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ORIGINAL RESEARCH

Flexural Strength and Morphological Characteristics of Resin-modified Glass-ionomer Containing Bioactive Glass

Sayed Mostafa Mousavinasab, Maryam Khoroushi, Fateme Keshani, Shirin Hashemi

ABSTRACT

Introduction: Recent advances in dental materials have led to the production of smart materials. Recently, addition of bioactive materials to glass-ionomer cements has resulted in new capabilities beyond the beneficial effects of fluoride release. This in vitro study compared the flexural strengths (FS) of a resin-modified glass-ionomer containing bioactive glass (RMGI-BAG) with that of a commonly used resin-modified glass-ionomer (RMGI).

Methods and materials: A total of forty RMGI and RMGI-BAG bars (20 × 4 × 4 mm) were prepared in stainless steel molds. Each of the RMGI and RMGI-BAG bars was set for FS test. FS values of the specimens were measured using three-point bending test at a crosshead speed of 0.5 mm/min. The surface changes and the amounts of elements on the materials' surfaces were also evaluated by SEM/EDS analyses. Data were analyzed using SPSS 11.5 and t-test ($\alpha = 0.05$).

Results: The means ± SD in the study groups were 61.46 ± 22.52 and 39.90 ± 9.11 MPa respectively. There were significant differences between FS of the two study groups ($p = 0.003$).

Conclusion: While adding 20 wt% of BAG to the RMGI powder evaluated in this study decreases FS of the material significantly, the mean value of FS is in the acceptable range of the reported FS values for conventional GIs and RMGIs that are commercially available for clinical use.

Clinical significance: While flexural strength of RMGI decreases subsequent to addition of bioactive glass, it is still clinically acceptable considering the flexural strength values reported for clinically used GIs and RMGIs. Further studies are recommended.

Keywords: Flexural strength, Resin-modified glass-ionomer, Bioactive glass.

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INTRODUCTION

Conventionally, the materials used in the human body, especially those used in the oral cavity, should be stable and passive, with no interactions with the materials in the surrounding environment. Amalgam, composite resins and cements mostly have those characteristics. Probably the first idea about producing active materials, which interact with the human body, originated from the fact that if dental materials can release fluoride, they will be able to bring about beneficial effects. The idea of producing smart materials in dentistry has gained momentum in recent years.[1,2]

Davidson, for the first time, paid attention to the smart behavior of glass-ionomer cements.[1] Glass-ionomer (GI) cements are widely used in restorative dentistry.[3] A major advantage of glass-ionomer over other restorative materials is the fact that they are placed in the oral cavity without any need for an additional bonding agent.[4] Glass-ionomer is also compatible with the pulp.[3,4] Although glass-ionomers are commonly used as cements in dentistry, they have disadvantages, the most important of which is inadequate strength and toughness. Resin-modified glass-ionomers (RMGI) were introduced in an attempt to improve the mechanical properties of conventional glass-ionomers; they contain hydrophilic polymers and monomers, such as HEMA.[3] According to a study, RMGIs have a significantly higher flexural strength compared to conventional glass-ionomers (71 MPa *vs* 11 MPa).[5]

Recently in some studies, bioactive glass (BAG) has been incorporated into GI structure to improve materials bioactivity, regeneration capacity and reconstruction.[3,5-8] There is increasing attention to the use of bioactive materials in dentistry with the aim of dentin remineralization. A number of studies have reported remineralization-inducing properties for such materials.[4,6-8] It is probable that the use of bioactive materials for tooth restoration procedures in open/closed sandwich techniques or restoration of root

surfaces is more beneficial than the use of RMGI or conventional GI, especially in patients at a high-risk for carious lesions. In addition, their use as cavity liners in deep cavities is of clinical significance.

Generally, biomaterials are synthetic materials which do not induce a toxic response when they contact human tissues.[9] When a material replaces a living tissue, different tissue responses are elicited depending on the material used, which include toxic, nearly biologically inert, bioresorbable and bioactive responses. When a biomaterial is placed in living tissues, some specific biochemical reactions occur at the biomaterial tissue interface and a process called bioactive fixation is initiated. Therefore, a bioactive material exhibits a behavior intermediate between a bioresorbable material and a nearly inert material and can create an environment capable of inducing a proper bond between living tissues and the material.[9] According to the definition above; bioactive materials induce a specific biologic response at tissue material interface.[9]

Bioactive glass contains silicon, sodium, calcium and phosphorus oxides; it was introduced by Larry Hench in 1969, as 45S5 Bioglass with the following weight distribution: CaO, 24.5%; Na_2O, 24.5%; SiO_2, 45% and P_2O_5, 6%.[10]

Clinically, this material was at first used as a biomaterial to replace lost osseous tissues in the human body. The material produces a hydroxyapatite layer and forms a chemical bond with collagen to produce a strong bond with bone without being rejected by the body.[10]

Several studies have used various chemical compositions of Bioglass. Xie et al[7] used Vivoxid with S53P4 formula (weight percentages of P_2O_5, 4%; CaO, 20%; Na_2O, 23%; SiO_2, 53%). Vollenweider et al[11] used NBG with 45S4 formula (weight percentages of SiO_2, 44.7%; P_2O_5, 4.9%; CaO, 27.6%; Na_2O, 22.8%) and Perioglass (NovaBone) with 45S5 formula and micron-sized particles. Marending et al[12] too, used 45S5 formula.

In this context, some researchers have studied the effect of these materials on tooth structures and some others have evaluated the physical and mechanical properties of these materials. Ana et al[8] evaluated the effect of incorporating bioactive glass into RMGI on its setting and mechanical properties and reported that its compressive strength decreases to some extent but it is still much higher than that of conventional GI containing bioactive glass. The results of the study showed a compressive strength of 148.7 MPa for RMGI and a compressive strength of 203.1 MPa for RMGI combined with 33 wt% of bioactive glass.[8] In a study by Urpo et al[3] BAG was added to glass-ionomer cement and the compressive strength, Young's modulus of elasticity and Vicker's hardness of the material were evaluated. This experimental material is bioactive in physiologic conditions and can mineralize human dentin *in vitro*. It also has anti-microbial properties.[3,13] Xie et al[7] used a polyacid produced by himself in order to improve the mechanical properties of a combination of glass-ionomer and bioactive glass. He measured compressive strength, diametral tensile strength and hardness and showed that this material has a strength comparable to that of Fuji II LC cement. However, some of the mechanical properties of this combination, including its flexural strength, have yet to be evaluated. Therefore, the aim of the present study was to evaluate flexural strength of combination of RMG and a type of bioactive glass (RMGI-BAG) in comparison with the flexural strength of a commercially available RMGI; in addition, SEM photomicrographs of both materials were evaluated.

MATERIALS AND METHODS

In the present experimental study, a commercially available RMGI (Improved Fuji II LC) (Batch: #0912011) (GC Corporation, Tokyo, Japan), which is a resin-modified, radiopaque, light-cured, restorative glass-ionomer, was used. It is available in a kit containing a liquid and powder (Table 1). In addition, NovaBone bioactive glass (NovaBone Products, LLC, Alabama, Florida, USA) was used, which is a synthetic bioactive graft material. It is a 45S5 bioglass with a chemical composition of SiO_2, 45%; P_2O_5, 6%; CaO, 24.5%; Na_2O, 24.5%, and a particle size of 90 to 710 µm. A pack of NovaBone contains 10 ml of the material, equal to 13.2 g of the material (Table 1).

Table 1: The materials used in the study, their compositions and manufacturers

Material	*Product name*	*Manufacturer*	*Composition*
Resin modified glass ionomer	Fuji II LC (improved)	GC Corporation, Tokyo, Japan	Powder: Fluoroaluminosilicate glass Liquid: Polyacrylic acid (20-25%), 2-hydroxylethyl methacrylate (30-35%), 2,2,4, trimethyl hexamethylene dicarbonate (1-5%), proprietary ingredient (5-15%)
Bioactive glass (45S5 bioglass)	NovaBone	NovaBone Products LLC, Alachua, Florida, USA	45% SiO_2, 24.5% Na_2O, 24.5% CaO, 6% P_2O_5

Preparation of RMGI Containing Bioactive Glass (RMGI-BAG)

Fuji II LC RMGI and NovaBone bioactive glass powders were mixed and milled manually in a mortar, with a 20 wt% of bioactive glass.[14,15] Fuji II LC liquid was used in the present study.[8] A metallic mold, measuring 4 mm × 4 mm × 20 mm, was custom-made to prepare RMGI and RMGI-BAG bars in order to measure flexural strength values of the materials. The mold was used to prepare 20 RMGI and 20 RMGI-BAG bars by separately placing the mixed materials in the mold. In order to prepare RMGI bars, a powder-to-liquid ratio of 3:2 was used according to manufacturer's instructions; in case of RMGI-BAG, a powder-to-liquid ratio of 2:7 was used based on previous studies.[3,4,7,8,16,17] The mixture was placed in the mold and gently pressed using a translucent matrix band and then light-cured for 40 seconds at a light intensity of 600 mW/cm^2 and a wavelength of 470 nm using a light-curing unit (Dr's Light, Doctors Co Ltd, Seoul, Korea). Each bar was carefully retrieved from the mold and again light-cured from the opposite direction for another 40 seconds. All the preparation procedures were carried out at a room temperature of 22 ± 1°C. Then all the specimens underwent a three-point bending test in a universal testing machine (DARTEC, Model HCIO, Southbridge, England) to evaluate flexural strength. The machine applied the force to the center of the specimens at a crosshead speed of 0.5 mm/min. Flexural strength (FS)[10,11,18,19] of each specimen was calculated using the following formula: $\frac{3pl}{2bd^2}$

In this formula, "p" is the maximum load or force which is applied to the center of the specimen to fracture it; "l" is the distance between the two rests on the surface under the tensile force; "b" is the width and "d" is the height of the specimen between the surfaces under the tensile and compressive forces. Data was analyzed by t-test using SPSS (version 11.5) software (α = 0.05). The surfaces of two specimens from each material were evaluated under a SEM (Seron Technology, Model AIS2300C, Korea). To this end, the specimens were dehydrated in a dessicator.[3] Then the specimens were sputter-coated by a 10 to 15 nm layer of gold-palladium in a sputter-coater (Model BAL-TEC SCD 005, Germany). Energy-dispersive X-ray spectroscopy (EDS) elemental analysis (IXRF systems, Inc 15715 Brookford Drive, Houston, USA) was used to characterize the compositions of the materials' surfaces. SEM evaluation and EDS analysis were carried out in a standard technique in vacuum using a voltage of 22 kV at a distance of 20 to 25 mm.

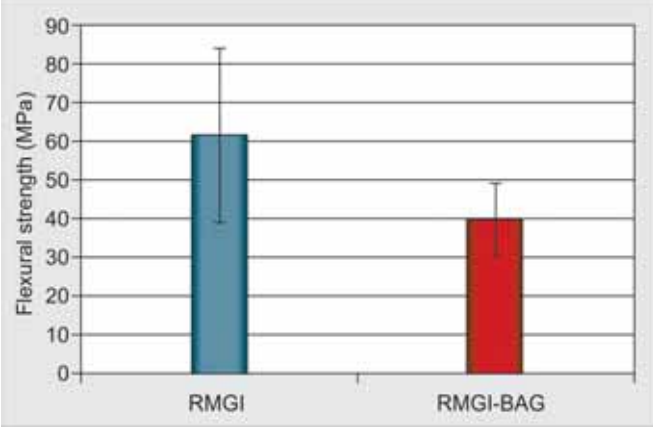
RESULTS

FS values for RMGI and RMGI-BAG are summarized in Table 2 and Graph 1. Statistically significant differences were observed between the two groups (p = 0.003). The results of EDS (energy dispersive X-ray spectroscopy) analysis, including intensity values (counts/second), phosphorus and calcium wt% and analysis of EDS spectrum of the materials surfaces are presented in Tables 3 and 4. SEM photomicrographs of both materials/EDS spectra are presented in Figures 1A and B, and Graphs 2A and B

Table 2: Flexural strength of the two studied materials (MPa)

Groups	*Mean ± SD*	*95% confidence Interval*		*Min*	*Max*
		Lower bound	*Upper bound*		
RMGI	61.4625 ± 22.51904	50.9233	72.0017	17.25	97.50
RMGI-BAG	39.9000 ± 9.11311	35.6349	44.1651	28.50	64.50

RMGI: Resin-modified glass-ionomer; RMGI-BAG: Resin-modified glass-ionomer containing bioactive glass.



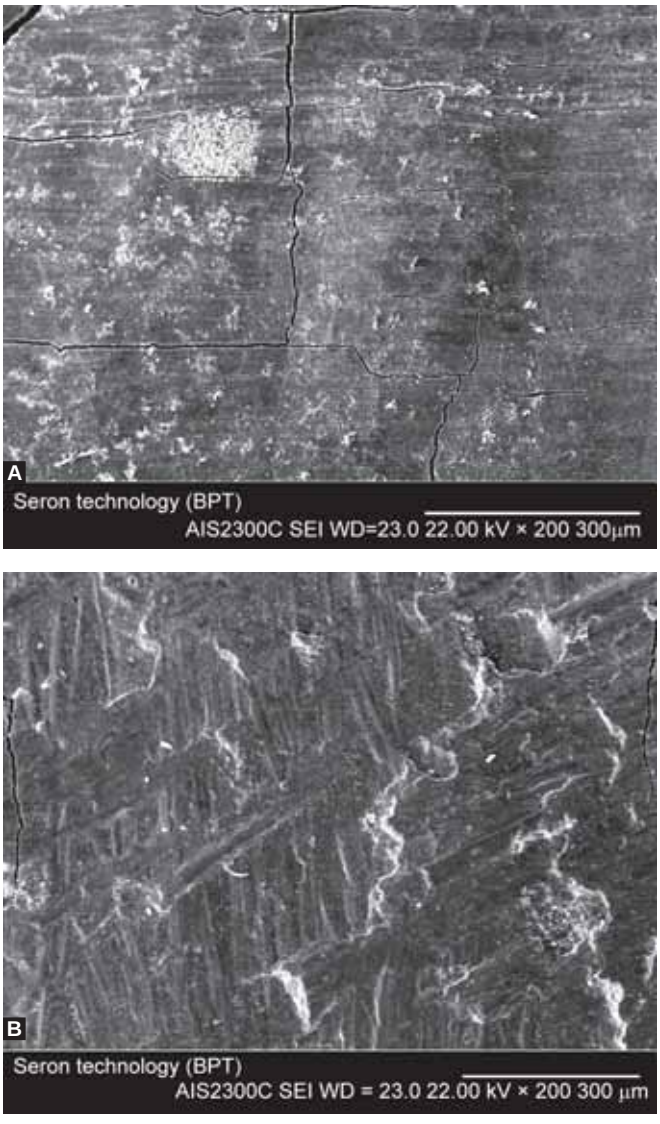
Graph 1: Flexural strength of the two studied materials (MPa) (RMGI: Resin-modified glass-ionomer; RMGI-BAG: Resin-modified glass-ionomer containing bioactive glass)

Table 3: The intensity and the percentages of different elements on the surface of RMGI according to EDS analysis

Element	*Intensity (C/S)*	*Concentration (wt%)*
Na	1.75	0.981
Al	30.36	12.269
Si	34.03	14.922
P	54.02	35.252
Ca	10.22	5.851
Sr	20.49	24.419

Table 4: The intensity and percentages of different elements on the surface of RMGI-BAG according to EDS analysis

Element	*Intensity (C/S)*	*Concentration (wt%)*
Al	25.72	12.719
Si	63.98	36.926
P	48.46	42.617
Ca	9.29	7.738



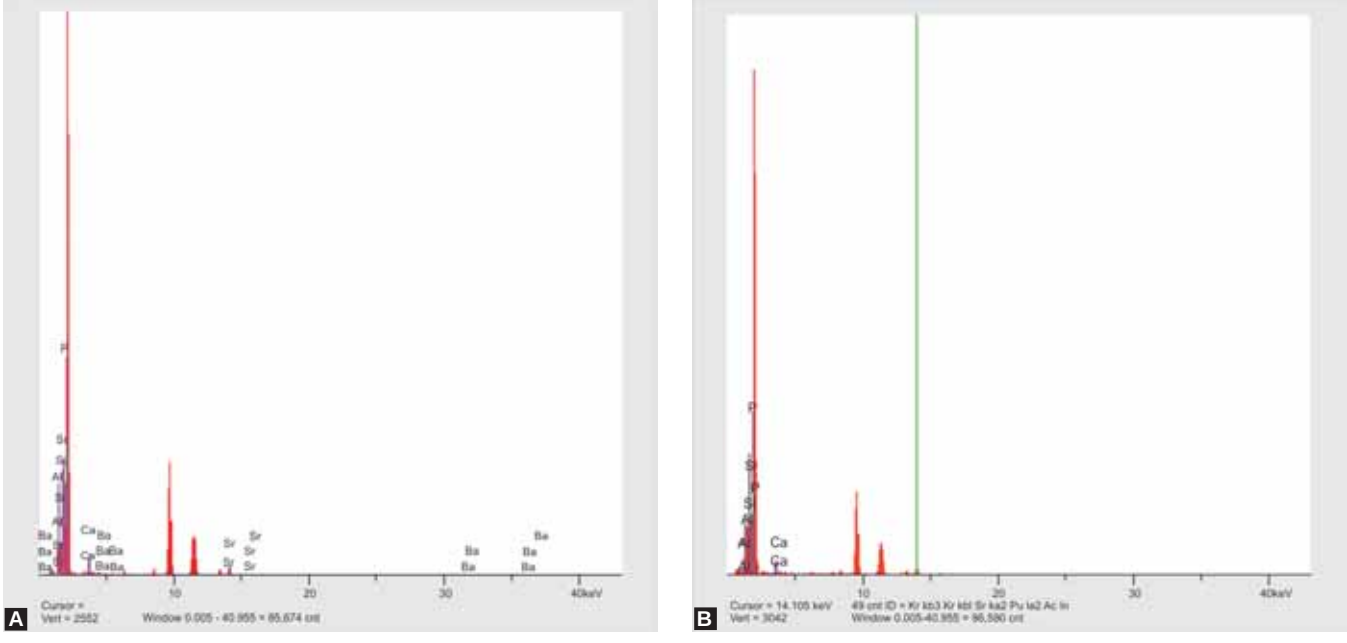
Figs 1A and B: Scanning electron photomicrograph of the RMGI and RMGI-BAG surface (Original magnification 200×)

respectively, which exhibit cracks on RMGI surfaces. However, deposits are visible on RMGI-BAG surfaces with no cracks.

DISCUSSION

The present study compared flexural strength (FS) values of RMGI and RMGI-BAG restorative materials. Three-point flexure test is used in specific ISO tests for dental restorative materials[18,19] and is clinically common because when these materials are used in the tooth cervical areas and root surfaces and also in small class I cavities, the material undergoes flexure. It appears fracture resistance of the material, which is determined with the flexural strength parameter[20], is one of the valuable parameters in evaluating this material.[21] The FS of RMGI (Fuji II LC) was 61.46 ± 22.5 MPa in the present study. Recently, Zhao and Xie reported a flexural strength of 35.8 ± 4.1 MPa for this material in a study.[21] Also, Xie et al in a separate study reported a FS value of 52.8 ± 1.9 MPa for the material.[22] FS values of RMGI have been reported to be 42 to 66 MPa,[19] 25 to 60 MPa[23] and 16.9 to 59 MPa[24] in various studies. Xie et al reported a FS value of 71.1 ± 3.6 MPa for RMGI (Fuji II LC).[5]

In the present study, an FS value of 39.90 ± 9.1 MPa was achieved for RMGI-BAG, which is higher than that of conventional GI. In previous studies, FS values of 20 MPa[25] and 11 MPa[5] have been reported for conventional GI, which is less than FS values reported for different RMGIs (42-68 MPa).[19] Moshaveirinia et al reported FS values of approximately 26 to 28 MPa for a combination of nanobioceramics of hydroxyapatite, fluoroapatite and conventional GI, which is higher than that for conventional GI.[15] In the present study, although incorporation of a bioglass component significantly decreased FS values for



Graphs 2A and B: EDS analysis of RMGI and RMGI-BAG surfaces

RMGI, it appears these FS values are clinically acceptable. In comparison, FS of RMGI-BAG in the present study (39.90 ± 9.1 MPa) was higher than that of a combination of hydroxyapatite and fluoroapatite nanobioceramics and conventional GI; it appears if optimization and incorporation of remineralization and bioactivity properties of GI cements are intended, combination of RMGI with bioactive glass is more appropriate than that of hydroxyapatite with conventional GI.

In the present study, the same procedure was followed for the setting reaction of RMGI-BAG as that of RMGI. Previously, Matsuya et al,[26] Ana et al,[8] Yli-Urpo et al,[3] Xie et al[5] and Chio et al[4] have reported that the setting reactions of RMGI and RMGI-BAG are similar. In addition, these studies have evaluated absorption and solubility, compressive strength, Young's modulus of elasticity, Knoop hardness, Vickr's hardness and diametral tensile strength values of this material.[3,4,7,8,17,26]

In the present study, 20 wt% of bioactive glass was used to prepare RMGI-BAG powder. According to previous studies, with an increase in bioactive glass content, the mechanical properties of the material decrease and bioactivity increases.[3,4,6-8,17] Yli-Urpo et al[3] and Chio et al[4] used 10 and 30 wt% of bioactive glass in their studies. According to a report by Kessler et al 20 wt% of bioactive glass is preferable.[14] Moreover, in the present study, powder-to-liquid ratios of 3:2 and 2:7 were used to prepare RMGI and RMGI-BAG, respectively. Yli-Urpo et al used a powder-to-liquid ratio of 3:2 for RMGI and lower ratios (2:5 and 2:7) to combine it with bioactive glass.[3,17] Chio et al reported that with an increase in the amount of bioglass (Sol-Gel glass) added to conventional glass-ionomer, setting time increases and with an increase in the amount of powder relative to liquid, setting time decreases.[4] They did not report a specific wt% in their study. Therefore, it appears it is not possible to compensate a delay in the setting reaction of this combination due to the incorporation of bioactive glass by increasing wt% of powder relative to liquid. Chio et al reported a delay in the setting reaction of combination of conventional glass-ionomer and bioactive glass but they believed the setting time is appropriate for dental applications.[4] In the present study in the SEM analysis of the surface of the specimens, RMGI surfaces had some cracks without any specific deposits; however, RMGI-BAG surfaces had a specific homogeneous layer of deposits without any cracks on the surface. Yli-Urpo et al evaluated some surface characteristics and mechanical properties of RMGI-BAG and reported a uniform and homogeneous layer of deposits on the surfaces; other specimens exhibited less deposits but there were cracks on the surface.[3,6] The mineral deposit in the case of light-cured RMGI with 30 wt% of BAG was visible only after a week but in specimens with 10 wt% of BAG the deposit was visible after 3 weeks. In the present study, RMGI-BAG consisted of 20 wt% of BAG, and SEM evaluations were carried out after a month.

In addition, EDS analyses of the surfaces of both materials were carried out in both groups, the values of which cannot be statistically compared due to a limited number of specimens but they are of significance from a descriptive viewpoint. Comparison of surface elements in RMGI-BAG and RMGI showed a higher wt% of silicon (36.92%) in the former compared to the latter (14.9%). In the study carried out by Yli-Urpo et al, the average wt% of silicon oxide on the oral surface of RMGI-BAG with a 10 wt% of bioactive glass and RMGI restorations were 24.9 ± 0.2% and 21.3 ± 4.7% respectively, which is consistent with the results of the present study, indicating a higher content of silicon in RMGI-BAG compared to RMGI.[6]

The phosphorus content of RMGI-BAG and RMGI were 42.9 and 35.2 respectively, in the present study. Yli-Urpo reported that in the case of RMGI, the phosphorus content is under the influence of the material itself and time. After a week, light-cured GI had a higher content of phosphorus compared to light-cured GI with 30 wt% of bioactive glass (LC30 BAG); however, after 6 weeks, LC30 BAG had a higher content of phosphorus compared to RMGI. [16,17] In the present study, EDS analysis was carried out after 4 weeks, confirming the results of a study carried out by Urpo et al.[16,17]

Recently some studies have been carried out regarding the introduction, a new type of polyacrylic acid and hope of that the use of this material will increase compressive strength so that the material can be comfortably used in occlusal surface cavities.[21,22] It might become an ideal restorative material to replace lost tooth structure, especially in patients at a high-risk for caries.

CONCLUSION

Under the limitations of the present study, it was concluded that flexural strength of RMGI-BAG is less than that of RMGI but it is still clinically acceptable considering the flexural strength values reported for clinically used GIs and RMGIs. Evaluation of other properties of these materials, especially their bond to tooth structures, is recommended.

CLINICAL SIGNIFICANCE

Recent studies, have reported remineralization-inducing properties for RMGI containing BAG. It is probable that the use of bioactive materials for tooth restoration procedures in open/closed sandwich techniques or restoration of root surfaces and as cavity bases is more beneficial than the use of RMGI or conventional GI, especially in patients at a high-risk for carious lesions. Based on the results of this study, the mean value of FS is in the acceptable range of the reported FS values for conventional

GIs and RMGIs that are commercially available for clinical use. More investigations are recommended.

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