The Effect of Magnesium Oxide Nanoparticles on the Setting Time and Properties of Glass-Ionomer Cement

Hanadia Almira[1], Ellyza Herda[2]*, Sunarso[2]

1. Postgraduate Student at Faculty of Dentistry Universitas Indonesia, Jl. Salemba Raya No.4, Jakarta 10430, Indonesia.
2. Department of Dental Materials Science Faculty of Dentistry Universitas Indonesia, Jl. Salemba Raya No.4, Jakarta 10430, Indonesia.

Abstract

The objective of this study was to determine the effect of adding magnesium oxide (MgO) nanoparticles to glass-ionomer cement (GIC) on setting time, hardness, fluoride release, and antibacterial properties.

This study was conducted on GIC GC Gold Label 9 Extra Shade A3 (GC Corporation, Japan) without addition as the control group and with the addition of MgO nanoparticles at 0.5%, 1%, 1.5%, and 2% (w/w) ratios. The data were analyzed using SPSS statistical software. The addition of MgO nanoparticles increased the setting time significantly ($P<0.05$). However, only GIC with less than 1.5% MgO nanoparticles remained within the ISO 9917-1 (2007) limits. Furthermore, the addition of 1.5% MgO nanoparticles to GIC significantly increased its hardness ($P<0.05$).

Glass-ionomer cement containing 1.5% MgO nanoparticles exhibited the most significant of fluoride ion release ($P<0.05$). Magnesium oxide nanoparticles at concentrations up to 2% could not impart antibacterial properties to GIC. The characteristics of the modified product were affected by the concentration of MgO nanoparticles as well as the composition of the GIC. Further research is required with concentrations of MgO nanoparticles between 1% and 1.5% to improve GIC characteristics, but the setting time is still within the standard range of ISO 9917.

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Introduction

Caries is one of the most prevalent diseases in society, affecting children and adults. Untreated dental caries can cause discomfort.[1] Atraumatic Restorative Treatment (ART) is a method to treat carious lesions with minimum pain and discomfort. Soft carious tissue is eliminated using a hand instrument during the ART procedure. The cavity is then filled with GIC.[2] The GIC restorative material consists of glass particles and polymeric acid liquid.[3] Mixing the powder with liquid GIC results in a setting reaction.[4] Glass-ionomer cement has the benefits of adhering to the tooth structure, releasing fluoride, and being biocompatible.[5,6] Despite this, GIC has inferior mechanical properties compared to composite resins.[4]

Hardness is one of the necessary mechanical properties of dental materials. Hardness is a material's resistance to indentation.[4] Opposing teeth in the oral cavity make an indentation during the process of mastication.[7] Indentation can lead to the formation of scratches, abrasions, and cracks.[8,9] Glass-ionomer cement is a brittle material that cannot withstand repetitive heavy occlusion loads.[5,6,10] These negative characteristics make the use of GIC in dentistry more difficult.[6]

Glass-ionomer cement is capable of releasing fluoride. Fluoride prevents secondary caries by supporting remineralization.[5] The amount of fluoride released by GIC is insufficient to inhibit bacterial growth beneath the restorative material.[11] Glass-ionomer cement releases a significant amount of fluoride in the first few days, swiftly decreasing over the first week. Caries are caused by the imbalance between demineralization and remineralization.[5] The development of secondary caries results in restoration failure.[12]

The researchers modified GIC by

*Corresponding author:
Prof. Dr. Ellyza Herda, DDS, M.Sc.
Department of Dental Materials Science
Faculty of Dentistry, Universitas Indonesia, Jakarta 10430, Indonesia.
E-mail: ellyza.herda@ui.ac.id

incorporating nanoparticles of metal oxide to enhance the positive properties without altering the existing GIC properties.[11] The size range of nanoparticles is 1 to 100 nm.[13] A previous study has demonstrated that metal oxide nanoparticles can enhance the mechanical properties of GIC.[11] Metal oxide nanoparticles may act as reinforcement by filling the voids between the GIC particles, enhancing mechanical properties.[14] Other studies have shown that adding metal oxide nanoparticles to GIC gives antibacterial properties.[15] The research on the addition of metal oxide nanoparticles to GIC produces diverse outcomes. Several metal oxides nanoparticles have been introduced to GIC, including zinc oxide, titanium dioxide, and magnesium oxide.[11,16–19]

Magnesium is one of the inorganic elements that compose tooth enamel.[20] Magnesium oxide nanoparticles are desirable due to their high boiling points, low solubility in water, thermal insulation characteristics, and non-toxicity.[21] The Food and Drug Administration has determined that MgO nanoparticles are safe. Magnesium ions, a by-product of MgO, are also biocompatible. Previous research has demonstrated that MgO nanoparticles can enhance the compressive strength, diametral tensile strength, and shear bond strength of GIC.[11] Various concentrations of MgO nanoparticles have been added to GIC under different brand names.[11,18] Modification of GIC composition can also influence setting time, hardness, fluoride release, and antibacterial activity.[11,17,18] The objective of this study was to determine the effect of adding MgO nanoparticles to GIC on setting time, hardness, fluoride release, and antibacterial properties.

Materials and methods

Materials and Specimen Preparation

Glass-ionomer cement GC Gold Label 9 Extra Shade A3 (GC Corporation, Japan) consisting of powder, liquid, and 50 nm-sized commercial magnesium oxide nanoparticles (Aldrich, USA) were prepared. Glass-ionomer cement without additions was used as the control group. Magnesium oxide nanoparticle powder was mixed with GIC powder in the experiment group at weight/weight ratios of 0.5%, 1%, 1.5%, and 2% using a Turbula shaker (Alphie, India).[11,22] Specimens were made by mixing GIC powder and liquid in the proportions specified by the manufacturer.[23]

Scanning Electron Microscope, Fourier Transform Infrared Spectroscopy, and X-Ray Diffraction Analysis

Powders of MgO nanoparticles and GIC were analyzed using X-ray Diffraction (XRD) (PANalytical, UK) operating at 45kV with a current of 40 mA and CuKα radiation (λ = 1,5406Å) in the 30° - 90° range. Glass-ionomer cement control and MgO nanoparticles-modified GIC were analyzed using Fourier Transform Infrared Spectrometer (FTIR) (Thermo Fisher Scientific, USA) with Attenuated Total Reflectance in the range of 400 to 4000 cm^{-1} and a Scanning Electron Microscope (SEM) with Energy Dispersive Spectroscopy (EDS) (Field Electron and Iron Company (FEI), USA) operating at 25kV to identify the loading of MgO nanoparticles on GIC.[11]

Setting Time Test

The setting time test used five 10×8×5 mm specimens.[11,24] A Vicat apparatus (Teguh Primatama, Indonesia) with a 400-gram indenter and a one mm-diameter flat-end needle was used to perform the setting time test.[24,25] The indenter needle was lowered to the specimen's surface after 90 seconds of mixing and let to remain for 5 seconds. Subsequent indents were made at 30-second intervals until the needle couldn't create a precise circular indentation in the test material. Indentations are made at 10-second intervals when the estimated time of setting approaches.[25] The net setting time of cement was measured according to ISO 9917-1 (2007) from the end of mixing until a needle indentation failed to form a well-defined circle in a specimen.[24]

Hardness Test

The hardness test used five specimens with a 6 mm diameter and 3 mm thickness.[26] The mylar strip, glass slide, and 200-gram load were then placed on top.[27] The specimens were left to rest for ten minutes before being removed from the mould.[28] The specimens were then placed in a container containing moist cotton and incubated for 24 hours at 37°C.[9] The hardness of specimens was tested using a Vickers hardness testing machine (Shimadzu, Japan) with a 50-gram load for 10 seconds.[28] Five indentations were recorded on each surface of the specimen.[29]

Fluoride Release Test

The fluoride release test used five specimens with a diameter of 6 mm and a thickness of 3 mm, which were produced using the same technique as specimens for hardness testing.[30] The specimens were immersed in 50 mL of deionized water at 37°C. The specimens were taken out, dried with absorbent paper, and transferred to fresh deionized water every twenty-four hours. Fluoride release testing was performed on deionized water on days 1, 7 and 14.[6] The deionized water was added in a 1:1 volume ratio to the Total Ionic Strength Adjusting Buffer (TISAB) (Horiba, Japan) solution, and the test was performed using a Fluoride Ion Selective Electrode (ISE) (Horiba, Japan).[31]

Antibacterial Test

Streptococcus mutans bacteria were cultured in Brain Heart Infusion (BHI) (Oxoid, UK) broth and incubated for 24 hours at 37ºC.[32] The antibacterial test used five specimens with a diameter of 6 mm and a thickness of 3 mm, which were produced using the same technique as specimens for hardness testing.[6] *Streptococcus mutans* bacteria were spread on BHI agar using a sterile swab. The specimens were placed on BHI agar plates, which were incubated at 37°C for 24 hours. The diameter of the inhibition zone was measured with a calliper at three distinct points. The size of the inhibition zone surrounding the GIC specimen was calculated by subtracting the diameter of the specimen from the average of three measurements of the inhibition zone's diameter.[18]

Statistical Analysis

The data were analyzed using IBM SPSS software, version 26 (SPSS Inc., USA). Shapiro-Wilk was utilized to assess normality (P>0.05). Kruskal-Wallis and Mann-Whitney post-hoc tests were utilized for comparing the setting time values and the concentration of the fluoride ion released between group on day 7. One-way ANOVA and Bonferroni post-hoc tests were utilized for comparing the hardness values. One-way ANOVA and Tamhane post-hoc tests were utilized for comparing the concentration of the fluoride ion released between group on day 1 and day 14. Friedman and Wilcoxon post-hoc tests were utilized for comparing the concentration of the fluoride ion released between various days on control group and 2% MgO nanoparticles-modified GIC group. General Linear Model Repeated Measure ANOVA and Bonferroni post-hoc test were utilized for comparing the concentration of the fluoride ion released between various days on 0.5%, 1% and 1.5% MgO nanoparticles-modified GIC group. Statistical significance was set at P<0.05.

Results

The peaks (111), (200), (220), (311), and (222) were at a diffraction angle of 36.9°; 42.8°; 62.2°; 74.5°; and 78.8°, which conformed to JCPDS No. 87-0653 and indicated that the material being examined is MgO, as shown in figure 1(a). The sharp peak on the diffractogram indicated that the MgO under analysis is crystalline.[11,18] The absence of a peak in figure 1(b), the GIC powder diffractogram, indicated that the tested material has an amorphous phase.[33,34]

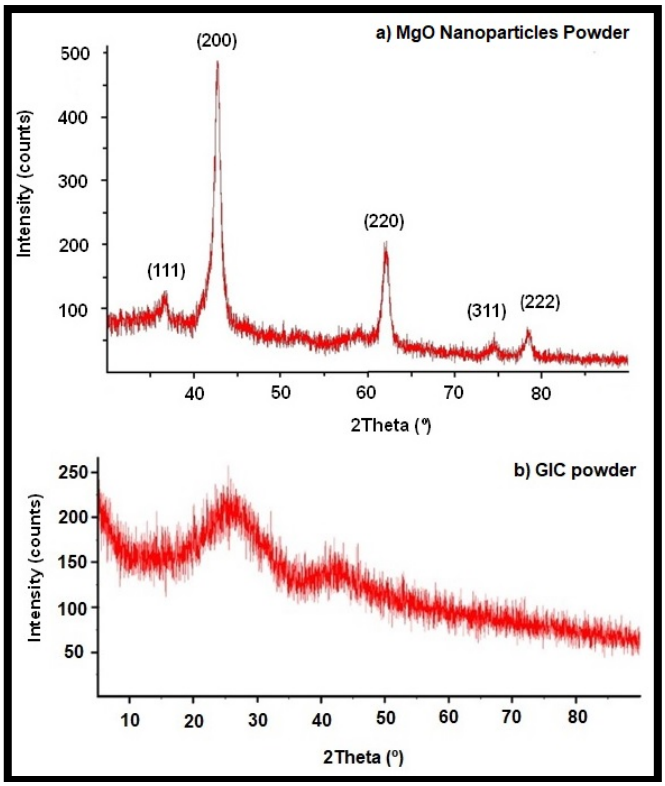

Figure 1. Diffractogram of (a) MgO nanoparticles powder and (b) GIC powder.

The peak at wave number 855 cm^{-1} in figure 2(a) of the FTIR spectrogram of MgO nanoparticles powder indicated stretching vibrations of Mg-O, which are characteristic of cubic MgO.[11,35] The FTIR spectrogram of GIC with the addition of MgO nanoparticles, as depicted in figure 2(b), had peaks that resembled

the unmodified GIC spectrogram, as depicted in figure 2(c). There was a peak at 3000–4000 cm^{-1}, which indicated stretching O-H vibration of water molecules; 2350 cm^{-1}, which indicated asymmetric stretching C=O of CO_2 in air; 1695 cm^{-1} and 1527 cm^{-1}, which indicated asymmetric stretching COO^- of polyacrylic acid with metal; 1030 cm^{-1}, which indicated asymmetric stretching vibration Si-O-Si; and 800 cm^{-1}, which indicated stretching vibration of metal-oxygen bonds related to the presence of MgO.[11,36–42]

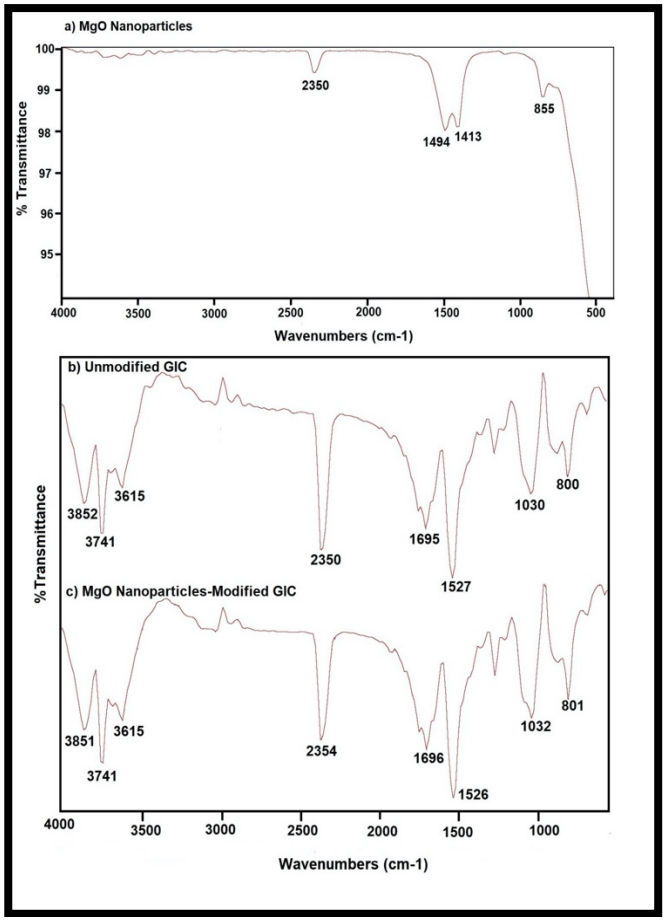

Figure 2. FTIR Spectrogram of (a) MgO nanoparticles, (b) unmodified GIC, and (c) MgO nanoparticles-modified GIC.

Figure 3 depicts the results of SEM-EDS mapping on unmodified GIC and MgO nanoparticles-modified GIC. It appeared that unreacted glass particles of varying sizes and shapes were dispersed throughout the matrix. Silicon, aluminium, strontium, oxygen, sodium, and fluoride were the chemical elements that had been identified. The distribution of elements on the GIC's surface, as revealed by elemental mapping, indicated that silicon, aluminium, and strontium were present in sufficient quantities. A small amount of magnesium appeared uniformly distributed on the surface of the MgO nanoparticles-modified GIC, indicating that the MgO nanoparticles were successfully mixed homogeneously.

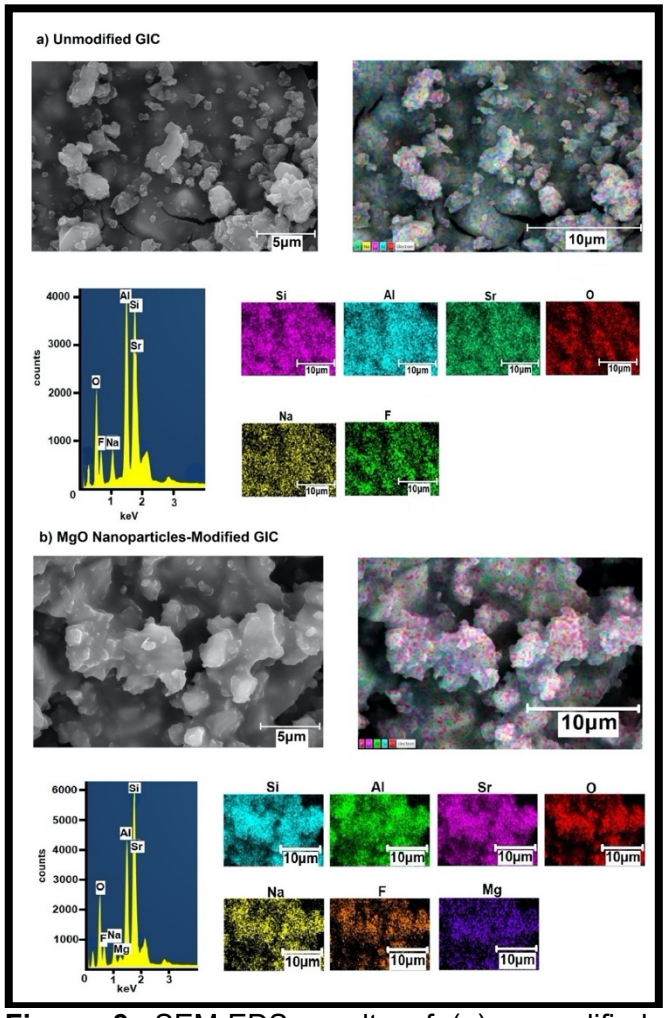

Figure 3. SEM-EDS results of (a) unmodified GIC and (b) MgO nanoparticles-modified GIC.

Table 1 demonstrated that increasing the number of MgO nanoparticles in the GIC significantly lengthened the setting time between the test groups (P<0.05). The setting period of GIC containing less than 1.5% MgO nanoparticles was still within the range specified by ISO 9917-1 (2007), which is 1.5 to 6 minutes.[24]

Table 2 shows that the addition of MgO nanoparticles increased the hardness of GIC compared to the GIC control. The hardness of modified GIC increased significantly upon adding MgO nanoparticles, starting from 1.5% (P<0.05).

Group	Setting Time (Minutes) ± SD	*P*
GIC Control	4.27 ± 0.09	
GIC + 0.5% (w/w) MgO	4.64 ± 0.08	0.006*
GIC + 1% (w/w) MgO	5.37 ± 0.09	0.007*
GIC + 1.5% (w/w) MgO	6.07 ± 0.09	0.007*
GIC + 2% (w/w) MgO	7.80 ± 0.07	0.006*

* Mann-Whitney post-hoc test with significant set at *P*<0.05

Table 1. The Results of The Mean Setting Time Test.

Group	Hardness (VHN) ± SD	*P*
GIC Control	61.18 ± 2.33	
GIC + 0.5% (w/w) MgO	63.20 ± 3.31	1.000
GIC + 1% (w/w) MgO	64.24 ± 3.27	1.000
GIC + 1.5% (w/w) MgO	71.52 ± 4.95	0.004*
GIC + 2% (w/w) MgO	76.20 ± 4.69	<0.001*

* Bonferroni post-hoc test with significant set at *P*<0.05

Table 2. The Results of The Mean Hardness Test.

Group	Fluoride Ion Concentration on Day 1 (mg/L) ±SD	Fluoride Ion Concentration on Day 7 (mg/L) ±SD	*P* between day 1 and day 7	Fluoride Ion Concentration on Day 14 (mg/L) ±SD	*P* between day 1 and day 14
GIC Control	1.287 ± 0.22	0.168 ± 0.02	0.043**	0.095 ± 0.01	0.043**
GIC + 0.5% (w/w) MgO	1.305 ± 0.13	0.172 ± 0.03	<0.001*	0.097 ± 0.01	<0.001*
GIC + 1% (w/w) MgO	1.603 ± 0.19	0.174 ± 0.02	<0.001*	0.099 ± 0.01	<0.001*
GIC + 1.5% (w/w) MgO	5.768 ± 1.75	0.573 ± 0.22	0.005*	0.353 ± 0.15	0.005*
GIC + 2% (w/w) MgO	3.164 ± 1.45	0.327 ± 0.15	0.043**	0.220 ± 0.11	0.043**

** Wilcoxon post-hoc test with significant set at *P*<0.05
* Bonferroni post-hoc test with significant set at *P*<0.05

Table 3. The Results of The Mean Fluoride Released Test.

Table 3 displays the results of the GIC fluoride release test. The addition of MgO nanoparticles increased the concentration of fluoride ions released by GIC, which improved significantly at 1.5% and 2% concentrations (*P*<0.05). The highest concentration of fluoride ions was detected in GIC when 1.5% MgO nanoparticles were added. The concentration of fluoride ions released by GIC on days 1, 7, and 14 significantly decreased over time (*P*<0.05).

The results of the antibacterial test indicated that after 24 hours of incubation, no inhibition zones had formed in any of the test groups. The addition of MgO nanoparticles had no antibacterial effect on GIC.

Discussion

This study showed that the addition of MgO nanoparticles to GIC at 0.5%, 1%, 1.5%, and 2% concentrations affected the setting time, hardness, and fluoride release. The incorporation of MgO nanoparticles increased the viscosity of the material during manipulation. The higher the concentration of the addition of MgO nanoparticles, the more difficult it was to manipulate due to an increase in viscosity, the consistency became more like rubber, and the colour became whiter than the control GIC. Changes in viscosity can occur because the addition of nanoparticles increases the powder's surface area, preventing the same quantity of liquid from wetting the powder particles.[43]

The surface topography, composition, and distribution of elements can be detected using SEM-EDS mapping. Scanning electron microscope operations are conducted in a vacuum, which evaporates the water from the GIC matrix, causing microcracks to form and altering the specimen's surface.[44] Figure 3 showed a surface with a rough texture, and the unmodified GIC displayed visible microcracks. The EDS of the MgO-modified GIC demonstrated that magnesium was homogeneously distributed on the specimen's surface, indicating the possibility for magnesium ions to form the matrix. This was also supported by the FTIR results depicted in Figure 2, which demonstrated that both unmodified GIC and MgO-modified GIC had a peak at a wavenumber of approximately 800cm^{-1}, corresponding to metal oxygen bonds, indicating that the MgO nanoparticles were successfully embedded in the GIC matrix.[11]

The presence of magnesium ions inhibits or interferes with the acid-base reaction, resulting in a lengthened setting time.[11,45] Initial setting results from the cross-linking of metal cations and poly anions, namely COO^- groups, to form a polysalt matrix.[40] Magnesium oxide slows down the conversion of COOH to COO^-.[46] The GIC setting process will appear on FTIR as the loss of carboxylic acid groups from the polyacrylic acid peak (COOH) at approximately 1700 cm^{-1}, resulting in a shoulder-shaped peak, along with the formation of polysalts (COO^-) in the range of 1350 – 1750 cm^{-1}.[40,47] Different study found that in the 1800s after the mixture process, the COOH peak decreases and becomes less prominent than COO^-.[39] Figure 2 showed a

shoulder peak at around 1700 cm^{-1}, which resulted from the loss of carboxylic acid groups, and peaks at 1695 cm^{-1} and 1527 cm^{-1}, which indicated the formation of polysalts. The addition of MgO nanoparticles to GIC 3M ESPE Ketac Molar Easymix Shade A3 (3M Deutschland GmbH, Germany) lengthens the setting time, but only up to a maximal concentration of 2.5% that still meets ISO limits.[11] However, the concentration of MgO nanoparticles added to GIC Gold Label 9 Extra Shade A3 (GC Corporation, Japan) in this study was still below 1.5%, which is within the ISO limits. The setting time of a 1.5% concentration exceeded the ISO limit by 1.1%. This difference in maximum concentration might be related to the use of two distinct GIC products with different chemical compositions. The composition influences the characteristics of the GIC that is produced.[4]

The addition of MgO nanoparticles increased the hardness of GIC. This happens because the nanoparticles fill the voids between the larger glass particles, making the cement more homogenous.[11,14,46] Additionally, nanoparticles provide additional bonding sites for polyacrylic polymers, thus hardening GIC.[11,14]

Powder manipulation with liquid GIC will result in the discharge of ions from glass particles, one of which is the fluoride ion.[48] Fluoride ions are not a part of the matrix and have a small size, allowing them to be released into the surrounding environment, which has a lower fluoride concentration.[5] Fluoride ions frequently bond to cations to make them more stable before being released in complex forms.[48] The magnesium ion is an example of a cation.[21] This study demonstrated that the presence of MgO nanoparticles on days 1, 7, and 14 increased the fluoride ion concentration released by GIC. The addition of 2% MgO nanoparticles decreased the concentration of fluoride ions compared to 1.5%. This may be due to the maximal number of metal oxide nanoparticles that are incapable of forming cross-links with polyacrylic acid. The reduction of monovalent ionic bonds linking polymer chains can decrease water transport within molecules and the release of fluoride.[48] This study also determined that the concentration of fluoride ions in GIC on the first day was the highest, followed by day seven and then day 14. Glass-ionomer cement will release significant amounts of fluoride in the first few days due to the initial superficial rinsing effect. Lesser quantities are diffused through the pores and cracks of the cement for long-term release after a few days.[3,5,6,49] The significant reduction in fluoride release with increasing age of GIC can be attributed to the slower dissolution of glass particles through the pores and the formation of the matrix during the setting process.[6,48] Fluoride release test results can be affected by the type and volume of immersion medium, research method, powder-to-liquid ratio, solubility, porosity, and specimen size.[48]

Fluoride released by GIC may be useful in remineralization, but the quantity is insufficient to provide an antibacterial effect.[5,50] Another study reported that 2.5% MgO nanoparticles with a size of 20 nm can impart antibacterial properties to GIC.[18] This study demonstrated that the addition of MgO nanoparticles with a size of 50 nm at concentrations as high as 2% did not provide antibacterial properties on GIC. This difference may be owing to the nanoparticle size of the MgO used. The size of MgO nanoparticles affects their antibacterial activity. The smaller the MgO nanoparticles are, the greater their antibacterial capability. MgO particles with a size between 45 - 70 nm exhibited an increase in antibacterial activity, whereas those smaller than 45 nm became more potent. Besides size, the concentration of MgO nanoparticles affects their antibacterial activity. A greater concentration will result in a more potent antibacterial effect. A concentration of MgO nanoparticles that is too low cannot possess antibacterial properties.[51]

Conclusions

Within the limits of this study, it was found that the addition of up to 2% MgO nanoparticles increased the hardness and fluoride release of GIC GC Gold Label 9 Extra Shade A3 (GC Corporation, Japan), but it lengthened the setting time and did not possess antibacterial properties. The characteristics of the modified product depend on the concentration of MgO nanoparticles and the composition of the GIC. Further research with concentrations of MgO nanoparticles between 1% and 1.5% is required to achieve improved GIC characteristics while maintaining a setting time within the ranges of ISO 9917.

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Declaration of Interest

The authors report no conflict of interest.

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