

AIN SHAMS DENTAL JOURNAL

Official Publication of Ain Shams Dental School March2024 • Vol. 33

Effect Of Surface Coating on Flexure And Roughness Properties of Glass Ionomers

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Aim: This study was conducted to evaluate the effect of different surface coating materials on flexural strength and surface roughness of two different glass ionomer restorative materials.

Materials and Methods: 80 rectangular bars $(12 \times 2 \times 2 \text{ mm})$ of glass ionomer materials; 40 highly viscous glass ionomer (HVGI) and 40 resin modified glass ionomer (RMGI) were prepared for flexure strength (FS) testing and divided into 4 groups (n=20) according to the applied surface coat; Group 0: no coat, Group 1: nano-filled resin coat, Group 3: total etch adhesive and Group 4: petroleum jelly. Another 80 cylindrical disks (6×2 mm) of the same materials were prepared for surface roughness (SR) testing and allocated to the same 4 groups as for FS testing (n=20). Samples were subjected to mechanical tooth brushing and then stored in distilled water at room temperature for either 24 hr. or 7 d. For FS testing, a 3-point loading test was performed using a universal testing machine running at a crosshead speed of 0.5 mm/min up to failure. For SR testing, mechanical profilometer was used with a cut-off value of 0.25mm. Statistical analysis was performed using One-Way ANOVA/ Turkey's HSD post hoc test.

Results: The results showed that the nano-filled resin coat exhibited the highest Statistically significant FS values and the lowest statistically significant SR values among all tested groups.

Conclusions: Immediate surface coating enhances the flexure and roughness properties of HVGI and RMGI. The light cured nano-filled resin positively influences the FS and the SR of glass ionomers.

Keywords: Glass ionomer, Surface coating, Flexure strength, Surface roughness, Nanofilled resin coat

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Introduction

Glass ionomer restorative materials (GIRM) have been widely used in dentistry for a long time with wide range of clinical applications for its well-known advantageous properties; fluoride release, biocompatibility, chemical adhesion to enamel and dentin, coefficient of thermal expansion similar to dentin, less technique sensitivity, dimensional stability at high humidity, and more esthetically pleasing than metallic restorations.¹

These positive properties are dwarfed bv inferior physical and mechanical properties, early moisture sensitivity and high surface porosity limiting their applications to low stress bearing areas. The immediate mechanical properties of the insufficient to GIRM are withstand masticatory forces. The weakness appears to be inherited in the matrix that is prone to crack propagation especially when it is subjected to water changes during setting. The propagation of these cracks may result in internal fragility of the set material and reduced wear resistance, leading to restoration failure.¹ Since their introduction, a lot of improvements have been performed to overcome these problems² eg; 1)Resin Modified Glass Ionomer (RMGI): addition of resin to glass powder that resulted in higher esthetics, less early moisture sensitivity with rapid strength development³ and 2)Highly Viscous Glass Ionomer (HVGI): chemical modification to the glass powder allowing higher powder-liquid ratios, optimizing polyacid and particle size distribution, resulting in a high cross-linkage matrix with superior physical and mechanical properties compared to their conventional type.⁴

The chemical reaction of GIRM is basically acid base reaction that starts when the polymeric acid attacks the glass particles resulting in formation of metal polyacrylate salts that serve as cross-links between the polyacid chains. Formation of these salts occurs in two steps in a diffusion-controlled process. First, the calcium ions react with the aqueous chains forming calcium polyacrylate salts that are responsible for immediate hardening process. Secondly, these salts are gradually replaced by the insoluble aluminum polyacrylate salts leading to maximum hardening of the material. This second step is much slower and may continue up to 24 hours. In this phase care should be taken to avoid water loss or gain, as this would lead to irreversible damages for the material's surface.⁴ If the GIRM is prematurely exposed to oral fluids; especially in the first hour, this will lead to consequent washing out of Ca+2 and Al+3 ions that impairs the setting reaction, leading to improper matrix formation with decreased mechanical properties.⁵ This can be clinically perceived as increased surface roughness with loss of translucency.² On the opposite side, if the reaction happened in a dry environment, material will tend to lose water which leads to dimensional changes, adhesion problems and formation of internal cracks.6

Surmounting these obstacles. researchers suggested protecting the material surface during its early stages of setting. Among these materials were using different types of varnishes (solvent based or resinbased varnishes), bonding resins, emollients like petroleum jelly, nail varnishes and paraffin wax. Studies have shown that this could improve the initial flexure strength and other mechanical properties of glass ionomers even for RMGI unlike most manufacturers which claimed that RMGI could be used without surface protection.⁵ This provided a gate for many studies to assess the effect of different coating materials on the overall properties of GIRM.

Studies showed that resin-based varnishes had a significant effect on increasing mechanical properties of GIRM

such as surface hardness⁷ flexure strength (FS)⁸ and surface roughness (SR).^{9,10}

On the other hand, it was found to severely affect their fluoride release due to associated reduction in water movement.⁵ Nowadays, newly marked varnishes contain nano-filled resin that combines extremely low viscosity with high hydrophilicity. This was claimed to provide perfect seal for the material by occluding any surface crack or porosity and preventing early water contamination.^{9,10}

Other practitioners chose to use different bonding systems owing to their higher retentive feature, higher surface increased mechanical protection with properties to GIRM.¹¹ Another coating material that gained clinical popularity was petroleum jelly^{6,8} which was considered a option due good to its safety, biocompatibility, promising results in enhancing the GIRM properties and its reasonable price.^{6,8} However; it could be easily washed away by oral masticatory wear.¹² Other variant materials mentioned in the literature like nail varnishes and paraffin wax showed superior results, however they lack biocompatibility and therefore their use was not recommended.¹³

Consequently, this study was conducted in an attempt to evaluate the effect of different coating materials on FS and SR of HVGI and RMGI restorative materials.

Materials and methods

A total of 160 samples were prepared for this study; 80 for FS testing and 80 for SR testing. Samples were divided into 16 groups (n=10) according to the three levels of the study; Level 1: Restorative material (HVGI (m1) or RMGI (m2)), Level 2: Surface coating (no coat, nano-filled resin coat, totaletch adhesive and petroleum jelly) and Level 3: Aging time (24 hr. or 7 days).

The materials used in this study are summarized in Table 1.

Table (1): Materials, Composition, Manufacturer and Lot No.

1	Material	Composition	Manufacturer		
			and Lot No.		
1	EQUIA Forte [®] Fil	Powder: Ultrafine highly reactive fluoro-	GC Corporation,		
	Encapsulated bulk	alumino-silicate glass particles	Tokyo, Japan		
	fill, highly viscous,	Liquid: High molecular weight polyacrylic	Lot#:1808163		
	fluoride releasing	acid, Polybasic carboxylic acid, Distilled			
	glass hybrid	water			
1	restorative material.				
	Shade: A3				
1	GC Fuji II LC®	Powder: Fluoro-alumino-silicate glass 100%	GC Corporation,		
I	CAPSULE	Liquid: Polyacrylic acid (24%), tartaric acid	Tokyo, Japan		
I	Encapsulated light-	(6%), HEMA (35%), UDMA,	Lot#:190403A		
	cured, resin	camphorquinone (0.10), distilled water (25%)			
1	modified glass	and pigments			
	ionomer restorative				
	material. Shade: A3				
	EQUIA ® Coat	MMA (40%-50%), UMA (30%-40%),	GC Corporation,		
-	Light cured, low	colloidal silica (10%-15%), Camphorquinone	Tokyo, Japan		
	viscosity self	(0.09%), phosphoric-ester monomer (1%-	Lot#:1804031		
1	adhesive nano-filled	5%).			
	resi <mark>n coa</mark> t.				
	Adp <mark>er ™</mark> Single	Bis-GMA, HEMA, copolymer of polyacrilic	3M-ESPE, Saint		
	Bond 2	acid, photoinitiator, 5nm colloidal silica filler	Paul, USA		
	Light cured total-	(10%)	Lot#: N980586		
	etch adhesive.				
	Vaseline [®] Pure	White petrolatum 100%	Unilever		
l	petroleum jelly				
	HEMA= Hydroxyethy	yl methacrylate, UDMA=Urethane Dimethacryla	te, MMA=		
	Methyl Methacrylate,	enol glycidyl			
	methacrylate				

Samples Preparation for FS Testing:

A specially fabricated split Teflon mold were used to prepare 80 rectangular bar samples $(12 \times 2 \times 2)$; 40 samples for each material. Materials were mixed for 10 sec according to the manufacturer's instructions, injected inside the mold with slight overfilling, covered with a celluloid strip (TOR VM Ltd, Moscow, Russia) and a glass slide (1 mm thickness) and gently fingerpressed to expel excess material. The m1 material was allowed to set under 1kg weight for 3 min while m2 material was subjected to 1kg weight for 20 sec then the weight and the glass slide were removed and the material was light cured directly through the celluloid strip using a light curing unit (3M Elipar Light Curing Unit, 3M ESPE, St Paul, USA) following the overlapping technique; central initial irradiation cycle followed by two subsequent cycles at both sides for 20 sec each (a total of 60 sec).^{14,15} Intensity of the light curing unit was periodically checked

using a radiometer (CM300-2000, Curing Light Meter, Apoza). After setting, samples were removed from the mold and flashes were cut away using scalpel blade no#11. Intensity of the light curing unit was periodically checked for every group using a radiometer (CM300-2000, Curing Light Meter, Apoza).

Samples Preparation for SR Testing:

A specially fabricated cylindrical Teflon mold was used to prepare 80 cylindrical disks (6mm×2mm); 40 disks for each material. Materials were mixed and injected inside the mold with the same technique used for FS except for m2 material that was light cured directly through the celluloid strip for only 20 sec.

Surface Coat Application:

The FS and SR samples of each material were allocated to 4 groups (n=20) according to the applied surface coat; Group 0 (G0): Samples were left uncoated as a control and stored immediately in distilled water. Group 1 (G1): Samples were covered by a thin layer of nano-filled resin coat using micro-brush (Voco, a medium sized Germany). Cylindrical disks were light cured for 20 sec while rectangular bars were light cured following the overlapping technique (a total of 60 sec). Group 2 (G2): Samples were covered by a thin layer of total-etch adhesive using micro-brushes and gently air thinned for 5 sec. Cylindrical disks were light cured for 10 sec while rectangular bars were light cured following the overlapping technique (a total of 30 sec). Group 3(G3): Samples were covered by a thin layer of white petroleum jelly using micro-brushes. Samples were then color coded and stored in distilled water at room temperature for either 24 hr or 7 days that was changed every day.

Mechanical tooth brushing:

Mechanical tooth brushing was performed to all samples using a custommade tooth brushing simulating device.¹⁶ 80 samples (40 of each material) were subjected to one brushing cycle after 24 hr for 13 sec under a weight of 200gm with a speed of 280 rpm giving a total number of 60 strokes corresponding to intraoral brushing twice per day. The other 80 samples were subjected to 7 brushing cycles; once per day for 13 sec each (a total of 420 strokes) corresponding to intraoral brushing twice per day for 7 days (17,18). A slurry mixture of moderately (Colgate abrasive dentifrice regular. Colgate[®], USA) and distilled water 1:1 was regularly injected over the samples to keep them wet throughout the cycle. Plane brush heads with soft rounded bristles (Fuchs® EKOTECTM, Egypt) were used. Samples were ultrasonically cleaned using an ultrasonic cleaner (CODYSON, MCS, CD-4830 Shenzhen Codyson electrical co., Ltd., China) for 10 min before testing.

Flexure strength (FS) Testing:

For FS testing; a 3-point bending test was used in a Universal Testing Machine (LR5K series, Lloyd Instruments, Ltd, UK). Each sample was supported by two rods parallel to each other with a 10mm distance between their centers. Load was applied at the center of each sample at a crosshead speed of 0.5mm/min up to failure. FS was calculated according to the following equation: FS = 3FL/2WT2, where F is maximum load in Newton (N), L is distance between supporting rods in mm, W and T are sample width and thickness respectively in mm. Values were expressed in Mega Pascal (MPa).

Surface Roughness (SR) Testing:

Surface roughness was analyzed using a mechanical profilometer (TR 220 Portable Roughness Tester, Time Group Inc, Beijing Time High Technology Ltd, PA,

USA), equipped with a diamond needle (5 μ m radius). The needle traversed the surface with a force of 0.7 mN at a constant speed of 0.135 mm/s. The cut-off value was set to be 0.25 mm.¹⁹ Three measurements were recorded for each sample at different areas then their mean value was calculated giving the average surface roughness value (Ra).

Statistical analysis was carried out using SPSS program (IBM®, SPSS®, Chicago, IL, USA, version 25). One-Way ANOVA followed by Tukey's HSD post-hoc test were performed to evaluate the effect of each coat on the FS and SR.

Results

FS testing results:

The mean of FS values and standard deviation for each group are presented in table 2 and figure 1. Generally, RMGI showed significant higher FS than HVGI after 24 hr. and 7 days. For HVGI, after 24 hr, G1 showed the highest FS values. There was no significant difference neither between G1 and G2 nor between G2 and G3. G0 showed the lowest FS value. After 7 days, G1 showed the highest FS value. There was no significant difference between G1 and G3.

For RMGI, after 24 hr., G1 showed the significantly highest FS value followed by G2 then G3 with no significant difference between G2 and G3. G0 showed the lowest FS value. After 7 days, G1 showed the highest FS value and G0 showed the significantly lowest FS value. There was no significant difference neither between Group 1 and 3 nor between Group 0 and 2.

SR testing results:

The mean of SR values and standard deviation for each group are presented in table 3 and figure 2. For HVGI, after 24 hr., G0 and G2 showed the highest SR values without significant difference between them

followed by G3. G1 showed the significantly lowest SR value. After 7 days, G0 showed the significantly highest SR value followed by G2. G1 and G3 showed the significantly lowest SR values without significant difference between them.

For RMGI; after 24 hr., there was no statistically significant difference between G0, G2 and G3 while G1 showed the lowest SR values. After 7 days, there was no significant difference between all groups.

Table (2): Means ± Standard Deviations for the effect of surface coating for each material and within each aging time on flexure strength

	Tr	G0	G1	G2	G3			
HVGI	24 hr	11±1.43°	20.07±0.58ª	18.24±1.12 ^{ab}	18.21±0.74 ^b			
	7 d	27.5±2.04°	39.32±3.78ª	32.56±2.08b	37.99±1.52ª			
RMGI	24 hr	33.52±2.25°	47.07±2.26ª	42.4±1.58 ^b	41.75±1.06 ^b			
	7 d	45.47±3.9 ^b	53.97±2.93ª	46.62±2.23 ^b	53.7±3.53ª			
Means with same superscript small letters within each row are statistically non-significant at P=0.05. n=5 samples/group								

Table (3): Means ± Standard Deviations for the effect of surface
coating for each material and within each aging time on surface
roughness.

-		1	c0	c1	c2	c3	
-	HVGI	24 hr	0.5 ± 0.9^{a}	0.15±0.02°	0.48±0.05ª	0.26±0.04b	
	0.	7 d	1.04±0.24ª	0.12±0.02°	0.45±0.08 ^b	0.21±0.03°	
-	RMGI	24 hr	0.29±0.05ª	0.12±0.01 ^b	0.37±0.05ª	0.36±0.07ª	
		7 d	0.97±0.16ª	0.82±0.05ª	0.93±0.06ª	0.93±0.11ª	
0	Means with same superscript small letters within each row are statistically non-significant at P=0.05, n=5 samples/group						

Discussion

GIRM is widely used in clinical dentistry but due to drawback of its early moisture sensitivity that disturb their setting reaction resulting in poor physical and mechanical properties⁵, clinicians tend to protect the material surface with different coating materials. For this reason, the objective of the current study was to evaluate the effect of different surface coating materials on flexural strength and surface roughness of GIRM.



Figure (1): Bar chart showing the effect of surface coating for each restorative material and within each aging time on flexure strength.



Figure (2): Bar chart showing the effect of surface coating for each restorative material and within each aging time on surface roughness.

In this study, the mini flexural test (MFT) (12x2x2) was chosen over the ISO flexural test (IFT) (25x2x2) according to Yap et al,²⁰ who stated that the sample length affects its flexural properties; the longer is the sample, the more flexible it is. Translated clinically; restorations with higher flexibility will have lower strength so will be more susceptible to marginal breakdown and bulk fracture. In addition, MFT has the advantages of ease of sample fabrication with more clinically realistic dimensions.

In the current study; mechanical tooth brushing was done once per day for 13 sec with (a total of 60 strokes) corresponding to intraoral tooth brushing twice per day according to the ADA recommendations.¹⁷ The brushing was done under 200gm load based on the ISO standardization of wear testing that ranged from 50 gm to 250 gm.²¹ The dentifrice used was of moderate abrasion (RDA~70) to be more gentle on the GIRM's surface.^{3,17} In addition, soft toothbrushes were used to promote low abrasion.¹⁷

According to our study, FS results after 24 hr. revealed that nano-filled resin coat showed the highest FS value for both HVGI and RMGI which comes in agreement with previous studies.²²⁻²⁴ This may be ascribed to presence of 30nm single phase dispersed nanofillers that share in microlamination effect with optimum wetting for the surface and uniform flow. This toughened thick laminated layer "about 35-40µm" possibly shares in protecting the surface against any moisture contamination, filling any surface porosities or cracks and consequently strengthen the material and enhance its FS.

For HVGI after 24 hr., there was no significant difference between nanofilled resin coat and total etch adhesive as it was believed that bonding agent shares in partial prevention of water movement across the surface of GIRM by forming a partially hydrophobic surface layer which was compliant with Hotta et al,²⁵. It was also found that the heat generated from the light curing unit accelerated the setting reaction in the surface layers leading to higher FS values.⁸ While another study²⁶ stated that not all light cured bonding agents were beneficial in protecting the glass ionomer surface which was attributed to differences in chemical and physical properties of the resin in each bonding agent.

However, no significant difference was found between petroleum jelly and total etch adhesive for HVGI after 24 hr., contrary to Brito et al, 2010^{13} where petroleum jelly resulted in significantly higher surface hardness than total etch adhesive after 24 hr. It worth noting that they were using the same total etch adhesive used in the current but with different challenging protocol, as Brito et al, exposed their samples to polishing with 1200grit silicon carbide paper for 3min with 600rpm while in this study samples were exposed to 60 strokes of mechanical tooth brushing for 13 sec with 280rpm. Different results may also be justified by testing different mechanical surface properties in both studies. Yet, this coat showed significantly lower FS values than nanofilled resin coat. This comes in agreement with several studies^{2,8,27}, where petroleum jelly led to lower FS and higher clinical wear than nano-filled resin coat. Another study²⁸ found jelly that petroleum led to higher microleakage than the resin coat with no difference from significant uncoated specimens. This may be attributed to the ease of washing away of the petroleum jelly leaving the surface unprotected. Results of petroleum jelly remained significantly higher than the uncoated group which comes in harmony with some studies^{5,29}, proving its ability to provide early protection to GIRM.

After 7 days of storage; FS of HVGI increased gradually reaching almost their double values which consents with some studies.^{30,31} This could be attributed to the

Effect Of Surface Coating on Flexure And Roughness Properties of Glass Ionomers | Noha Mohamed Abd El-Kader Anany et al. MARCH2024.

long time needed by the GIRM to complete their setting reaction and reach their final strength. On the other side, one study³² found that FS decreased after one week which was explained by the water penetration that led to degradation resulting hydrolytic progressively weaker material. Results of total etch adhesive for both HVGI and RMGI after 7 days were lower than nanofilled resin coat and petroleum jelly which could be attributed to the separation of the thin adhesive layer from the restoration's surface after being subjected to multiple tooth brushing cycles and before reaching the maximum strength of GIRM.

Generally, RMGI showed higher FS values than HVGI which could be attributed to the fact that RMGI are less brittle and less prone to bulk fracture. This came in agreement with many studies^{32,33} where higher results of RMGI was justified by their dual cure nature, dual crosslink, plastic deformation behavior and the integrated interface between the polymer matrix and the glass particles. Results also showed that RMGI benefited from surface protection especially the nanofilled resin coat that led to the highest FS which is compliant with several studies^{33,34}, in contrast to most manufacturers who claimed that RMGIs could be used without surface protection While others³⁵ found that this effect is temporary and limited to the first hour after setting without significant difference from uncoated specimens after 24 hr.

As for SR results, surface coating of HVGI especially the nanofilled resin coat led to decreased SR values compared to uncoated samples which was compliant with many previous studies.^{10,36} This could be justified by the ability of the coat to occlude any surface cracks or porosities giving a high surface polish with lower SR.² Contrary to that; others⁹ found that there was no significant differences in SR of coated and uncoated specimens of either HVGI or RMGI

which was contributed to the immediate finishing procedure that led to premature moisture contamination. However, SEM analysis showed that the nanofilled resin coat provided a continuous interface along the entire surface of the glass ionomer with no air bubbles.

The current study showed that RMGI didn't benefit from total etch adhesive or petroleum jelly after 24 hr. unlike nanofilled resin coat that succeeded in decreasing SR of RMGI by formation of a continuous surface layer with no porosities. While after 7 days, there was no significant difference between all groups. This could be attributed to the mechanical tooth brushing that led to discernible material loss from the surface due to insufficient coherence between the cross linked polyacrylate network and the polymer chain of the RMGIs resulting in increased SR which was previously proven by De Paula et al,³⁷ and Hassanien et al,¹⁶.

Conclusions

Within the limitations of this study, the following conclusions could be suggested:

1- Immediate surface coating is very beneficial for both HVGI and RMGI.

2- The light cured nano-filled resin coat led to the highest FS and lowest SR values among other tested materials.

Conflict of interest

The authors declare that they have no conflict of interest regarding the materials used in this study.

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