COMPRESSIVE STRENGTH AND SURFACE ROUGHNESS OF CERAMIC REINFORCED GLASS IONOMER SUBJECTED TO CHEMICAL CHALLENGE

Randa M. Hafez*, Maha E.Elkorashy** *and* Mayada S. Sultan***

ABSTRACT

Objective: This research was conducted to investigate the effect of chemical challenge on the compressive strength (CS) and surface roughness (Ra) of a ceramic reinforced glass ionomer in comparison to a nanofilled resin composite.

Methods: A total of 60 disc specimens (6mm height x 4mm diameter) were prepared for the compressive strength testing and another 60 disc specimens (5mm diameter x 2mm thickness) for the surface roughness testing. Specimens were divided into 2 groups (n=30) according to the material used; ceramic reinforced glass ionomer (Amalgomer CR, Advanced Health Care Ltd, Tonbridge, Kent, UK) and nanofilled resin composite (Filtek™ Z350 XT, 3M ESPE, St.Paul, MN,USA). Each group was divided into 3 subgroups (n=10) according to the storage media; distilled water (control), 0.02N citric acid and 50% ethanol. Specimens were stored for 7 days at 37°C. After storage period, they were subjected to compressive loading using a universal testing machine and surface roughness testing using white light interferometer. Data were tabulated and statistically analyzed using Two-way ANOVA followed by Bonferroni's post-hoc test.

Results: Amalgomer CR recorded a significantly lower CS and higher Ra than nanofilled resin composite under different storage media. Citric acid revealed the lowest CS of Amalgomer CR followed by ethanol in comparison to distilled water with significant difference between them. For nanofilled resin composite, both citric acid and ethanol significantly decreased CS. Ethanol showed the highest Ra values for both restorative materials.

Conclusions: The performance of Amalgomer CR under different storage media was inferior to nanofilled resin composite regarding compressive strength and surface roughness. Citric acid severely affected compressive strength of Amalgomer CR. Nanofilled resin composite was able to preserve its surface roughness within the clinically acceptable threshold after chemical challenge in contrary to Amalgomer CR.

KEY WORDS: ceramic reinforced glass ionomer, citric acid, ethanol, compressive strength, surface roughness.

* Professor, Conservative Dentistry Department, Faculty of Oral and Dental Medicine, Cairo University
** Lecturer, Operative Dentistry Department, Faculty of Dentistry, Fayoum University
*** Lecturer, Operative Dentistry Department, Faculty of Dentistry, Assiut University

INTRODUCTION

Glass ionomer cements (GICs) are one of the most attractive restorative materials. They are characterized by interesting properties as chemical bonding ability, high biocompatibility, anti-cariogenic property due to fluoride release and coefficient of thermal expansion and contraction matching to that of the tooth structure [1,2]. On the other hand, they suffer from poor mechanical properties as low fracture strength, toughness and wear resistance which limits their use in stress bearing areas[3]. Based on the poor mechanical properties of glass ionomer cements, numerous attempts have been done in order to improve their strength properties [2,4-6].

Amalgomer™ CR is a recent ceramic reinforced posterior restorative glass ionomer where zirconia fillers were incorporated to achieve strength properties comparable to that of amalgam. As stated by the manufacturer, it has low wear rates and excellent resistance to fracture and cracking [7].

Nanofilled resin composite is considered one of the most commonly used restoratives nowadays. It fulfills the clinical requirements as a posterior restoration in terms of high strength properties and high resistance to abrasion. This is achieved via the incorporation of filler particles in the nanoscale which allow high filler loading [8].

Mechanical properties describe the behavior of the material under functional loading [9]. During clinical service, restorations are subjected to combination of forces resulting in development of different types of stresses as compressive, tensile and shear stresses[10]. Compressive strength testing is one of the most commonly employed testing methods for evaluation of the strength properties of dental restoratives [9-11].

The surface texture of tooth-colored restorative materials plays an important role in their clinical performance. Restorations with surfaces irregularities facilitate colonization of bacteria and biofilm maturation with increasing risk of development of dental caries and periodontal diseases [12].

In the oral environment, exposure of dental restoratives to saliva, food components and beverages can degrade and age dental restorations. It was reported previously that resin matrices of dental composites become softened with exposure to organic acids and various constituents of food and drinks [13]. Also, glass ionomer cements showed leaching out of inorganic components with great susceptibility to hydrolytic degradation at the matrix/filler interface under different environmental conditions [14]. Therefore, the chemical environment in the oral cavity might have a critical influence on the properties of dental restoratives and subsequently their clinical performance.

Hence, evaluation of the compressive strength and surface roughness of a ceramic reinforced posterior restorative glass ionomer under chemical challenge of the oral environment in comparison to a nanofilled resin composite might be of value.

MATERIALS AND METHODS

Two tooth colored restorative materials were used in the current study; ceramic reinforced glass ionomer (Amalgomer ™ CR Posterior Restorative glass ionomer) and a nanofilled resin composite (Filtek ™ Z350 XT Universal Restorative) as shown in (table 1). Three storage media; distilled water (control), 0.02N citric acid and 50% ethanol were also used in the study.

Specimens' preparation and grouping

A total of 120 specimens were prepared for the present study. Sixty cylindrical specimens were prepared for the compressive strength test, 6 mm in height x 4 mm in diameter, and 60 disc specimens for surface roughness testing, 5mm in diameter x 2 mm in height. Specimens for each test were prepared using a specially designed split teflon

TABLE (1) The materials used in the study

Product	Description	Composition		Manufacturer
		Matrix	Fillers	
Amalgomer™ CR	Ceramic reinforced GIC	70-80% Glass powder 10-20% Acrylic acid polymer	- Zirconia fillers	Advanced Health Care Ltd, Tonbridge, Kent, UK
Filtek™ Z350 XT Universal Restorative material	Nanofilled resin composite	Bis-GMA UDMA TEGDMA PEGDMA Bis-EMA	- Non- agglomerated 20 nm silica filler. -Non-agglomerated 4-11 nm zirconia filler. - Aggregated zirconia/silica cluster filler 0.6-10μm. - 72.5 wt% (55.6 V%)	3M ESPE, St.Paul, MN,USA
Bis-GMA: Bisphenol A diglycidylmethacrylate **Bis-EMA:** Bisphenol-A polyethylene glycol dietherdimethacrylate **UDMA:** Urethane dimethacrylate **TEGDMA**: Triethylene glycol dimethacrylate **PEGDMA:** poly(ethylene glycol) dimethacrylate				

mold. The samples were divided into 2 groups (n=30) according to the type of restorative material used; Amalgomer™ CR conventional glass ionomer and Filtek Z350 XT Universal Restorative. Each group was further divided into 3 subgroups (n=10) according to the storage medium; distilled water (control), 0.02N citric acid and 50% ethanol.

For specimens' preparation, a glass slide covered with a mylar strip (Stripmat, POLYDENTIA, CH-6805 Mezzovico, Switzerland) was used. For Amalgomer™ CR specimens' preparation, the powder and the liquid (distilled water) were proportioned according to the manufacturer' instructions and mixed until a homogenous mix was obtained. The mix was then packed into the intended mold in one increment, covered with another mylar strip and a glass slide then pressed for 10 sec to extrude the excess material and achieve a uniform smooth surface. The mix was left in the mold till complete setting. The specimens were then coated with a light cure glaze (AHfil LCG, Advanced Health Care Ltd, Tonbridge, Kent, UK) as recommended by the manufacturer and light cured for 10 sec using LED light curing unit with an intensity of 1200mW/cm^2 (Elipar™S10, 3M ESPE, St. Paul, USA). Regarding the resin composite specimens, they were prepared in the same manner as mentioned before and light cured for 40 sec from top surface and 40 sec from bottom surface. All specimens were prepared by the same operator.

Specimens were stored for 24h at 37ºC in distilled water to ensure complete setting. They were then immersed in one of the three storage solutions; distilled water (control), 0.02N citric acid or 50% ethanol/water solution in labeled containers for 7 days at 37°C in an incubator [15].

Compressive strength testing

After the storage period, specimens were subjected to compressive loading using a universal testing machine (Lloyd LR 5K, Lloyd Instruments Ltd, Hampshire, UK) operating using Nexygen software version 4.6. The load was applied along the long axis of the specimens with a load cell 5KN at a cross head speed 0.5 mm/min. The maximum load at fracture was recorded and the compressive strength was calculated in MPa by dividing the load with the cross sectional area of the specimen.

Surface roughness testing

Surface roughness was measured using a white light interferometer. This was carried out using ZYGO Maxim-GP 200 profilometer, which is a general purpose surface optical profiler that measures the microstructure and topography of surfaces in three dimensions. Computerized phase stepping interferometry (PSI) upgraded with scanning white light interferometry (SWLI) and advanced surface texture software which analyzes areas as well as profiles and step height. A white light from a Halogen lamp incident on an interference filter with Full Width at Half Maximum (FWHM) ≈3 -15 nm was used depending on the measuring technique. Three readings were recorded for each sample (2 readings from the peripheries and 1 reading form the center) and an average reading was calculated to represent the surface roughness for each specimen in μm.

Scanning Electron Microscopic Observation

Two representative samples from each subgroup were evaluated by an environmental scanning electron microscope (Quanta FEG 250, FEI Company, Netherland). The surfaces to be evaluated were mounted on metallic stubs and assessed by SEM at magnification 1500X to scan surface topography.

Statistical Analysis

Numerical data were explored for normality by checking the data distribution using Kolmogorov-Smirnov and Shapiro-Wilk tests. All data showed normal distribution. Data were represented by mean, standard deviation (SD), range and 95% Confidence Interval (95% CI) values. Two-way ANOVA test was used to study the effect of material, storage media and their interactions on compressive strength (CS) and surface roughness (Ra). Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test revealed significance. The significance level was set at P ≤ 0.05. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

RESULTS

Compressive Strength Results

Two-way ANOVA test showed that material, storage media and their interaction had a statistically significant effect on mean compressive strength (table 2). Descriptive statistics of compressive strength were presented by mean, standard deviation (SD), minimum, maximum and 95% confidence interval (95% CI) values in Table (3).

TABLE (2) Two-way ANOVA results for the effect of material, storage media and their interaction on mean compressive strength (CS) in MPa

Source of variation	Sum of Squares	df	Mean Square	F-value	P-value
Material	231193.5	1	231193.5	3289.6	<0.001*
Storage medium	39188.6	2	19594.3	278.8	<0.001*
Material x Storage medium interaction	14666.7	2	7333.3	104.3	<0.001*

df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05

TABLE (3) Descriptive statistics of compressive strength (CS) values in MPa

Material	Storage medium	Mean	SD	Minimum	Maximum	95% CI Lower bound	95% CI Upper bound
Amalgomer CR	Distilled water	148.8	6.0	140.4	156.9	142.5	155.1
	Citric acid	30.3	4.4	24.6	37.2	25.7	35.0
	Ethanol	116.3	8.3	105.6	128.9	107.6	125.0
Resin Composite	Distilled water	289.9	5.5	280.8	295.4	284.1	295.7
	Citric acid	246.7	12.1	230.8	262.7	234.1	259.4
	Ethanol	239.6	11.0	226.0	257.7	228.0	251.1

Bonferroni's post-hoc test (table 4) showed that Amalgomer CR revealed lower compressive strength with all storage media in comparison to resin composite. Regarding the effect of the

storage media, Amalgomer CR showed the lowest compressive strength results with citric acid followed by ethanol then distilled water with statistically significant difference between them. For resin composite, both citric acid and ethanol showed significantly lower compressive strength in comparison to distilled water with no significant difference between them.

TABLE (4) Mean, standard deviation (SD) values and P-value of the effect of material and storage media on compressive strength (CS) in MPa

Storage medium	Amalgomer CR		Resin Composite		P-value
	Mean	SD	Mean	SD	
Distilled water	148.8[A]	6.0	289.9[A]	5.5	<0.001*
Citric acid	30.3[C]	4.4	246.7[B]	12.1	<0.001*
Ethanol	116.3[B]	8.3	239.6[B]	11.0	<0.001*
P-value	<0.001*		<0.001*		

*: Significant at P ≤ 0.05, Different superscripts in the same column are statistically significantly different

Surface Roughness Results

According to Two-way ANOVA test, results showed that material and storage media had a statistically significant effect on mean Ra. The interaction between the two variables had no statistically significant effect on mean Ra (table 5). Descriptive statistics of surface roughness (Ra) values in μm are presented in Table (6).

TABLE (5) Two-way ANOVA results for the effect of material, storage media and their interaction on mean surface roughness (Ra) in μm

Source of variation	Sum of Squares	df	Mean Square	F-value	P-value
Material	0.148	1	0.148	133.4	<0.001*
Storage medium	0.070	2	0.035	31.7	<0.001*
Material x Storage medium interaction	0.003	2	0.001	1.2	0.321

df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05

TABLE (6) Descriptive statistics of surface roughness (Ra) values in μm

Material	Storage medium	Mean	SD	Minimum	Maximum	95% CI Lower bound	95% CI Upper bound
Amalgomer CR	Distilled water	0.17	0.03	0.13	0.21	0.14	0.20
	Citric acid	0.23	0.04	0.17	0.29	0.18	0.27
	Ethanol	0.30	0.05	0.22	0.36	0.25	0.35
Resin Composite	Distilled water	0.06	0.02	0.04	0.10	0.04	0.09
	Citric acid	0.10	0.02	0.08	0.13	0.08	0.11
	Ethanol	0.15	0.02	0.12	0.18	0.13	0.17

Table (7) showed that Amalgomer CR showed significantly higher mean surface roughness (Ra) compared to resin composite in different storage media. For Amalgomer CR, ethanol showed the highest mean Ra value followed by citric acid then distilled water with statistically significant differences between them. In resin composite, ethanol showed the highest mean Ra while no statistically significant difference was recorded between citric acid and distilled water.

TABLE (7) Mean, standard deviation (SD) values and p-value of the effect of material and storage media on surface roughness (Ra) in μm

Storage medium	Amalgomer CR		Resin composite		P-value
	Mean	SD	Mean	SD	
Distilled water	0.17[C]	0.03	0.06[C]	0.02	<0.001*
Citric acid	0.23[B]	0.04	0.10[BC]	0.02	<0.001*
Ethanol	0.30[A]	0.05	0.15[A]	0.02	<0.001*
P-value	<0.001*		<0.001*		

*: Significant at P ≤ 0.05, Different superscripts in the same column are statistically significantly different
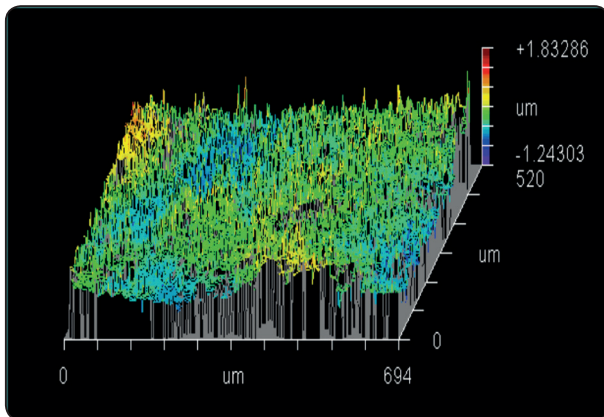
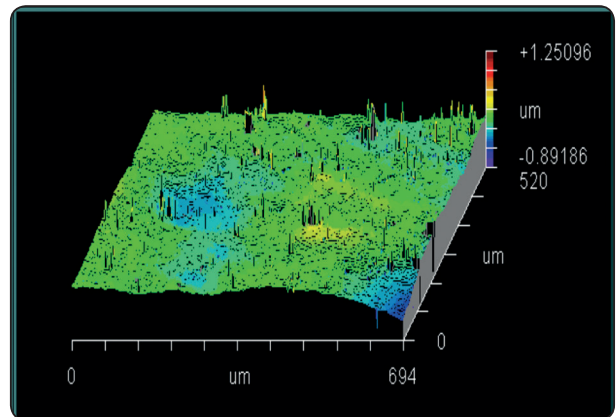Fig. (1a) Interferometer image of the Amalgomer CR after storage in distilled H2O

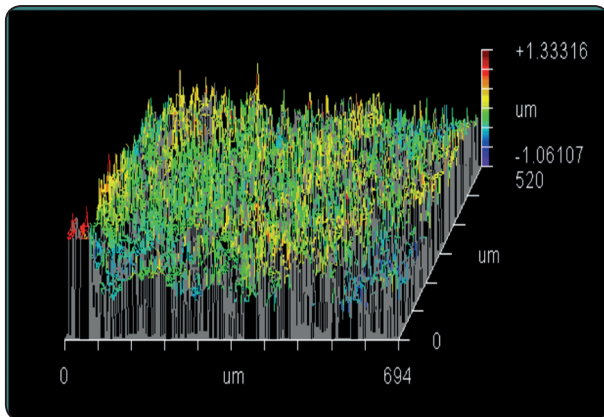Fig. (1b) Interferometer image of the resin composite after storage in distilled H2O

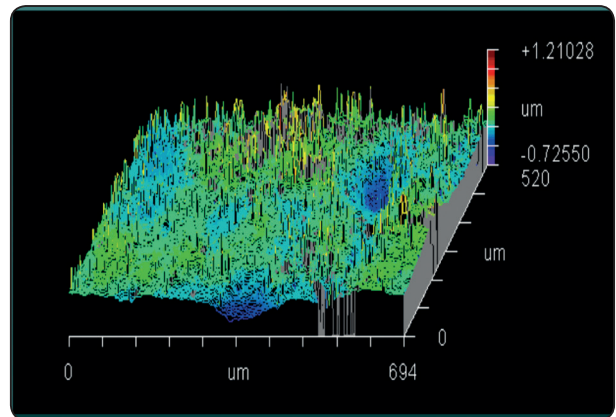Fig. (1c) Interferometer image of the Amalgomer CR after storage in citric acid

Fig. (1d) Interferometer image of the resin composite after storage in citric acid
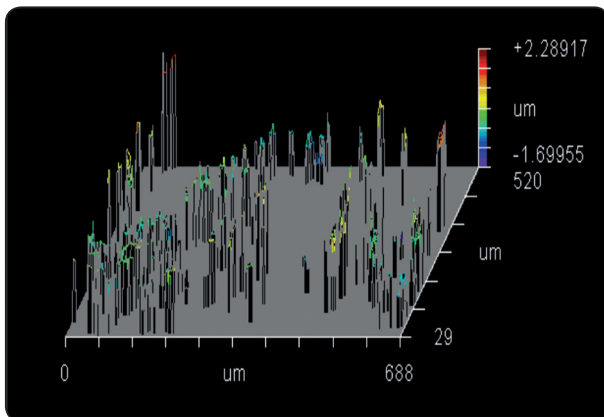
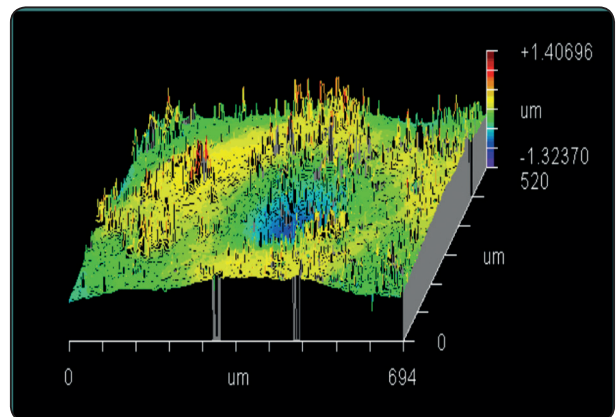Fig. (1e) Interferometer image of the Amalgomer CR after storage in ethanol

Fig. (1f) Interferometer image of the resin composite after storage in ethanol

Fig. 1 (a–f): Interferometer images of Amalgomer CR and nanofilled resin composite after immersion in different storage media

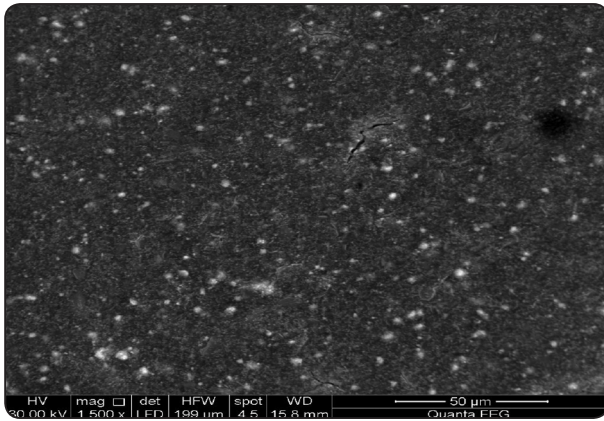## Scanning Electron Microscopic Observations

Fig. (2a) SEM image of the Amalgomer CR after storage in distilled H2O
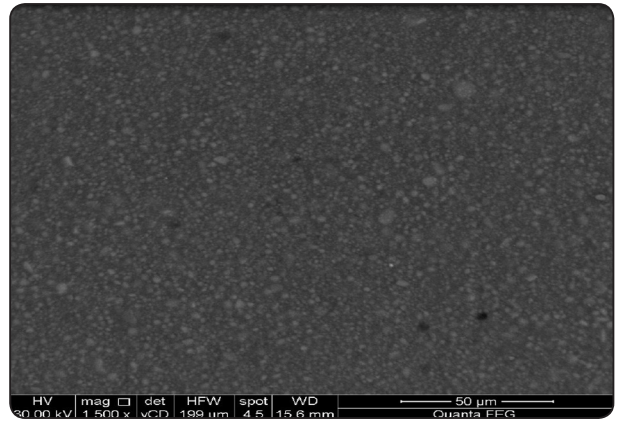

Fig. 2b: SEM image of the resin composite after storage in distilled H2O
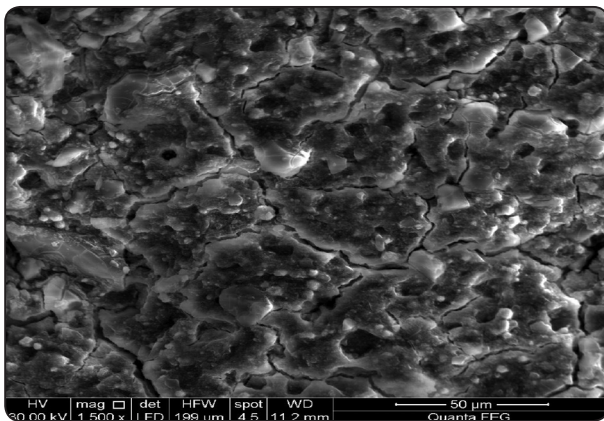

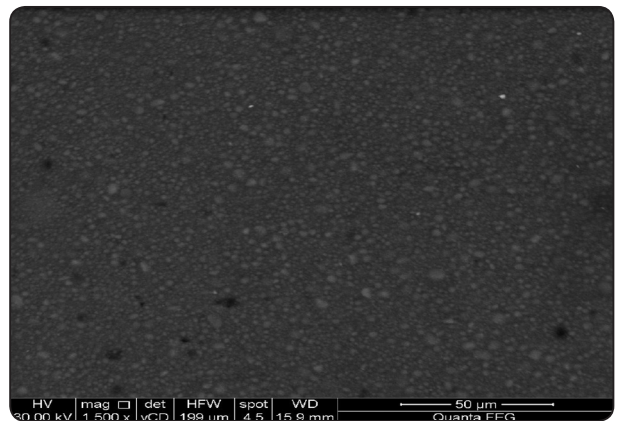Fig. (2c) SEM image of the Amalgomer CR after storage in citric acid

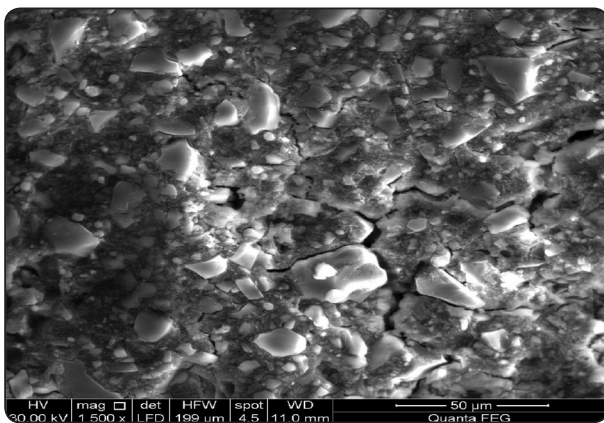Fig. (2d) SEM image of the resin composite after storage in citric acid

Fig. (2e) SEM image of the Amalgomer CR after storage in ethanol
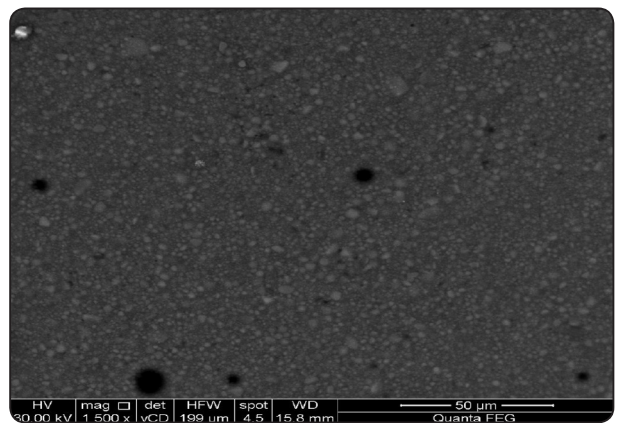

Fig. (2f) SEM image of the resin composite after storage in ethanol

Figure 2 (a-f): SEM images of Amalgomer CR and nanofilled resin composite after immersion in different storage media

Scanning electron microscopic images showed that Amalgomer CR specimens immersed in distilled water (fig 2a) revealed a uniform surface layer while those immersed in citric acid (fig 2c) showed surface alterations due to matrix degradation with observed cracks, irregularities and empty spaces probably due to detached fillers. Specimens stored in ethanol (fig 2e) showed irregular surface with multiple cracks and prominent fillers of different sizes. Regarding resin composite, SEM images showed uniform surface in the specimens immersed in distilled water (fig 2b). Specimens immersed in citric acid (fig 2d) revealed slight alteration in surface topography due to filler dislodgement in some areas. Such topographical feature was more pronounced with larger concavities in specimens stored in ethanol (fig 2f).

DISCUSSION

The current study was conducted to evaluate the compressive strength and surface roughness of ceramic reinforced glass ionomer after immersion in distilled water, citric acid and ethanol in comparison to a nanofilled resin composite.

Chemical challenges are one of the most important degrading factors of the oral environment which could be in a continuous or intermittent mode. In case of poor oral hygiene patients or inaccessible areas in the oral cavity, chemicals could be absorbed by remaining food debris or accumulated plaque on restorations' margins. This acts as a continuous reservoir for such chemicals. On the other hand, eating or drinking of chemicals represents the intermittent exposure [16,17].

The US Food and Drug Administration recommended some food simulating fluids to be used during testing of materials' aging which were followed in the current study. Distilled water simulates the wet oral environment provided by the presence of saliva and water. Consumption of acidic foods such as certain beverages, fruits and vegetables was represented by citric acid solution. Aqueous ethanol water solution simulates alcoholic liquids [18].

In the present study, no finishing and polishing procedures were done to provide standardization and avoid surface modifications of the specimens [19]. The specimens were immersed in the recommended solutions for 7 days at 37°C. This period of immersion was chosen to accelerate the effect of the food simulating liquids in accordance with previous methods described in the literatures [13,15,16].

In the current study, Amalgomer CR recorded lower compressive strength in comparison to nanofilled composite under different storage media. The manufacturer of Amalgomer CR did not report any information about the filler size and filler percentage of its zirconia fillers. However, it seems that the mechanical performance of the used nanofilled resin composite relays on the fact that it uses nanoscaled filler particles which allows higher filler loading[10]. The difference in filler size and distribution between Amalgomer CR and nanofilled composite could be observed in the SEM images especially in specimens stored in citric acid and ethanol (figure 2). This was in accordance with *Souza et al, 2016* [11] who found that nanohybrid composite has higher compressive strength than zirconia and alumina reinforced glass ionomer cement.

The inferior performance of Amalgomer CR might be attributed to its moisture sensitivity, being a water based material that consists of ion-leachable glass and water soluble polymeric acids[20]. A previous study[20] found that Amalgomer CR recorded higher water sorption value than resin composite. This might in turn affect its mechanical strength properties.

The weak bond between zirconia fillers and glass ionomer matrix might be another explanation for the lower compressive strength of Amalgomer CR. *Yli-Urpo et al, 2005* [5], reported a massive

reduction in compressive strength of glass ionomer after addition of bioactive glass and attributed this finding to the loose attachment of these particles to glass ionomer matrix.

Immersion of Amalgomer CR in citric acid recorded a major drop in its compressive strength. This might be attributed to the severe dissolution and degradation of GIC by citric acid as described by *Kumar et al, 2014 & Maganur et al, 2015* [21,22] They demonstrated that hydrogen ions of citric acid diffused into the glass ionomer components and replace metal cations in the matrix in a continuous process causing progressive dissolution of the GIC. *Kooi et al, 2012* [15], reported a significant degradation of giomer by citric acid and attributed this to the great susceptibility of fluorosilicate glass fillers to degradation by weak acids. Another research [23] demonstrated severe erosion of GIC by citric acid with considerable bulk loss of the material. This was also revealed in the current study where an obvious reduction in the specimens' size has been observed. This bulk loss of the material could explain the diminished mechanical properties.

Compressive strength of nanofilled resin composite recorded a significant decrease after immersion in ethanol and citric acid in comparison to distilled water. Ethanol is characterized by having a solubility parameter matching with that of BisGMA[15]. It also has high ability to extract unreacted monomers as it can penetrate through the resin matrix causing swelling of the polymeric chains[24,25] and degradation of filler-matrix interface[26] which consequently reduces it mechanical properties. Citric acid could affect resin composite by causing ionic dissolution of filler particles [14].

Regarding surface roughness results, Amalgomer CR recorded a significantly higher surface roughness than nanocomposite with different immersion solutions. Surface roughness is greatly influenced by size of filler particles and percentage of fillers occupying the restoration surface [27]. *De Paula et al,*

2015 [28], reported that there is a direct relationship between size of filler particles and material loss. This result was in accordance with *Kantovitz et al, 2009* [14], who reported higher surface roughness of ionomer materials in relation to resin sealant materials under acidic conditions. They attributed this result to fluoride release of glass ionomer cements. Also, *Hamouda, 2011* [29], reported the same finding and explained it by the larger particle size and moisture sensitivity of GIC.

Although, surface roughness of nanofilled resin composite significantly increased after immersion in citric acid and ethanol, it remains within the threshold of surface roughness for bacterial retention which is 0.2 μm [21]. This might be due to the high filler percentage and the nano-sized filler particles with minimal inter-particle spacing [8]. The good bonding of fillers to the resin matrix due the presence of silane coupling agent might be another explanation [29]. In addition, exfoliation of fillers from the restoration surface results in formation of very small surface defects [28].

Despite the aggressiveness of citric acid which results in massive reduction in compressive strength especially with GIC, it recorded lower surface roughness values than ethanol for both materials (table 7). This high aggressiveness might result in softening of resin matrix [13] in association with the ionic dissolution and loss of filler particles [14] which probably leads to the removal of a uniform layer of the material with bulk loss. However, ethanol results in matrix degradation without loss of filler particles. SEM images of Amalgomer CR after immersion in ethanol (fig 2c) shows the presence of prominent filler particles of various sizes which might justify their higher surface roughness values.

Based on the results obtained in the present study, the addition of ceramic fillers to glass ionomer cement did not improve the material performance in comparison to nanofilled resin composite. Ceramic reinforced glass ionomer cements still have

limitations regarding their resistance to chemical degradation as revealed in the compressive strength and surface roughness results. Nevertheless, further investigations are recommended to improve the performance of glass ionomer cements under different chemical environmental conditions.

CONCLUSIONS

Despite of the limitations of the current study, the following could be concluded:

1- The performance of Amalgomer CR under different storage media was inferior to nanofilled resin composite regarding compressive strength and surface roughness.

2- Citric acid severely affected compressive strength of Amalgomer CR.

3- Nanofilled resin composite was able to preserve its surface roughness within the clinically acceptable threshold after chemical challenge in contrary to Amalgomer CR.

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