: ISO [24]. Polytetrafluoroethylene (PTFE) mold ($2 \times 2 \times 25\ \mathrm{mm}^3$) was used to prepare specimens for the FS test. According to the manufacturer's recommendations, the cement was manually mixed and quickly loaded into the middle of the mold cavity, causing the flow to be outward, overfilling the mold and making no effort to level it. These steps were performed within the working time of the material (2.30 min) according to the manufacturer's recommendation.

The experimental protocol included promptly filling the PTFE mold with a cement mixture. A polyester film was used to cover the mold and glass plates on both sides to ensure the production of a level and parallel end specimen. A 500 g load was applied to facilitate the extrusion of any extra material. Afterward, the assembly was placed into 37 ± 2°C distilled water for 1 hr.

After curing, the specimens were taken out of the mold without damaging the specimens, and then the abrasive paper with 320 grit was used to remove the flashes by abrading them. Then, in distilled water of 37 ± 2°C, all these specimens were stored. After that, each specimen was taken out from the water bath, and the dimensions of each specimen were accurately evaluated with an accuracy of 0.01 mm by using a micrometer, which had been placed at the center of the specimen. The FS test was conducted after 1, 28, and 180 days of storage; for each of the five groups, the 24 samples were arbitrarily subdivided into three subgroups based on the duration of storage: subgroup 1 for 1 day, subgroup 2 for 28 days, and subgroup 3 for 180 days ($n = 8$ for each period).

The test was done using a universal testing machine (Larry compressive machine/China); each specimen was positioned in the center of the testing jig, perpendicular to the three rods; the support distance was 20 mm.

Within 10 s of specimen removal from the distilled water, the specimen was subjected to the load using 0.75 ± 0.25 mm/min crosshead speed, which was applied until fracture of the specimen [25]. The specimen's maximum force was measured, and the FS was estimated in MPa using Equation (3).

$$\sigma = \frac{3FL}{2bh^2}, \tag{3}$$

where F represents the greatest force applied to the specimen, measured in newtons; L represents the distance between the supports, measured in millimeters with a precision of 0.01 mm; b represents the width, measured in millimeters, in the center of the specimen prior to testing; h represents the height in millimeters at the center of the specimen prior to testing.

2.5. Setting Time (ST) Test. The ST was determined by the ISO standard 9917-2:2017 for water-based dental cement [24]. ST is defined as the time elapsed between the initiation of mixing and the point at which the flat-end needle (with a diameter of 1.0 ± 0.05 mm) attached to the indenter (weighing 400 ± 0.5 g) of a Gillmore apparatus (manufactured by UTEST-Material, Ankara, Turkey) failed to produce an entire circular indentation on the surface of the cement. The Gillmore apparatus is comprised of a pair of needles. The needle used for measuring the initial setting should have a tip diameter of 2.12 ± 0.05 mm and a mass of 113.4 ± 0.5 g. The mass of the final ST needle should be within the limit of 400 ± 0.5 g, while the tip diameter should be 1.0 ± 0.05 mm. The mold used for setting the time test contains a square hole (10 ± 2 mm) within a mold with a minimum thickness of 5 ± 2 mm cut in a Teflon block at least $16\ \mathrm{mm}^2$. The mold was conditioned to 23 ± 1°C and placed on the aluminum foil; at this moment, after starting the timer, the cement was mixed and poured into the mold until it reached a uniform level.

The indenter, with a mass of 400 ± 0.5 g, was lowered vertically onto the cement's surface after a 10-s interval of the manufacturer's specified ST. It was then left in place for 5 s. Subsequently, the indenter was disengaged from the surface. This process was repeated at regular intervals of 10 s until the indenter could no longer produce a completely formed circular indentation in the cement. The examination was conducted with a magnification of ×2. The average setup time was determined by conducting five replicate tests for each group.

2.6. Film Thickness (FT) Test. The RMGIC test was conducted according to ISO 9917-2:2017 [24]. Two visually square flat glass plates with a $200 \pm 25\ \mathrm{mm}^2$ contact surface area. Each plate was produced with a uniform thickness of at least 5 mm. Shaped glass plates were placed in contact and measured four times with a digital micrometer (Digimatic, Mitutoyo Europe GmbH, Neuss, Germany) to an accuracy of 1 μm. This reading was assigned Reading A. According to the manufacturer's recommendations, each group's ($n = 5$) cement was mixed and placed between the glass plates. Using a universal testing machine, the upper glass plate was loaded with 150 ± 2 N. The total thickness of the glass plates with the cement in-between was measured 10 min later, and this measure is considered Reading B. The total FT of the test specimens represents the difference in thickness between the plates with and without the intervening substance.

2.7. Water Sorption and Water Solubility. Five control and modified cement disks were made according to IOS specification 4049:07:2019 [26]. Specimens were made in a cylindrical Teflon mold (15 ± 0.1 mm in diameter by 1 ± 0.1 mm in thickness) at 23 ± 1°C. The mold was slightly over-filled with the material, which was placed on a glass slide with a thickness of 50 μm, covered by a piece of transparent polyester film. Subsequently, a second layer of polyester film was applied to the mold, concurrently subjected to pressure by an additional glass slide.

The silicon carbide paper (1,000 grit) was used to finish the specimens to eliminate flash and provide a consistent thickness throughout. Using a digital electronic caliper (Mitutoyo Corporation, Japan), we determined the diameter and thickness of each specimen at two and four sites, respectively. Each specimen's volume was expressed as an mm^3 mean value.

The incubation of samples was done at 37 ± 1°C. After 22 hr, the samples were moved and kept at 23 ± 1°C for 2 hr. Then, the electronic analytical balance calibrated to a precision of 0.01 mg and an accuracy of 0.1 mg was used to weigh the samples (Ohaus Corporation, USA; Ohaus Analytical Plus). This cycle was repeated to confirm that polymerization and dehydration were complete until each specimen's mass change in any 24 hr was less than 0.1 mg. This constant mass ($m1$) served as the specimen's initial mass.

Five cement specimens were immersed in separate glass containers containing 10 mL of distilled water. The specimens were left immersed for 30 days. Daily weight assessments were done throughout the first week and on the 14th, 21st, and 30th days. Gentle drying of the specimens was done on filter paper until all apparent moisture was removed. Following this, they were exposed to air for 15 s and then weighed after 1 min with an accuracy of ±0.01 mg. Finally, the specimens were placed back into distilled water-filled glass containers. The mass recorded was indicated as $m2$. Following a period of complete immersion lasting 30 days, all five specimens were subjected to a drying process until a consistent mass was achieved. The cement specimens underwent a drying procedure until they reached a consistent mass ($m3$). This was done using a fresh silica gel to place the specimens in desiccators (Clear Glass Vacuumed Desiccator/China). The drying process lasted for 30 days, following the described cycle.

Calculation of SO (W_{SL}) and water sorption (W_{SP}) in μg/mm^3 is done by the following equations:

$$W_{\mathrm{SP}} = \frac{m1 - m2}{v}, \tag{4}$$

$$W_{\mathrm{SL}} = \frac{m1 - m3}{v}, \tag{5}$$

where the mass of spacemen before water immersion is $m1$ in μg, the after-immersion mass in water for 30 days is $m2$ in μg, the reconditioned mass is $m3$ in μg, and the volume of the specimen is v in mm^3.

2.8. Scanning Electron Microscope (SEM) Analysis. The investigation included the analysis of tiny structural alterations in specific cement compositions (2-MEP 0%, 2-MEP 20%, and 2-MEP 30%). This was achieved using SEM of fractured specimens collected from CS and FS testing. The micrographs were captured at various magnifications (×2000, ×500, and ×250) and a working distance of 50 μm, 300, and 500 μm, respectively. Before examination, these surfaces were subjected to a drying process. Subsequently, a coating of gold nanoparticles was applied to the surfaces using a current of 45 mA for 2 min. The coated surfaces were then seen using an SEM (Inspect F50, FEI company Holland) with an accelerating voltage of 30 kV [27].

3. Statistical Analysis

The data analysis was performed using the SPSS Statistical software for Windows of version 25, developed by (SPSS Inc., based in Chicago, Illinois, USA). The data values' normality was evaluated by implementing the Shapiro–Wilk test, with a significance level of $p > 0.05$. The statistical study included a one-way analysis of variance (ANOVA) to examine the data. Subsequently, post hoc tests, namely Dunnett T3 and Tukey's honest significant difference (HSD), were applied to determine significant differences among the groups.

4. Results

4.1. Mechanical Properties. The CS of the eRMGICs (2-MEP 10%, 20%, 30%, and 40%) at 24 hr showed no differences compared to the control 0% of 2-MEP. The enhancement in CS was evident in all groups after 28 days of storage in 37°C distilled water, compared to the values of the early storage period ($p < 0.001$). The higher 2-MEP-containing cements (2-MEP 30% and 40%) continued gaining strength after 4 weeks of aging (114.57 ± 1.5 and (112.12 ± 2 MPa, respectively) and were statistically higher than the 2-MEP 0% (99.18 ± 1.2 MPa) ($p < 0.001$). On longer-term aging 180 days, there was evident deterioration in CS of the control cements compared to all other modified cements (eRMGICs), which maintained their CS with statistically significant differences from the baseline values of commercial cement ($p < 0.001$) and their reference values ($p < 0.001$). The higher the 2-MEP content, the higher the CS values. Prolonged aging in distilled water raised the CS of the 2-MEP 20%, 30%, and 40% cements (Table 2 and Figure 3).

For FS, when the values of modified cement were compared to the control cement, these values were increased by double $p < 0.001$ through the different storage periods, and this increase coincided with increasing the percentage of 2-MEP. Among the experimental groups, aging had varying effects on the results, while 2-MEP 20% and 30% continued to have higher FS after 180 days of storage (Figure 4 and Table 2).

4.2. Setting Time. The mean values of ST are shown in Table 3. The incorporation of 2-MEP reduced the ST of the resultant

TABLE 2: CS and FS of the experimental eRMGICs (0–40 wt%) at 1, 28, and 180 days aged in distilled water at 37°C, shown as mean (SD), $n = 8$.

Days	2-MEP 0%	2-MEP 10%	2-MEP 20%	2-MEP 30%	2-MEP 40%
	Compressive S	Compressive S	Compressive S	Compressive S	Compressive S
1	85.75 (2.2) ^{Aa}	83.07 (1.4) ^{Aa}	83.98 (1.9) ^{Aa}	83.78 (3.6) ^{Aa}	83.42 (2.4) ^{Aa}
28	99.18 (1.2) ^{Ba}	100.27 (1.6) ^{Ba}	105.78 (2.0) ^{Bb}	114.57 (1.5) ^{Bc}	112.12 (2.0) ^{Bc}
180	81.85 (2.3) ^{Ca}	98.24 (2.2) ^{Bb}	103.28 (2.2) ^{Bc}	113.07 (1.0) ^{Bd}	107.87 (3.0) ^{Ce}
	Flexural S	Flexural S	Flexural S	Flexural S	Flexural S
1	12.67 (2.5) ^{Aa}	22.51 (2.3) ^{Ab}	28.44 (1.2) ^{Ac}	24.16 (1.7) ^{Ab}	21.303 (1.6) ^{Ab}
28	10.5 (1.2) ^{Aa}	23.8 (2.1) ^{Ab}	30.0 (3.2) ^{Ac}	32.3 (2.0) ^{Bcd}	28.8 (3.0) ^{Bce}
180	11.81 (1.4) ^{Aa}	21.63 (1.2) ^{Ab}	26.24 (2.2) ^{Bc}	30.126 (1.4) ^{Bc}	23.395 (2.7) ^{Ac}

Note. Similar lowercase letters in the same rows across groups indicate no significant differences (ANOVA, post hoc Tukey test at a significant threshold of $p = 0.05$). Similar uppercase letters in columns show that aging within the groups has no significant effect on values from day one. CS, compressive strength; FS, flexural strength; SD, standard deviation.

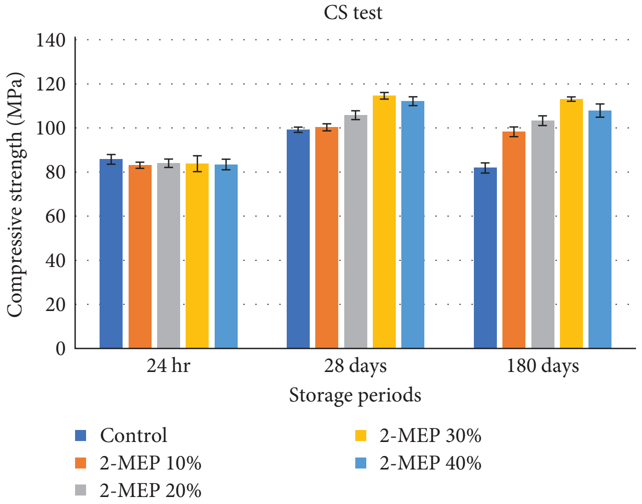


FIGURE 3: Bar graph showing the mean compressive strength of modified eRMGICs through different storage periods (24 hr, 28 days, and 180 days) compared to control cement.

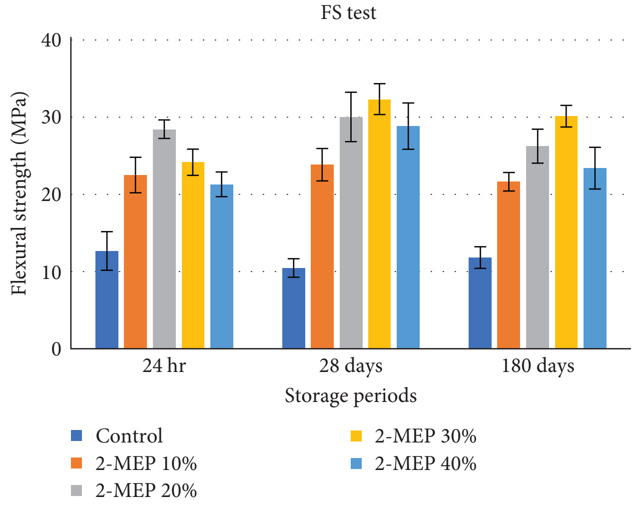


FIGURE 4: Bar graph showing the mean flexural strength of modified eRMGICs through different storage periods (24 hr, 28 days, and 180 days) compared to control cement.

TABLE 3: Mean values and standard deviations of experimental cement's setting time (min) and film thickness (μm).

Groups ($n = 5$)	Film thickness (μm)	Setting time (min)
2-MEP 0	14.5 ± (0.2)	5.65 ± (0.3)
2-MEP 10	14.7 ± (0.1)	5.50 ± (0.2)
2-MEP 20	15.0^{*} ± (0.1)	5.33 ± (0.1)
2-MEP 30	16.7^{*} ± (0.2)	5.00^{*} ± (0.08)
2-MEP40	17.2^{*} ± (0.2)	4.78^{*} ± (0.30)

Note. *Represents the mean values, which are statistically significant compared to the control group at $p < 0.05$.

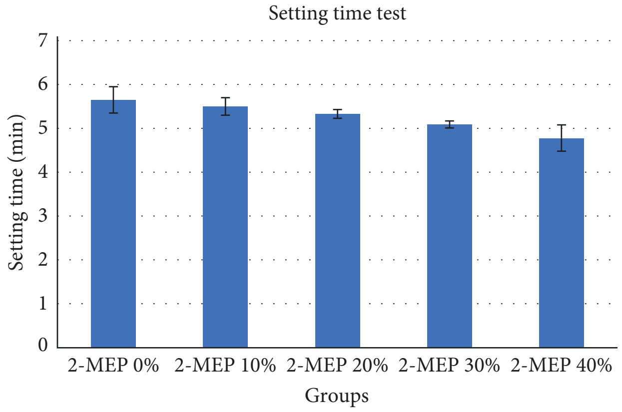


FIGURE 5: Bar graph showing the mean setting time of experimental eRMGIC compared to control cement.

cements compared to the control one; the statistical result showed that the reduction in ST is directly related to increasing the weight percentage of the 2-MEP monomer. The multiple comparison test (Dunnett T3 post-hoc) showed that the eRMGICs (10% and 20%) had nonsignificant differences compared with control cement. Moreover, the eRMGICs with high 2-MEP percentages (30% and 40%) showed significant differences from control cement (Figure 5). However, all these results were within the specification given by ISO slandered 9917-2:2017 of resin-modified water-based cement [24], which is no more than (8 min.)

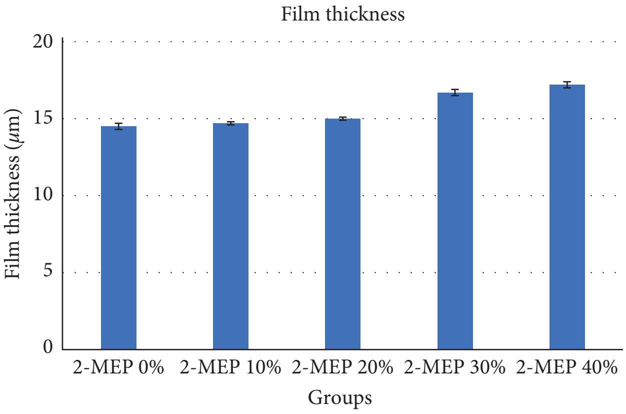


FIGURE 6: Bar graph showing the mean film thicknesses of experimental eRMGICs compared to control cement.

TABLE 4: Mean values and standard deviations of water uptake and the solubility of the commercial and experimental RMGICs for a total immersion time of 30 days in distilled water ($n = 5$).

Days	Water uptake (μg/mm^3)	Solubility (μg/mm^3)
2-MEP 0	62.7 ± (2.8)	5.3 ± (1.6)
2-MEP 10	63.0 ± (1.8)	3.8 ± (1.3)
2-MEP 20	65.9 ± (1.1)	2.9* ± (0.3)
2-MEP 30	68.3* ± (1.9)	1.8 ± (0.2)
2-MEP 40	69.1* ± (1.3)	1.9* ± (0.4)

Note. *Represents the mean values, which are statistically significant compared to the control group at $p < 0.05$.

4.3. Film Thickness. The FT values of the Fuji PLUS cement were analyzed using a one-way ANOVA, which showed a statistically significant difference between the groups ($p < 0.05$). The data is shown in Table 3. The most significant FT was produced by the 2-MEP 40%. Subsequent analysis using the post hoc Tukey HSD test indicated that all experimental groups exhibited significant differences compared to the control group, except the 2-MEP 10% group. The 2-MEP 10% group displayed no deviation from the control group and showed no difference from the 2-MEP 20% group. The 2-MEP 40% exhibited the highest FT ($p < 0.05$) (Figure 6).

4.4. Water Sorption (WS) and Water Solubility (SO). Compared to the control cement (RMGIC 0%), the modified cements (eRMGIC 10%–40%) showed a higher percentage of water uptake, according to the statistical results. It was also evident that the proportional increase in water uptake was directly correlated with increasing 2-MEP content in the matrix (Table 4 and Figure 7). Simultaneously, the water uptake of all cement's samples achieved the equilibrium state within a week. The water uptake values for the eRMGIC cements varied from 63 to 69.1 μg/mm^3, whereas they were 62.7 μg/mm^3 for the control cement (2-MEP 0%). Regarding the SO in μg/mm^3, it was generally lower in eRMGICs than in the control cement (2-MEP 0%), and 2-MEP 20%, 30%, and 40% showed a highly significant reduction in SO compared to conventional RMGIC cement ($p < 0.001$) (Table 4 and Figure 8). The findings (3.8–1.9 μg/mm^3) were less than the 7.5 μg/mm^3, the limit that the ISO 4049 recommends [26].

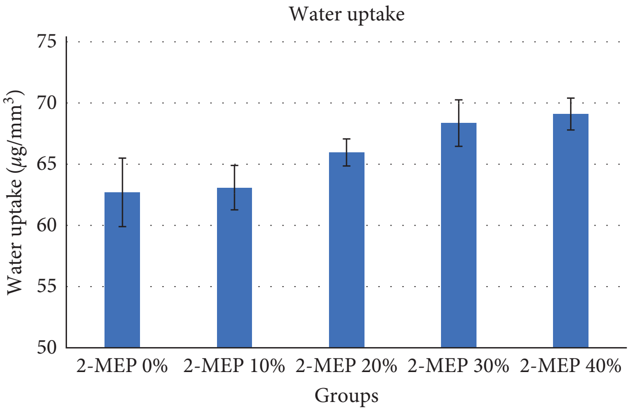


FIGURE 7: Bar graph showing the mean water uptake in μg/mm^3 of experimental eRMGICs compared to control cement for a total immersion time of 30 days.

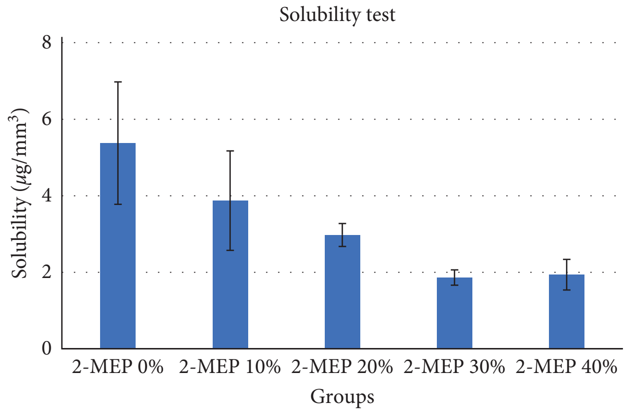


FIGURE 8: Bar graph showing the mean solubility in μg/mm^3 of experimental eRMGICs compared to control cement for a total immersion time of 30 days.

4.5. FTIR. Cement FTIR spectra (both standard and experimental) are shown in Figure 9. The stretching vibration of SiO_4 tetrahedral with a varied number of bridging oxygen atoms was responsible for the appearance of a new shoulder at 972 cm^{-1} above the peak at 1,030cm^{-1} in the experimental cements with phosphate groups (eRMGIC 30%), marking them distinguished from the control cements (RMGIC). There is considerable overlap between this intense and wide peak and P–O stretching modes [28]. However, 2-MEP 30% in the eRMGIC has a shoulder centered at about 972 cm^{-1} because of the $V1$ stretching vibrations in the PO_4 tetrahedral structure of the phosphate group. Peaks at 1,456 and 1,639 cm^{-1}, representing the symmetric and asymmetric –COO stretching bands, respectively, are characteristic of carboxylate salt formation [10, 29]. The ester group in HEMA, 2-MEP, and the COOH group in polyacid all have $C=O$ stretching vibrations, reflected in an absorption band at 1,720 cm^{-1} [30]. The broad band at about 3,445 cm^{-1} is linked to water's O–H stretching vibrations [31].

4.6. SEM Analysis Results of the Failed Samples. The SEM results of eRMGICs cement showed a denser microstructure

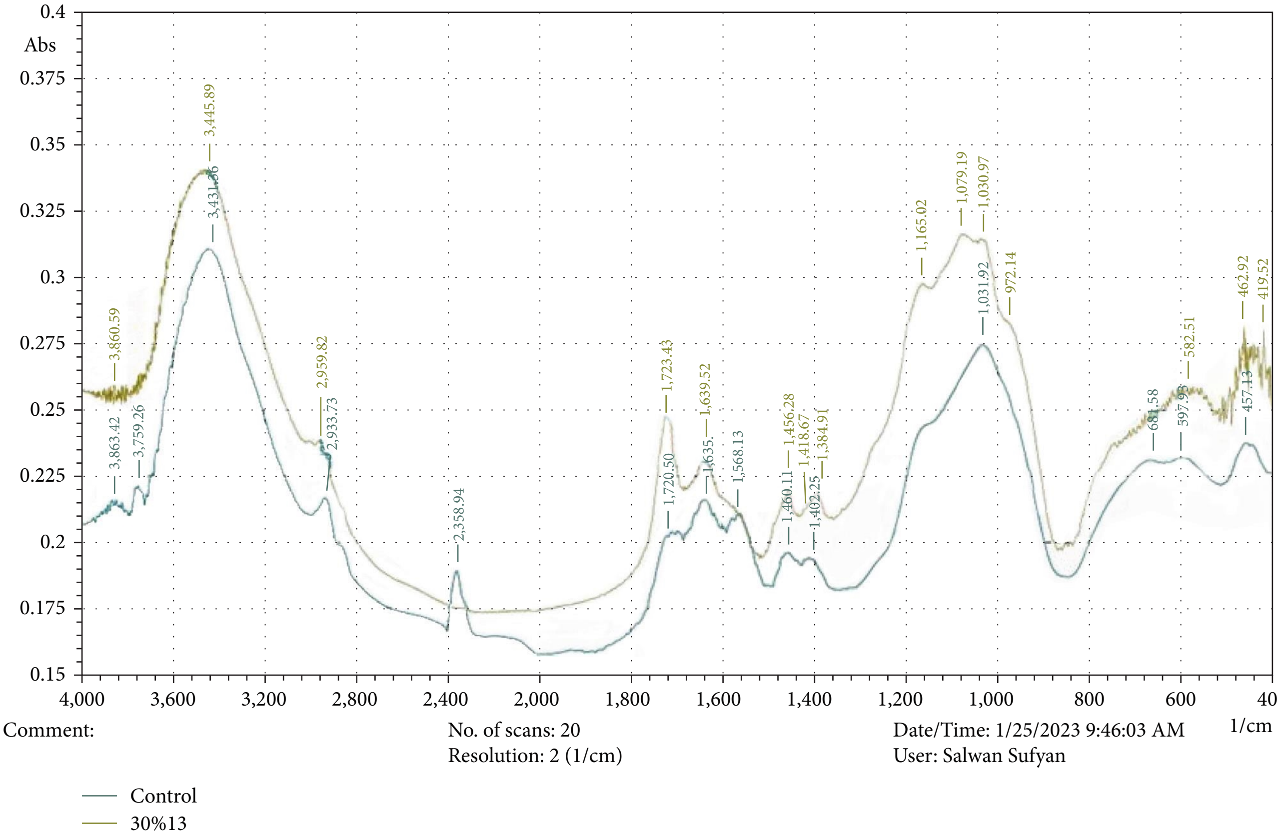


Figure 9: FTIR charts characterizing RMGIC (2-MEP 0%) and eRMGICs (2-MEP 30%). Observe the intensity of the 1,030 cm^{-1} peak and the new 972 $V1$ shoulder peak in modified eRMGIC cement.

with smaller-sized pores with fewer numbers compared to the control one, and this effect is directly related to increasing the weight percentage of 2-MEP (Figure 10).

5. Discussion

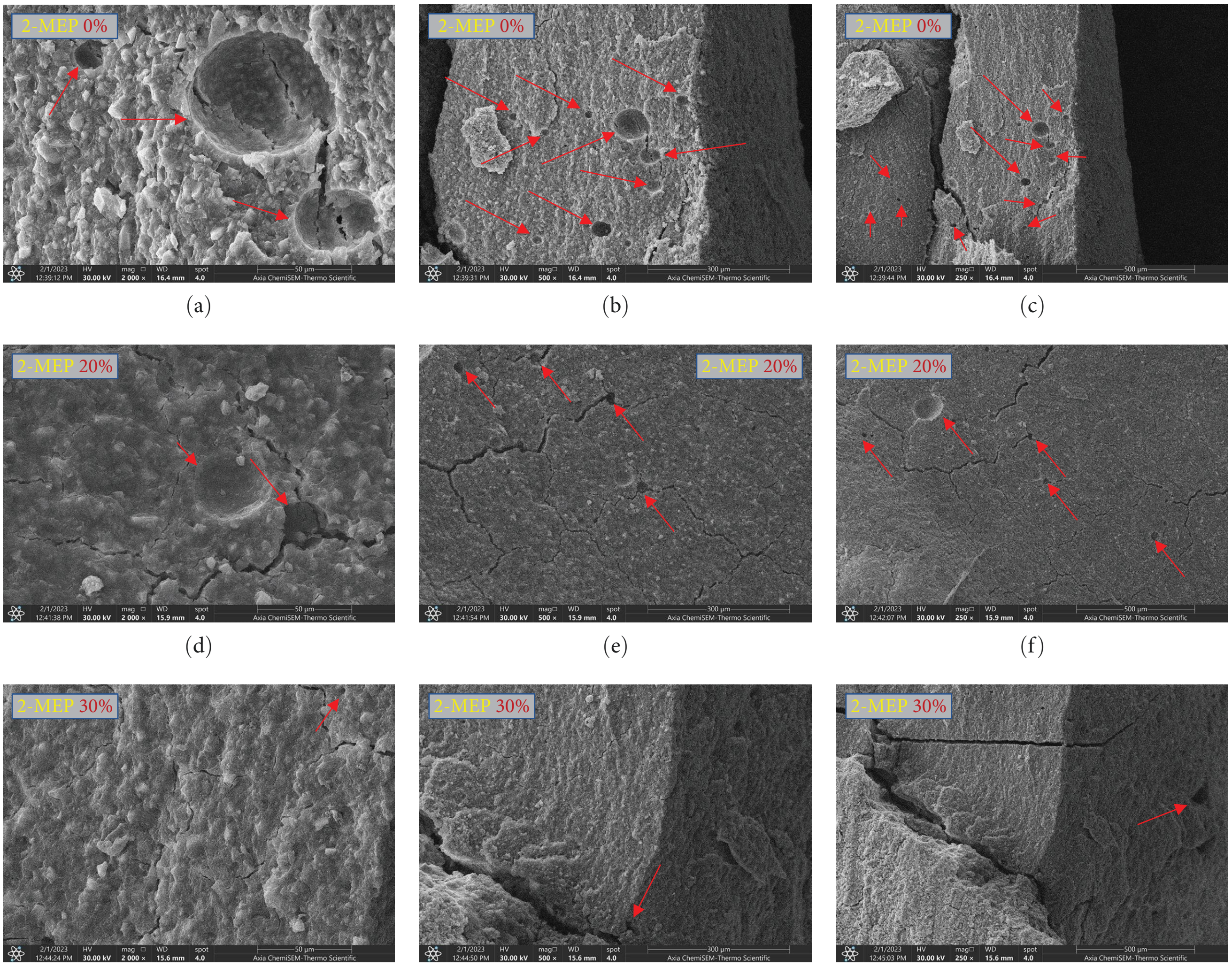
Indirect restorations have been kept in place using dental luting cements. The durability of the cement is essential for the success and endurance of these restorations. Bonding to the tooth tissue, releasing therapeutic ions, and minimal toxicity are all features of RMGIC cements [32, 33]. In the 1980s, researchers attempted to develop this cement to eliminate the two major issues associated with traditional GIC cement: poor early strength and high SO [34]. Despite their benefits, RMGIC cement materials still lack the mechanical qualities of resin cement due to their susceptibility to moisture (presence of HEMA) and poor strength [3].

Improving the physical and mechanical characteristics of RMGIC cements by functionalizing the acrylic acid copolymers with functional monomers is not a new approach [17]. The strong chemical interactions between the functional monomers (due to their reactivity and acidity) and hydroxyapatite minerals in the tooth structure result in enhanced physicomechanical characteristics of these cements [13, 14].

According to the results of the present study, there was no statistically significant difference between the STs of the control cement and the eRMGICs at 10% and 20% 2-MEP ($p > 0.05$), as indicated in Table 3. In addition, eRMGIC at 30% and 40% 2-MEP showed a statistically significant reduction ($p < 0.05$) compared to the control cement; nonetheless, these findings met the specifications stated in the ISO standard 9917-1:2007, which is relevant to water-based cement [21]. This effect might be interpreted based on the idea that incorporating resin monomers modifies the chemical properties of the setting process in RMGIC [35].

Mixing of powder and liquid starts the acid–base reaction, forming a complex network of poly-salts. This reaction persists for several minutes, with further maturation occurring over time [35]. This process involves the simultaneous acid–base reaction and free-radical-driven polymerization of methacrylate monomers. Depending on the initiator system, the monomer polymerization may be initiated photo-chemically or chemically. The structure of set RMGIC exhibits a complex configuration characterized by interconnected networks of polyacrylate-salts and poly (HEMA), within which the remnants of unreacted glass particles are embedded [35]. During the initial phase of the setup process, network-forming reactions have a competitive characteristic, and a sensitive balance is present between these two processes [36, 37].

Considering the mechanism above, it is clear that functionalization of cement with 2-MEP (phosphate-containing monomer) improves early-stage competent reactions and promotes interaction within the glass matrix between the ions from one side and the ligating phosphate groups on



(a) (b) (c)

(d) (e) (f)

(g) (h) (i)

Figure 10: The scanning electron micrographs of the broken CS and FS test surfaces were seen at magnifications of ×50, ×300, and ×500. A comparative analysis reveals that 2-MEP 20 (d), (e), and (f), as well as 2-MEP 30 (g), (h), and (i), exhibit a more integrated uniform and homogeneous surface structure characterized by a decrease in pore size and density, in contrast to the surface properties found in 2-MEP 0 (a), (b), and (c). The red arrows shown in 2-MEP 0, 2-MEP 20, and 2-MEP 30 indicate the presence of pores at varying concentrations.

the other side, at the same time the incorporation of 2-MEP in the presence of 2-HEMA enhances acid–base reaction and forms covalently linked matrix within the eRMGIC hence shortening the eRMGIC cement's curing time [17].

The eRMGIC cements' setting was confirmed by FTIR spectra (Figure 9). A shoulder peak at 972 cm^{-1} indicates the successful incorporation of 2-MEP into the matrix. This observation explains the compatibility of 2-MEP with the liquid phase of Fuji PLUS and HEMA comonomer, as shown by the absence of any signs of phase separation [16].

WS and SO serve as essential criteria in assessing luting materials; these properties are also related directly to the durability of cement [38]. The WS studies measure the net weight obtained due to water dilution and the release of monomers and other small molecules [39].

At the end of the immersion time, both the control and modified groups in the WS test showed water gain; compared to the control group, the 2-MEP 30% and 2-MEP 40% exhibited a statistically significant increase. However, compared to the control group, 2-MEP 10% and 20% showed a statistically insignificant difference.

Water uptake measurements were performed per the ISO standard 4049:07:2019 for resin-based products [26]. However, the specimens were immediately submerged in distilled water at 37°C for the sorption cycle without desiccation. The process of desiccation in the case of RMGICs may result in the removal of one or both types of water: the "loosely bound" water, which plays an essential role in the acid–base reaction progression, and the "tightly bound" water, which contributes to the structural integrity of the cements. Additionally, the RMGIC cement utilized in this study might include air gaps from the hand mixing procedure, which speeds up WS by expanding the cement's surface area exposed to water [40, 41].

All kinds of cement in the current study gained water over the first week before achieving equilibrium, and this finding comes in consistent with the literature by Zankuli et al. [42]. The hydration level exhibited a clear correlation with the amount of the additional monomer (2-MEP) present in the cements, resulting in significantly higher hydration levels compared to the control group. In cements containing 2-MEP, more water is attracted by the functional group's polarity. A study by Kemal et al. [16] demonstrated the hydrophilic ability of the phosphate functional monomer-; they found a proportionate relationship between the quantity of phosphate monomer copolymer and the equilibrium water content of a hydrogel comprising HEMA-co-phosphate functional monomer.

Additionally, as shown in SEM pictures in Figure 10, these functional monomers may chelate with the metallic cations to create complexes that result in a dense and more homogeneous matrix. However, water absorption may significantly support the development of a stable ionic connection and the long-term persistence of the acid–base process.

Theoretically, the density of the polymeric network, the quantity of polar sites capable of forming hydrogen bonds, the polarity of the polymers, and the polar interactions occurring within the matrix might potentially impact water uptake and loss [42, 43].

For the SO, when compared to the control type of cement, all modified types of cement had significantly lower SO ($p<0.001$), and it was immediately apparent that this was directly related to the amount of 2-MEP monomer present in the matrix; SO decreases as monomer weight percentage increases.

This finding explains that the strongly crosslinked polymers are more resistant to degradative processes because the polymeric structure has only a few pores and spaces for solvent molecules to permeate into and out of cement [44]. Additionally, a dense matrix had been created due to the interactions and the polarity of the functional phosphate groups; this matrix is more resistant to water penetration, slowing the water diffusion rate and drastically lowering SO. It is also seen that the percentage of the 2-MEP monomer inside the matrix directly impacts the coefficient of diffusion and SO. Specifically, a higher concentration of monomer leads to a decrease in the coefficient of diffusion and SO. Compared to the control group, this reinforced cement matrix restricts the release of degradative residuals and unreacted components, decreasing the SO of the cement containing 2-MEP. Moreover, this finding is consistent with Al-Taee et al. [17].

While it is challenging to mitigate the development of artificial cracks in RMGICs caused by desiccation under vacuum, a visual examination of the surface revealed the lack of significant cracks in the modified cement, compared to the control RMGIC cement (Figure 10). This "bushy" matrix was correlated with the weight percentage of the 2-MEP monomer within the matrix; the higher the proportion of monomer, the less the cracks and voids formation, thus reducing the surface area exposed to moisture, and this also might be another explanation of less SO of modified cements compared to control one [45].

Additionally, the cement matrix undergoes specific changes, including water retention, by converting the loosely bounded water into tightly bounded one with time, which might compensate for the mass loss due to dissolution [46].

When choosing a durable and effective luting agent, FT is an essential rheological feature to consider. Manipulation variables highly influence FT, and the most important one is cement's consistency, which means the viscosity [47].

The FT and proper adaption of the restoration are both directly impacted by the consistency of the luting agent. More time and force are required for proper restoration seating when using a luting agent with a high viscosity [48].

The FT assessment was conducted per the guidelines outlined in ISO 9917-2:2017 [24]. All groups have less than 25 μm FT, which the IOS standard requires [24]. The mean FT values of the experimental group (2-MEP 10%) and the control (2-MEP 0%) did not vary significantly, and the same result between the 2-MEP 10% and 2-MEP 20%.

The mean FT values for 2-MEP 30% and 2-MEP 40% were noticeably higher than the other groups. The findings were consistent with those of other studies [49, 50]. Because 2-MEP monomer is a very viscous liquid, its addition may directly affect the viscosity of the modified cement; the observed results coincide with the conclusions established in a previous study conducted by Marcondes et al. [51].

It was also noticed that the incorporation of 2-MEP monomer into the liquid phase of cement might increase the rate of conversion through the chemical cure polymerization, which occurs synergistically with the acid–base reaction of RMGIC; this impact directly on the rheology of cement and accelerates the time needed to reach the optimal viscosity which is 10 s before the end of working time by the cement's manufacturer to perform the FT test according to the ISO 9917-2:2017 [24].

The CS and FS of the cement are used not only to estimate the function and survival of new luting cement over time but also to predict the properties and internal structure of formulated luting cement used in this study, which directly influences the behavior of luting cement under load.

The early CS values of eRMGICs at 24 hr are similar to those of the control. However, aging of these cements for 28 days in distilled water at a physiological temperature, all cements showed a statistically significant increase ($p<0.001$) compared to those tested at 24 hr (Table 2).

Mixing polyalkenoic acid and calcium aluminofluorosilicate glass in the presence of water results in the liberation of protons from the acid. This, in turn, triggers the glass hydrolysis and causes the release of Al^{3+}, Ca^{2+}, PO_4^{3-}, and F^-ions. Consequently, polyacrylates are generated, and the glass particles become enveloped by a siliceous layer, which impedes further degradation. The mechanical characteristics of RMGICs tend to enhance with time due to the maturation process of cement [52].

Following a 6-month aging period, it was observed that all modified cements exhibited a significantly higher value in CS when compared to the control reference and their corresponding values after 24 hr ($p<0.001$). The CS values increased directly in proportion to the higher 2-MEP concentration (30% and 40%) (Table 2). The 2-MEP in cement composition serves as spacer molecules within the polyacid environment. These molecules facilitate the mobility of carboxylic acid groups bonded to the rigid polymer structure, enhancing the motion of carboxylate ions.

During the setting reaction, this might facilitate a higher level of carboxylic acid conversion into a complex of metal carboxylate via the salt-bridge formation mechanism. Therefore, the strength of the resultant cement is increased by decreasing the amount of unreacted groups of carboxylic acid present due to the steric hindrance [8].

Furthermore, the 2-MEP monomer, which includes a residue of methacrylates, undergoes radial polymerization through a free-radical initiation mechanism. This process produces a matrix of covalently bonded homopolymers in a random arrangement. Alternatively, the 2-MEP monomer may be copolymerized with HEMA, forming 2-MEP-HEMA copolymers [16]. These polymers can reinforce the matrix, creating types of cement with improved properties.

The synergistic effect is achieved by creating a double-network structure facilitated by the matrix's presence of (ligating-phosphate) groups. The SEM findings (Figure 10), which showed a denser structure with fewer and smaller infrastructural pores, complement the high CS values obtained by the functionalized groups [17].

Regarding FS, the impact of 2-MEP monomer on FS is shown in Table 2, which presents the effect of 2-MEP monomer on FS, indicating up to a double-fold increase in value ($p < 0.001$) in comparison to the control cement at all intervals of time. The FS test is used to predict the elastic property of the material [53].

A material's elasticity property is attributed to the interatomic or intermolecular forces present inside the substance. Materials with effective primary attraction forces exhibit higher FS and elastic modulus values, resulting in increased rigidity and stiffness [54].

Since the FS is significantly influenced by the intermolecular or interatomic forces present in the material, strong hydrophilic domains, such as the 2-MEP, may prevent the detachment of the atoms' planes inside the matrix [55]. They function as a hydrophilic center that supports different hydrophilic domains, perhaps enhancing the bipolar–bipolar forces of dipole molecules and influencing the material's behavior to the FS test [56]. Our finding confirms that increasing the concentration of 2-MEP causes double increases in FS values with little impact on aging.

Physicochemical interactions may influence the cement matrix's strength, as the presence of carbonyl, phosphate, and hydroxyl groups within the matrix may facilitate the formation of hydrogen bonds [17].

Undoubtedly, enhanced bonding between the inorganic and organic networks within the cement matrix increased the set cement's mechanical strength. As a result, these networks can fill the spaces inside the glass-ionomer particles and enhance their overall strength [17].

Based on the methodology, the limitations of the present study were: (1) mechanical testing protocols for the water-based cements in this research were based on the procedure described in ISO 9917-1 and 9917-2. Changes in the composition of RMGICs seem to affect the physical attributes measured by this technique; (2) the current study's recorded failure load values are not intended to have clinical relevance; instead, the relative survival probabilities of the compared luting cements are clinically relevant; (3) the variations in powder/liquid ratios may also affect the early viscosity of the mixture and long-term and short-term properties. However, this study was conducted to simulate the clinical application of RMGIC systems according to the manufacturer's instruction; hand-mixed properties (RMGICs) may be compromised due to inconsistencies in the powder/liquid ratio. These inconsistencies can arise from variations in the densities of packed powder when filling the scoop, how the bottle is grasped, and dispensing liquid drops. Additionally, air bubbles incorporated inside the bottle can further impact the drop amount, exacerbating the issue. It is recommended to weigh both the amount of powder and the liquid to eliminate these discrepancies; (4) using similar approaches, it is advised to investigate the luting agents' behavior attached to the tooth structure and restorative material in vivo by testing their sorption and SO with correlations to their mechanical performances.

The suggestion for future work includes (1) the utilization of mechanical mixing, which exhibits enhanced physical characteristics compared to manual mixing, which was employed in this study. In addition, the improved physical properties of cements containing 2-MEP served as a foundation for developing and examining properties using an auto-mixing technique involving an encapsulated system that eliminated variability caused by operators and dispensing; (2) evaluate the bonding strength of eRMGIC 30% utilizing the microshear bond strength test to different dental substrates; (3) to determine this innovative cement's therapeutic efficacy and remineralizing capability, quantitative and qualitative evaluation of the chemical and physical changes in tooth tissues using the nanoindentation test, second-Harmonic imaging microscopy, and Raman spectroscopy; (4) future studies will use this study's findings to assess the system's viability and clinical performance, as well as the clinical importance of using this material on the long-term survival of fixed prostheses and the evaluation of fluoride-release of experimental cement.

According to the results above of cement's mechanical and physical properties, the null hypothesis that incorporating different weight percentages of 2-MEP (10–40 wt%) into a commercial resin-modified glass ionomer cement has no significant effect on their mechanical and physical properties must be rejected.

6. Conclusions

Within the limitations of this study, the functionalization of RMGIC with phosphate-containing comonomer (2-MEP) could draw many conclusions:

(1) The present study yielded novel eRMGIC cements with superior compressive and FS with low SO compared to the control one, and this effect was directly related to the incorporation of a monomer with phosphate moieties into the liquid phase of control cement.

(2) The modified cements showed a reduction in the ST and an increase in the FT but still met the specifications given by ISO standards 9917_1_2007 and 9917_2_2017.

(3) The recommended weight percentage of eRMGIC is 30%, which exhibited superior strength values over storage durations of up to 6 months.

Data Availability

The dataset that supports the findings of this study is available from the corresponding author (RJK) upon reasonable request.

Ethical Approval

This research has an exemption as it is a routine treatment (no new materials were used).

Disclosure

Both authors thoroughly evaluated and approved the article's final version. They share the responsibility for the text's content and similarity index.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

RJK and AMW conceived and designed the study. RJK made the study samples, performed all the experiments, and collected the data. AMW contributed to the supervision, selection of the different weight percentages of the study, acquisition, and interpretation of results. RJK performed the statistical data analysis, drafted the manuscript and abstract of the article, and provided logistic support.

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